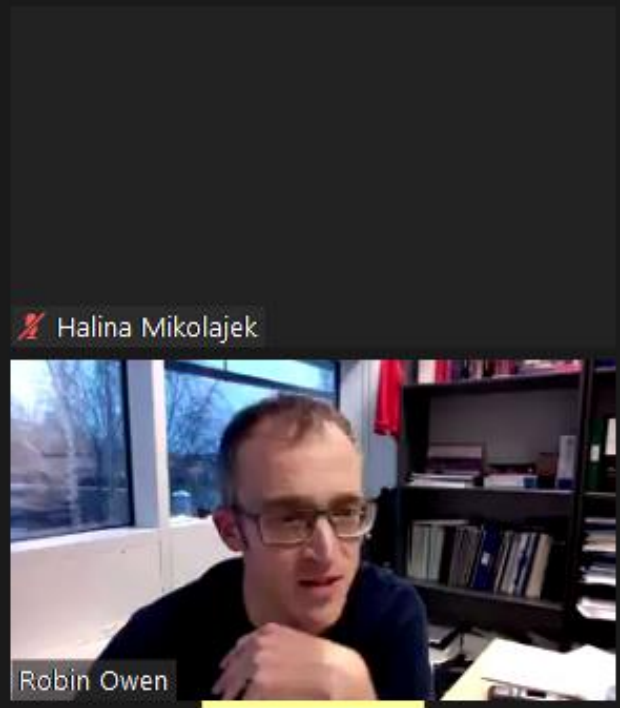
I24 update

December 2021

diamond



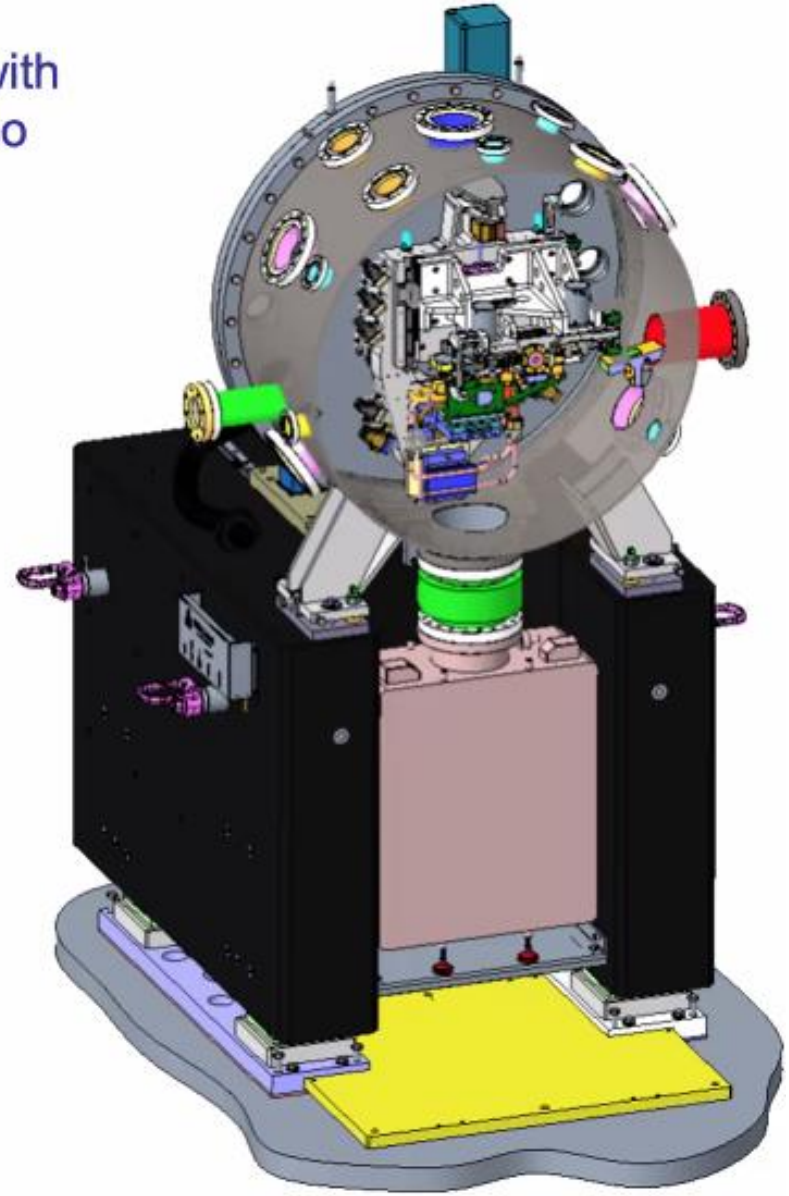
What's new - hardware

(I) Monochromator

Old mono was great but was showing its age with several last ditch repairs over the last year or so

