APPLICATION NOTE

Higher flux from the XtaLAB Synergy-DW leads to improved data for small crystals and challenging sample types

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Introduction

Single crystal X-ray diffraction is the preferred method for structure determination of small molecules as well as protein and larger biological macromolecules. The complexity of samples that can be studied using X-ray techniques has improved significantly over recent years. Samples that were previously too small to be considered for single crystal X-ray analysis are now fully amenable to study. Highly unstable and weakly diffracting samples may be studied immediately, samples do not need to be discarded or undergo further recrystallization attempts.

An X-ray source with higher brightness can often reveal additional features of a particular crystal sample: for instance modulation, diffuse scattering or missed space group assignments, etc. Taking proper care of these anomalies can clarify particular subtle physical properties of the materials that were not previously understood. Furthermore, with a brighter X-ray source much fast data collections can be conducted: so allowing decomposing samples and real-time phase transitions to be investigated in finer detail.

The Cu Kα (1.54 Å) and Mo Kα (0.71 Å) characteristic X-ray wavelengths each have their benefits: depending on the crystal sample to be investigated. With the ready availability of both wavelengths in a single instrument, the widest range of materials and applications can be handled. Cu Kα can be generated in higher intensities, and is ideal for investigating, for instance, very weakly diffracting metal-organic frameworks (which are often porous and/or highly solvated), small chiral organic crystals, proteins and larger biomolecular structures. Mo Kα radiation is more optimal for strongly absorbing inorganic materials, high resolution 'charge density' measurements or high pressure crystallography using diamond anvil cells.

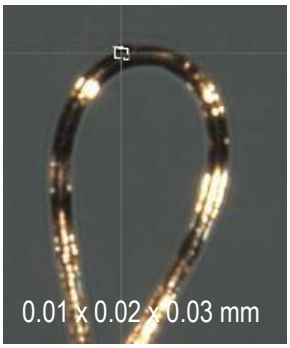
The XtaLAB Synergy-DW

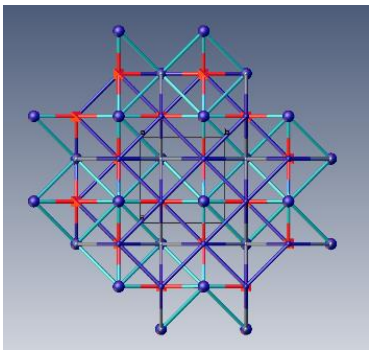
XtaLAB Synergy-DW

The XtaLAB Synergy-DW diffractometer includes a dual wavelength rotating anode source which produces a much higher flux than the microfocus sealed-tube diffractometers. With this advantage, the most challenging crystal sample types and applications can be examined 'in-house'. Accordingly, the XtaLAB Synergy-DW is the perfect instrument for institutions where several research groups may share a single instrument to cover a range of research interests.

Small, yet measurable inorganic sample

A small crystal of a lithium iron hydroxide selenide superconductor was provided by Dr. Daniel N. Woodruff from the University of Oxford, UK. Due to their small size, these particular crystals are normally collected at synchrotron facilities. Table 1 summarises results from data collected on a sealed-tube source microfocus system and the Synergy-DW rotating anode system: both using Mo Kα (0.71 Å).





Tetragonal, *P* 4/*n* m m
3.7901(6), 3.7901(6), 9.127(3) Å
90, 90, 90 °
131.11(6) Å^3
Chemical Formula:
$Fe_{1.16}Li_{0.84}OHSe$

