

Empyrean MultiCore

Unbeatable ease-of-use and automation for popular XRD applications



A new standard in XRD automation

Empyrean MultiCore is a multipurpose X-ray diffractometer offering a winning combination of effortless automation and top-notch performance for the most popular XRD applications in industry and academia.

Since 2010, Empyrean has set the standard in X-ray diffraction, scattering and imaging, giving analysts the best possible performance for the widest possible variety of measurements – for everything from powders to thin films, and from nanomaterials to solid objects.

Empyrean MultiCore is a tailored configuration for everyday use that allows you to carry out a wide range of routine experiments without manual intervention, thanks to its fully-automated incident beam and diffracted beam optics (iCore and dCore) and easy-to-use software.



With precision optics and unbeatable ease-of-use, Empyrean MultiCore offers everything you need to analyze routine samples, from the thinnest epitaxial film to the rockiest geological sample – all in a spacious, easily accessible cabinet.

At the heart of Empyrean MultiCore are the **iCore** and **dCore** modules, which allow you to automate the acquisition of a wide range of measurement types, all within in a single batch. And thanks to automatic component setting, they're fully compatible with regulated environments, too.

So whether you're an industry lab needing rapid turnaround on large batches, or an academic department wanting an easy-to-use error-free setup, you can rely on Empyrean MultiCore to give you the performance you need – day in, day out.

Should I prioritize intensity or resolution?

If you're struggling with this question on your existing instrument, then we've got you covered.

With Empyrean MultiCore, you can easily adapt to situations where the measurement priority changes – choose high resolution for data quality, or high intensity for fast measurements. It's up to you!



The optics and sample stage at the heart of MultiCore provide fully automated, reliable operation and outstanding performance for a wide range of the most popular XRD experiments.

The heart of Empyrean MultiCore

Whether you're working with Cu or Co X-rays, the iCore and dCore modules in the new Empyrean MultiCore make it easy to use advanced techniques, even if you're new to XRD.

This ease of use is only possible because their interactive optics allow the system to automatically switch from one measurement configuration to another. All the complexity of manually changing separate components is eliminated, and instead you get the same performance for a large application range, with none of the hassle.

Automated component changeover for effortless performance

Below we show all components in each module, and highlight the five component technologies (1-(5)) that make the biggest difference to effortless everyday performance.

iCore

Incident-beam optics



dCore

Diffracted-beam optics



1 Mirror

Empyrean MultiCore uses Malvern Panalytical's tried-andtested full-sized **Bragg–Brentano^{HD} multilayer mirror**, to ensure that only monochromatic K α radiation is incident upon the sample.



This means that:

- Background levels are reduced and minimum detection limits are improved
- Fluorescence background is eliminated
- Multiple measurement types can be accommodated

Figure 1. Analysis of the NIST instrument response standard SRM1976b, using two optical configurations (chosen to create matching peak shapes): a conventional programmable divergence slit (PDS) set to ¼ ° (red), and Empyrean MultiCore Bragg–Brentano^{HD} mirror (blue). The latter clearly shows a higher intensity and lower background, demonstrating the benefit of using perfectly monochromatic K α radiation.

2 Programmable mask set

The programmable divergence mask set used in the iCore module of Empyrean MultiCore allows the mask widths to be controlled independently (down to 0.1 mm). This creates a beam tunnel that provides precise control of the width of the incident beam.

This means that:

- The beam width perfectly matches the sample width
- Scattering from air and sample holder is minimized
- Signal-to-background ratios are improved



Figure 2. The programmable mask sets used in Empyrean MultiCore ensure a perfect match for every sample, whatever the size.



Figure 3. This 2D diffraction image of an SBA-15 mesoporous silica powder (using a PIXcel^{3D} detector and an SAXS/WAXS stage) shows perfect Debye-Scherrer rings at low angles. The 0.1 mm minimum size of Empyrean MultiCore's programmable mask makes it possible to obtain these types of image easily and even to perform 'micro-mapping' over the sample surface.

(3) Programmable divergence and anti-scatter slits

Unlike conventional asymmetric slits, the **programmable divergence slit (PDS)** and **programmable anti-scatter slit (PASS)** used in Empyrean MultiCore each have two blades that can move independently.



This means that:

- The irradiation length and observation length can be controlled to exactly match the sample length
- The intensity can be increased by up to 20% compared to a symmetric slit design



Figure 4. With a conventional symmetric slit setup (left), part of the sample does not contribute to the measurement. With the PDS and PASS technology used for MultiCore (right), the measured area of the sample is maximized.