New monochromator offers

- Reliability
- Stability
- Upgrade path to multilayer



Installation – last shutdown

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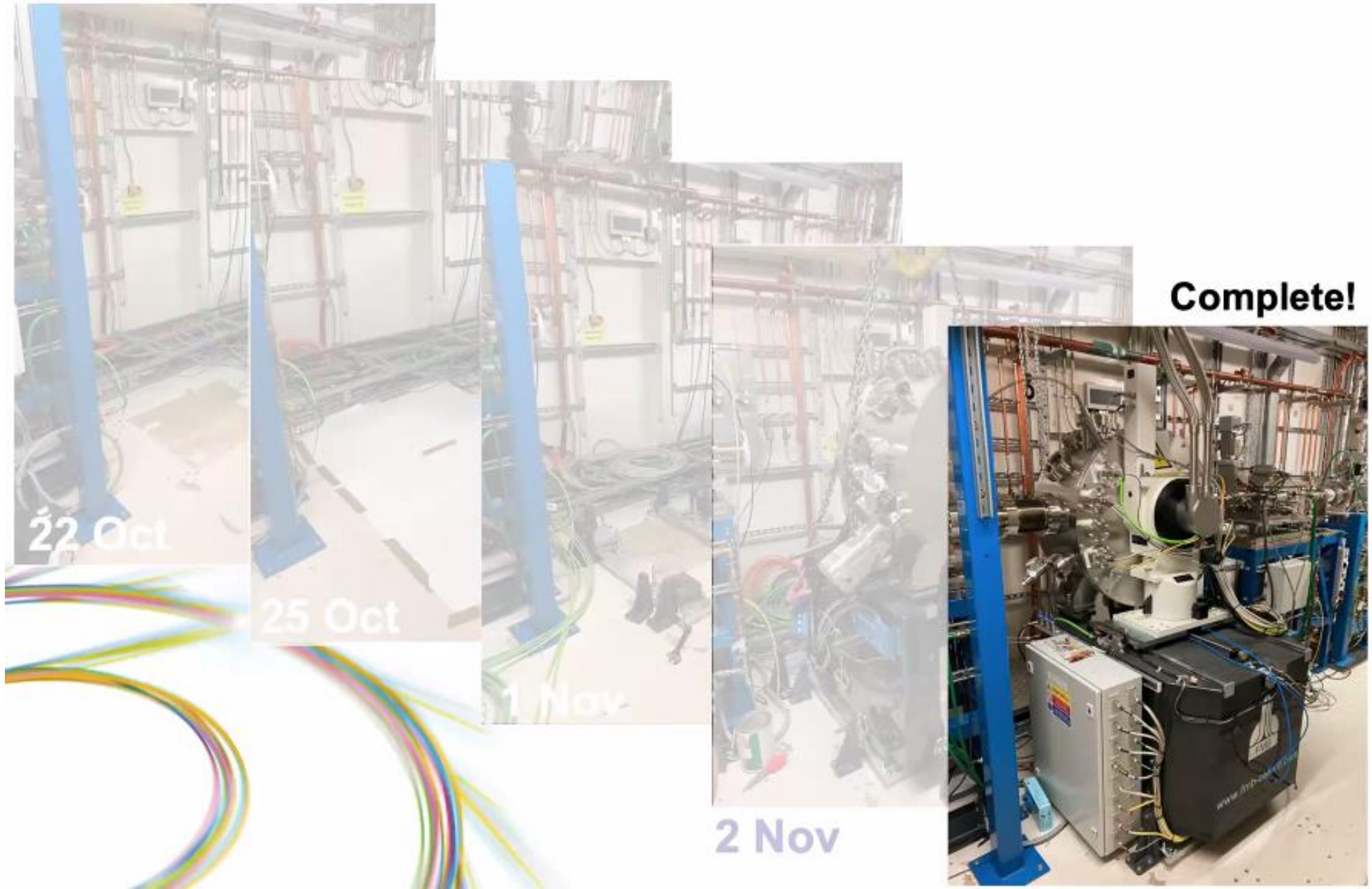
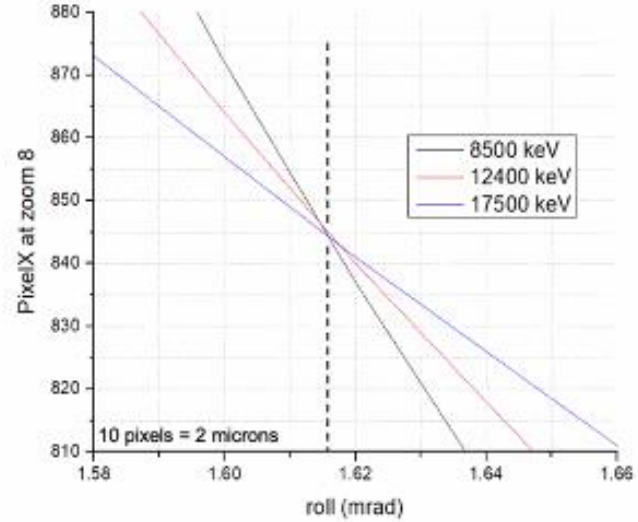
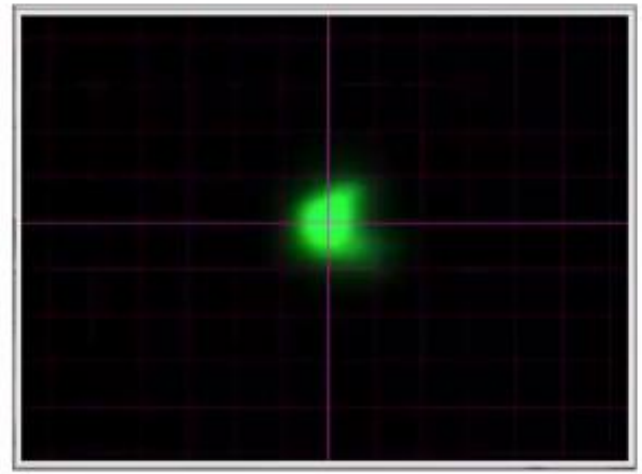


