XtaLAB PRO series

Single crystal diffractometers

Hybrid pixel array detector based diffractometers

Rigaku

Better measurements. Better confidence. Better world.

A diffractometer based on an outstanding detector

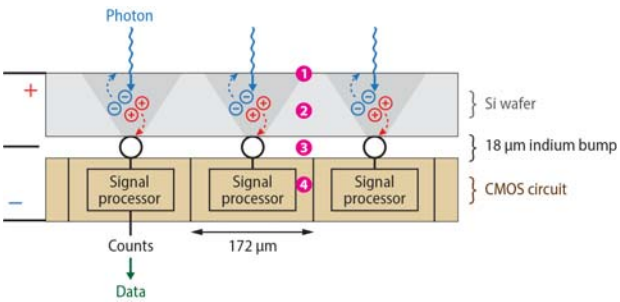
The XtaLAB PRO series

The XtaLAB PRO series of diffractometers addresses a wide range of sample types, from small molecules to MOFs, to biological macromolecules. The key component that is common for this series of diffractometers is the use of a HPAD (hybrid pixel array detector) detector. HPAD technology produces an almost perfect detector and greatly expands the capabilities of a single crystal diffractometer in terms of speed of data acquisition and more accurate measurement of weak data. The standard detector in the XtaLAB PRO series is the PILATUS 200K, which is well proven in the field and based on the same technology adopted by synchrotron beamlines around the world. The outstanding characteristics of these detectors ensure that every XtaLAB PRO diffractometer will produce the best data possible for the X-ray source selected.

At the core of the XtaLAB PRO is the detector

There are basically four types of detectors currently being used with single crystal diffractometers: HPAD, CCD, CMOS, and IP. The HPAD (Figure 1) is the newest technology and is unique in that it is a photon counting detector that directly detects X-ray photons without the intermediate step of converting the photons to light with a phosphor. The HPAD detector allows for shutterless data collection due to the combination of extremely fast readout and high dynamic range. In addition the extremely low detector noise means that you can count poorly diffracting samples for a long period of time without swamping the signal with electronic noise. The combination of these features means that you will collect better data faster.

HPAD detector



1. **X-ray photon absorbed in the Si wafer.**
2. **Electron hole pairs are generated and funneled towards Indium bump.**
3. **Si wafer is connected to CMOS circuit by Indium bump for effective charge transfer.**
4. **Pixel signal processing unit, shapes and amplifies then compares vs reference value. If above, it is counted, if below not counted.**

Figure 1. How an HPAD detector works

How shutterless data collection reduces data collection times

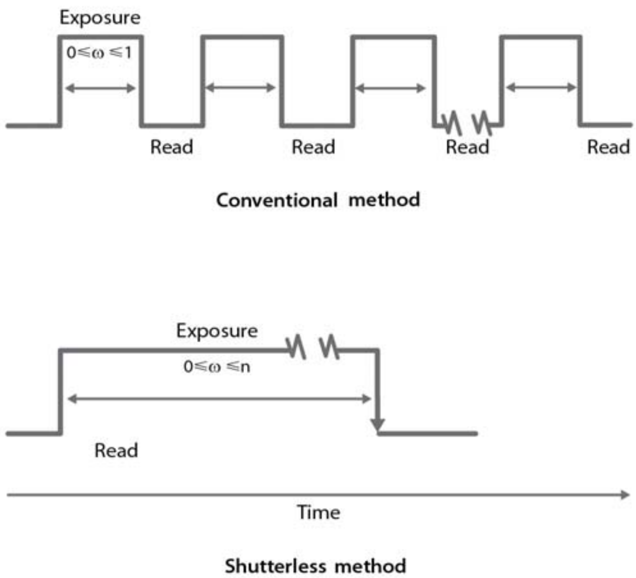


Figure 2. Comparison of conventional data collection versus shutterless data collection