Figure 5. Whether you prefer work with fixed divergence slit (FDS) setting (top) or programmable divergence slits (PDS) setting, Empyrean MultiCore provides the flexibility allowing you to define the best measurement strategy for your sample types.



Figure 6. Comparison between scans of a metalorganic framework using an FDS setting (red) and an PDS setting (blue). Using PDS, the irradiation and observation areas are constant across the scan range, allowing the weaker diffraction peaks to be more easily seen at higher angles.

Beam shaping - A unique advantage of Empyrean MultiCore

The possibility to automatically adapt the beam shape to fit the sample is unique to Empyrean MultiCore. By controlling the beam size in two dimensions using this 'beam shaping' technology, diffraction intensity is increased and background signals are minimized, compared to traditional setups.



LaB6_PPC 0.28° LaB6_PPC 0.11°

(counts)

Intensity

14400

10000

6400

3600

1600

400

0

Empyrean MultiCore features full-sized **high-resolution & high-intensity parallel-plate collimators** (PPCs) to control the **equatorial** divergence of the beam along the 2theta direction.

This full size means that:

- Peaks are sharper, providing higher resolution
- You can decide whether to optimize for intensity or resolution, according to the sample type and required measurement speed

20.6 20.7 20.8 20.9 21.0 21.1 21.2 21.3 21.4 21.5 21.6 21.7 21.8 21.9

Figure 7. Comparison between measurements of LaB_6 powder using the parallel-plate collimator set at 0.11° (blue) and 0.28° (red). High intensity or high resolution.



(5) Soller slits

The full-sized **high-resolution & high-intensity Soller slits** in Empyrean MultiCore control the **axial** divergence of the beam perpendicular to the beam direction.



Figure 8. Comparison between measurements of LaB_6 powder using Soller slits at 0.02 rad (blue) and 0.04 rad (red).

This means that:

2Theta (°)

- Peaks are sharper, providing higher symmetrical profiles especially at lower 2theta angles
- You can decide whether to optimize for intensity or resolution, according to the sample type and required measurement speed

Can I use PPC and Soller in one measurement?

If you're running XRR, GI-XRD, stress or texture measurements, then yes – the combination of PPC and Soller slits enable you to achieve the best resolution, peak-to-background ratio, and counting statistics for these experiments. What's more, the dCore module in Empyrean MultiCore offers nine ways to integrate these components, providing unrivalled speed and resolution for your experiments.

Optimum performance, minimum time

With Empyrean MultiCore, there's no compromise between performance and efficiency – and you'll benefit from a number of exciting features not offered by other systems. On the next three pages, we've summarized why we think you'll love using Empyrean MultiCore.

Fully flexible automated experiment changeover

Conventional XRD systems involve manual intervention to switch optics. This is not only time-consuming in its own right, but you often have to choose between optimizing every experiment manually (which incurs significant downtime), or using an overnight batch run (which inevitably means running some experiments on suboptimal settings).

Empyrean MultiCore eliminates these compromises and difficult choices, by allowing you to run a range of fully automated experiments and sample types in whatever order you like.



Figure 9. The advantages in time-saving and experimental optimization achievable with Empyrean MultiCore, compared to a conventional XRD system.



Seeing is believing.

Watch one of our Application Specialists provide a demo of Empyrean – without leaving your desk!



Figure 10. To complement the automated configuration options, the 15-position sample magazines for batch measurements on Empyrean MultiCore support a vast range of sample types, from powder to nanoparticles, and solid to thin films.

Great low-angle performance

Low-angle experiments are important for measuring larger structural features, but a very narrow, high-intensity X-ray beam is essential.

As illustrated in Figure 11, Empyrean MultiCore provides excellent performance for these experiments, thanks to the wellcontrolled beam and patented low-angle sample holder. Importantly, the narrow beam is achieved without using a beam knife, which allows transmission, stress and grazing-incidence data to also be acquired.



Figure 11. A sample of mesoporous silica (SBA-15), containing uniform hexagonal pores of diameter ~10 nm, and measured using Empyrean MultiCore. This data shows the high quality of the analysis at very low angles (the first data point is at 0.3° 20, and the intensity is on a square-root scale), thanks to the tight, high-intensity X-ray beam.

Effective fluorescence supression

For samples that exhibit fluorescence, it's vital to remove background fluorescence, and the superior energy resolution of the 1Der detector is a great solution, as demonstrated in Figure 12.



