

Diffractometers for modern X-ray crystallography: The XtaLAB Synergy X-ray diffractometer platform

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1. Introduction

The modern X-ray diffractometer has drastically changed in performance compared with those from even only 10 years ago. With Hybrid Photon Counting (HPC) detector technology, brighter sources and more intelligent software, modern diffractometers enable higher quality, faster research and open new areas of study possible on smaller and more challenging samples. The XtaLAB Synergy is a range of diffractometers from Rigaku which are designed to meet the needs of modern crystallographic researchers as multipurpose versatile diffractometers sharing a common platform. The range covers instruments from entry level microfocus systems to high performance rotating anode instruments. The instrumentation is modularised such that the core platform supports different combinations of components to make up a comprehensive range covering the needs of many different classes of user. All are powered by CrysAlis^{Pro}, which has an intuitive user interface which provides a consistent environment to enable users to feel at home on any instrument in the range. This article endeavours to describe the XtaLAB Synergy diffractometer range and its capabilities.

2. The XtaLAB Synergy Diffractometer Platform

The most recognisable feature of the XtaLAB Synergy instrument family is the radiation protection enclosure. The primary function of the enclosure is to ensure the safety of the user and as such fully isolates the user from exposure to X-rays while at the same time containing all of the core instrument components including the goniometer, sources and detector, with the high voltage generator, chiller, system interface and safety module contained in the electronics rack below. The cabinet features 2 hinged doors on the front as well as removable side and rear panels. In all cases the panels are interlocked with encoded magnets to ensure door panels are in place before X-ray generation is allowed to commence. The interlocks are connected to an electromechanical safety system which cannot be influenced or overridden by software. Communication is “read-only” so that status may be read for the purposes of fault finding and determining operational readiness.

The cabinet is designed to be compact enough to fit through most standard single doors whilst containing all instrument components with the exception of larger chillers required for higher powered sources, and some



Fig. 1. The XtaLAB Synergy-S diffractometer system.

third party device control electronics. This allows the instrument to be mounted on wheels for easy repositioning or transportation should future relocation be necessary.

All XtaLAB Synergy instruments are designed for reliability and serviceability. Hardware control electronics (e.g. X-ray generator, system interface, etc.) are modularised into rack mounted units each with custom firmware and communicating through a CAN bus network. This allows for containment of faults and easy replacement should modules become faulty. In most cases modules can be replaced by users under instruction though this is typically performed by trained service engineers. Each unit provides a status feed readable by the diffractometer control software such that logging of status is possible either to remotely determine or set instrument status or for fault finding. XtaLAB Synergy instruments thus allow many issues to be diagnosed and in some cases fixed without an engineer visit on-site. Compartmentalisation into single purpose modules also allows for simplification of hardware modules and their firmware to ensure the highest reliability is achieved.

2.1. The universal goniometer

At the centre of the XtaLAB Synergy instrument

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is the universal goniometer. This is a 4 circle Kappa geometry design with axes described briefly in Fig. 2 and in more detail by Meyer *et al.*^{(1), (2)}. The design allows for high accessibility to reciprocal space without occluding portions with bulky hardware. All axes, omega, theta, kappa, and phi are driven by stepper motor technology for accurate and precise positioning. Each axis has both relative and absolute encoders for positional feedback so that the goniometers position and angles can be known and recorded at any time of the user's choosing including during data collection. The use of absolute encoder technology allows positional feedback even following power outages which has been known to cause instrumentation to forget it's last known position requiring a re-homing procedure. The goniometer is a robust field proven design which has been in use over several iterations with long term reliability over several decades. Upgrades to this design have been made to improve speed and reliability to match enhancements in other components and also to support modern techniques and attachments.

Kappa geometry with a β angle (angle between Kappa and Phi in Fig. 2(a)) of 50 degrees has been chosen to offer the most efficient data collection and highest coverage, whilst using a simple rotation design as opposed to a partial track for easier maintenance and better long term reliability and precision. In comparison to fixed chi instruments, a Kappa goniometer can reduce the number of frames and runs required for a given data collection goal and may provide higher completeness for cases in which use of symmetry equivalents do not offer a route to observing all reflections or selected important reflections, e.g. $P1$ symmetry. This can save valuable time when experiment time or sample dose is a factor such as high throughput applications and

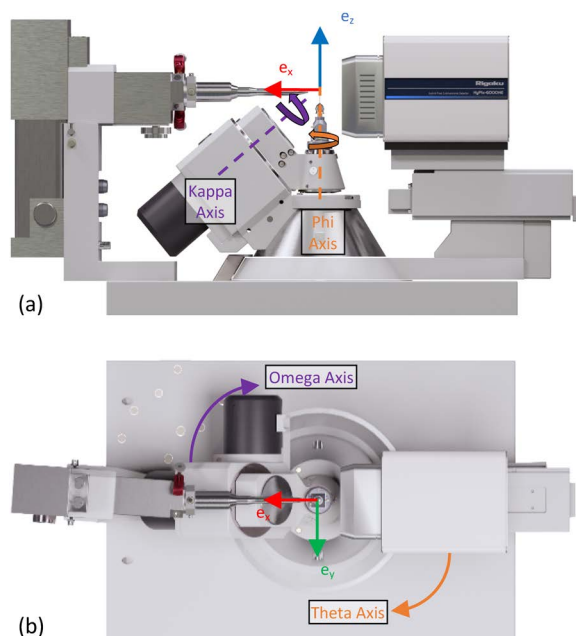


Fig. 2. Goniometer axis system showing side view (a) and top view (b).

studies of metastable species. For the XtaLAB Synergy diffractometers, motor speed has been upgraded to allow 10°/s movement on omega theta and kappa axes with 20°/s available on phi. This ensures the maximum benefit can be gained from the current generation of high performance sources and detectors.

As the characteristic (pixel size and point spread function) of the current generation of HyPix detectors are different from previous detectors e.g. CCD, Pilatus, the detector drive has been designed to allow an appropriate range of detector distances without compromising theta motion. To do this, it uses a unique telescopic arm so that a compact profile is achieved at short detector distances with long sample-to-detector distances still achievable.

