XtaLAB P200

Small molecule systems based on hybrid pixel array technology

The highest quality data of any small molecule system in the world

Rigaku

The future is here

The field of chemical crystallography is constantly evolving but the needs of the chemical crystallographer remain the same: high quality data in as little time as possible. Rigaku uniquely addresses this issue by approaching the problem from two directions.

First the detector. Rigaku employs the PILATUS 200K hybrid pixel array detector (HPAD) manufactured by DECTRIS®, Ltd. as the primary detector in small molecule systems. The HPAD directly detects X-rays and, since its introduction, has revolutionized data collection at synchrotron beamlines all over the world.

The technology behind the PILATUS 200K is dramatically different from what is used in phosphor-based CCD detectors and phosphor-based CMOS detectors. In phosphor-based detectors, X-rays are converted to light, which then travels through a glass stub or taper and finally reaches a CCD or CMOS chip where it is accumulated until read. The final step in the signal processing chain is an analog to digital conversion. Each step in the detection process adds to the noise of the signal and hurts the ability to measure weak signals.

With a hybrid pixel array detector, the X-rays are measured directly with a solid state sensor. Each X-ray photon that hits the detector creates a large number of electron-hole pairs, with the charge being directly transferred to the CMOS chip and measured without the need for analog to digital conversion. Hybrid pixel array detectors feature very high dynamic range, extremely low noise, and hence achieve optimal signal-to-noise ratio at short readout time and high frame rates.

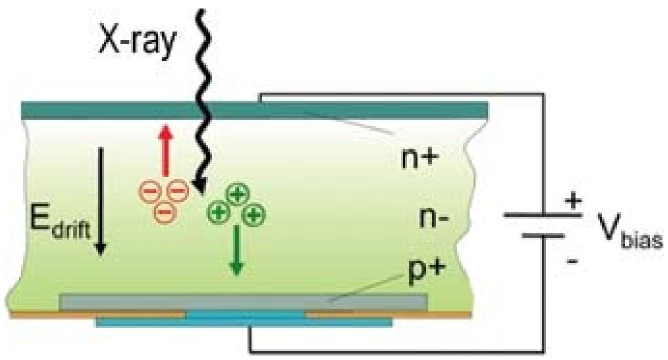


Figure 1. Principle of direct detection of X-ray photon in a solid-state sensor.

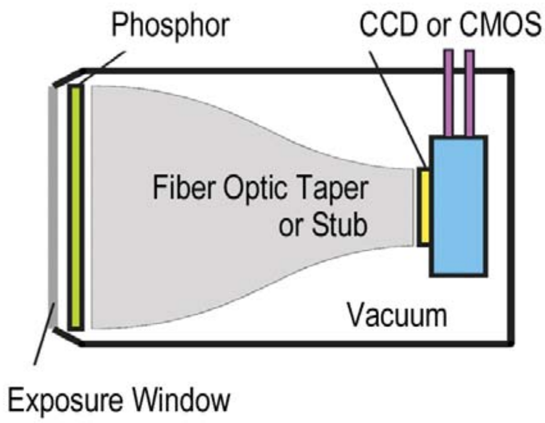


Figure 2. Principle of a phosphor-based detector. X-rays hitting a phosphor are converted to light, some of which travels down a glass connector to an integrated circuit (CCD or CMOS).

The benefits to the chemical crystallographer are very significant. The low-noise characteristics of the detector mean that the ability to measure weak reflections accurately is improved. This can be seen in the measurement of higher resolution reflections for weakly diffracting crystals. One of the most important classes of compounds being studied today are Metal Organic Frameworks (MOFs). These structures tend to be large and the crystals often diffract poorly. A PILATUS 200K detector is the perfect device to optimize the weak diffraction and improve the success of structure solution. Another example of where the accurate measurement of weak data is important occurs with superlattice structures, structures that involve weak interstitial reflections that must be accurately measured to properly describe the special structure.

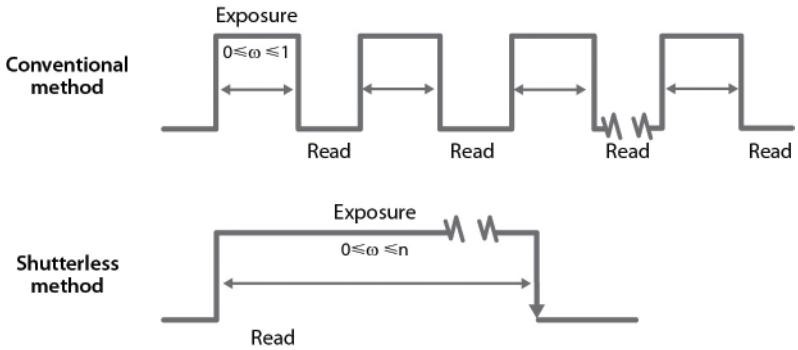


Figure 3. Comparison of conventional data collection versus shutterless data collection.