Figure 12. Analysis of a mixture of Cr₂CuO₄, Cr_2O_3 and $CrCuO_2$, using a single scan on Empyrean MultiCore (the main peak is normalized). (A) Data collected with a fixed slit (FDS) and a conventional line detector, showing high level background intensity caused by Cr and Cu fluorescence. (B) Data measured with FDS and the high energy resolution 1Der detector, showing a significant reduction on background intensity level as the fluorescence from Cr is removed by the detector. (C) The combination of both the iCore and the 1Der detector results in complete exclusion of all (Cu and Cr) fluorescence, improving the signal-tobackground ratio by a factor of 90. Weak peaks at 40°, 48° are now clearly visible.

High-resolution X-ray reflectivity measurements

Measuring layer thickness of thin films is an increasingly important application of XRD, and the non-destructive X-ray reflectivity (XRR) technique has emerged as the 'gold standard' thanks to its ease of use and reliability.

Using Empyrean MultiCore, layers as thick as 300 nm (or more) can be resolved.



Figure 13. X-ray reflectivity curve of a 282 nm layer of SiO_2 on Si. The inset shows a comparison of data acquired on the iCore (red) and a parabolic mirror, also known as a Goebel mirror (blue), showing a much higher resolution of the iCore.

Experimental options for every application

For well over 100 years, X-ray diffraction has been a fundamental analytical method for materials analysis – so wherever materials are being researched or processed, you'll find an X-ray diffractometer nearby.

Empyrean MultiCore goes one step further than many diffractometers, by providing easy-to-use, automated analyses of a wide range of sample types across all application areas. As suggested by the chart below, navigating the wide range of experiments can be daunting at first. But with our experts on-hand to help you, you'll soon be confidently setting up and running automated batch measurements for all your experiments.



Figure 14. The suite of experiments available with Empyrean MultiCore provides you with everything you'll ever need from an XRD, across the full range of application areas.

Compatibility with non-ambient stages

In non-ambient studies, a sample is measured across a wide range of controlled temperatures, humidities, or electro-chemical conditions – and Empyrean MultiCore can easily accommodate these requirements.

For example, during temperature-controlled studies, thermal expansion can lead to changes in sample height. The MultiCore optics scan across the surface of the sample with the programmable Z stage, therefore eliminating these height shift errors.

Not only that, but the motorized components and optimized settings ensure high data quality, providing accurate position shifts for temperature-dependent peaks and reliable data on phase transitions.





Figure 15. This 2D plot illustrates how diffraction intensity (a scale from blue to red) for a sample of the ferroelectric material $(NH_{a})_{2}SO_{a}$ varies with both 20 angle (x-axis) and temperature (y-axis). Upon cooling from room temperature, an abrupt peak shift (indicating a crystalline phase transition from paraelectric to ferroelectric) is observed at –61 °C, and with further cooling the peaks continue to shift gradually down to -260 °C. Data was acquired on Empyrean MultiCore using a PheniX closedcircle cryostat from Oxford Cryosystems (shown above).



Figure 16. Relationship between cell parameter a and temperature for the same material as in Figure 15. With the help of the smart user batch process in HighScore Plus, the cell parameters from this large dataset are exported automatically once a structure fit is achieved.

Polycrystalline powders

The analysis of polycrystalline materials is probably the most common application of XRD, and Empyrean MultiCore can provide detailed information about the identity, distribution and properties of the phases present in both powders and solids. Samples can be supported in flat holders or in capillaries, while special stages allow non-ambient measurements.

Multiphase quantification

Empyrean MultiCore offers both high data quality and high speed for quantification of crystalline phases in mixtures. Detection limits can be lower than 0.1 wt%, making it an ideal setup for assessing purity of materials in academia and in pharmaceutical applications.

Routine measurement of any powder samples

In routine work, samples are commonly measured as part of a sequence, either manually or as an automated batch. However, the surface size and volumes of adjacent



samples can be very different, and on a conventional system you have to set the divergence slit and masks manually to produce the best data quality. With Empyrean MultiCore, the optimum beam size and shape is set automatically, streamlining routine acquisitions.



Intensity (counts)

7000

6000

5000

4000

3000

2000

1000 iCore

0

В

Slag

Fixed

10

20



Figure 17. Within a single sequence, Empyrean MultiCore can routinely adapt to samples of completely different size, within a single sequence. A good example of this adaptability is the pair of analyses shown here: (A) a small quantity, yellow powder in the middle (see arrow), of an unknown mineral from a geological site, mounted on top of a Si zero-background holder; (B) a large quantity of slag, mounted in a standard backloading holder. To illustrate the performance of the Empyrean MultiCore for these very different samples, uninterrupted data acquired using a classical fixed slit (red) and the motorized iCore module with a beam knife (blue) are shown.