2.2. XtaLAB Synergy models

The XtaLAB Synergy X-ray diffractometers are generally defined by the source technology used within. There are four different models in the standard range with the XtaLAB SynergyCustom, a special case for more demanding users, making up a fifth.

2.2.1. The XtaLAB Synergy-S

The XtaLAB Synergy-S (Fig. 3) features the PhotonJet-S source which is a high performance microfocus X-ray tube coupled to a multilayer confocal optic.

The X-ray source (Fig. 4) is built in a compact form with single and dual source configurations available in Cu, Mo and Ag wavelengths. The low power tubes run at 50 W and are coupled to confocal multilayer optics to produce intense beams of 110 and 120 μm FWHM depending on source wavelength. The high voltage generator units supply 50 W of power (50 kV, 1.0 mA) with a stability of 0.05% or better per 8 hours after 30 minutes' warm-up resulting in unparalleled reliability and ease of use. As with any high voltage X-ray source, heat is produced during X-ray generation as an unwanted by-product and is detrimental to X-ray flux e.g. through thermal expansion of tube materials and other mechanisms. Conversely for maximum flux the highest achievable power density is desired making it essential to firmly control and limit anode temperature



Fig. 3. The XtaLAB Synergy-S.

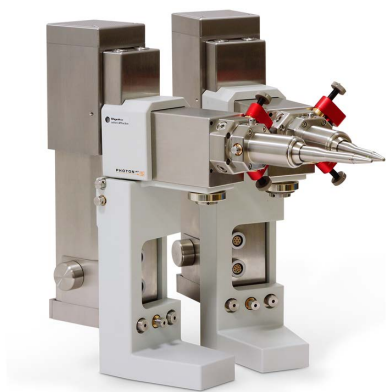


Fig. 4. Dual PhotonJet-S microfocus X-ray sources.

for optimal, stable flux output and to prevent anode degradation.

Water cooling offers the highest thermal stability of the X-ray beam, and so the PhotonJet-S incorporates a closed circuit water cooling system. Additional benefits of this are that it: allows heat to be efficiently removed from the cabinet and sample environment; is more resistant to fluctuations in ambient temperature and; avoids the need for small high-speed fans and their associated vibration attached directly to the source. Air-cooling in a confined space also means the cooling medium, air, is heated by the sources heat sink, exhausted into the cabinet then recycled through the heat sink of the source (and other heat loads) creating a potential for runaway heating, particularly in hotter environments.

2.2.2. The XtaLAB Synergy-*i*

The XtaLAB Synergy-*i* is also based on microfocus technology with an HPC detector to provide a cutting edge solution. With dual or single source configurations the XtaLAB Synergy-*i* shares many design principles with the XtaLAB Synergy-S but is designed to meet the needs of modern crystallography while remaining cost-effective. The goniometer differs from the other XtaLAB Synergy diffractometers having slower movement speed at half that of the universal goniometer. The XtaLAB Synergy-*i* features the HyPix-Bantam hybrid photon counting detector for instantaneous single X-ray photon.

2.2.3. The XtaLAB Synergy-R

The XtaLAB Synergy-R instrument (Fig. 5) is built around the PhotonJet-R rotating anode X-ray source. Offering 1.2kW of X-ray generation and custom designed optics the PhotonJet-R provides our highest performance source in a self-contained compact cabinet instrument. Rigaku rotating anodes are supported by numerous patents⁽³⁾⁻⁽⁹⁾ and years of research and experience.

Rigaku rotating anodes have been a core Rigaku product for most of its history and as such many reliability issues which plague other rotating anodes have been addressed and solved. Generally, source flux is limited by the ability of the anode material to



Fig. 5. XtaLAB Synergy-R, a rotating anode diffractometer.

dissipate heat. Achieving higher power density on the anode surface without melting the anode material can be achieved in several ways. By moving the anode surface under the electron beam, applying higher heat load to the anode is possible. This presents challenges in maintaining vacuum and mechanical reliability both of which are problems Rigaku has solved. For a typical rotating anode source the anode requires refurbishment only annually, allowing higher throughput and access to more challenging samples. The only additional scheduled maintenance for the source during this annual maintenance cycle are filament replacements which can easily be performed by the user approximately every three months or less with only a few hours of downtime. This enables a laboratory equipped with a rotating anode to achieve up to 5 times the output of an equivalent laboratory equipped with only microfocus sealed tube technology. In recent times a new type of source, the diamond backed anode has seen a rise in popularity. Typical diamond backed anode sources only offer twice the power loading of an ordinary microfocus sealed tube whereas a rotating anode offers 24 times higher power loading.

2.2.4. XtaLAB Synergy-DW

The XtaLAB Synergy-DW (Fig. 6) offers a unique solution to the problem of wavelength choice.

Since their introduction in 2004, dual source instruments have become the standard choice for sealed tube instruments. The potential of choosing source wavelength to suite the sample or experiment at hand was quickly recognised by the crystallographic community.

For sealed tube instruments, the small form-factor of the source allows co-mounting of two sources on the instrument without a significant reduction in the symmetric accessible theta range. For rotating anode solutions, the size of the source makes co-mounting unrealistic without accepting a significant reduction in symmetric accessible theta range. To solve this problem, the PhotonJet-DW was developed which contains two wavelengths in one source.

The anode used in the PhotonJet-DW has two target tracks (Fig. 7) which can be positioned under the electron beam with a high precision hydraulic system.

The source optics are also mounted within a motorised chamber which allows the rotation of the appropriate optic into position. By achieving this single source form-factor solution, higher theta access is possible with two wavelengths than for co-mounted sealed tubes. The versatility of having two rotating anode power wavelengths sources in a single cabinet offers the potential for multidisciplinary instruments shared between research groups or high throughput service instruments capable of working with many different sample compositions.

