XtaLAB

Synergy

Single crystal X-ray diffractometer

Advanced technologies applied to single crystal diffraction

Rigaku
oxford diffraction

XtaLAB Synergy – Fast, Precise, Intelligent

CONTINUITY AND INNOVATION

The common goal of any single-crystal experiment is to efficiently and accurately measure reciprocal space data. This is true whether you are determining the structure of a novel chemical compound, screening a crystal before a synchrotron trip or measuring highly redundant, high-resolution data for a charge density study. In all cases, the quality of data generated by your diffractometer, as well as the speed and ease by which you can measure the data, is paramount to the success of your research. With your success utmost in our mind, we have produced the XtaLAB Synergy diffractometer, a combination of leading edge components and user-inspired software tied together through a highly parallelized architecture to produce fast, precise data in an intelligent fashion.

New micro focus X-ray sources incorporate new X-ray tubes, new optics and improved alignment mechanism.

KEY FEATURES

- User-inspired CrysAlis^{Pro} software tightly integrated with new goniometer features and highly parallelized for speed and throughput
- "What is This?" software function for small molecule structure determination in minutes
- New, third generation microfocus X-ray sources with longer tube life, higher-performance optics, improved alignment mechanism and, best of all, higher flux
- Single or dual source options (Cu, Mo or Ag) with 3-year tube warranty
- Wide range of detector options including various sizes of Hybrid Photon Counting (HPC) and CCD detectors
- New goniometer includes faster motor speeds, new telescoping 2θ arm, electronically controlled brightness of cabinet and crystal lighting, and ±2θ accessibility for more efficient data collection strategies
- New enclosure conforms to the most stringent of X-ray safety guidelines and offers ample experimental workspace and accessibility
- True shutterless data collection when HPC detector selected

GAP-FREE RECIPROCAL SPACE

There are two types of gaps in reciprocal space, and each type of gap must be accounted for in an ideal instrument. The first gap relates to the completeness of data collection and is a function of the strategy algorithm that is utilized and the hardware's ability to efficiently execute a strategy. The second gap relates to systematic under-measurement of a specific class of reflections which can occur when the noise level of a detector swamps the signal in weak reflections, for example.

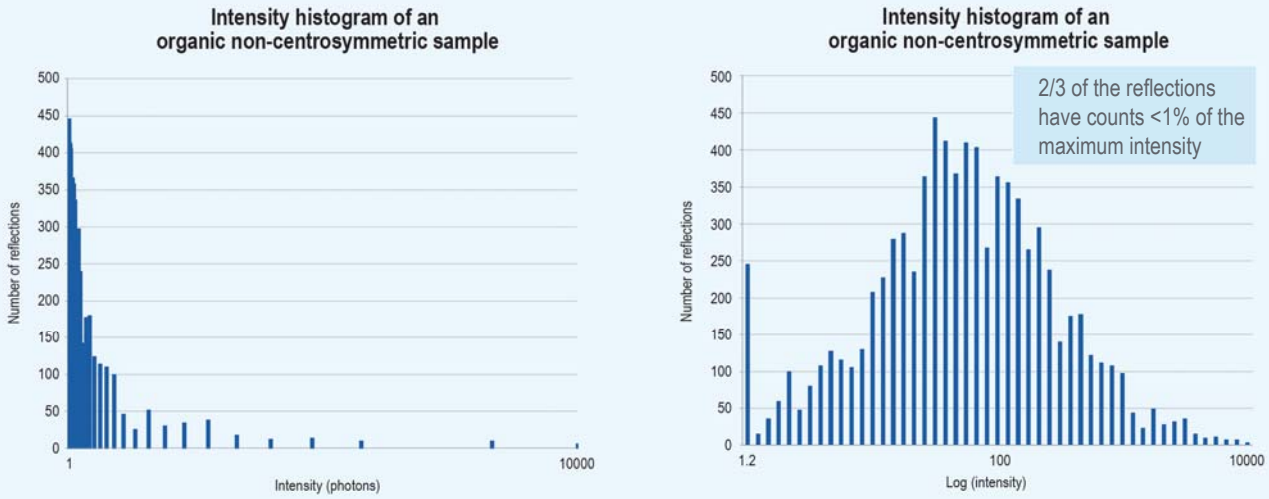
For some manufacturers' instruments, the process of calculating an efficient strategy is so arduous that users often resort to a canned set of scans that is known to always produce a complete set of data, albeit in an inefficient and time-consuming manner.

