Benchtop X-ray diffractometer system with molybdenum X-ray source and hybrid photon counting detector

XtaLAB mini II

1. Introduction

Riding on the success of the XtaLAB mini as a compact, low-maintenance, benchtop system providing high-quality crystal structures, the new XtaLAB mini II has been developed. The XtaLAB mini II differs to its predecessor by the inclusion of a Hybrid Photon Counting (HPC) detector, called the HyPix-Bantam. HPC detectors are at the cutting edge of diffraction technology, and are widely used at synchrotron beamlines due to their high sensitivity and fast read-out speeds. The whole system is controlled by the popular CrysAlis^{Pro} software, which offers users the ability to complete their structure determination with one software package.

Fig. 1. The XtaLAB mini II cabinet design.

2. Features

2.1. Benchtop Design

The XtaLAB mini II cabinet (Fig. 1) is compact and robust. With dimensions of just 560 × 395 × 674 mm and a weight of approximately 100 kg, the XtaLAB mini II is the world's smallest diffractometer, making it easy to install almost anywhere. The control buttons on the front display are clearly labelled, and the fail-safe radiation enclosure means that the system is safe and easy to use for even inexperienced users. This lends itself well to being used as a teaching tool. Whilst the door is opaque, users are still able to view the inside of the cabinet via a camera linked to the PC.

Should low temperature data collection be required, the XtaLAB mini II is compatible with various cryo devices, such as Oxford CryoSystems' CryoStream 800 attachment.

2.2. X-ray source

The XtaLAB mini II is equipped with a molybdenum fine focus X-ray source and coupled with SHINE optics for enhanced flux. For easy maintenance and low cost of ownership, the XtaLAB mini II uses a standard X-ray tube, which is readily available should a replacement ever be required.

2.3. Goniometer

The XtaLAB mini II has just three moving parts: the

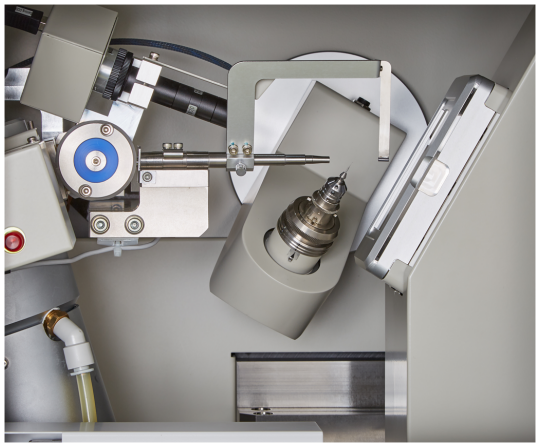

Fig. 2. View inside the XtaLAB mini II.

shutter and the phi and omega axes of the goniometer (Fig. 2). The latter of these were designed specifically for ease of mounting—useful when training new users. The simple design also means minimal maintenance and no special infrastructure requirements, meaning that the system can run with very little downtime for servicing. This is important for universities or service laboratories with a high throughput of samples.

2.4. HPC Detector

The new HyPix-Bantam detector is a next-generation two-dimensional semiconductor detector designed specifically to meet the needs of the home lab diffractionist. The HyPix-Bantam is a Hybrid Photon Counting (HPC) detector with an active area of approximately 3000 mm^2, a small pixel size of 100 μm $\times$100 μm, and a high count rate of greater than 10^6 cps/pixel.

A big advantage of HPC detectors is that each pixel is independent and the overall dynamic range of a detector is a sum of the dynamic range of each individual pixel. Each pixel of the HyPix-Bantam has two 16-bit counters, and these can be combined to work as a single 31-bit counter achieving very wide dynamic range. Another benefit of HPC detectors is the extremely low background noise and high sensitivity meaning that even low photon counts are detected.

The combination of all these features means that images with both strong and weak reflections present are measured extremely accurately without the need to do a second pass of reciprocal space in order to recollect the image at a lower exposure time.

2.5. Software

The highly regarded CrysAlis^{Pro} software package is the nerve center of the XtaLAB mini II, tying together all the new detector features through a highly parallelized architecture, resulting in a reliable system for generating 3D structures of crystalline materials. CrysAlis^{Pro} is a comprehensive but user-friendly, semi-automated software package, ideal for both researcher and student level users. It links directly to Olex^2, a freely available software package for structure solution and refinement.

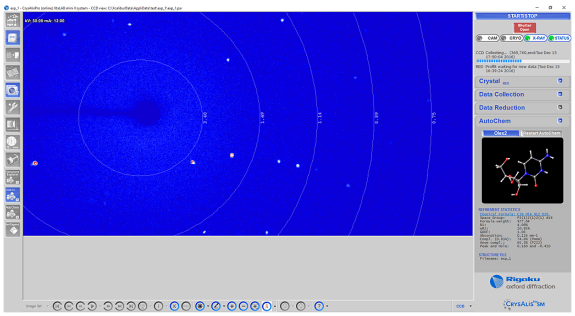

Fig. 3. CrysAlis^{Pro}: Data collection and reduction software.

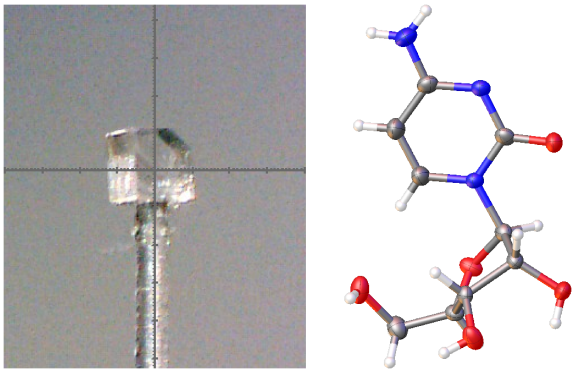

Fig. 4. Image of the crystal sample and refined structure.

Table 1. Refinement details.

Space group	$P\,2_12_12_1$
Cell parameters	a=5.1265(6) Å b=13.9969(15) Å c=14.7637(13) Å V=1059.37(19) $Å^3$ Z=4
Chemical formula	$C_9H_{13}N_3O_5$
Completeness to 0.77 Å	99.6%
Redundancy to 0.77 Å	3.6
R_{int} to 0.77 Å	0.0249
Final R factors [I>2σ (I)]	R_1=0.0320, wR_2=0.0813
Largest residual peak/hole/e$Å^{-3}$	0.18/−0.20

3. Applications

Data was collected on the XtaLAB mini II at room temperature on a crystal of cytidine with dimensions 0.167×0.171×0.231 mm. The exposure time was set to 10 seconds per frame with 648 images collected in just under 1 hour 50 minutes.

Excellent data was collected on this light organic sample in less than two hours using the XtaLAB mini II. The new HyPix-Bantam detector allows data to be collected very quickly due to its high sensitivity and dynamic range.