2.2.5. XtaLAB SynergyCustom

Contrary to the other systems described in this paper, the XtaLAB SynergyCustom (Fig. 8) is not installed within the XtaLAB Synergy cabinet. Typically mounted on an optical table, the XtaLAB SynergyCustom is the

most flexible member of the XtaLAB Synergy line-up and the only member supporting our most powerful X-ray source, the FR-X. For applications where bulky or non-standard equipment is required, a large amount of space is provided around the goniometer.

The FR-X source operates at 2.97kW providing approximately 2.5 times higher flux output than the PhotonJet-R making it the highest flux X-ray source currently available for X-ray crystallography. The FR-X also supports single or dual wavelength similar to the PhotonJet-DW but with more flux.

The XtaLAB SynergyCustom may also be fitted with a PhotonJet-R source for applications which do not demand the highest flux, but where a flexible arrangement is required for the support of additional equipment or more specialised experimental setup. Additionally, dual port operation is supported by the XtaLAB SynergyCustom should two end-stations from a single X-ray source be required.

2.3. Beam conditioning

Most XtaLAB Synergy sources offer beam conditioning options⁽¹⁰⁾. Through software control, the divergence and intensity of the X-ray beam can be controlled. With continuous control the user is free to optimise the loss of intensity against the lower divergence to ensure peaks may be spatially resolved with enough intensity for high data quality (Fig. 9).

With beam slits positioned inside the X-ray source and motorised, the user does not need to physically interact with the source such that there is no potential for source alignment to be disturbed. A motorised continuously variable slit also provides higher mechanical reproducibility than is possible when e.g. collimator tips are replaced and offers a much wider range. In combination with HPC detectors, beam slits offer the ability to measure far longer axes than any other available solution without demanding long sample-to-detector distances as has been the traditional



Fig. 6. The XtaLAB Synergy-DW with HyPix-Arc150° detector.

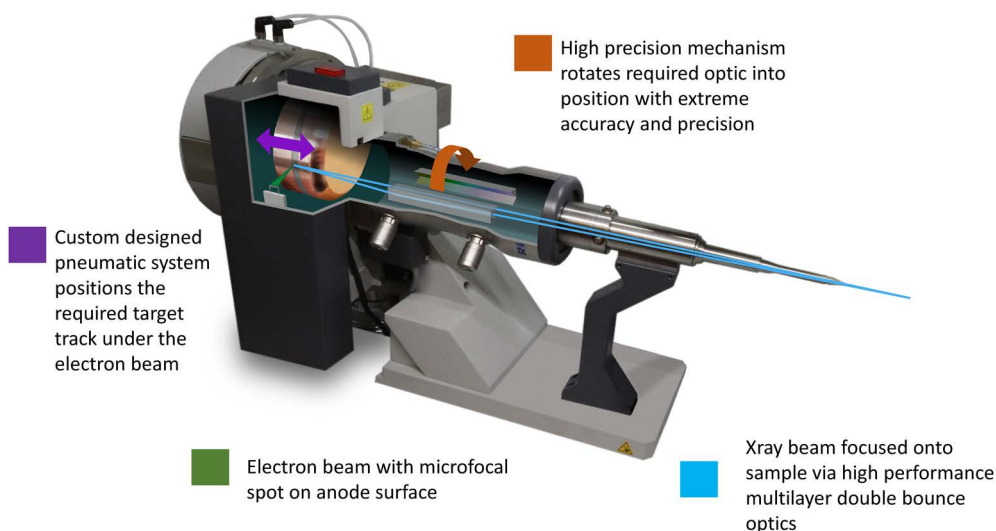


Fig. 7. The PhotonJet-DW X-ray source.



Fig. 8. XtaLAB SynergyCustom with FR-X high flux X-ray source and HyPix-6000HE detector.

solution.

3. HyPix Hybrid Photon Counting Detectors

3.1. Features of Rigaku HyPix detectors

All Rigaku HyPix X-ray detectors use Hybrid Photon Counting (HPC) technology to directly detect X-rays. HPC technology was initially devised at CERN⁽¹¹⁾ for use at the large hadron collider and have been achieved widespread acceptance for synchrotron use in single-crystal diffraction applications in the years since. Rigaku first adopted third party HPC technology 2007 and began offering the Pilatus detector⁽¹²⁾ for single crystal X-ray diffraction in 2013. Rigaku has since introduced its own HyPix family of HPC detectors for single crystal applications in 2016 with the HyPix-6000HE (see section 3.2.2).

The HyPix detector technology uses $100 \times 100 \mu\text{m}$ pixels which are defined by the structure of the bump-bonded application specific integrated circuit (ASIC) counting electronics behind the photodiode substrate. Incoming photons are directly absorbed by a photodiode substrate immediately generating hole-electron pairs. The voltage associated with the charge generated is compared to a threshold and a count registered if higher. Since the X-ray photon is directly converted to electrons, the X-ray photon energy is preserved in the charge. The signal is also highly localised within a pixel giving a single pixel top-hat shaped point spread function (Fig. 10). Note that traditional X-ray detectors, those which integrate and use a scintillator, lose the ability to discriminate X-ray photon energy since visible light photons are detected instead and integration prevents assignment of charge to individual X-ray photon events.