Our approach is different. Your time is valuable. Our strategy algorithm is quick, highly efficient and easy to use, and thus is a standard part of the workflow. The goniometer plays a key role in efficient data collection; thus we only offer the kappa-geometry goniometer. The kappa design is so much more efficient that it can measure reciprocal space up to 33% faster than with a fixed-chi design.

Gap-free reciprocal space refers not only to measuring a data set to IUCr acceptable completeness, but also to the proper measurement of weak reflections so that they have sufficient information content for use in refinement. A diffractometer that does not allow for accurate measurement of weak reflections introduces a gap in your measurement of reciprocal space. Detectors with a high-noise threshold are the biggest sources of such a gap. We avoid this problem with proper detector selection and proper adjustment of data collection parameters.

Any diffractometer can measure strong reflections well, but the true test of an X-ray detector is the ability to measure weak reflections well. By far the most important factor for accurately measuring weak reflections is the inherent DQE (Detective Quantum Efficiency) of the detector in the area of low count rates. The DQE for low level measurements (<100 photons) can drop off dramatically for certain detector types based on inherent noise limitations of the detector technology. Other detector types have the same DQE for one photon as for 10,000 photons.

THE IMPORTANCE OF WEAK DATA



Linear (left) and Log (right) plots of the intensity distribution for an organic structure, illustrating that the majority of reflections are weak, with counts less than 1% of maximim intensity.

To understand the importance of weak data in structure analysis, it is informative to look at a typical example. A data set was calculated for an organic non-centrosymmetric structure with an average $I/\sigma = 10$. As shown in the above plots of the intensity distribution as a function of reflection intensity, weak data dominate. The intensity distribution is strongly biased toward low intensities, with 2/3 of the data having intensities lower than 1% of the maximum intensity. Since the majority of reflections are weak, even in a well-diffracting crystal, proper measurement of these weak reflections is crucial to achieve a better refined structure.†

† Hirshfeld, F.L.; Rabinowich, D. T*reating Weak Reflexions in Least-Squares Calculations.* Acta Crystallogr. 1973, A29, 510–513.; Arnberg, L.; Hovmöller, S.; Westman, *S. On the Significance of 'Non-Significant' Reflexions.* Acta Crystallogr. 1979, A35, 497–499.

Thus, it is most important to give careful consideration to the choice of detector when configuring a diffractometer, as one should select the detector with characteristics that best suit your experimental needs. In order to give you that flexibility, Rigaku Oxford Diffraction offers two detector types with the XtaLAB Synergy systems: CCD and HPC detectors.

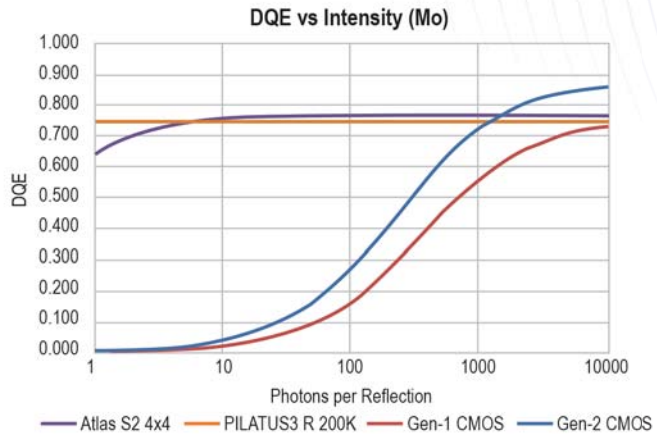
CCD detectors are classified as integrating detectors, and have been used in crystallography very effectively over the last 20 years. These detectors require a scintillator to convert X-rays to light and then a glass taper to transfer the light to the CCD sensor. By varying the taper size, CCD detectors can be built with different size active apertures, with the tradeoff that a larger aperture means the gain and sensitivity are reduced, but dynamic range will increase. With the innovative S2 Smart Sensitivity Control, which boosts our detector sensitivity by 50%, Rigaku Oxford Diffraction's CCD sensitivity is enhanced to allow for better data up to 2X faster.