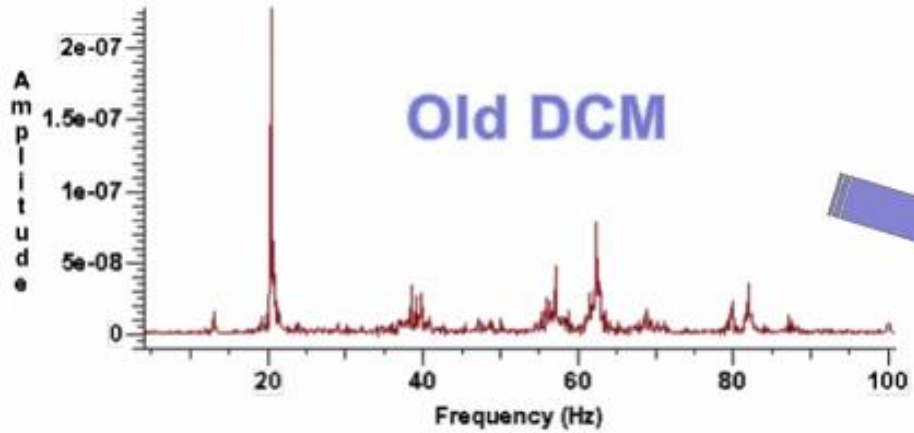
Initial performance

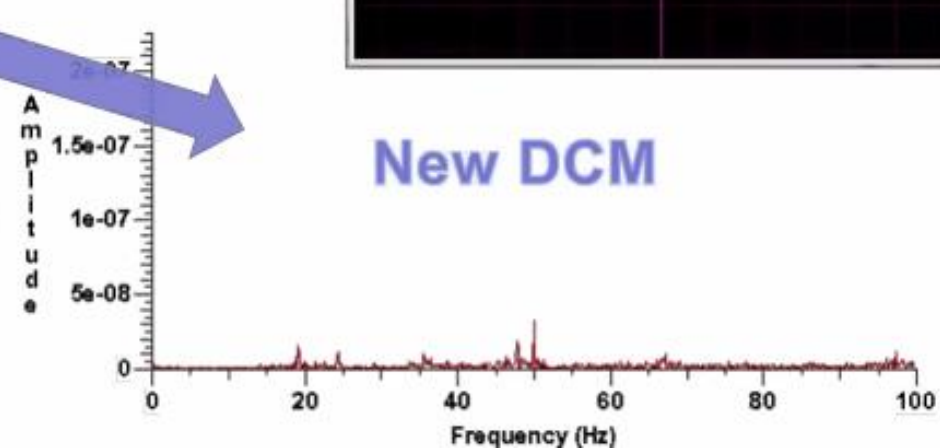
Extremely encouraging

- Straightforward to align and commission: over whole energy range horizontal drift of beam at sample << 0.25 micron
- Vibration much less. Shown below measured in optics hutch immediately after DCM and in video at sample right.





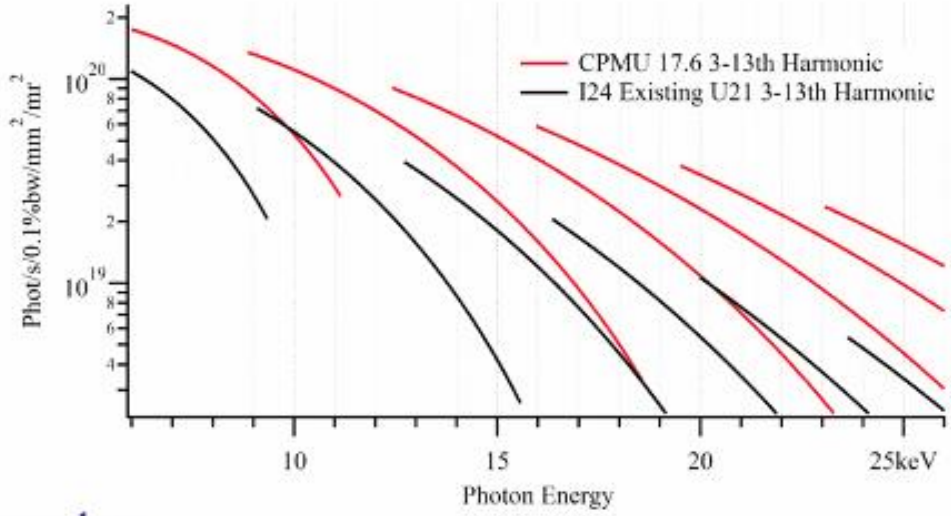




What's new - hardware
(II) insertion device

Cryocooler permanent magnet undulator (CPMU) was installed in 2020
- Higher fluxes at higher energies

Has performed well but we have had problems limiting the min. gap we can utilise
- CPMU will be swapped out in upcoming shutdown
- Should see immediate step change in flux 17keV plus