Direct-detection of X-rays with an energy threshold method gives several benefits. Firstly, only photons greater than a desired energy are counted. Electronic noise or dark current—a big problem for integrating technologies—is not significant enough to overcome the threshold thus it does not affect digital counts. Secondly counting pulses over the threshold as they happen eliminates readout noise. Lastly this can be used to selectively count photons matching the X-ray source

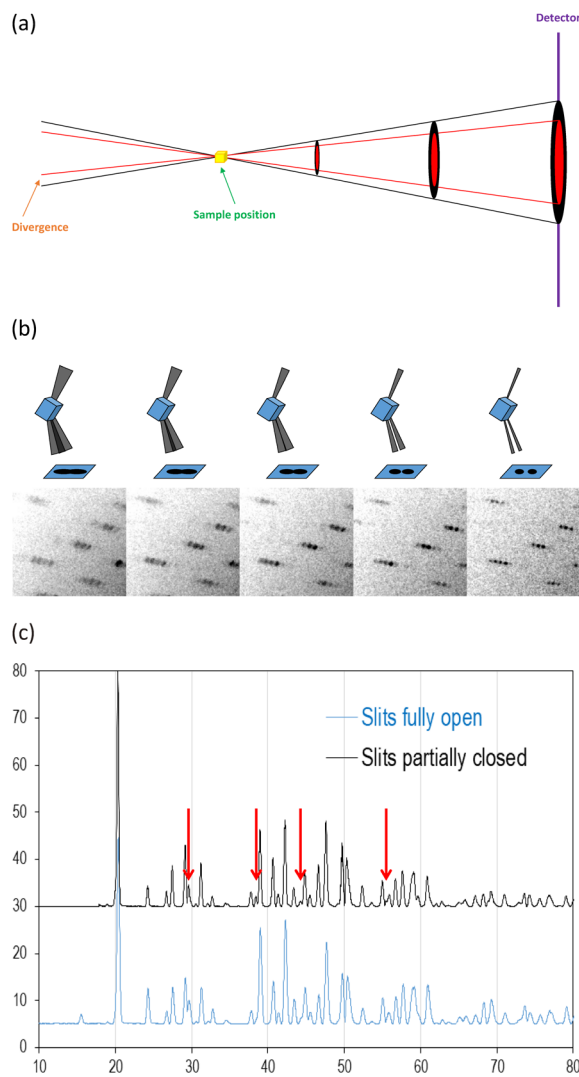


Fig. 9. (a) Divergence control principle and its effect on peaks size, red peak uses low divergence setting to reduce peak size. (b) Decreasing divergence from left to right reduces peak overlap shown both schematically and in real diffraction images. (c) Improved powder pattern resolution is achieved when using low divergence settings (blue).

energy e.g. to suppress fluorescence signal by adjusting the threshold at the small cost of slightly reduced counts for sample peaks (Fig. 11). This counting method therefore eliminates the two most common and difficult noise sources, readout noise and dark current and means only diffracted photons counts appear in saved images when appropriate settings are used.

3.1.1. Shutterless operation

As described above, HyPix detectors differ from integrating detectors in that they digitally count photons as they arrive. Unlike integrating technologies, since digitisation has already occurred at photon arrival, readout noise and therefore speed is no longer a factor. HPC detectors can therefore readout entire images in a few milliseconds. As with all other detector technologies however, during readout photons arriving at the pixel, in this case the counter, cannot be detected. Most

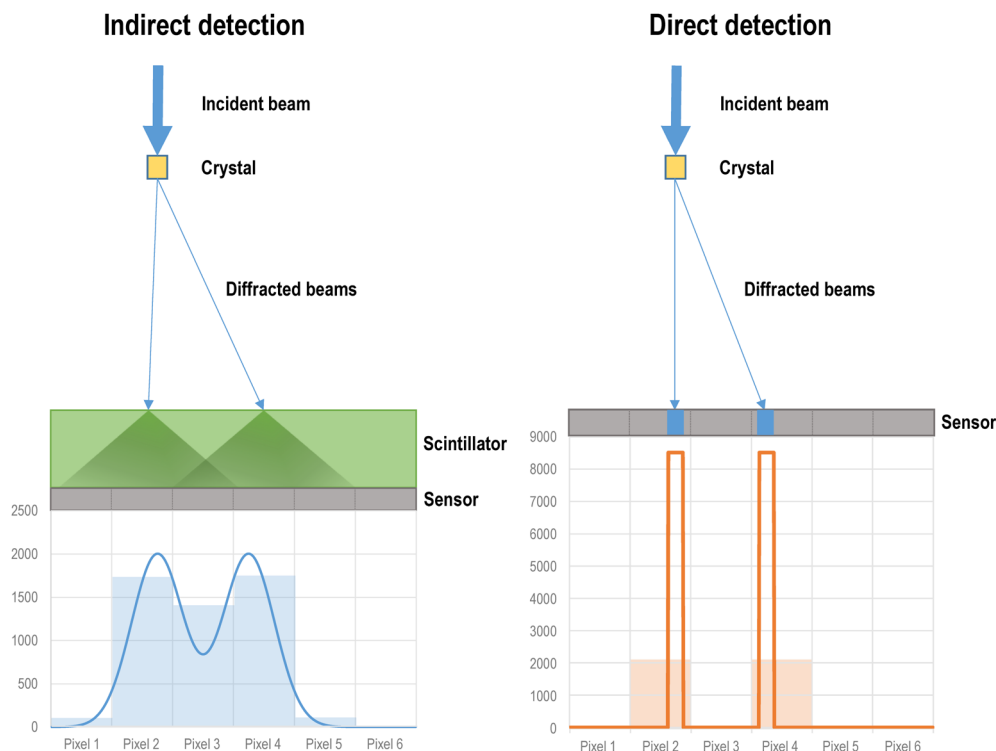


Fig. 10. The effect of a single-pixel Gaussian point spread function on peak separation vs a top-hat single-pixel point spread function for the same incoming X-ray signal and pixel size.

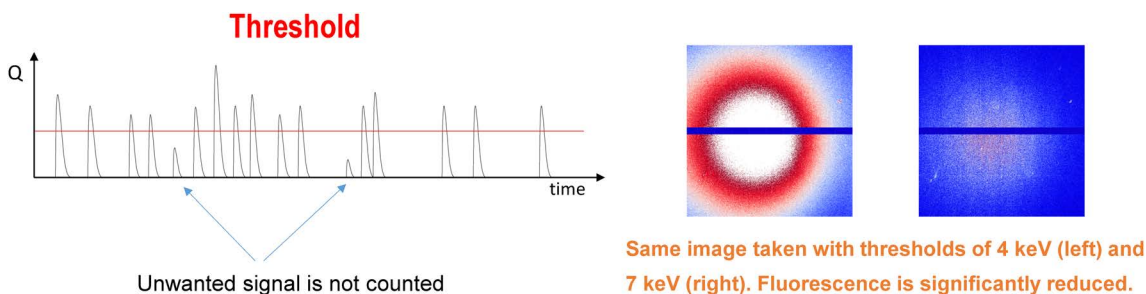


Fig. 11. Energy thresholding in HPC detectors.

modern detectors feature a dead time of up to several milliseconds which is considered acceptable loss and so operate in a shutterless mode. This avoids the need for goniometer repositioning for each frame and allows a continuous scan giving considerable speed benefits. Additionally, as readout is simultaneous for all pixels (global shutter), there is no shutter skew a temporal distortion effect described in Fig. 12.