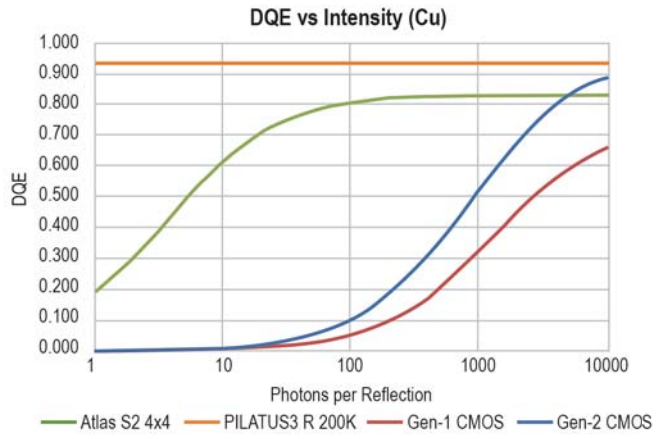
The Importance of Detector Selection

PILATUS3 R 200K

Eos S2

HPC detectors are characterized as hybrid detectors because they are comprised of both a silicon sensor and silicon electronics, connected at each pixel by a bump bond. HPC detectors are direct detection devices as opposed to integrating; X-ray photons are directly converted into electric charge in the solid-state sensor, counted and stored immediately. HPC detectors are event driven so noise is not accumulated.





The figures to the left compare DQE values of HPC, CCD and CMOS detectors. CMOS detectors, also referred to as Charge-Integrating Pixel Array Detectors (CPADs), are integrating detectors. The plot shows that typical CPADs have much worse DQE for weak reflections due to their high intrinsic noise compared to CCD and HPC detectors. This is especially true for the intensity range of data common to diffraction experiments.

In the case of HPC detectors, the high gain dominates the DQE equation and allows for high DQE for weak reflections. For CCD detectors, the read noise, though small, starts to become significant at 10 photons or less. Finally, for CMOS (CPAD) based detectors, the DQE rapidly falls off below measurements involving <1% of maximum intensity, the threshold below which 2/3 of the reflections fall in the in the example on page 2. The very rapid fall off is the result of the much higher noise—both read noise and dark current noise—inherent to the CMOS design.

DETECTOR OPTIONS

HPC or CCD: The choice is up to you

Atlas S2

Titan S2

RIGAKU OXFORD DIFFRACTION CCD DETECTORS offer a wide range of aperture sizes depending on your experimental needs—from the extra-high sensitivity Eos S2 to the Titan S2, a monolithic detector with the largest active area available today. These modern detectors are fast and sensitive over a wide range of X-ray energies, and are well suited for Ag radiation. They also have the capability to measure data with a very high dynamic range due to the instant binning control and software-controlled self-optimizing sensitivity. The actual effective dynamic range available with the S2 technology (two images with different sensitivity settings combined automatically) is 1 : 8,000,000.

HPC-BASED DETECTORS are direct-detection, photon-counting devices that have essentially no noise, and thus are well suited for measuring weak reflections. The fast read time and high dynamic range allows for true shutterless data collection—there is no need to measure one scan for strong reflections and a second scan for weak reflections. HPC detectors have revolutionized data collection at beamlines around the world due to their unique characteristics of high speed, high dynamic range and low noise.