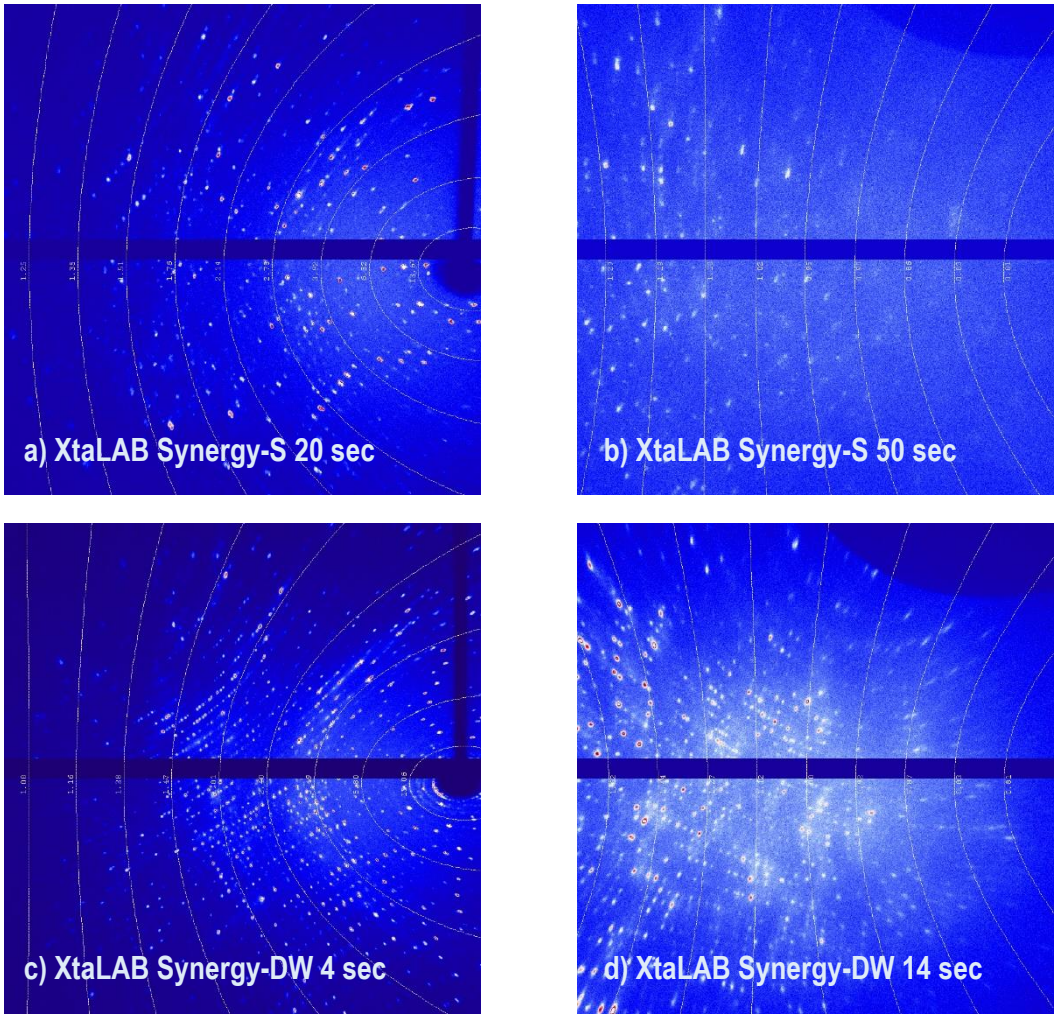
Table 1. Data collection details and refinement parameters

	XtaLAB Synergy-S	XtaLAB Synergy-DW
Generator settings	50 W (50 kV, 1 mA)	1.2 kW (50 kV, 24 mA)
Temp	100 K	100 K
Detector distance	30 mm	32 mm
Exposure Time	80 seconds	10 seconds
Total Time	7.5 hours	1 hour
Total Frames	336	336
I/σ(I) (merged)	19.90	41.51
Redundancy	6.8	6.9
R_{int}	7.9 %	3.6 %
R_1	4.3 %	3.0 %

The results clearly show the benefit of the higher flux generated from the rotating anode source. Nonetheless both instruments performed well and illustrate the excellent performance that can be obtained from home laboratory based instruments.

The power of Cu Kα radiation for a protein-sized inorganic cluster

A very difficult to handle protein-sized inorganic cluster material from Prof. Lee Cronin and co-workers (University of Glasgow, UK) was investigated using Cu Kα (1.54 Å) on the XtaLAB Synergy-S and XtaLAB Synergy-DW systems. Representative low and high angle diffraction images from the XtaLAB Synergy-S (a and b) and XtaLAB Synergy-DW (c and d) data collections are depicted with their respective exposure times.



*Detector distance: Synergy-S 45 mm and Synergy-DW 34 mm

For this centrosymmetric, triclinic material a 36 hour data collection on the XtaLAB Synergy-S was not sufficient to obtain a complete dataset for publication purposes (95.8% complete; resolution inf → 0.90 Å). In contrast, within just 13 hours using the XtaLAB Synergy-DW system a publishable dataset was obtained (99.2%, complete; resolution inf → 0.83 Å). The high level of disorder and large pores within these protein-sized inorganic clusters make them exceptionally difficult to measure.

The XtaLAB Synergy-DW diffractometer has all the necessary hardware and software features to make the most challenging crystal samples amenable to study in the home laboratory.

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