Figure 2 illustrates the difference between the conventional data collection mode of a CCD detector and that of an HPAD detector. The readout speed of the HPAD is so fast that for a given angular scan, the shutter is opened, the scan angle rotates, and the detector is read at appropriate time intervals to generate a set of scan images. The high dynamic range of the HPAD detector eliminates the need to treat overloaded reflections, and is one of the reasons it is a popular technology at synchrotrons. With a CCD detector, an angular segment is measured with the shutter open. The shutter is then closed and the CCD is read out. The relatively low dynamic range of a CCD detector means that either a fast pre-scan must be performed to check for overloaded reflections so that the appropriate CCD binning can be set or, in case an overload is detected, the angular segment must be rescanned at a higher speed. Either way, the relatively high readout speed and relatively low dynamic range render CCD detectors incapable of shutterless data collection and leads to longer data collection times.

In principle CMOS detectors are capable of performing shutterless data collection. Due to the skewed nature of the detector readout (one line at a time), there appears to be inherent problems in processing such data and it does not appear to be a data collection method that can be routinely used to produce higher quality data. With HPAD detectors, shutterless data collection always produces equal or better data as compared to a conventional data collection method.

Accurate measurement of weak data

Another important characteristic of the HPAD detector is the fact that the noise level is essentially zero (no detector is perfect and so it is not correct to call it a noiseless detector, but the HPAD is the closest thing to a noiseless detector available today). A detector with extremely low noise is important because it means that you can measure weak reflections more accurately because backgrounds are as low as possible. Any crystal will have a combination of relatively weak and strong reflections but crystals that most benefit from the accurate measurement of weak reflections are crystals that scatter poorly. With an HPAD detector, the low noise characteristic means that you can expose a crystal for significantly longer periods of time than either a CCD or a CMOS based detector. There is no significant build up of noise with the HPAD detector and you can measure diffraction from poorly diffracting crystals that would be swamped with electronic noise in other types of detectors.

Peak shape defined by the pixel size

People are often confused when they first view the peak shape of a reflection measured with an HPAD detector. Because the X-rays are detected directly, you do not see the "blooming" associated with phosphor based detectors (Figure 3a). Integrating detectors such as CCDs and standard CMOS detectors, require a phosphor and glass stub assembly or fiber optic taper for converting X-ray photons to light. Light loss occurs at the interfaces between the phosphor-stub and stub-sensor as well as in the glass material itself. Diffusion of light in the phosphor and loss of light through the fiber optic stub means CCDs and standard CMOS detectors have a point spread function with longer tails than a Gaussian such that data is spread across many pixels (Figure 3b). As a result of no light diffusion within the detector, PILATUS spots have a 'top-hat' point spread function of one pixel rather than the long tail PSF seen with CMOS and CCD detectors (Figure 4). The lack of the long tail point spread function means that HPAD based detectors can easily resolve large unit cells. For example, a unit cell length of 462Å was resolved for a crystal of mouse angiotensinogen using Cu radiation.

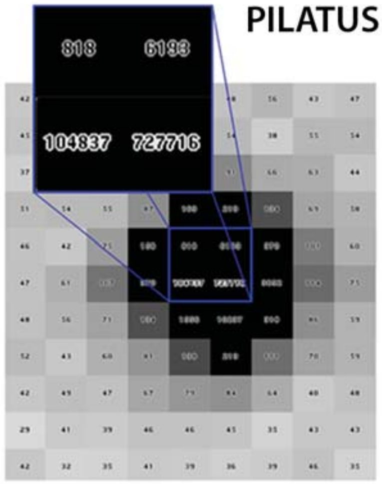


Figure 3a. Typical peak shape from an HPAD detector

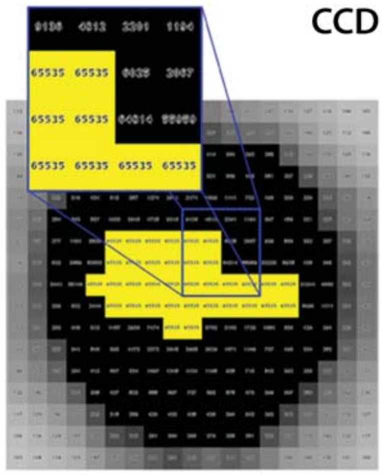


Figure 3b. Typical peak shape from a CCD detector

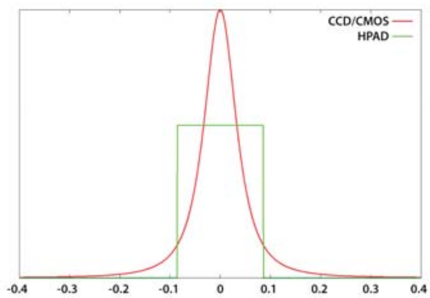


Figure 4. Comparison of a "top-hat" point spread function and a phosphor based detector

