

EMPYREAN XRD WITH THE 1Der DETECTOR

Empyrean brings new wonder to XRD



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The Empyrean platform just got better with a new detector: 1Der. Building on our strong track record of pioneering X-ray diffraction technology, this completely redesigned 1D detector dramatically improves performance, opening up a new world for wonderful X-ray diffraction (XRD).

Ultimate exclusion of fluorescence interference

In an X-ray diffraction measurement, fluorescence from some elements can be strong enough to create unwanted background in the measured data. The 1Der's superior energy resolution, a major advantage over previous detectors, enables users to exclude this background from their measurements.

As an example, *Figure 1* shows a periodic table, showing the elements that fluoresce strongly when illuminated with a Cu X-ray source. The 1Der's programmable energy window enables the exclusion of all these fluorescence energies from the measured scan.



Figure 1. The periodic table showing elements that are easily excited by X-rays from a Cu X-ray tube. 1Der alone, or together with Empyrean's unique incident beam optics, excludes unwanted fluorescence from any X-ray source.

Figure 2 shows the excellent capability of the 1Der detector alone to exclude the fluorescence background during measurement of an Fe₂O₃ sample using Cu radiation. The blue line shows a Bragg-Brentano scan using a fixed divergence slit and standard detector settings. The background is high due to considerable Fe fluorescence. The red line shows the same scan using the 1Der detector with narrow energy window. The main intensity peaks are normalized and the low background (increased signal level) can be clearly seen.



Figure 2. Two Bragg-Brentano scans of an Fe₂O₃ sample, using fixed-slit incident beam optics: Blue line: FDS with standard detector settings. Red line FDS with 1Der narrow energy window.

The lowest background ever

The 1Der's superior energy resolution enables dramatic data improvements in crystallography. This is shown in *Figure 3* for data collected on copper chromium oxide, which is used in advanced catalysis. Chromium is a well-known fluorescing element in this kind of material.

Figure 3 also shows that when the 1Der is combined with either the Malvern Panalytical iCore or the Bragg-Brentano^{HD} incident beam PreFIX optics, background caused by fluorescence can be dramatically excluded.

The Empyrean system with the 1Der detector provides the highest sensitivity X-ray powder diffraction data available today.



Figure 3. Data collected on a mixture of $Cr_2CuO_4 + Cr_2O_3 + CrCuO_2$ with various configurations: in blue, data collected using slits and a conventional energy resolution configuration showing background caused by Cr and Cu fluorescence. In green, data measured with fixed slits and 1Der detector; fluorescence from Cr is completely removed but the angle independent Cu fluorescence remains. In red, the combination of both the iCore and the 1Der detector results in complete exclusion of all fluorescence. As shown here the main peak is normalized for all three scans. Overall the signal to background ratio is improved by a factor of ~50.

The widest range of X-ray source compatibility

The 1Der is compatible with all X-ray sources that are used on the Empyrean. With all Empyrean X-ray tubes, users can quickly change the source themselves, enabling them to select improved performance for unique applications. This kind of multi-source system opens possibilities for measuring applications previously inaccessible on a single-source system.

For example, when used in combination with higher energy or 'hard' radiation (such as Mo or Ag), the 1Der detector can measure the total scattering data suitable for Pair Distribution Function (PDF). Hard radiation is also used in powder diffraction transmission experiments, for example, to penetrate solid objects such as metal components or pouch cell batteries.

Soft radiation (such as Cr) is often used to spread the diffraction pattern and improve the resolution of closely spaced peaks, such as those seen in clays and ceramics. *Figure 4* compares scans of a sugar sample, using Cr radiation (in blue) and Cu radiation (in green). The Cr radiation clearly provides sharper and more easily resolved peaks.

With 1Der you have freedom to use any X-ray source.



2Theta (°)

Figure 4. Comparison between Empyrean data collected using $Cu-K_{\alpha}$ radiation (in red) and $Cr-K_{\alpha}$ radiation (in blue) at the same optic (divergence and soller slit) settings. In the lower graphs the Cr data are reproduced and wavelength-converted to allow favourable comparison with Cu radiation peak widths.

Simplify your data analysis with K-beta monochromation!

Traditionally, many powder diffraction experiments use $Cu-K_{\alpha}$ radiation because this produces the highestintensity peaks. However, for the more intrepid user, there may be occasions when this analysis could be improved by taking a more tailored approach.

Specifically, the Cu- K_{α} peak is a doublet of two peaks known as Cu- $K_{\alpha 1}$ and Cu- $K_{\alpha 2}$. In some circumstances, these doublet peaks can cause confusion when identifying phases, or they can lead to a reduction in precision for analytical tasks such as peak indexing or lattice parameter refinement.

Figure 5 shows that, whilst Cu-K_β is a lower-intensity beam, its single peak can provide clarity in complex data analysis cases. By using the wavelength selection functionality in the integrated detector control, users can easily select Cu-K_β in a measurement program.

Talk to us about how K_{β} measurement could benefit your data analysis!

Technical specifications: 1Der	
Window size	9 mm x 15 mm
Efficiency Cu K _a	> 94%
Efficiency Mo K_{α}	38%
99% Linearity range	0 – 1.7 10 ⁶ cps – Overall
Energy resolution around Cu \ensuremath{K}_α	<5% (typical performance ~340 eV)
Maximum count rate	38.4 10 ⁶ cps - Overall
Maximum background	<0.4 cps - Overall
Strip size	70 µm
Total number of strips	127
Active length	9 mm
Smallest step size	0.0021° 2θ at 240 mm goniometer ra
Supported wavelengths	Ag, Mo, Cu, Co, Fe, Mn, Cr
K_{α} to K_{β} switch	Yes



Figure 5. Peak profile comparison between classic Cu-K_{$\alpha1$} and Cu-K_{$\alpha2$} radiation (in blue) and monochromatic Cu-K_{β} radiation (in red).

Summary

The addition of the 1Der detector to the Empyrean XRD solutions portfolio is the next great step in a program of continuous product development.

Together with Empyrean's suite of optical configurations and sample stages employing PreFIX, the fast exchange of pre-aligned components, the 1Der combines ease of use with unrivalled flexibility and data clarity for all powder diffraction applications.



Talk to us now to see how our latest Empyrean solutions can bring real benefits to your research and processes!





WHY CHOOSE MALVERN PANALYTICAL?

We are global leaders in materials characterization, creating superior, customerfocused solutions and services which supply tangible economic impact through chemical, physical and structural analysis.

Our aim is to help you develop better quality products and get them to market faster. Our solutions support excellence in research, and help maximize productivity and process efficiency.

Malvern Panalytical is part of Spectris, the productivity-enhancing instrumentation and controls company.

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