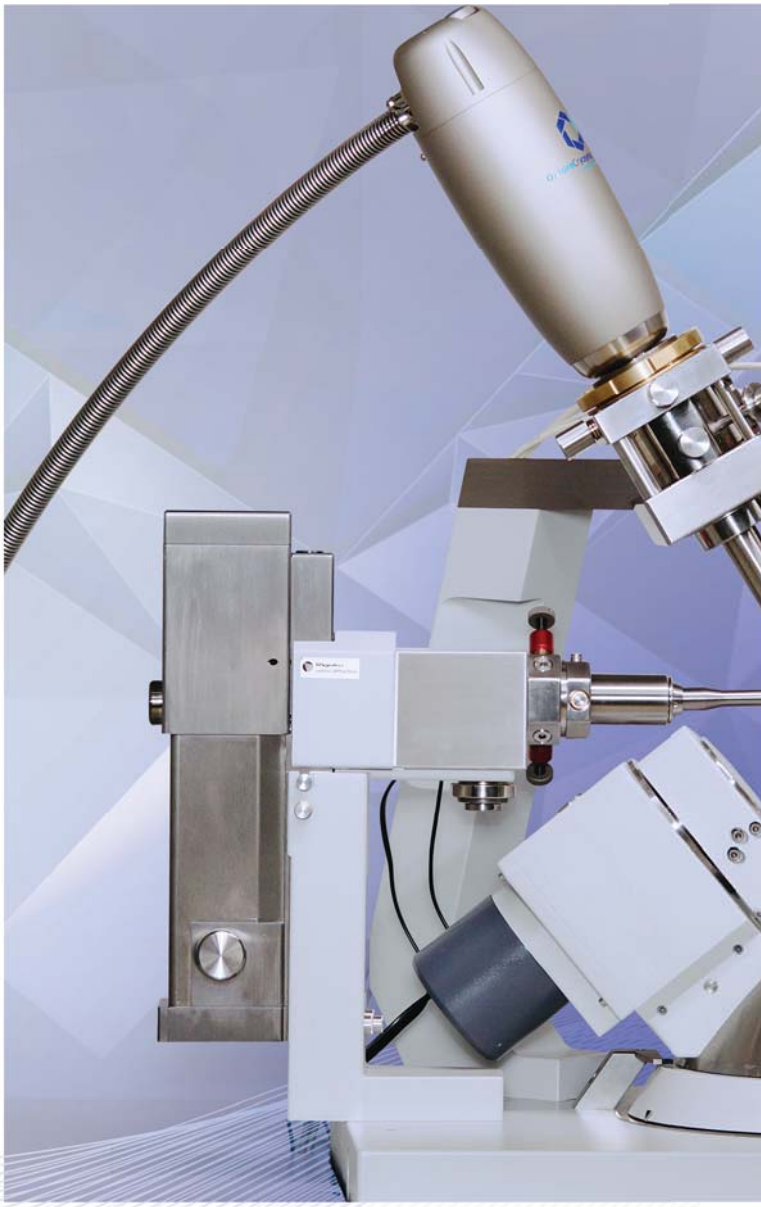
XtaLAB Synergy – Designed for Your Success

NEW microfocus sources – third generation microfocus X-ray sources with longer tube life, higher-performance optics, improved alignment mechanism and, best of all, higher flux.

Single or dual source configurations from a selection of three targets: Cu, Mo, Ag.

NEW goniometer – with motor speeds which have been **doubled** in order to minimize the time between scans and provide for extremely fast data collection.

User-inspired cabinet design that features room for your microscope and tools.



Dual-source configuration for the NEW microfocus X-ray sources

Target	Power	Voltage	Increase in fluence (ph/sec/mm^2)
Cu	50W	50 kV	>100%
Mo	50W	50 kV	>75%
Ag	44W	65 kV	>100%

X-ray source options for XtaLAB Synergy.

NEW electronically controlled brightness of cabinet and crystal lighting results in optimum video imaging for all types of crystal samples.

The widest range of available detectors to suit the needs of any research project. CCD or HPC? Your choice.

Unique telescoping 2θ arm provides total flexibility for your diffraction experiment.

Enhanced kappa goniometer design with symmetrical 2θ positioning to allow the maximum in efficiency for data collection strategies.

Detector name	Active area	Readout times
Eos S2 CCD	92 mm diagonal	0.22 sec.*
Atlas S2 CCD	135 mm diagonal	0.22 sec.*
Titan S2 CCD	165 mm diagonal	0.22 sec.*
PILATUS3 R 200K	83.8 mm x 70 mm	7 msec.
PILATUS3 R 300K	83.8 mm x 106.5 mm	7 msec.

Detector options for XtaLAB Synergy.
*refers to 4x4 binning

PILATUS3 R 200K

CrysAlis^{Pro} – the Nerve Center of the XtaLAB S

INTELLIGENCE WHERE IT COUNTS

XtaLAB Synergy is controlled by CrysAlis^{Pro} software, one of the world's most popular data collection and processing packages. CrysAlis^{Pro} is often referred to as "user-inspired software". Rigaku Oxford Diffraction's software team makes a concerted effort to incorporate features and areas of functionality based on our customers' ideas and feedback.