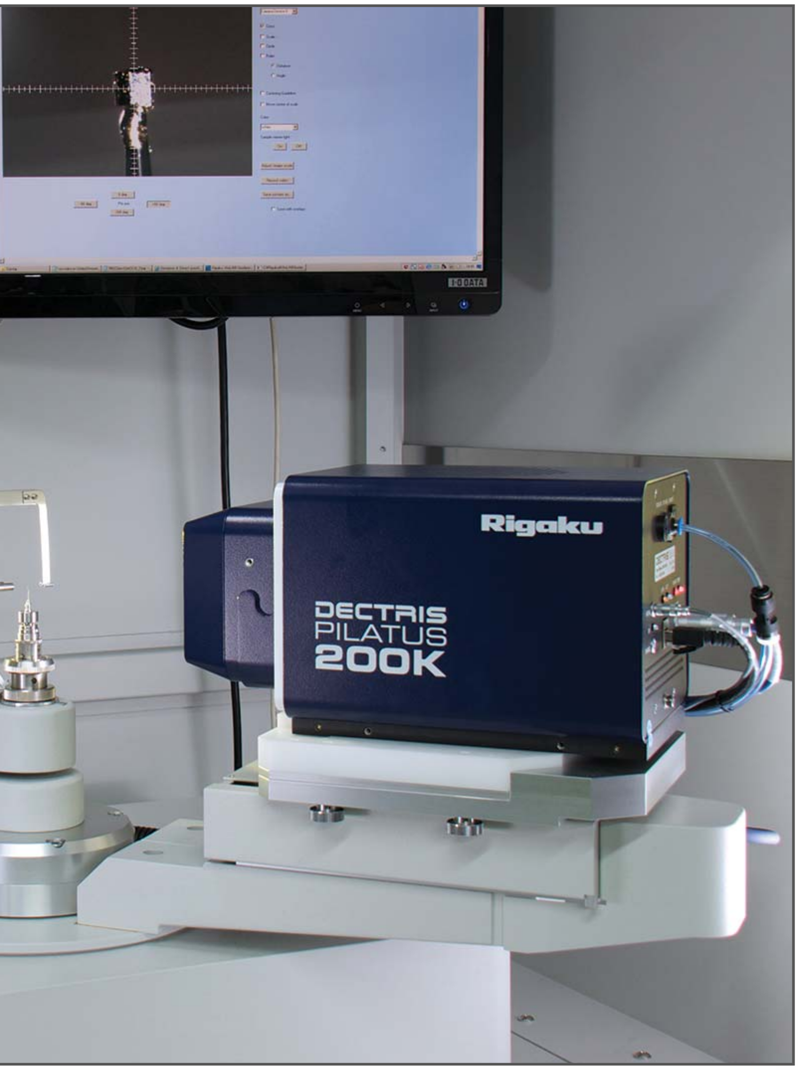
The standard XtaLAB PRO configuration: Ultimate flex

The Hybrid Pixel Array Detector features

- Extremely low noise
- High dynamic range
- Direct detection of X-rays
- Fast readout speed
- Shutterless data collection

The kappa goniometer a

xibility for your research needs



The dual source configuration provides the ability to switch between Mo and Cu radiation easily. Other X-ray source options include the MicroMax™-007 HF rotating anode (single or dual wavelength) or the FR-X rotating anode generator.

allows easy access to the crystal

The XtaLAB PRO series is housed in a newly developed enclosure that was designed to improve the workflow of mounting air-sensitive and temperature sensitive samples. There is space inside the XtaLAB PRO enclosure for a microscope and a dewar. No matter what type of samples you are working with, the ability to identify and mount crystals in the proximity of the diffractometer can be a true time saver. In the case of air-sensitive and temperature-sensitive crystals, having close proximity of the mounting station to the goniometer can mean the difference between a crystal that diffracts and a crystal that dies.