Nanomaterials

A variety of properties of nanomaterials – including size distribution, surface-to-volume ratio and aggregation properties – play roles in their behavior, and therefore their applications. Empyrean MultiCore makes it easy to determine both the purity of the phase, and shed light on its nanostructural characteristics using information-rich small-angle X-ray scattering (SAXS) technique.

Phase identification and microanalysis

Regular powder diffraction provides information on phase identity and phase purity, along with details of nanocrystal size and microstrain. As a complementary method, wide-angle X-ray scattering (WAXS) can also be used.

The optics in Empyrean MultiCore work in conjunction with the reflection and transmission spinner, to enable fully automated acquisition of powder XRD, WAXS and SAXS data.

Size distribution using SAXS

Small-angle X-ray scattering (SAXS) experiments are carried out in transmission mode close to the direct beam, and so require tight control of the beam path, together with low background scatter from the optical components.

Empyrean MultiCore not only meets both of these criteria, but provides sensitive detection, courtesy of the high dynamic range and high resolution of the PIXcel detectors.





Figure 18. WAXS result for $Ce_{0.5}Zr_{0.5}O_2$ nanoparticles. The inset shows a Williamson–Hall analysis using HighScore Plus, which gives an average grain diameter of 83 ± 10 Å.





Figure 19. SAXS results for the same $Ce_{0.5}Zr_{0.5}O_2$ nanoparticles: (A) Comparison of the (logarithmic-scale) SAXS data (red) with the system background (blue); (B) Subsequent determination of the average particle radius (R_{50}) as 41 Å, using EasySAXS software, consistent with the WAXS analysis shown in Figure 18.

Solid materials



For modern advanced engineering, additive manufacturing, and archaeological research, samples are often a solid piece that should be examined non-destructively. Those samples often have a non-even surface that can only be analysed by parallel beam geometry for the best data quality and accuracy. With the flexible parallel plate collimators in the MultiCore optic, residual stress and texture analysis can be made faster or more accurately.

Residual stress

Analyzing residual stress in a material is useful for ensuring the safety and durability of a wide variety of polycrystalline materials, especially hardened steels, welded joints, ceramics and novel materials.

Residual stress analyses can be carried out in a variety of ways, and Empyrean MultiCore supports all the conventional techniques, using a wide range of sample stages.

For example, using the either high-resolution or high-intensity parallel plate collimator in the dCore module, you can quickly and accurately determine the variation between lattice spacings in a material, and so determine if any residual stress is present. Moreover, with Stress Plus software, the analysis routine can be fully automated.

Texture measurement

Texture analysis is critical in the production of metals for controlling properties such as mechanical strength and formability. These analyses are carried out by tilting and rotating a sample to generate the intensity distribution of a given reflection over all possible orientations (a 'pole figure').

Empyrean MultiCore simplifies this process by using a (3- or 5-axes) cradle to collect the data automatically. Subsequent data analysis using our 'Texture' software module is then used to produce an orientation distribution function (ODF) for the crystallites.



Figure 20. Residual stress of a tungsten carbide (WC) sample in two perpendicular directions, showing a multi-axial compressive stress component of -1864 ± 90 MPa at φ = 0° and -2184 ± 54 MPa at φ = 90°.



Figure 21. Texture measurement for a Ti alloy. (A) Four pole figures (B) ODF analysis

Polycrystalline thin films

Analysis of thin films used to be a non-trivial task, but Empyrean MultiCore offers a broad range of thin-film experiments on a single platform, enabled by an optimized combination of high-intensity or high-resolution components for speed and/or accuracy. In addition, predefined measurement templates handle all measurement types at the click of a button, including sample alignment routines.

Measurement of density, thickness and interface characters

X-ray reflectometry (XRR) is a highly sensitive method that measures the scattering from thin films and surfaces to obtain information such as film density and thickness, and the roughness and lateral homogeneity of interfaces. The example shown is notable for having an intensity range spanning 8 orders of magnitude, collected within just 5 minutes.

Phase identification and residual stress measurement

As well as understanding the identity of the phases present in a thin film, for engineered materials it is important to check their structural integrity using a residual stress measurement.

You can either interpret the data directly, or ask Empyrean MultiCore to perform a dedicated 'GI-stress' measurement (which can be automated using our 'Stress Plus' software).

Excellent parallel beam performance

Analyzing thin films requires use of small incident beam angles, to maximize the interaction of the X-rays with the surface layers instead of the substrate.

