XtaLAB mini II

Crystal structure analysis by X-ray diffraction

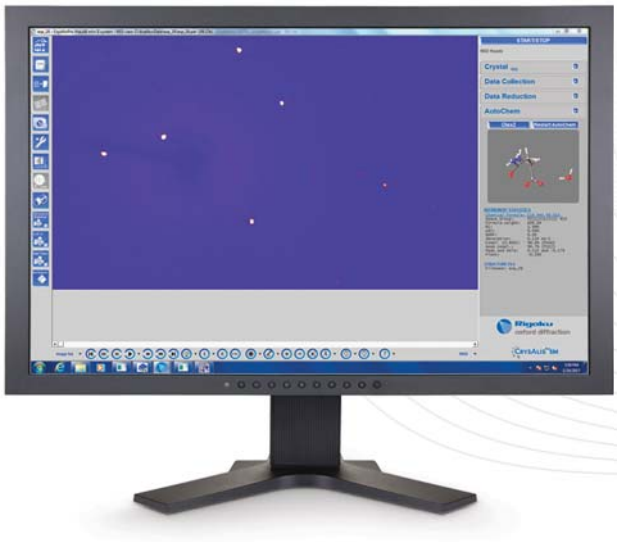
Benchtop single crystal X-ray diffractometer

Rigaku
oxford diffraction

XtaLAB mini II

A DESKTOP DIFFRACTOMETER

With its compact design, the XtaLAB mini II can be placed on a workbench within any laboratory for easy access by synthetic chemists or for use in advanced teaching laboratories. The XtaLAB mini II does not have any special power or water requirements, making it simple to install anywhere.



The system as a whole is designed to be as low maintenance as possible. With very few moving parts, this compact diffractometer is robust and needs very little technician support.

Designed to collect data for publication, the geometry of the X-ray source, goniometer and detector has been carefully calculated to achieve a maximum resolution of 0.66 Å. This is a higher resolution than you need for publication, as most journals ask for a minimum of 0.8 Å.

TECHNICAL DETAILS

X-RAY SOURCE

The XtaLAB mini II diffractometer is equipped with a fine-focus Mo $K\alpha$ X-ray tube with SHINE graphite monochromater to deliver monochromatic X-rays onto the sample. The X-ray tube rarely needs changing but when it does, tubes are readily available and the process is quick and easy.

X-RAY DETECTOR

X-rays are detected using the very latest HPC (Hybrid Photon Counting) technology detector called the HyPix-Bantam. These detectors are built by Rigaku Oxford Diffraction, renowned for their expertise in building great detectors.

Unique benefits of the XtaLAB mini II

- Benchtop diffractometer providing publishable data quality
- User-friendly and semi-automated
- Robust enough for students to operate in a teaching environment
- Latest low noise HPC detector technology
- Researcher and student friendly, comprehensive CrysAlis^{Pro} software

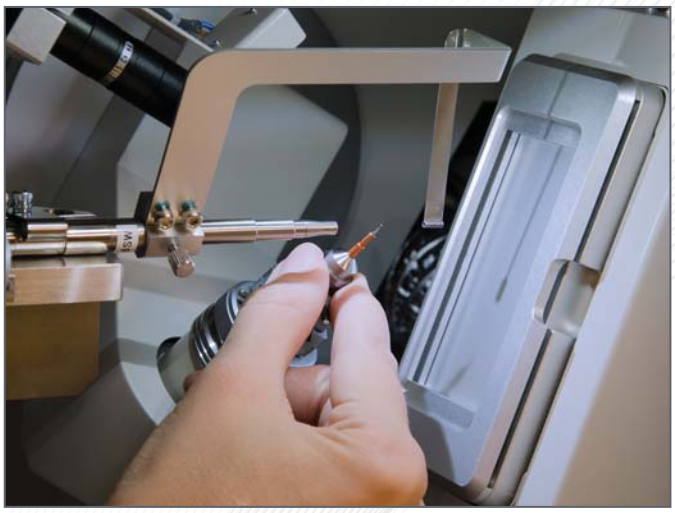
SIMPLE TO USE

After crystallizing a sample, the user mounts one of the single crystals on a loop supported by a pin and places it on the goniometer head. The crystal is aligned and centered on the goniometer using the video displayed on the PC screen. Only once the door is shut and locked do X-rays reach maximum power, making it a very safe system.