X-ray source configurations

Ultimate source flexibility

In the XtaLAB PRO series, the selection of X-ray sources is quite diverse and allows one to select an X-ray source configuration based on the radiation type or types as well as the level of flux desired. The standard configuration for the XtaLAB PRO includes a Cu MicroMax-003 microfocus sealed tube source and Mo standard sealed tube source with a SHINE (curved graphite monochromator) optic. However, the XtaLAB PRO can also be configured with a single sealed tube X-ray source in the beginning and a second source can be added in the field at a later date. The XtaLAB PRO can also be configured with two microfocus sealed tube sources (Cu and Mo) or a number of different rotating anode sources if higher flux is important.

Microfocus sealed tube versus standard sealed tube sources

The selection of a microfocus Cu source and a standard focus Mo source is based on the difference in efficiency between Cu and Mo radiations when coupling micro focal spots with multilayer optics as well as the experimental advantages of a Mo standard focus source coupled with a curved graphite monochromator. Figure 5 shows the relative flux through a 100 µm aperture at the crystal position for both a standard sealed tube Cu source with graphite monochromator and a microfocus Cu X-ray source with a multilayer optic. The 12 fold improvement in flux at the sample makes the use of the Cu microfocus X-ray source an easy decision.

Figure 6 shows the relative flux through a 100 µm aperture at the crystal position for a standard sealed tube Mo source with graphite monochromator, a standard sealed tube Mo source with a curved graphite monochromator (SHINE optic) and a microfocus Mo X-ray source with a multilayer optic. It is easy to see that the standard Mo source with the SHINE optic produces equivalent X-ray flux through a 100 µm aperture as the microfocus Mo source. However if you look at the relative flux at the sample for crystals larger than 100 µm (Figure 7) you will see that the standard Mo source will provide more flux at the sample due to the larger beam size. In other words, the SHINE optic makes the standard Mo X-ray source equivalent to the microfocus Mo source for small samples, but for larger samples, the standard Mo source with a SHINE optic outperforms the microfocus Mo source. For laboratories that routinely see samples larger than 100 µm, the standard Mo source with a SHINE optic is the best choice. While the XtaLAB PRO can be equipped with a Cu and Mo microfocus source, the performance will not be improved and for larger crystals the performance will go down.