HyPix detectors (excluding HyPix-Bantam) offer 2 counters per pixel which may be used in different combinations to achieve several different counting methods.

3.1.2. High Dynamic Range mode

Firstly, the two counters may be combined as a single 31 bit counter which offers extremely high counter depth. In this mode the counter can register from a single count to an essentially inexhaustible count limit of over 2 billion counts during the exposure. This is essential for measurements where strong and weak data are expected in the same image, a typical situation

for crystallographic experiments (Fig. 13). Note that photon counting is possible across the entire range of the detector.

3.1.3. Zero dead-time mode

The zero dead-time mode uses the counters in an alternating sequence. While one counter is active and counting photons, the other may be readout and reset. This means that the only light insensitive period is when switching from one counter to another rather than throughout the readout process.

Switching between counters takes only a few nanoseconds instead of milliseconds which reduces the light insensitive time by six orders of magnitude. This is naturally most useful when the exposure times are very short and highest frame rates are in use. Under these circumstances readout time would represent a bigger proportion of the total frame time and might accumulate to large counting losses. In the zero dead time mode, these losses are less than 0.000001% of maximum signal for the shortest sustainable exposure time possible

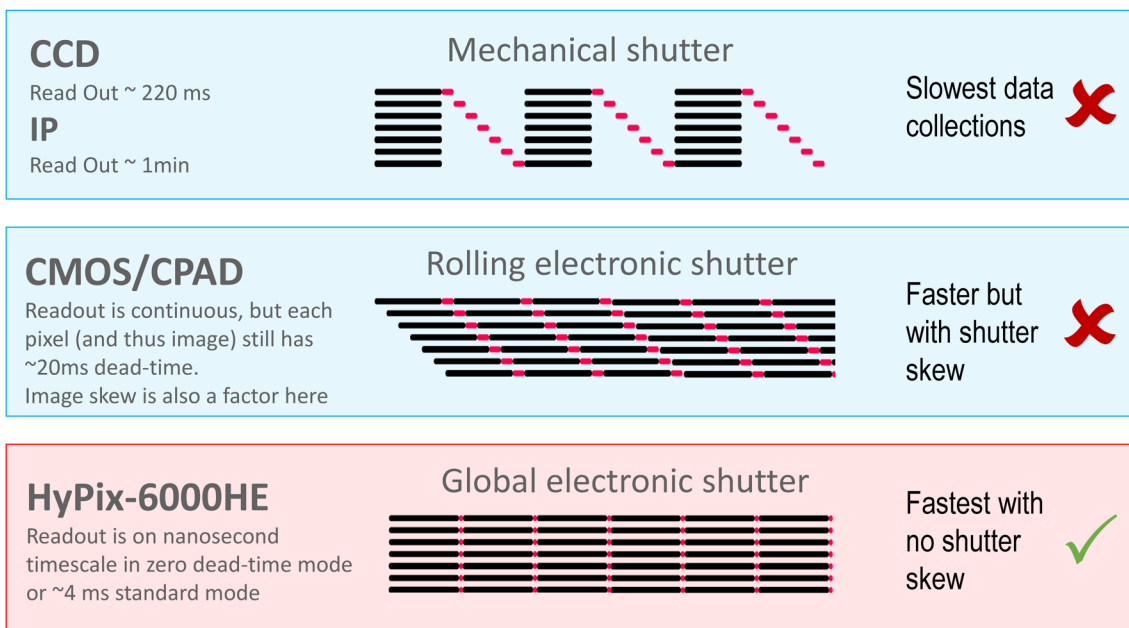


Fig. 12. Detector readout modes and their relative advantages and disadvantages.

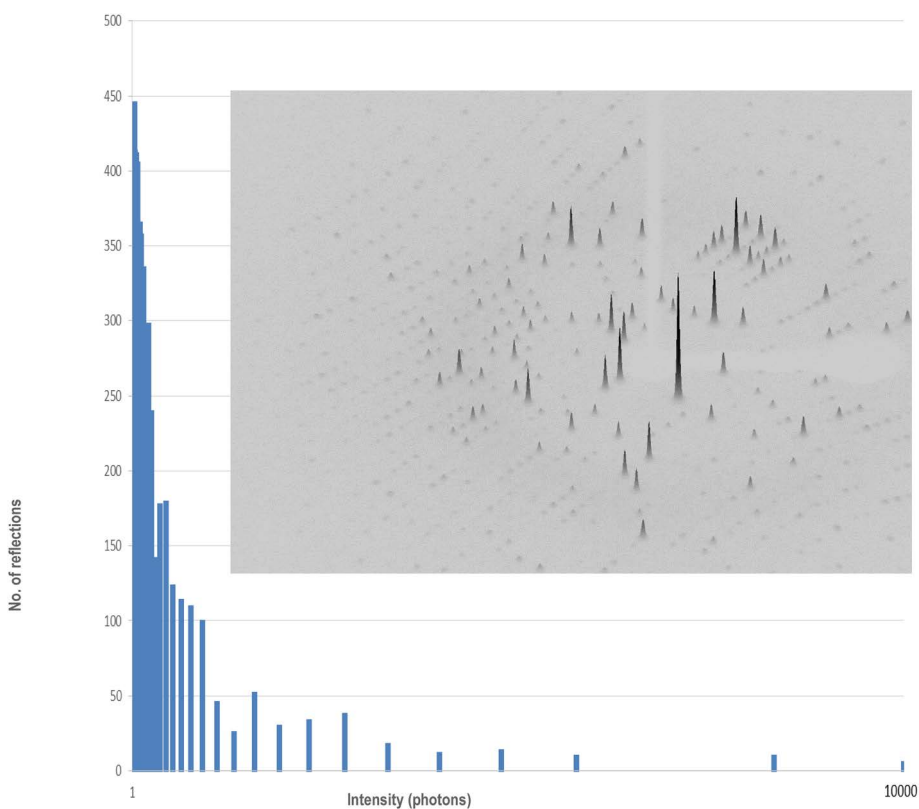


Fig. 13. Distribution of intensities in a typical X-ray crystallography experiment showing the vast majority of peaks are ‘weak’.