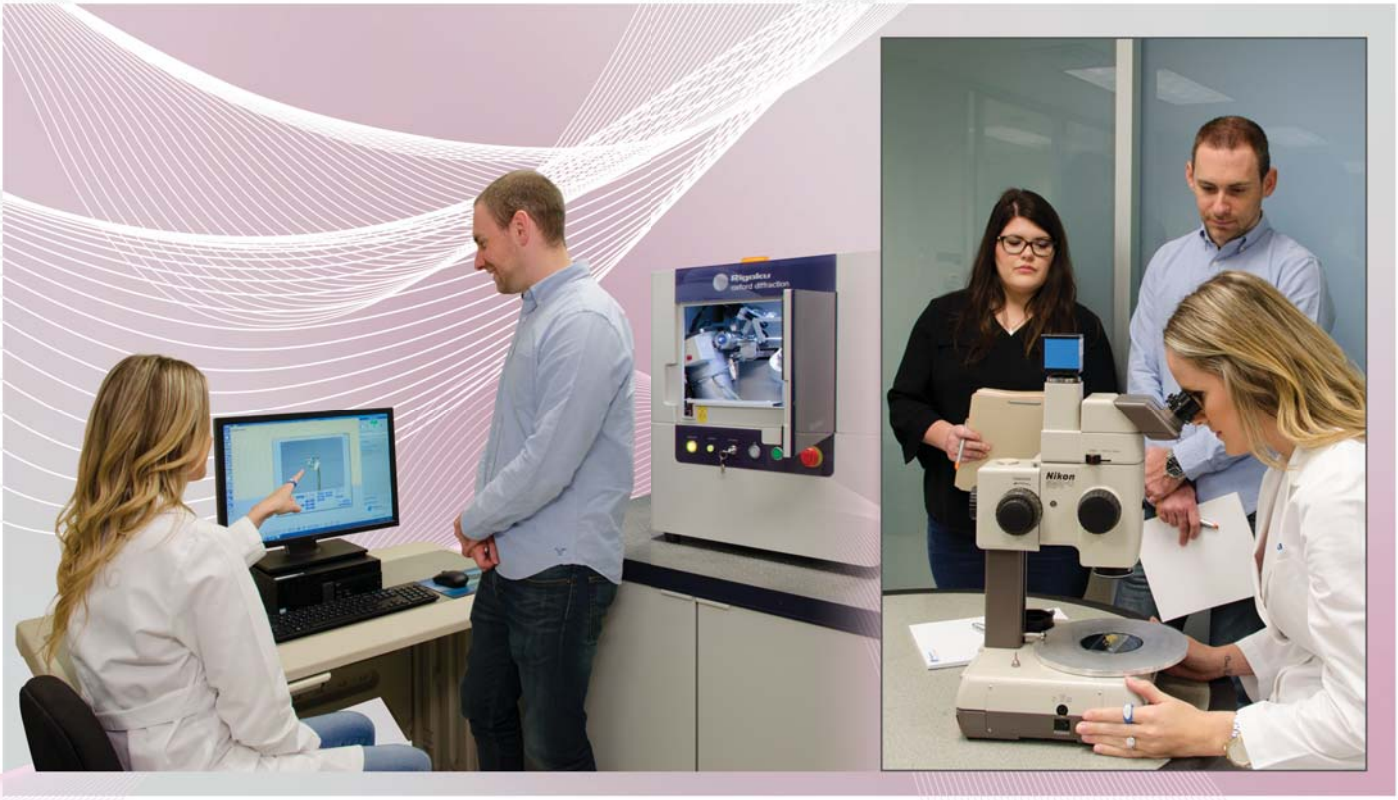
The XtaLAB mini II is controlled by Rigaku Oxford Diffraction's in-house software CrysAlis^{Pro}. The software also processes the data collected. CrysAlis^{Pro} is freely available to all users of Rigaku Oxford Diffraction's equipment and can be installed on any Windows® PC, making it convenient for processing data offline.

The easy-to-follow software leads the user through the experiment pathway, optimizing the experimental parameters for efficient data collection. Data integration and processing is all automated, with only structure solution required at the final step.

Where further input is required along the experiment pathway, for example in the case of a twinned sample, expert features in CrysAlis^{Pro} software can be readily accessed.



Mounting the crystal on the goniometer head.