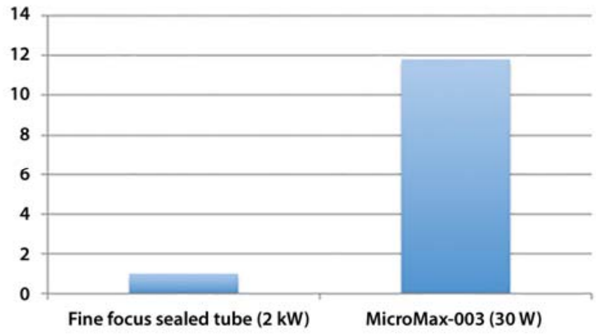


Figure 5. Comparison of Cu sealed tube sources

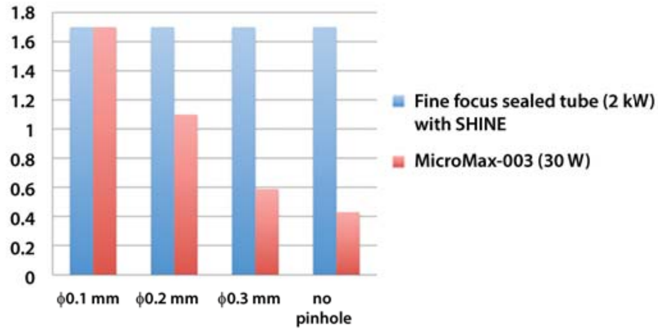


Figure 7. Relative flux through different size pinholes

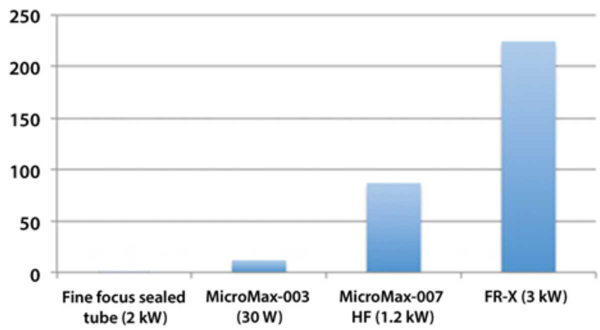


Figure 9. Comparison of all Cu sources

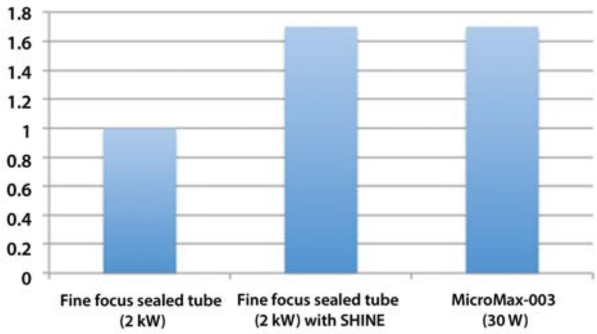


Figure 6. Comparison of Mo sealed tube sources

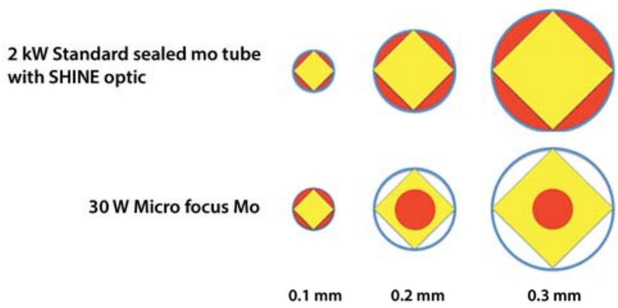


Figure 8. Beam diameter versus crystal size. The yellow squares indicate crystals of various sizes and red indicates the beam diameters.

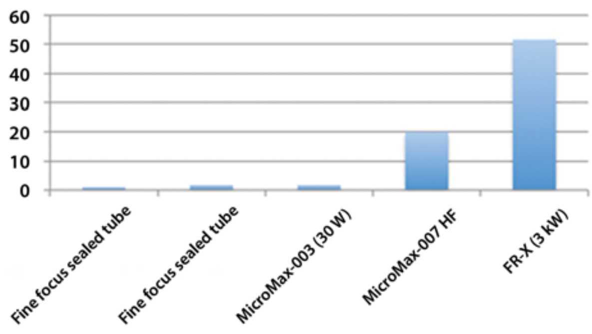


Figure 10. Comparison of Mo sources

Figure 8 illustrates the impact of using a beam diameter that is different than the size of the crystal. In the case of a Mo microfocus sealed tube and a crystal that is 300 µm in size, the X-ray beam only illuminates the center of the crystal. This means the scattering power of the crystal is reduced and that the diffracted X-ray reflections will be attenuated in ways not easily corrected for by absorption techniques. In contrast, a 300 µm crystal irradiated using a standard Mo X-ray source with a SHINE optic, will be fully bathed in the X-ray beam and variations in intensities of equivalent reflections will be easily modeled as absorption effects.

When crystals diffract poorly or highest throughput is a requirement

Beyond the standard configuration of the XtaLAB PRO, it is possible to increase the flux at the sample significantly by utilizing a rotating anode based X-ray source and Rigaku offers a number of models and configurations. The MicroMax-007 HF is a microfocus rotating anode generator and the most popular rotating anode source utilized for single crystal analysis around the world. It is available in both a single wavelength configuration as well as a unique double wavelength configuration. In the double wavelength configurations (Mo/Cu or Cu/Cr), the wavelength to be used can be selected automatically.