The design of CrysAlis^{Pro} features a multi-threaded environment. This means that all hardware and software modules run in parallel to achieve the highest efficiency and speed. This design maximizes instrument use by automating mundane tasks, leaving you more time to work out the difficult problems that today's crystallographers face.

Selecting the proper crystal is one of the most important parts of a single crystal experiment. The XtaLAB Synergy's multi-threaded architecture allows an incredibly fast initial crystal investigation. For example, once a crystal is mounted you typically know about the diffraction quality in less than ten seconds.

By simply clicking on the CrysAlis^{Pro} logo on the main screen the user can select one of the two GUI modes: "SM" for small molecule experiments and "PX" for macromolecular data collection. Each of these modes gives access to customized menus and workflows with default parameters specific for the type of experiments the user wants to carry out.

CrysAlis^{Pro} SM

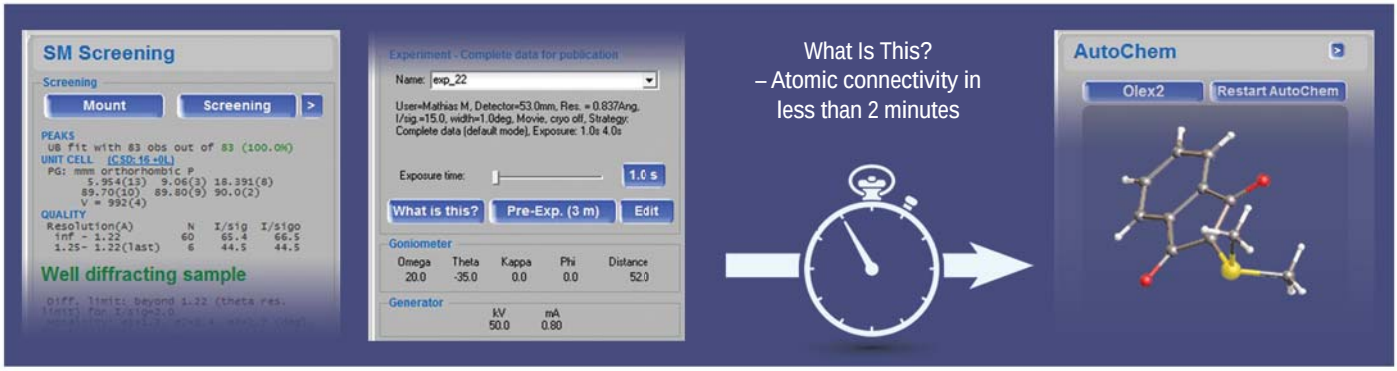
For small molecule samples, CrysAlis^{Pro} combines automated crystal screening, the fastest, most accurate strategy software available, concurrent data reduction and automatic structure solution with refinement by AutoChem, giving you visual feedback in the shortest time possible.

STRUCTURES IN SECONDS WITH "WHAT IS THIS?"

If the initial diffraction pattern indicates that you can continue, a new feature called WIT, or "What Is This?", collects a rapid data set, indexes the cell, determines if it has been previously published (by searching the Cambridge database) and simultaneously attempts to solve the structure. For well diffracting crystals, it is possible to collect data, process it and see the atomic connectivity in one or two minutes. WIT relies on parallelized data collection and data reduction threads as well as the new AutoChem pipeline taking full advantage of a variety of structure solution and refinement programs with built-in support for multi-core speed ups.

A quick structure determination serves two purposes. First, it tells you whether this is a molecule or structure of interest, and whether you want to continue or not. Second and perhaps more important, improved structure information allows the software to do a better job of calculating the strategy for a better, publishable data set.

CrysAlis^{Pro} links into Olex2, the world's most popular small molecule structure solution system, via AutoChem, Rigaku Oxford Diffraction's automatic structure solution interface. After the first 25 images of data are collected, AutoChem will try to solve the structure and, as more data are collected, build upon or refine the solution.



Synergy

At this point the user may want to interact with the experiment after evaluation of these results to further add to the strategy in the middle of the experiment, without disturbing the current data collection.