(0.01 s).

3.1.4. Differential counting modes

As there are two counters per pixel this also means each has its own threshold. Normally these are set to the same value but this architecture allows the counters to independently count different photon energies. These can subsequently be subtracted from one another to offer different possibilities. Firstly, with one threshold

set low and the other set high, it becomes possible to eliminate low energy contamination and high energy contamination, truly focusing in on the energy band containing the photon energy of the X-ray source. In this regime one counter eliminates spurious low energy photons and counts the diffracted photons and any spurious high energy photons. The second counter only counts the spurious high energy photons and thus its

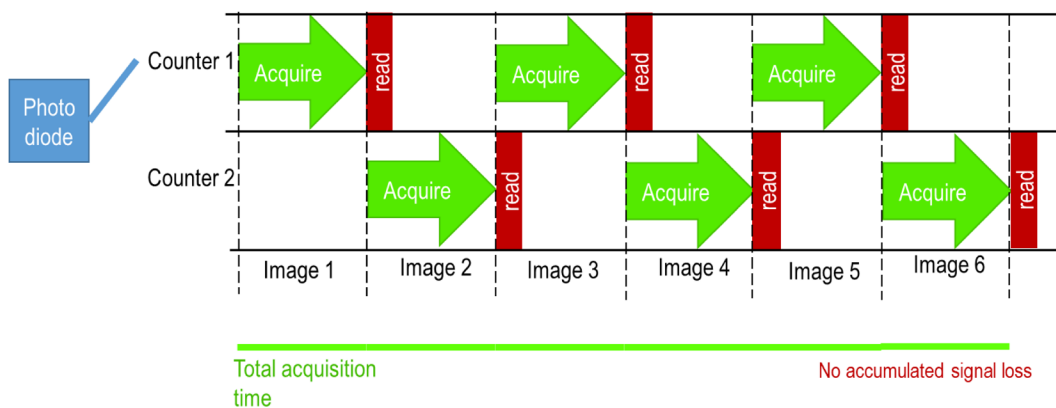


Fig. 14. Zero dead time operating mode in which counters are alternated rapidly to reduce dead-time to the nanosecond timescale.

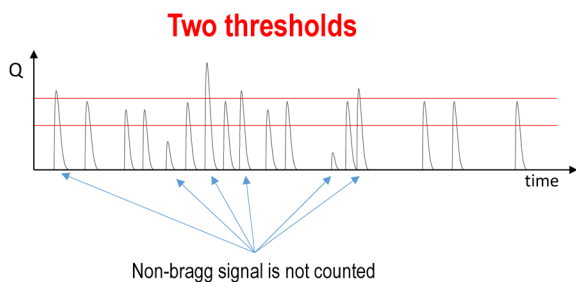


Fig. 15. Dual thresholding for elimination of low and high energy contamination.

counts may be subtracted out of the final result giving only photons within a band of energy defined by the low energy threshold and the high energy threshold.

Another possible application of differential counting is the potential for simultaneous dual wavelength experiments. In such an experiment, both X-ray sources in a dual source system are active simultaneously with the energy thresholds used to separate the signals.

3.2. Detector models and specific features

3.2.1. HyPix-Bantam

The HyPix-Bantam is a 38.5 × 77.5 mm detector which features air cooling and a simple Ethernet connection to the diffractometer control PC. The HyPix-Bantam uses the same HPC counting method though features simplified electronics and engineered to provide a high performance HPC detector whilst remaining affordable.

3.2.2. HyPix-6000HE

The HyPix-6000HE detector is 77.5 × 80.3 mm and features a fast 100 Hz frame rate and is also air cooled. To support high frame rates, a frame-grabber PC is used to collect and transfer images to the diffractometer control PC. The HyPix-6000HE frame-grabber performs noiseless image pileup on the frame-grabber, to provide the benefits of fine sliced data collection whilst reducing the dataset footprint and network transfer demands.

3.2.3. HyPix-Arc150°

The HyPix-Arc150° is a large area HPC detector. Offering 3 times the active area of the HyPix-Bantam



Fig. 16. The HyPix-Bantam HPC X-ray detector.



Fig. 17. HyPix-6000HE 100 Hz X-ray detector.

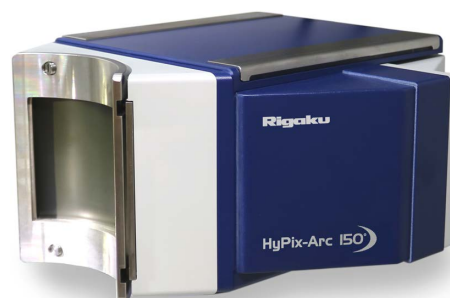


Fig. 18. The curved HyPix-Arc150° detector.

and 1.5 times the area of the HyPix-6000HE, it operates with the same approach as the HyPix-6000HE, using a frame grabber for image acquisition, pileup and transfer. With a larger area and more electronics, the detector is

water cooled, fully hermetically sealed and offers 70 Hz frame rates.

The HyPix-Arc 150° is the first home lab HPC detector that is arranged on a curve to provide higher coverage than flat detectors. This enables more intelligent use of the available active area and outperforms an equivalent area flat detector, performing similarly to large flat detectors offering >25,000 mm² active area with less than half the size.