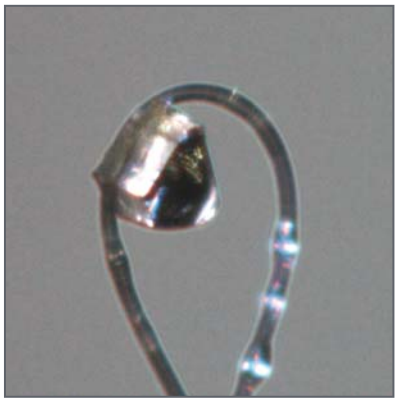


Achieving great data

APPLICATIONS

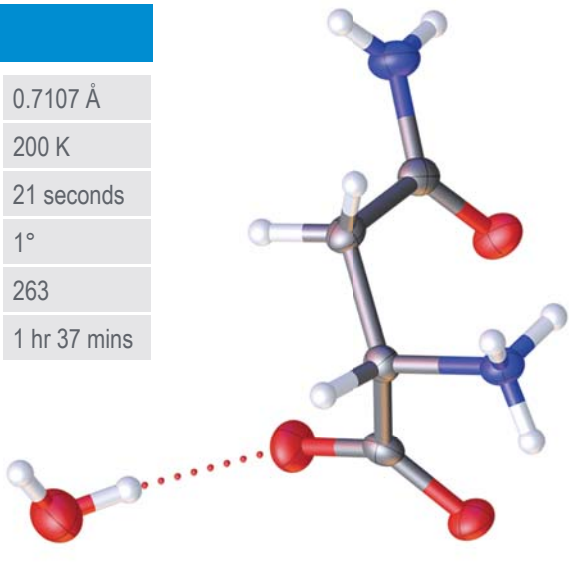
The XtaLAB mini II produces journal quality, publishable results across a range of molecule classes including organic, organometallic and inorganic materials. Data collected on a crystal of the amino acid asparagine is an example of the XtaLAB mini II achieving great data on a light atom, organic compound.

Asparagine sample details		
Space group	$P2_12_12_1$	
Cell parameters	a b c v	5.6180(11) Å 9.8714(17) Å 11.874(3) Å 658.5(2) Å^3
Sample dimensions (mm)	0.14 × 0.16 × 0.24	
Chemical formula	$C_4H_{10}N_2O_4$	



The crystal was mounted on a nylon loop with a small amount of mineral oil and centered on the XtaLAB mini II goniometer. Using data collection parameters suggested by the CrysAlis^{Pro} software, the exposure time was set to 21-seconds per image for a total experiment time of 1 hour and 37 minutes.

Experiment details	
Wavelength	0.7107 Å
Temperature	200 K
Exposure time	21 seconds
Scan width	1°
Total frames	263
Total experiment time	1 hr 37 mins

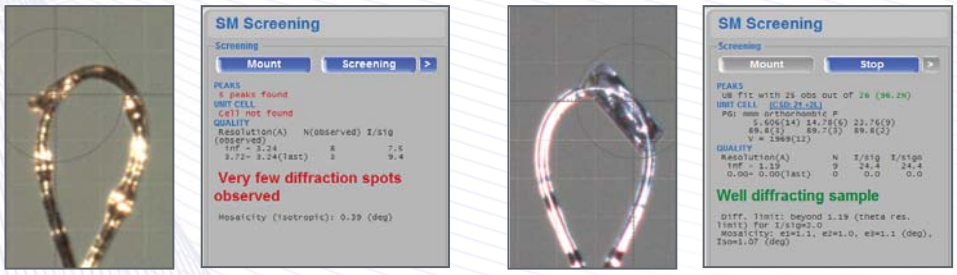


Unmerged data was 98.8% complete to 0.8 Å with 2.5 fold redundancy. The structure was solved and refined to 2.98% using freely available Olex^2 software.

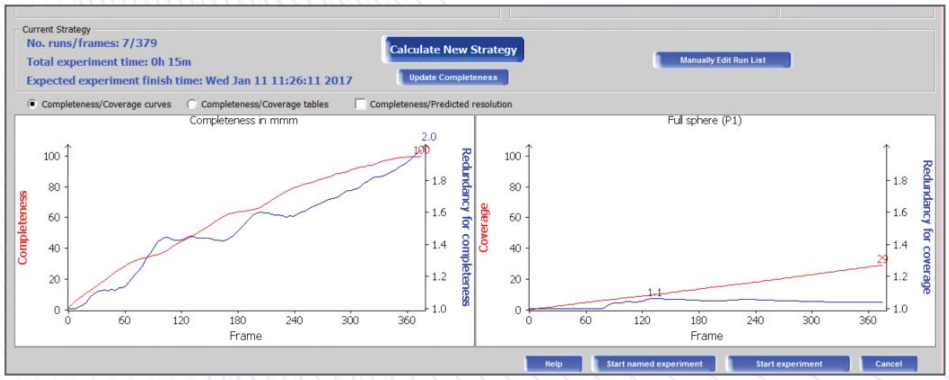
Refinement details	
Final R factors [I>2σ (I)]	R_1 = 0.0298, wR_2 = 0.0785
Goodness of fit	1.060
Largest residual peak/hole / eÅ^{-3}	0.13 / -0.18