In addition to the outstanding low-noise characteristics, the PILATUS 200K allows you to collect data very quickly without having a negative impact on your data quality. The PILATUS 200K is a photon counting device, which means that it does not accumulate data before being read out. There is essentially no deadtime during readout so the most efficient way to collect data is to open the shutter, spin the crystal and measure data. The time savings are illustrated in Figure 3. In conventional data collection mode, the shutter is opened, the angle scanned, the shutter is then closed while the detector is read out. Each shutter open/close operation, coupled with an oscillation axis start/stop, introduces error into the data. Shutterless data collection removes those errors as well as removing the deadtime from the experiment. The result is better quality data in a shorter period of time.

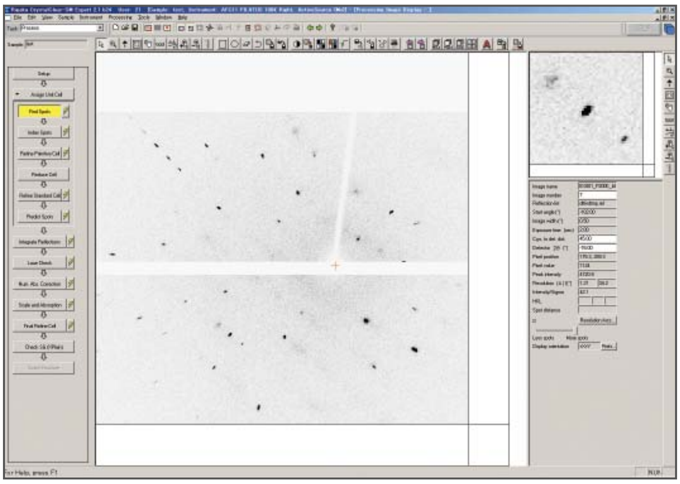


Figure 4. CrystalClear™ data collection and processing software displaying an image from the PILATUS 200K.

Data quality and speed

An X-ray system is only as good as the data collected and the structures solved and published. The current technologies of imaging plate, CCD and phosphor-based CMOS detectors have all been proven to measure good, publishable data. Is there a better way? Can you measure weak data accurately? Can you improve data quality while actually reducing data collection time? The answer is "yes". With a system based on a direct detection detector, you can improve data quality and significantly improve data collection times.

Table I. Comparison of Saturn 724 HG CCD and PILATUS 200K

	Saturn 724 HG	PILATUS 200K
Rmerge	2.10%	1.60%
R1	2.76%	2.76%
Total time	2H27M	0H25M

Data sets were collected on a crystal of cytidine utilizing a CCD detector (Saturn 724 HG) and a HPAD detector (PILATUS 200K). Two scans were performed on a crystal size of 0.22 x 0.22 x 0.22 mm and the exposure time of 4 sec/image.

As seen in Table I, the merging statistics for the PILATUS 200K data are significantly better than from the data measured with the Saturn 724 HG. This is exactly the sort of improvement that you would expect from an improvement in weak data measurement and the subsequent improvement in sigmas. R1 is the same for the two data sets, again this is to be expected from a well diffracting crystals where strong reflections will dominate. Finally, the full advantage of the PILATUS 200K detector can be seen in the data set that only took 17% as long to measure for equivalent or better data.

The world's best detector coupled with the wi

Unique Advantage #1

True shutterless operation

Continuous acquisition of full images reduces the total measurement time, because there is no need to open/close the shutter during measurement.

Unique Adv

Direct detect

Direct detection sensor technology
from being introduced by scintillators
gain variations

FR-X microfocus rotating anode source

MicroMax-007 HF microfocus rotating anode source

MicroMax-003 microfocus sealed tube source

Unique Advantage #4

Excellent point spread function

A sharp point-spread function (one pixel) means that reflections are sharper and easier to resolve.

How is the cycle ti

- **No dark image**
- **No overload imag**
- **No dezinger imag**
- **No shutter dead ti**
- **No significant read**

idest range of X-ray sources and goniometers

vantage #2

tion of X-rays

prevents a number of image artifacts and fiber optics, such as fixed patterns, and distortion.

Unique Advantage #3

Extremely low noise

Since the PILATUS 200K is a photon counting device, it avoids noise problems that are inherent in the design of integrating detectors. CCD and CMOS active pixel detectors, accumulate undesired signal during the integration process.