Like XRR, these grazing-incidence X-ray diffraction (GI-XRD) experiments require very precise control of the beam divergence. The iCore module on Empyrean MultiCore produces a very high-quality parallel beam that fulfils this requirement, while the PPCs in the dCore module offer high-intensity and high-resolution options, giving full flexibility without compromising on speed or intensity.





0.01 0.02 0.03 0.04 0.05 0.06 0.07 0.08 0.09 0.10 0.11 0.12 0.13 0.14 0.15 0.16 0.17 0.18 0.19 0.20 0.21 0.22 0.23 0.24 0.25 0.28 0.27 0.28 0.29

Figure 22. Three consecutive measurements of a 50 nm layer of indium tin oxide on a glass sample following automated sample alignment. (A) XRR simulation and fit to determine the layer and interface thickness, density and roughness; (B) GI-XRD and phase identification, showing a single composition of the layer (blue markers); (C) GI residual stress analysis, showing the layer has tensile stress.

Epitaxial thin films

Epitaxial thin films, in which crystalline layers have well-defined orientations with respect to the substrate, are the basis of many electronic devices. With Empyrean's unique dual-beam path design, MultiCore offers a range of specialist methods to simplify and streamline routine analysis of epitaxial thin films – without the need for manual intervention.



Ultra-fast 2D XRR mapping

Classic 1D X-ray reflectometry (XRR) curves offer useful information on layer thickness, density and roughness, but 2D XRR maps – constructed from hundreds of 1D XRR curves with different offset angles – provide additional knowledge on interface morphology.

Thanks to the perfectly parallel beam profile of the iCore module, combined with the small pixels and extremely low noise level of the PIXcel^{3D} detector, Empyrean MultiCore provides outstanding resolution for this experiment type.

It's worth noting that the collection time for a 2D XRR map is the same as for a classic 1D XRR curve, making this option highly appealing when you need to maximise your knowledge of the sample.





Figure 23. Analysis of a bilayer film (26.7 nm C + 253 nm SiO_2) on a Si substrate using Empyrean MultiCore. Using AMASS software, the 2D Ultra-fast XRR map (A) can be used to derive both a specular reflectivity curve (B) and a diffuse scattering curve (C). The latter shows that the C-SiO₂ interface follows a fractal model with a correlation length of 130 nm.

High-resolution rocking curves and reciprocal space maps

By employing the dual-arm configuration with a 3-bounce (TA) analyzer, the resolution of iCore optics can be further enhanced to provide classic rocking curves and reciprocal space maps (RSMs) for high-quality epitaxial strained-layer structures.

From the smooth fit provided by the AMASS software module, you can easily determine the superlattice period, and the composition ratio of individual elements. And for RSM acquisition, the unique dual-beam path configuration with a PIXcel^{3D} detector provides flexibility and ease of use, allowing fast, automated alignment and data collection.



Figure 24. Rocking Curve data acquired using iCore with a TA analyzer for an InGaAIAs–InGaAsP superlattice grown on an InP substrate, with highresolution rocking-curve data for the (004) reflection (red) and the resulting AMASS-enabled smooth fit (blue).



Figure 25. Examples of ultra-fast RSM (URSM) collected within 5 min using the PIXcel^{3D} detector with Empyrean MultiCore: (Left) Plot of the (004) reflection of a InGaAIAs–InGaAsP superlattice thin film; (Right) Plot of the (105) reflection for a GaN-based high-electron-mobility transistor. (In both maps the $K\alpha_2$ component has been removed using Highscore software).

Options for Empyrean MultiCore

To help you decide what you need from Empyrean MultiCore, here's a quick summary of the additional options that we offer, and an overview of the experiment types you might want to perform. You can find out more in our full technical specification – or by asking one of our specialists.



Got any questions about Empyrean MultiCore?

Talk to our global/regional sales representatives or application scientists for a tailored solution for your specific requirements!

A 100-year obsession for XRD

XRD is a long-established technology – and from X-ray tubes to automated optics, we've been at the forefront all the way.

So yes, we've had over 100 years to get pretty good at XRD. And we'd like to help you get the best out of this technology too – which is where Empyrean comes in.

Empyrean is now installed in over 1000 industry labs, research centers and universities around the world. If you'd like to be our next customer, then good news – our global network of 100+ regional contact centers means there's one not far from you.

And if (or when) you do become a proud owner of an Empyrean MultiCore, then you'll be reassured to know that our training sessions, consumables packages and service offerings will help it running perfectly for many years to come. Perhaps even 100!







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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