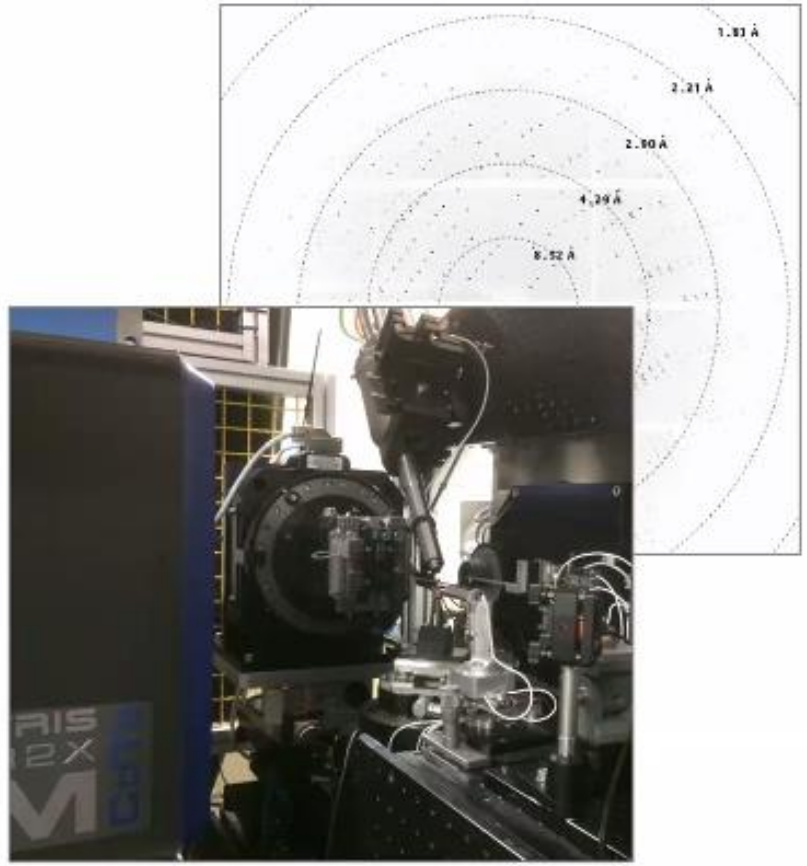


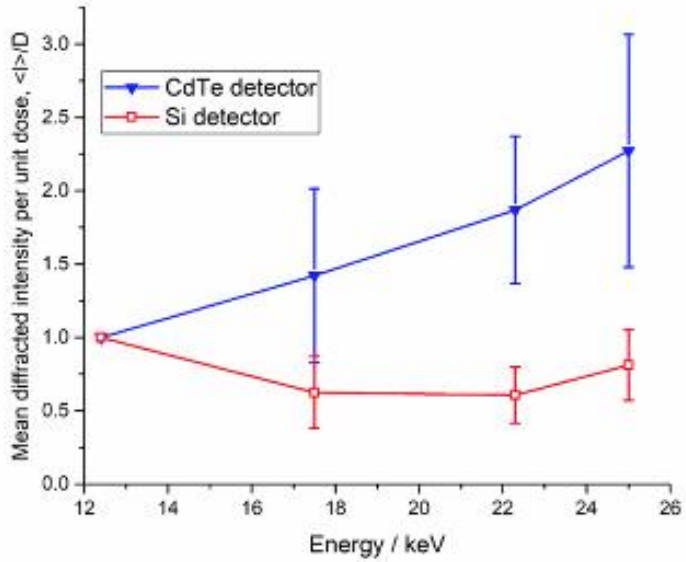
Main Parameters

Beamline operating range	7-30 keV
Device Period	17.6 mm
Number of Periods	≤ 117
Nominal Device Magnetic Length	≤ 2 m
Minimum Operating Gap	4 mm

What's new - hardware
(III) CdTe Eiger 9M

- Radiation damage is limiting in MX and crystals can only survive a finite dose before crystalline order is lost.
- This limiting dose is approximately constant for all proteins and viruses
- At higher X-ray energies it may be possible to collect more data from each crystal.
- A CdTe detector makes high energy data collection efficient and routine.

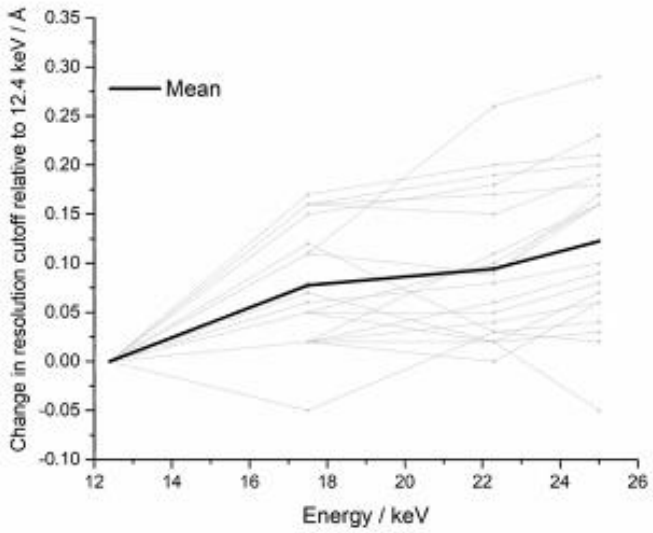




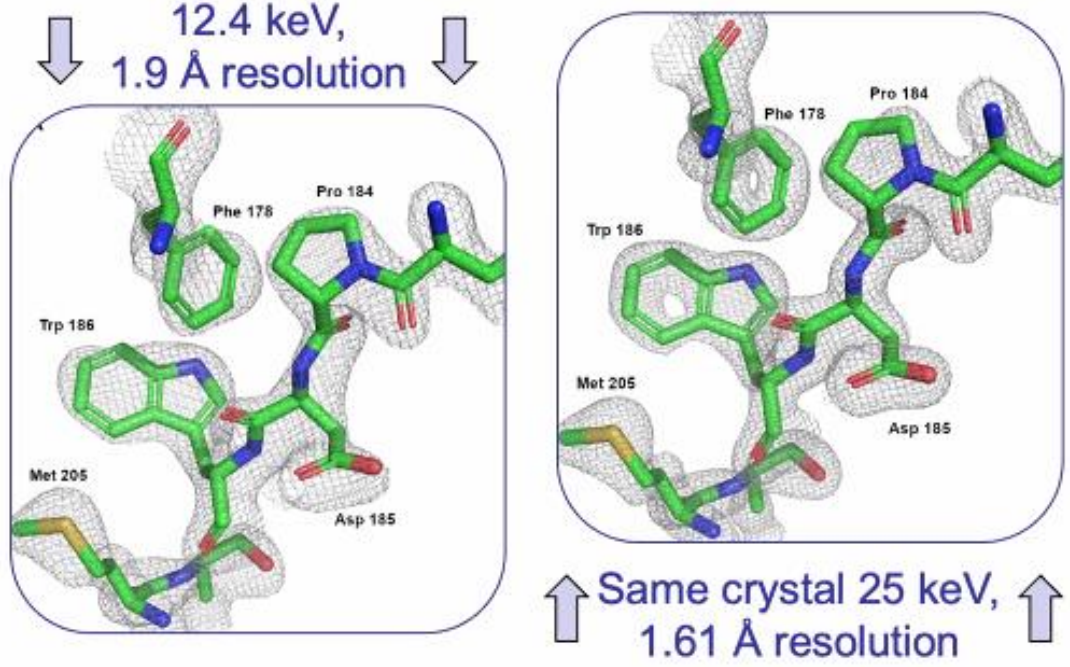
As the X-ray energy increases the diffracted intensity per unit absorbed dose increases significantly when using a CdTe based detector (blue line).