The HyPix-Arc 150° can see data from 0° up to >150° from a single position (Fig. 19) with lower obliqueness (Fig. 20) for diffracted beams arriving at the detector compared with large flat detectors. This range is sufficient for collecting data up to IUCr guideline resolution for Cu K α as well as high resolution quantum crystallography measurements with molybdenum or silver radiation. This single theta approach allows data to be collected with as few frames as possible meaning lower X-ray dose on the sample. Corrections are also reduced with all data measured on the same scale and with a short time window for minimising sample decay, changes in sample conditions, and avoiding issues

associated with longer term experiments such as icing of cryo-devices.

4. Experimental Results

To demonstrate the capabilities of the XtaLAB Synergy diffractometers and HyPix detectors, an extremely challenging case was selected. Recent work in electron diffraction has pushed towards micron sized crystals and suggestions have been made⁽¹³⁾ that such sample sizes are not possible with current X-ray diffractometer technology. To demonstrate the cutting edge performance of the XtaLAB Synergy diffractometer, a small crystal of paracetamol (also known as acetaminophen) measuring only 3 × 2 × 1 μm was selected for measurement. In order to successfully measure such a crystal, the properties describe previously: high flux source, accurate X-ray photon counting detectors and a precise, stable goniometer.

The XtaLAB SynergyCustom with an FR-X Cu X-ray source with the HyPix-Arc150° detector was used for the first measurement. To demonstrate capability, a

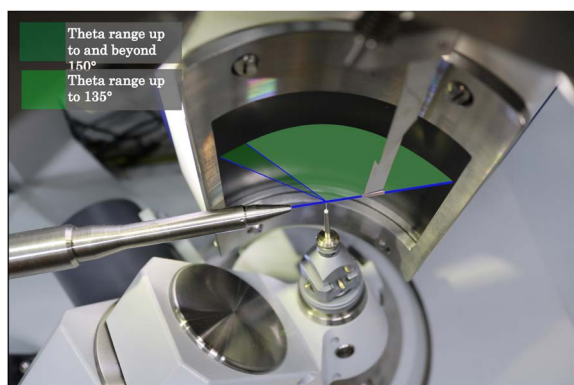


Fig. 19. High angular coverage offered by the HyPix-Arc150°.

Table 1. Experimental results on small crystals with XtaLAB Synergy diffractometers.

Instrument	FR-X + HyPix-Arc150°	Synergy-R	Synergy-S
Measurement temperature	100K	100K	100K
Sample size (μm)	3 × 2 × 1	4 × 4 × 3.5	30 × 16 × 6
Experiment type	WIT	WIT	WIT
Total experiment time	34 m 20 s	2 h 38 m	7 m 33 s
Final R1	7.21	4.45	4.88

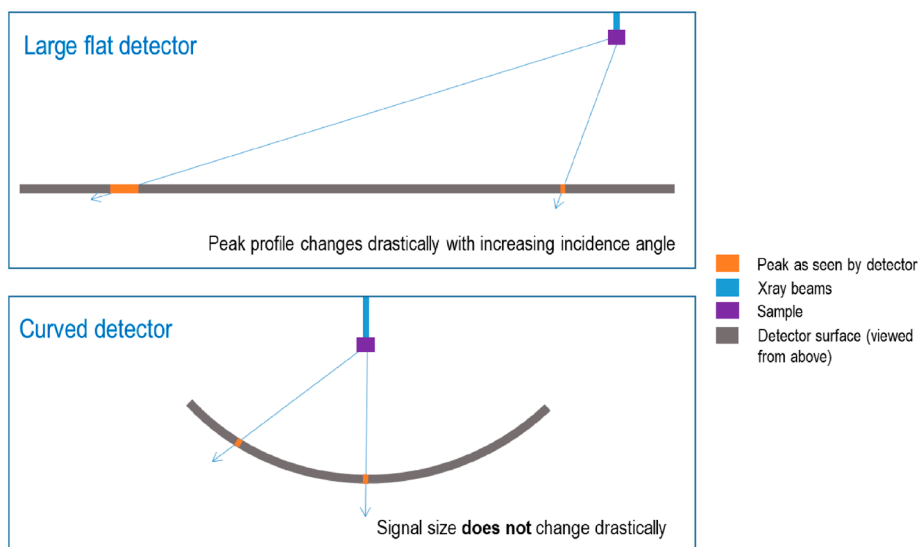


Fig. 20. Flat detectors suffer from higher oblique angles at their edges vs a curved detector. Lower obliqueness means fewer corrections are required for better data quality.