HELPFUL SOFTWARE

The XtaLAB mini II is controlled by CrysAlis^{Pro}, an intelligent software program designed and produced specifically for single crystal X-ray diffraction. CrysAlis^{Pro} has brilliant features, including on-the-fly cell indexing and automatic data reduction, which guide you toward getting the best possible data from your unique sample. There is also helpful and clear feedback to assist you in selecting the best crystal from your bulk sample.



Once you have enough information to determine the unit cell parameters of the crystal, a strategy calculator automatically produces the best data collection parameters, including exposure time, scan width, redundancy and coverage. The calculator tells you how long your data collection will take or, if you choose, you can alter the parameters to suit your requirements.



Once data collection and automatic data reduction are complete, you can select the Olex^2 button within CrysAlis^{Pro} to start the structure solution process. The single button click will not only open the structure solution software, e.g. Olex^2 on your Windows PC, but also copy the relevant structure files into the working folder so you can begin solving the structure immediately.

CrysAlis^{Pro} licenses are not limited to sites with instruments installed, so data may be processed offline on any Windows PC, freeing up the instrument for subsequent data collections. After structure solution is completed, the resulting files will contain all the experimental information required to submit to your favorite journal for publication.

Single crystal X-ray diffraction in education

XTALAB MINI II IN THE TEACHING LAB

A large proportion of today's chemical research relies on single crystal X-ray diffraction as the ultimate in structure solution techniques. The technique is used throughout academia and industry.

A hands-on approach to learning is the most powerful method for educators in both classroom settings and teaching laboratories. Direct experience at collecting single crystal data and solving structures, whether for structural confirmation or for physical properties studies, is extremely valuable for all students of chemistry.

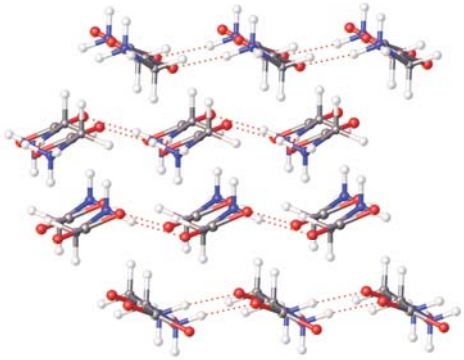
The challenges	Solutions provided by the XaLAB mini II
Diffractometers are expensive so we are unlikely to obtain funding	Has a low purchase cost and operational cost
X-rays pose a significant hazard to the students	Impossible for students to be exposed to X-rays due to system safety interlocks
Traditional diffractometers are large and take up valuable space within the laboratory	Benchtop diffractometer needs only space on lab bench and requires no special power or water supply
Maintaining a diffractometer takes a lot of time and expertise	Very little maintenance required due to only three moving parts
It's not easy to show a student how to use the system and software	CrysAlis^{Pro} is a user friendly software. Online videos and manuals are available
It takes too long to collect data	With a sensitive detector, samples can be measured in just a couple of hours

AN EXAMPLE OF AN UNDERGRADUATE X-RAY COURSE

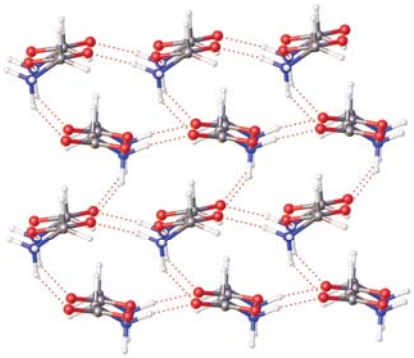
POLYMORPHISM IN THE SOLID STATE

Crystallography, using Rigaku Oxford Diffraction's XtaLAB mini, is one of the key analytical methods covered in an advanced undergraduate teaching course designed for chemistry students at the University of Southampton, UK†. In the practical lab, students begin by crystallizing their own samples, on which they then collect diffraction data using the Rigaku XtaLAB mini single crystal and MiniFlex powder diffractometers.