FULLY INTEGRATED WORKFLOW

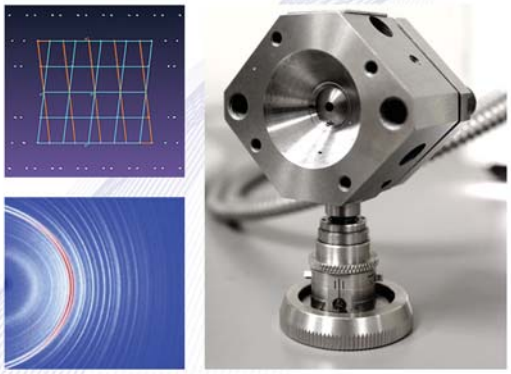
StructureExplorer, based on the AutoChem/Olex2 pipeline, is an integral part of CrysAlis^{Pro} that combines data reduction and finalization with structure modelling and refinement. Key features include:

- AutoChem and AutoComplete support
- Simple intuitive viewer
- One-click twin handling
- Support of SHELXL2014, SHELXS, SIR, Superflip, SHELXD, SHELXT and olex2.solve/refine
- Pre-publication “Checklist” feature with direct links to re-finalization window, for face indexing, and to strategy window, if additional data needs to be collected

WIDE RANGE OF CRYSTALLOGRAPHIC APPLICATIONS

In addition to standard structure determination from single crystals, a wide range of crystallographic experiments can be performed including:

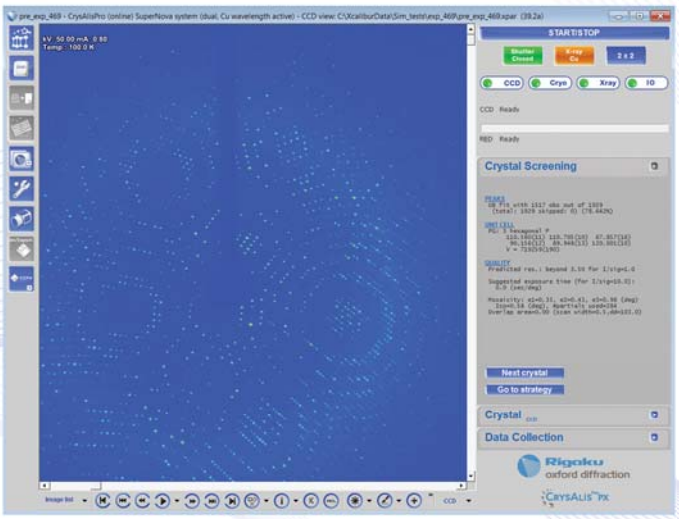
- Powder diffraction experiments, incorporating a Gandolfi motion to remove preferred orientation
- Multi-temperature and multi-wavelength experiments fully controlled by CrysAlis^{Pro}
- High-pressure data collection with strategy and data reduction optimized for diamond anvil cells
- Incommensurate structures and studies of quasi-crystals



CrysAlis^{Pro} PX

The XtaLAB Synergy is the perfect low-maintenance home lab instrument for collecting high-quality data as well as screening of crystals for synchrotron experiments. Dedicated protein features include:

- Data collection, processing and scaling in a single package
- Crystal screening tool for testing crystals in quick succession
- The fastest, most accurate strategy software available as well as processing and scaling algorithms optimized for macromolecular crystallography
- Support for importing and processing data from synchrotrons and third-party detectors, including imaging plate, CCD, CMOS and HPC detectors
- Export of images to MOSFLM and XDS
- Automatic data scaling and merging using AIMLESS to prepare data for structure determination and refinement by CCP4 and PHENIX programs



CrysAlis^{Pro} – Graphical User Interface with intuitive workflows. Shown in PX (macromolecular) mode.