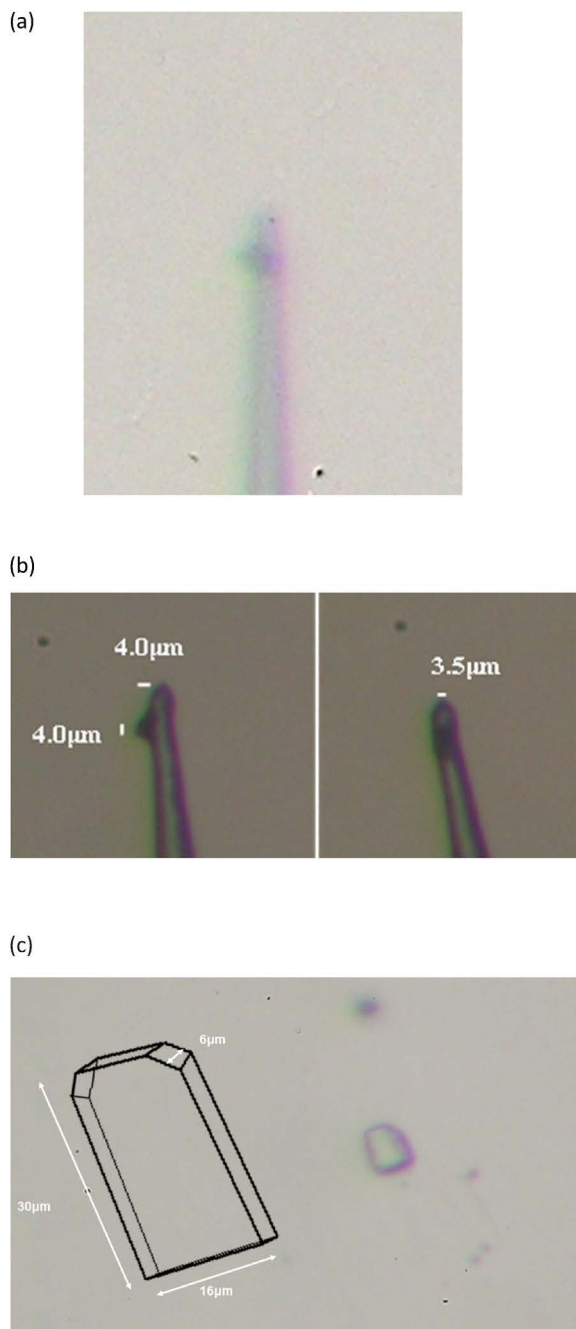


Fig. 21. The crystal samples used for (a) XtaLAB SynergyCustom, (b) XtaLAB Synergy-R, and (c) the XtaLAB Synergy-S.

“What is this?” experiment was conducted. This aims to get structure connectivity as fast as possible. Despite the measurement taking only 34 minutes and 20 seconds, a complete structure with an R-factor of 7.21% was possible. This result clearly demonstrates that micron sized crystals are measurable on cutting edge X-ray diffractometer equipment.

Similar experiments on paracetamol were conducted for Synergy-R and Synergy-S though due to lower source flux more suitable crystal sizes were selected, increasing with decreasing source brilliance. The results are tabulated in Table 1.

The results show, that micron scale crystals are

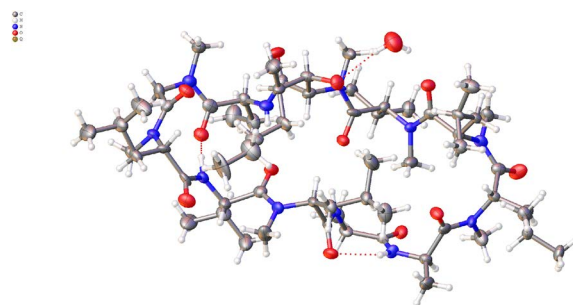


Fig. 22. Structure of cyclosporine A as measured from a $25 \times 10 \times 6 \mu\text{m}$ crystal on a XtaLAB SynergyCustom.

Table 2. Results of data collection on cyclosporine A collected with a XtaLAB SynergyCustom.

Total data collection time	1 h 53 m
Resolution	1 Å
Redundancy	5.6
I/σ (last)	12.87 (2.51)
Rint	0.094
R1	5.80%

possible on all XtaLAB Synergy instruments.

Further to these proof of principle measurements, a full experiment was conducted on a small cyclosporine A crystal measuring $25 \times 10 \times 6 \mu\text{m}$. A goal was set to collect the best possible dataset on this crystal in under two hours. The sample diffracted to 1.00 \AA and data were processed with a resolution cut-off set a 0.95 \AA . The quality of diffraction was good enough for hydrogen atoms to be visible but in this case they were not refined. The structure is presented in Fig. 1 and shows good atomic displacement parameters which are physically reasonable.

5. Conclusions

The XtaLAB Synergy diffractometers are built with on the combined knowledge and expertise of Rigaku and Oxford Diffraction independently developing diffractometers for decades. The combination of these two independently evolved diffractometers and the intellectual property behind them has allowed for the selection of the best component in each case resulting in an overall higher quality and performance solution. Rigaku diffractometers are designed to give the highest quality results by first establishing a stable and reliable platform and then building the highest performance components onto it.

References

- (1) M. Meyer: *Construction of a multi-purpose X-ray CCD detector and its implementation on a 4-circle kappa goniometer* (1998).
- (2) M. Meyer, G. Chapuis and W. A. Paciorek: *Acta Crystallographica A*, **55** (1999), 543–557.
- (3) M. Kitada and Y. Shimazaki: *Magnet proof magnetic fluid sealing device* U.S. Patent 6,247,701, 2001-06-19.
- (4) M. Nonoguchi and M. Kuribayashi: *X-ray tube*. U.S. Patent

- 7,333,592, 2008-02-19.
- (5) N. Osaka and T. Kobayashi: *Rotating-Anode X-ray Tube*. U.S. Patent 5,579,364, 1996-11-26.
- (6) M. Kuribayashi, M. Nonoguchi, N. Osaka and Y. Kobayashi: *Filament for X-ray tube and X-ray tube having the same*. U.S. Patent 7,352,846, 2008-04-1.
- (7) M. Kuribayashi and T. Chaki: *Rotary current-collecting device and rotating anode tube*. U.S. 7,005,774, 2006-02-28.
- (8) Y. Shimazaki and K. Akiyama: *Magnetic fluid sealing device*. U.S. Patent 7,950,672, 2011-05-31.
- (9) M. Sakata, T. Chaki, M. Okazaki, Y. Kusaka, S. Umegaki, A. Hamanaka and M. Nonoguchi: *Rotating anode X-ray tube and X-ray generator*. U.S. Patent 7,197,117, 2007-03-27.
- (10) B. Verman and L. Jiang: *X-ray optical system with adjustable convergence*. U.S. Patent 7,245,699, 2007-07-17.
- (11) P. Delpierre: *Journal de Physique IV*, **4** (1994), 11–18.
- (12) B. Henrich, A. Bergamaschi, C. Broennimann, R. Dinapoli, E. F. Eikenberry, I. Johnson, M. Kobas, P. Kraft, A. Mozzanica and B. Schmitt: *Nuclear Instruments and Methods in Physics Research Section A*, **607** (2009), 247–249.