Fixed χ three-axis

XtaLAB P200 MM003

Partial χ four-axis

Kappa goniometer

me reduced?

es

es

me

d time of detector

Unique Advantage #5

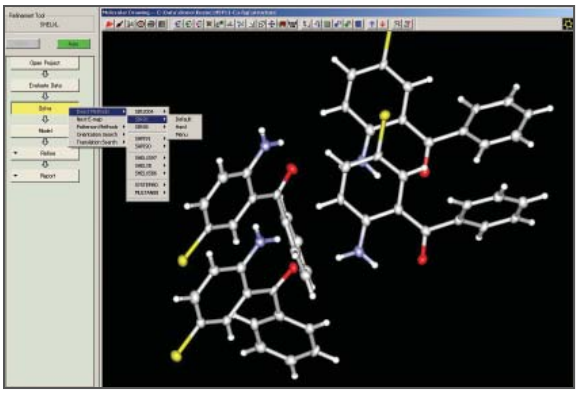
Wide choice of configurations

With three different goniometer choices and ten different source choices, the perfect system can be configured to meet the needs of any research lab.

Unprecedented flexibility

Structure solution software

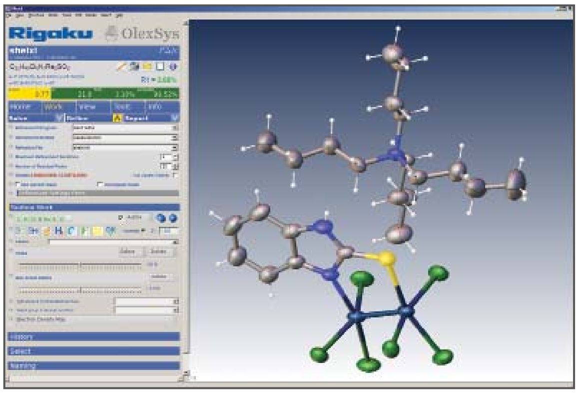
Structure solution and analysis are provided by Rigaku's CrystalStructure™ program suite. This flexible package includes multiple structure solution programs so that you can try various techniques when faced with a difficult problem. Both the CRYSTALS and SHELX programs (including the new SHELX 2013) are supported so that you are ensured of working with software that is accepted around the world.



CrystalStructure

In addition to CrystalStructure, Rigaku provides a special version of the popular small molecule package, Olex2. Olex2 is an easy-to-use program for small-molecule structure solution and refinement. It also includes many useful tools for structure analysis, archiving and report generation. All aspects of the structure determination and publication process are presented in a single, workflow-driven package. An active online user community ensures access to Olex2 experts.

Olex2 was created by Oleg Dolomanov and Horst Puschmann at Durham University, UK. Olex2 uses smtbx, a toolbox for small molecule crystallography. This development is driven by Luc Bourhis along with Richard Gildea, who also helps with maintaining the GUI. Olex2 is an open source project and is widely supported by the crystallographic community.



Olex2

XtaLAB P200 MM007HF

XtaLAB P200 DS

X-ray sources

Rigaku developed the first rotating anode generator in 1952 and has been pioneering X-ray source technology ever since. Researchers should carefully evaluate the best X-ray source to use in their specific research. An X-ray source is more than just the X-ray generator; an X-ray source is the expertly selected X-ray generator beam size coupled with the appropriate synthetic multilayer optic. From the low-maintenance microfocus sealed tube X-ray sources to the highest power rotating anode generators, Rigaku has the technology that allows you to customize your X-ray system to suit your research needs. Single source and dual source systems are available and dual wavelengths and high total flux are both considerations in selecting a source. Both micro-focus rotating anode generators are available with dual banded anodes, providing both the flexibility of a dual wavelength system, with the extraordinary flux of a micofocus rotating anode generator.

Hardware configurations

Table II. PILATUS 200K	
Detector type	Hybrid pixel array detector
Active area	83.8 x 70.0 mm^2
Point spread function	1 pixel
Pixel size	172 x 172 μm^2
Sensor thickness	1 mm
Dynamic range	1.04 x 10^6:1
Cooling system	Air-cooled

Table III. Goniometer specifications

Goniometers	Fixed χ goniometer	Partial χ goniometer	Kappa goniometer
ω-axis	2θ ±90°	2θ ±90°	2θ ±90°
χ-axis	45°	0 to 60°	N/A
φ-axis	-20 to +300°	-360 to +360°	-360 to +360°
κ-axis	N/A	N/A	-180 to +180°

Rigaku offers a number of X-ray sources to couple with the XtaLAB P200:

Standard sealed tube X-ray sources

- Fine Focus sealed tube with standard graphite monochromator (Cu or Mo)
- Fine Focus sealed tube with SHINE optic (curved graphite), Mo only