The Proof of Any Good Instrument: Great Applicatio

SIGNIFICANTLY IMPROVE DATA QUALITY AND SPEED

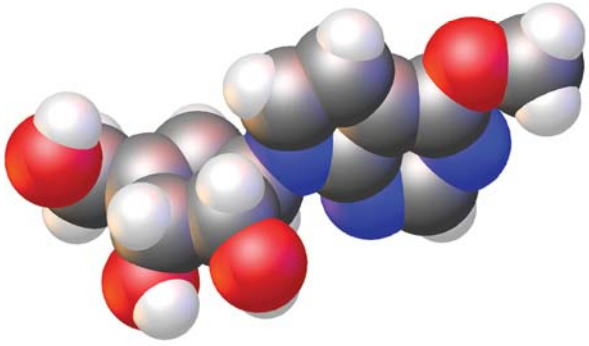
Improve your data quality and data collection speed for small molecule crystals using the XtaLAB Synergy. Not only are the sources brighter, but the increased goniometer speed and the ability of the detector to move close to the sample mean that there are great improvements over its popular predecessor, the SuperNova.

NEW SOURCES, HIGHER FLUX

A careful redesign of the entire source, from tube to optics, gives fantastic improvements in intensity. With the same crystal-to-detector distance and the same data collection speed, the XtaLAB Synergy provides more than a 50% increase in I/σ compared to the SuperNova. The improvement in I/σ subsequently means that the R_{int} and R_1 of the final structure are considerably lower.

Experiment parameters	XtaLAB Synergy AS2	SuperNova AS2
Crystal to detector distance (mm)	52	50
Exposure time (sec./deg)	1	1
Completeness	98.6	97.3
Redundancy	2.1	2.4
I/σ to 0.84 Å	39	25
Dose time	6 min. 29 sec.	7 min. 5 sec.
R_{int}	0.018	0.026
R_1 (%)	2.74	4.14

Comparison of the Nova source and the XtaLAB Synergy microfocus Cu source.



Refined structure of a light atom organic sample.

FASTER GONIOMETER AND CLOSER DETECTOR DISTANCE

Increasing the speed of the goniometer and decreasing the minimum distance of the detector can make huge differences in the total data collection time. Alternatively, this extra time gained could be used to collect more data.

The new goniometer in the XtaLAB Synergy moves twice as fast as the SuperNova and the detector is allowed to approach much closer. These factors, coupled with the advanced strategy calculation, allow you to optimise data collection to suit your requirements as summarised below.

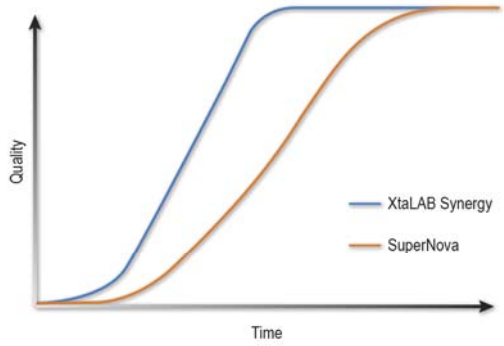


Chart representing the improvements of the XtaLAB Synergy over the SuperNova for the same crystal. Higher quality data can be measured in a shorter time.

Experiment parameters	SuperNova AS2	XtaLAB Synergy AS2 very fast	XtaLAB Synergy AS2 using extra data
Crystal to detector distance (mm)	50	35.5	35.5
Completeness to 0.84 Å	99.2	98.6	99.8
Redundancy	2.7	2.1	2.7
Relative goniometer speed	x1	x2	x2
I/σ to 0.84 Å	26	39	59
Experiment time	12 min. 48 sec.	7 min. 38 sec.	11 min. 17 sec.
R_{int}	0.036	0.021	0.016
R_1 (%)	3.97	3.00	2.54

Experiment details on a light organic chemical sample measured on a SuperNova and the XtaLAB Synergy. These results highlight the benefits of the new, faster goniometer, the closer detector distance and increase in source flux of the microfocus source with the Atlas S2 detector.

n Results

FAST SAD PHASING

The true test of any diffraction system is the ability to accurately measure high-quality data that is suitable for structure solution. In particular, it is especially important to have a system with the high accuracy and DQE offered by XtaLAB Synergy for measurement of the weak anomalous signal from S atoms in proteins. In this example, we illustrate SAD phasing for a low redundancy lysozyme data set.