CdTe Eiger detector

a new detector for high energy MX



Furthermore, data collected at higher energies are useful to a higher resolution even though the dose deposited in the crystal is the same in each case (mean change for twenty crystals shown right).



research papers

IUCrJ

BIOLOGY | MEDICINE

Experimental evidence for the benefits of higher X-ray energies for macromolecular crystallography

Selina L. S. Storm,‡ Danny Axford and Robin L. Owen*

X-ray-induced radiation damage is a limiting factor for the macromolecular crystallographer and data must often be merged from many crystals to yield complete data sets for the structure solution of challenging samples. Increasing the X-ray energy beyond the typical 10–15 keV range promises to provide an extension of crystal lifetime via an increase in diffraction efficiency. To date, however, hardware limitations have negated any possible gains. Through the first use of a cadmium telluride EIGER2 detector and a beamline optimized for high-energy data collection, it is shown that at higher energies fewer crystals will be required to obtain complete data, as the diffracted intensity per unit dose increases by a factor of more than two between 12.4 and 25 keV. Additionally, these higher energy data can provide more information, as shown by a systematic increase in the high-resolution cutoff of the data collected. Taken together, these gains point to a high-energy future for synchrotron-based macromolecular crystallography.

1. Introduction

Synchrotron-based macromolecular crystallography (MX) is

These data point the way to a new high energy future for MX.

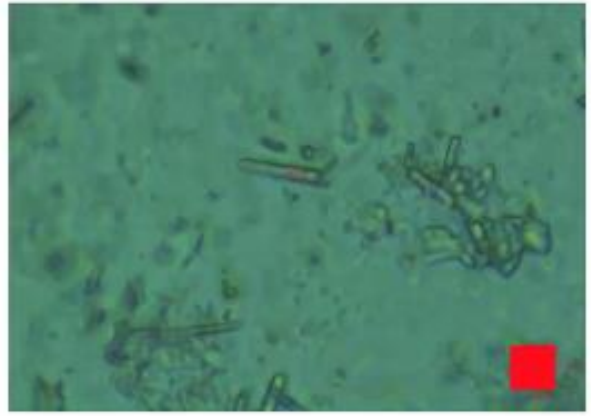
Data collection

(I) ‘standard’ MX

Just want to highlight one new-ish processing tool: multiplex

Example:

- 5-10 μm crystals. Needed a room temp dataset
- Crystals mounted on thin films
- Iteratively optimised parameters for 2.5° data collection

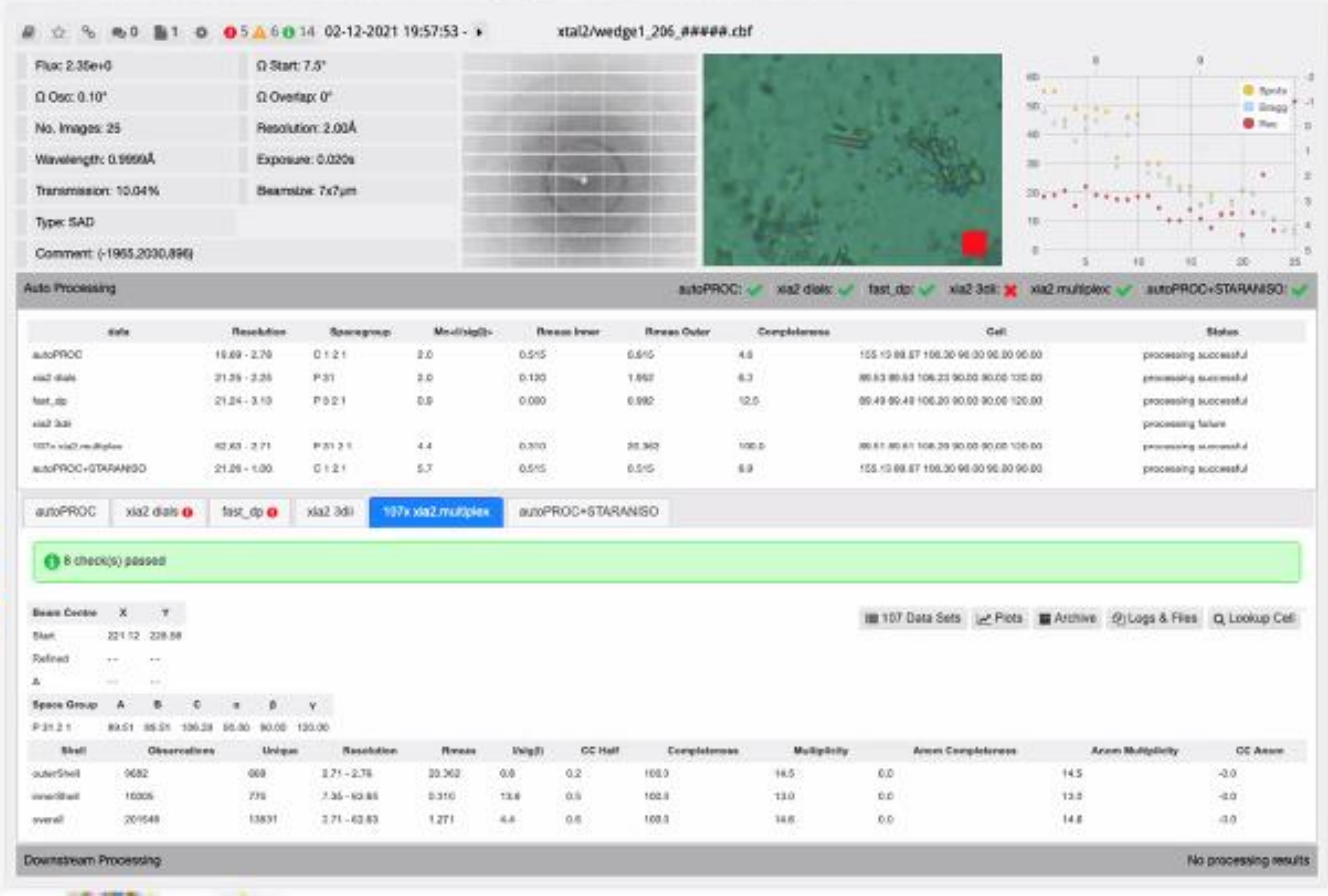


Data collection
(I) ‘standard’ MX

Just want to highlight one new-ish processing tool: multiplex

Example:
- 5-10 µm crystals. Needed a room temp dataset
- Crystals mounted in [illegible]
- Iteratively optimis[illegible]