The students initially prepare their own samples of two polymorphs of glycine under different recrystallization conditions, which they then analyze via a series of techniques, including both powder and single crystal XRD. A full set of instructions for the whole practical is available online‡. The students thereby gain an appreciation for the differences between the polymorphs, and by using these complementary techniques they gain a thorough understanding of how the structure of a material relates to its properties.



α-glycine packing polymorph



β-glycine packing polymorph

When using the XtaLAB mini, the students select their crystal using an optical microscope, understanding what makes a good or appropriate sample, and then mount the crystal onto the goniometer. Due to the ease of operation of the XtaLAB mini, students are able to conduct their own data collection with minimal supervision. During the pre-experiment, sample quality is assessed and students are allowed to choose the appropriate exposure time for the experiment. Typical run times are 1-2 hours for a complete data set.

Structural analysis of the packing and interactions shows some clear differences between the polymorphs. This can be related to data gathered from other analytical techniques and used to rationalize different properties and observations. The students gain an appreciation for the use of a combination of analytical techniques and how structural data can help justify and inform fundamental physical property behavior.

† S.J. Coles & L.K. Mapp, "Conducting Reflective, Hands-On Research with Advanced Characterization Instruments: A High-Level Undergraduate Practical Exploring Solid-State Polymorphism" *J. Chem. Educ.*, 2016, *93*, 131–140. DOI: 10.1021/acs.jchemed.5b00071

‡ http://www.edshare.soton.ac.uk/13584/

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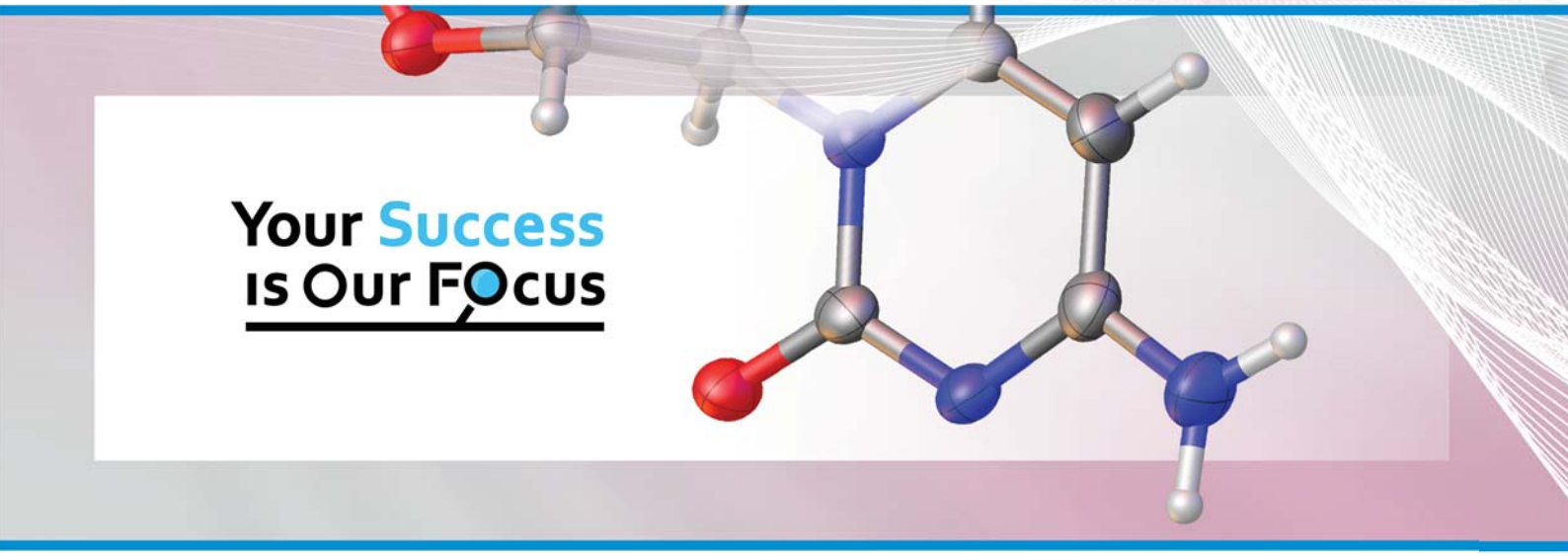
www.Rigaku.com
www.Rigaku-OD.com



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