Standard rotating anode X-ray sources

- MultiMax9 fine focus rotating anode generator (5.4 kW) with standard graphite monochromator, Mo or Cu
- MultiMax9 fine focus rotating anode generator (5.4 kW) with SHINE curved graphite monochromator, Mo only

Microfocus sealed tube and standard sealed tube X-ray sources

- MicroMax-003 microfocus sealed tube generator (Cu or Mo) with multilayer optics
- Dual Source configuration with two MicroMax-003 sources – one Mo and one Cu

Combined microfocus sealed tube and standard sealed X-ray sources

- Dual Source configuration with a Cu MicroMax-003 source and a Mo fine focus sealed tube source with SHINE optic

Microfocus rotating anode X-ray sources

- MicroMax-007 HF microfocus rotating anode generator and multilayer optics. Various target materials available
- MicroMax-007 HF DW microfocus rotating anode generator, dual target anode and dual wavelength multilayer optics (Different combinations are available, e.g. Cu/Mo or Cu/Cr)
- FR-X ultra high brilliance microfocus rotating anode generator with multilayer optics
- FR-X DW ultra high brilliance microfocus rotating anode generator, dual target anode and dual wavelength multilayer optics (Different combinations are available, e.g. Cu/Mo or Cu/Cr)

Table IV. Relative flux at sample for a 100 μm sample

	Fine focus sealed tube (2 kW)	Fine focus sealed tube (2 kW) with SHINE	MultiMax9 fine focus (5.4 kW)	MultiMax9 fine focus (5.4 kW) with SHINE	MicroMax-003 (30 W)	MicroMax-007 HF DW with VariMax™ DW (1.2 kW)	FR-X (3 kW)
Cu	1.0	–	–	–	11.8	87.1	224.3
Mo	1.0	1.7	4.7	8.0	1.7	20.0	51.6

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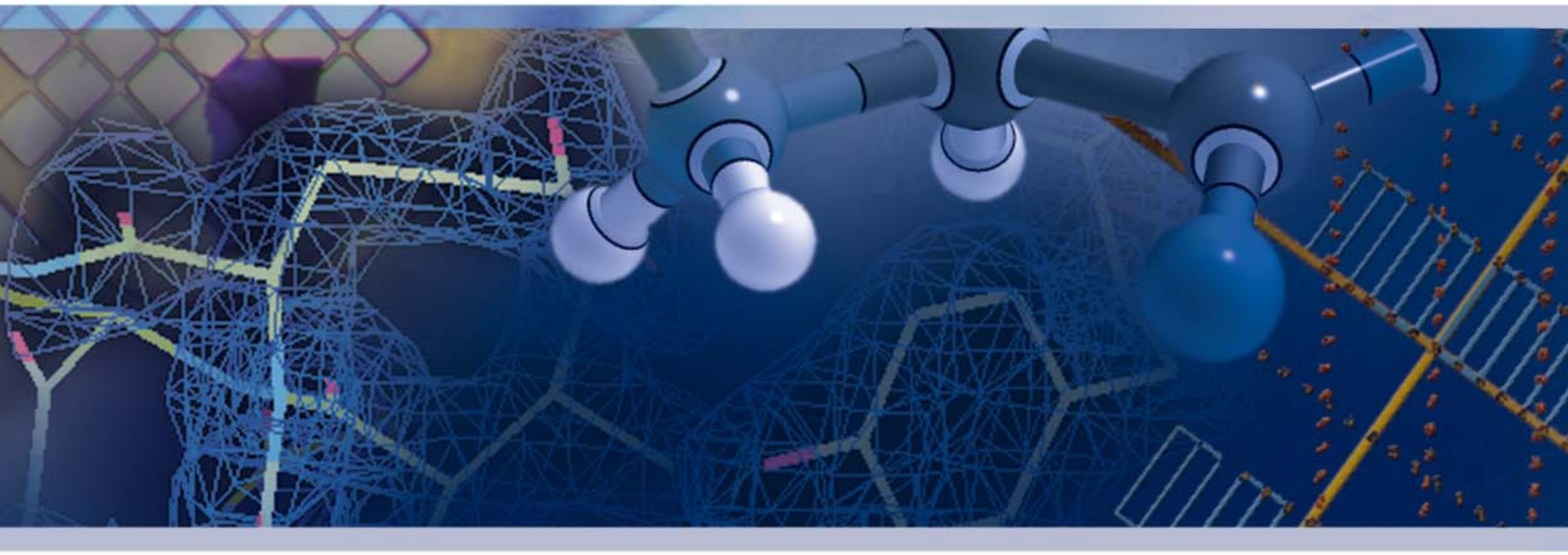

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