Data automatically merged from 229 crystals
Results available in ispyb



After manual rerun with confirmed spacegroup

- Excluded outlier crystals – 165 crystals used in final merge
- Reduced data range of 1.5° per crystal used

xia2.multiplex report

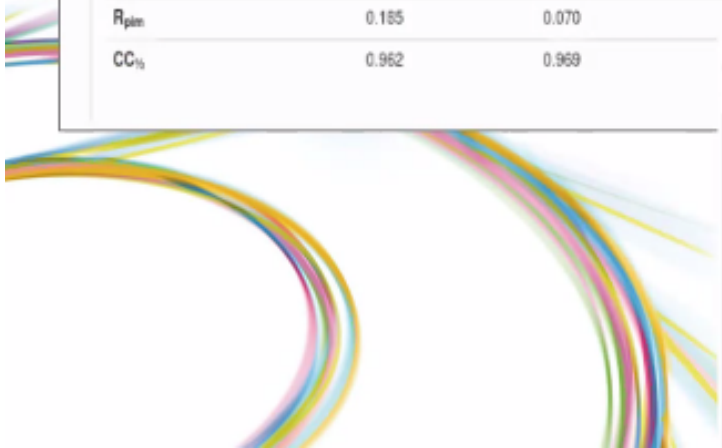
Summary | All data

Detailed statistics for dataset All data

Overall

	Overall	Low resolution	High resolution
Resolution (Å)	106.51 - 2.46	106.61 - 6.68	2.50 - 2.46
Observations	252605	12376	12786
Unique reflections	18518	1023	935
Multiplicity	13.6	12.1	13.7
Completeness	100.00%	100.00%	100.00%
Mean I/σ(I)	4.4	13.4	0.7
R_{merge}	0.676	0.237	14.407
R_{meas}	0.703	0.248	14.955
R_{pim}	0.185	0.070	3.851
$CC_{½}$	0.962	0.969	

More info and instructions on I24 webpages (need to be careful with filename and directory choice)
Best contact: Richard Gildea



MX / I24 / News and Current Status / Multiplex data merging

In This Section

News and Current Status

- Grid scan features
- I24-serial
- Multiplex data merging

I24 Manual
SSX at I24
Beamline Schematic
Staff
Latest I24 Structures

Multiplex data merging

Automatic multi crystal processing

Please see the multi-crystal pages in the manual here. This page gives an extremely brief introduction running the relatively new multiplex tool for merging datasets manually. As it evolves this info will probably also move to the manual but is here for now to increase visability

Multiplex

Multiplex is part of Dials and is written by Richard Gildea. For detailed info see

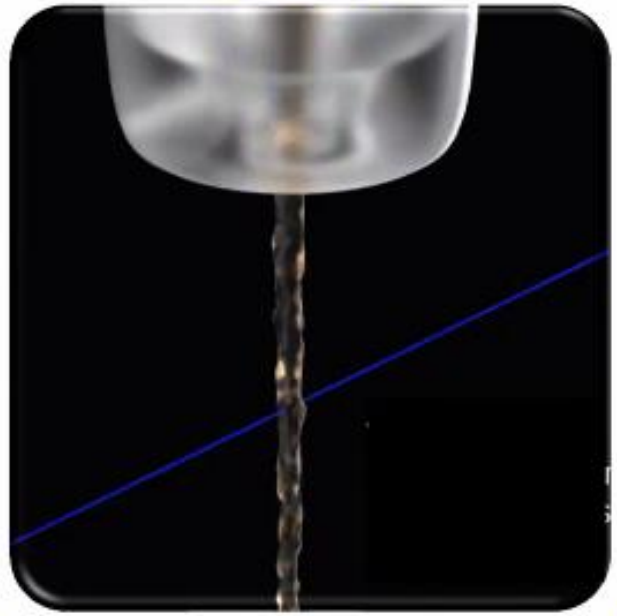
https://dials.github.io/documentation/tutorials/multi_crystal_symmetry_and_scaling.html