For even more flux at the sample, the FR-X microfocus rotating anode generator is available and is the most powerful rotating anode X-ray source for single crystal analysis available today. These two rotating anode sources are well proven in the field and offer a low-maintenance regimen compared to rotating anodes of the past. While the ongoing maintenance of a rotating anode generator is more than that of a sealed tube generator, the increase in flux at the sample is significantly higher and will allow you to measure samples that could only previously be measured at a synchrotron. Figures 9 and 10 show the relative flux through a 100 µm aperture at the crystal position for a standard sealed tube source, a microfocus sealed tube source, the MicroMax-007 HF, and the FR-X for Cu and Mo radiation respectively.

For Cu radiation, the 7 and 19 fold increases in flux for the MicroMax-007 HF and FR-X compared to the MicroMax-003 (microfocus sealed tube) is a clear indication of the performance improvements that are possible when using rotating anode based X-ray sources. It is even more significant for Mo radiation where 12 and 30 fold improvements can be gained from the MicroMax-007 HF and FR-X compared to a Mo sealed tube source with a SHINE optic or a Mo microfocus sealed tube source. Laboratories around the world are routinely using these high-flux rotating anodes to measure data on poorly diffracting crystals and for significantly increasing throughput and laboratory efficiency.

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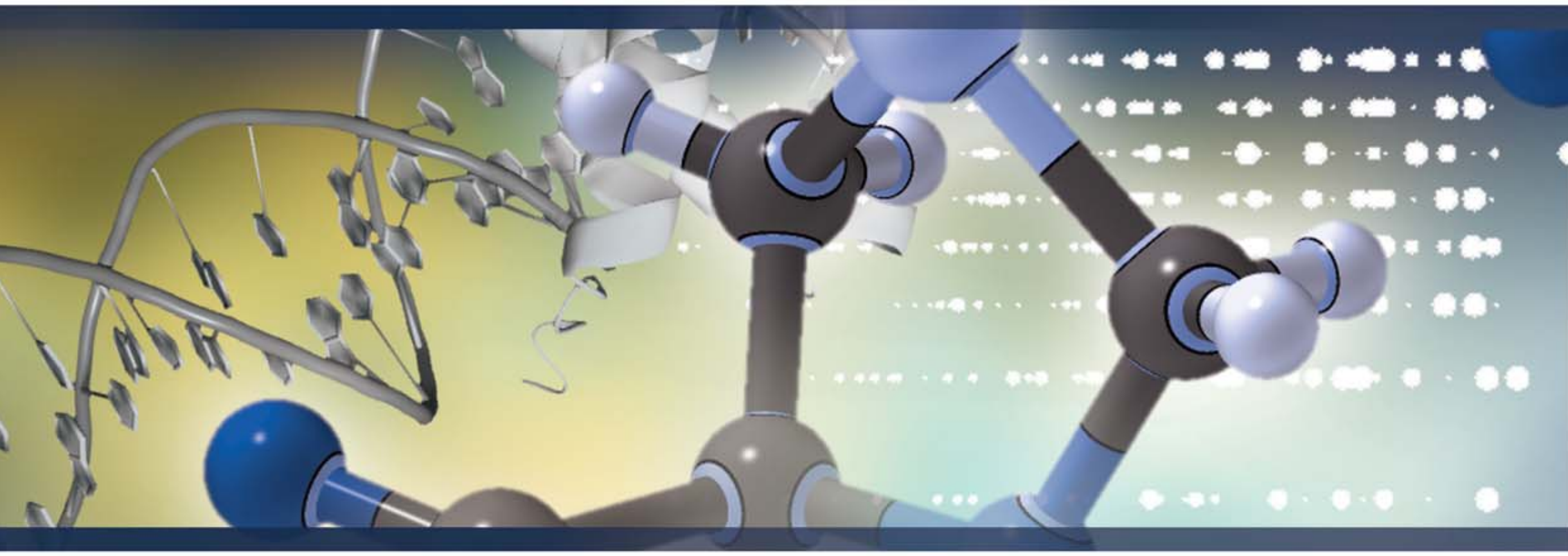

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