Details for integration and scaling	
Space group	$P4_32_12$
Unit cell	77.46 Å, 77.46 Å, 38.01 Å 90°, 90°, 90°
Total # reflections	110,989
# unique reflections	13,699
Completeness (%) (Friedel pairs unmerged)	84.6 (11.5)
Redundancy (Friedel pairs unmerged)	4.5 (1.1)
$\langle I/\sigma I \rangle$	34.1 (1.95)
R_{fac} / R_{meas} (%) (Friedel pairs unmerged)	2.5 / 2.8
Chi^2 (last shell) (Friedel pairs unmerged)	1.28 (0.92)

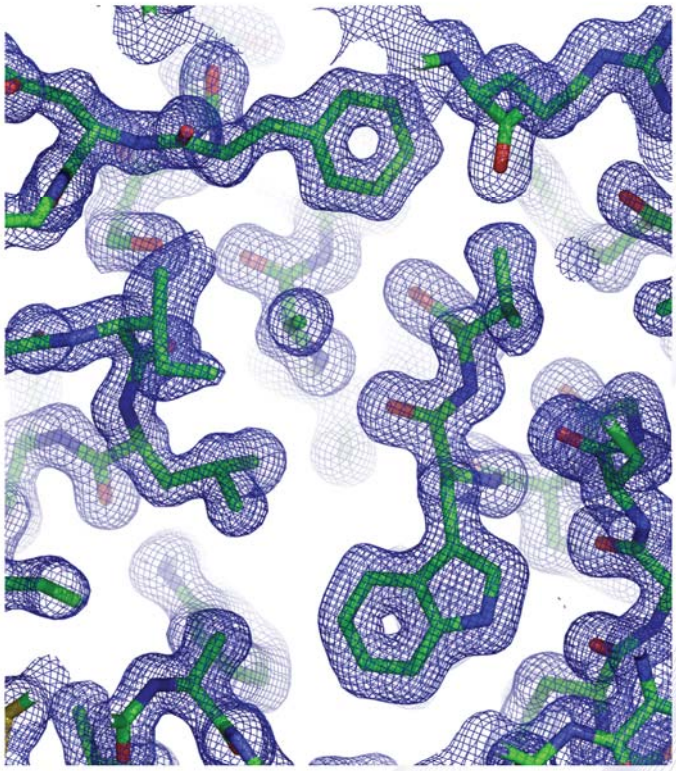
Note: Values in ( ) are for the last resolution shell.

A data set containing 690 images was collected to 1.6 Å using exposure times of 10 sec. / 0.3° on a PILATUS3 R 200K. Statistics from data processed with XDS, shown above, indicate that the redundancy for this data set is 4.5 overall and 1.1 in the highest resolution shell.

The structure was subsequently solved by S-SAD phasing methods. First, the data were analyzed with SHELXC to check the anomalous signal. Then, SHELXD was used to find the heavy atom (S) sites. Subsequently, the anomalous sites were used in phasing with SHELXE and MLPHARE followed by density modification with DM. The Figure of Merit after density modification was 0.79.

Details for structure solution and refinement	
Resolution for heavy atoms search	1.8 Å
Corr. Coeff. for heavy atoms solution (all data/weak data) # heavy atom sites selected from solution	33.4 / 18.3 10 out of 10
Resolution used for phasing and density modification	1.6 Å
FOM after density modification	0.79
# residues built / # waters added	120 (93%) / 125
R_{fac} / R_{free} after REFMAC refinement	17.5% / 22.5%

Following phasing and density modification, ARP/wARP was used to autobuild 93% of residues. Then, the structure was refined with REFMAC to a final R_{fac} and R_{free} of 17.5% and 22.5%, respectively.



REFMAC σA-weighted 2*m*Fo-DFc electron density map, contoured at 1σ, for lysozyme solved using S-SAD methods.

RIGAKU OXFORD DIFFRACTION X-RAY FORUM

JOIN THE BIGGEST USER COMMUNITY OF CRYSTALLOGRAPHERS

www.rigakuxrayform.com

Here you can find discussions about software, general crystallography issues and more. It's also the place to download the latest version of Rigaku Oxford Diffraction's CrysAlis^{Pro} software for single crystal data processing.

XtaLAB Synergy

Single crystal X-ray diffractometer

www.Rigaku-OD.com

Your Success is Our Focus

The employees of Rigaku Oxford Diffraction are dedicated to providing the best solutions for single crystal analysis, whether it be small molecule or macromolecule related research. Our inspiration comes from helping you solve your difficult experimental problems and our personal satisfaction derives from helping you achieve your research goals.

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