Data collection

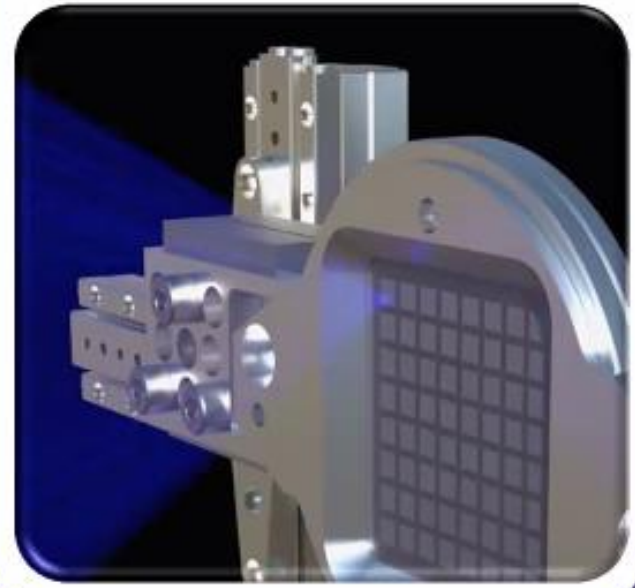
(II) Serial Crystallography

Multiple modes of sample delivery

Extruder

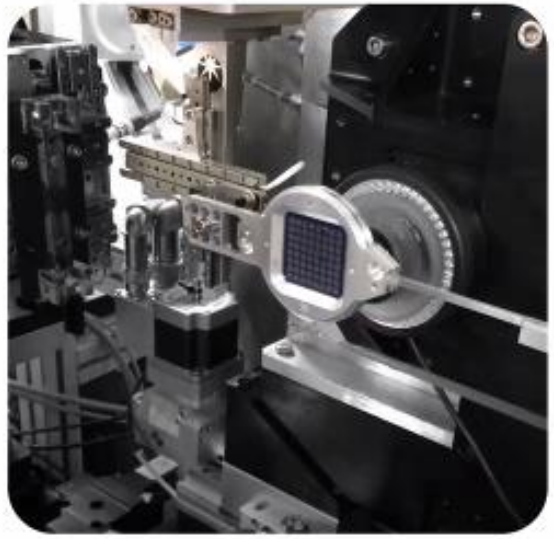


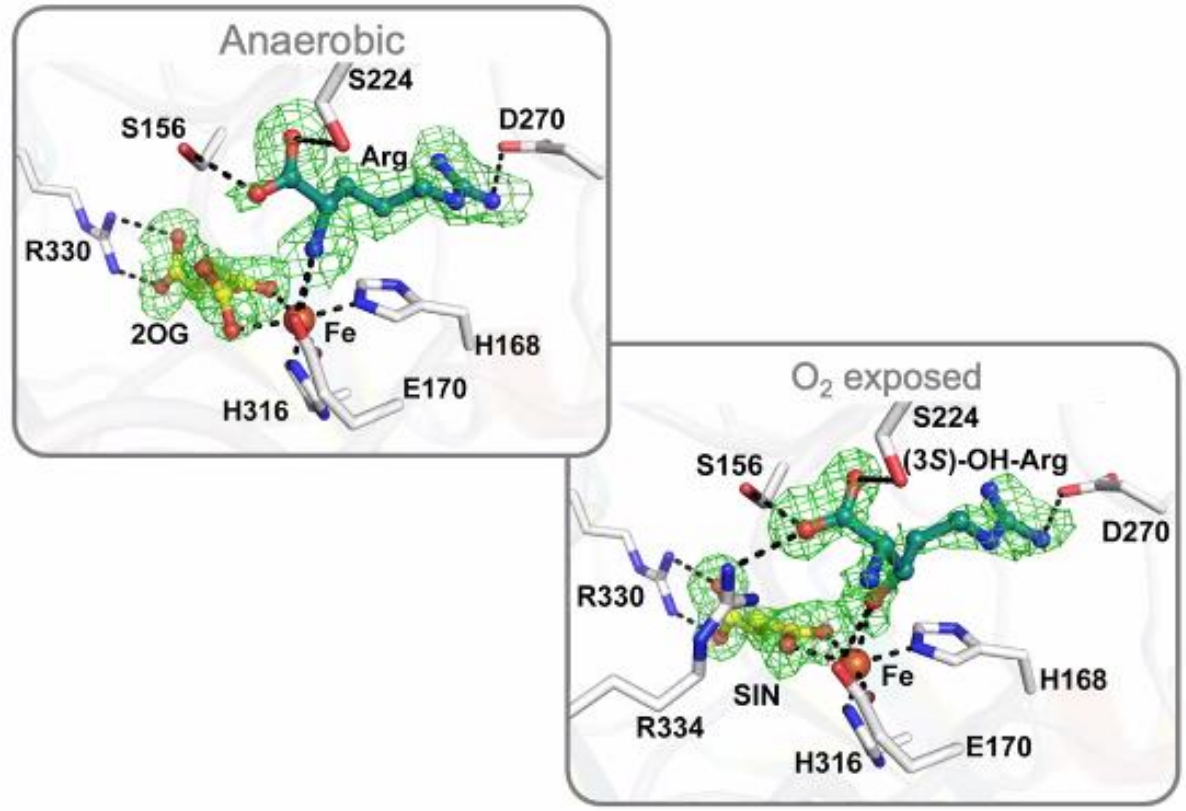
Fixed target



Data collection
(II) Serial Crystallography

New sample environments





- By modifying fixed target loading protocol can maintain modified sample environment
- Useful, for example, for studying oxygen sensitive enzymes at room temperature

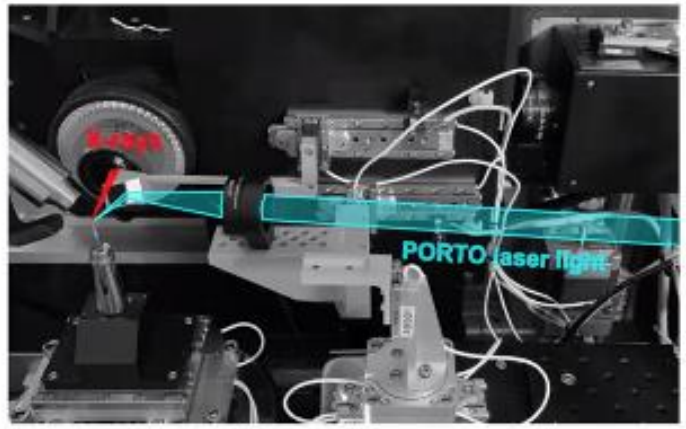
Anerobic and oxygen exposure structures of radiation sensitive oxygenase VioC
Rabe et al. IUCrJ (2020), **7**, 901-912

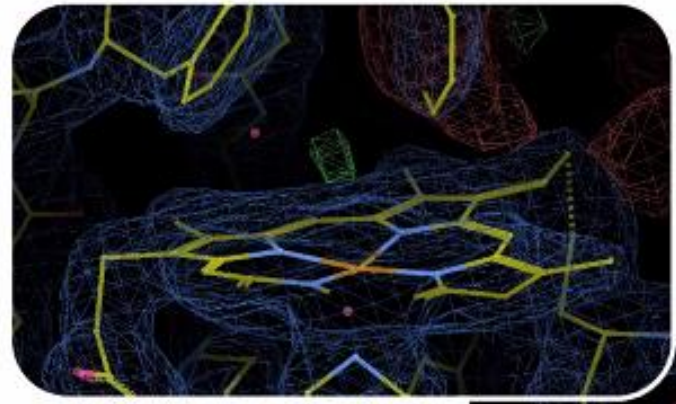
Data collection

(II) Serial Crystallography

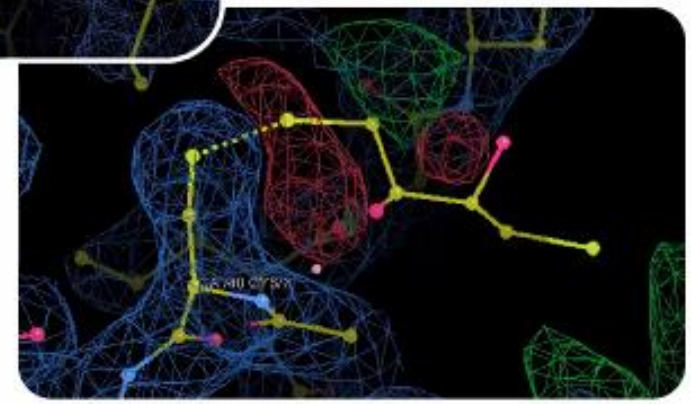
Dynamics

PORTO high power pulsed laser and coolLED available for triggering light driven reactions





Non-light sensitive protein plus photocage after laser flash

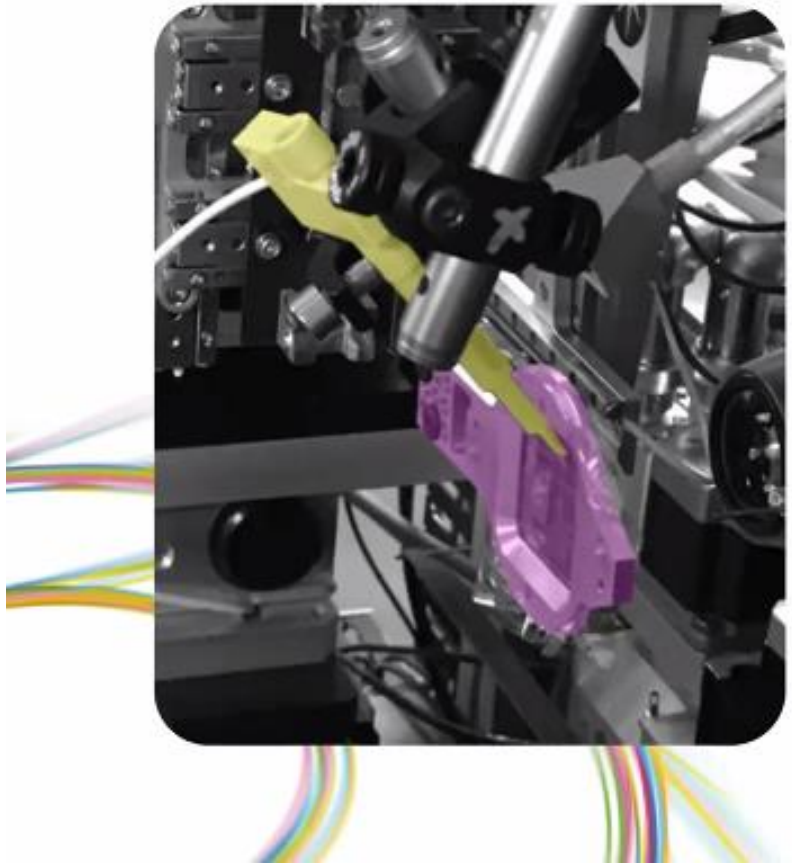


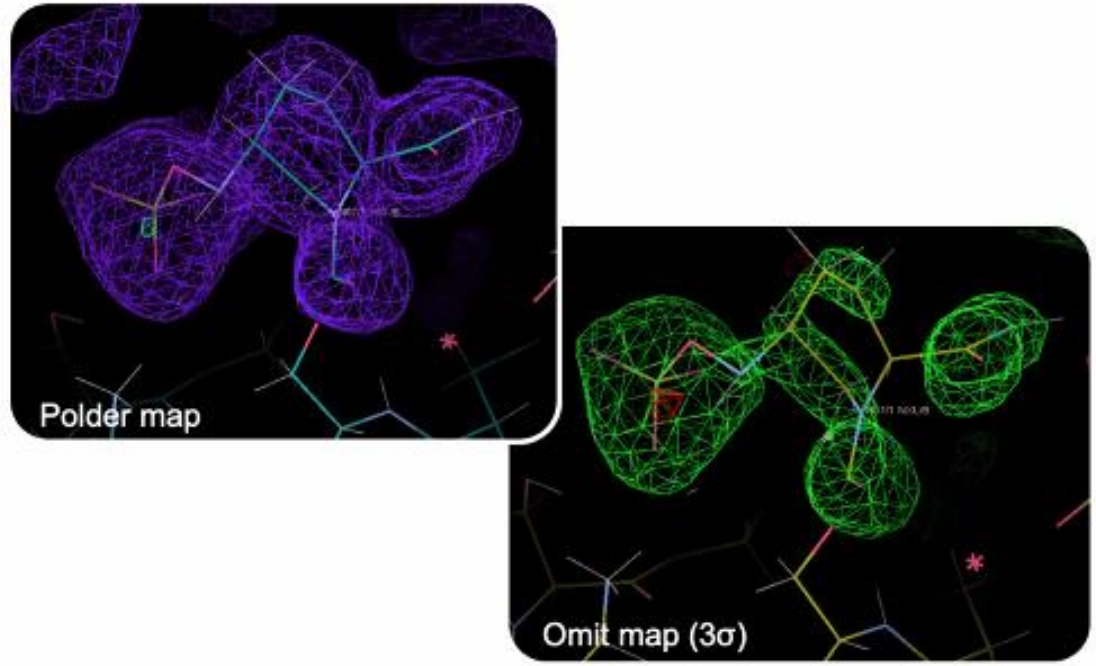
Light sensitive protein after laser flash

Data collection
(II) Serial Crystallography

Dynamics

Microdrop ejector (yellow in image) available. Delivers picolitre droplets of substrate directly onto crystals for triggering substrate driven reactions.





Bound inhibitor at 1.73 Å resolution

Keep in touch…

It is much easier for us to answer questions well ahead of beamtime

- Always happy to provide advice, possible data collection strategies, set up the beamline in a specific way or assist with difficult crystals
- On-site users welcome

Use your local contact and feel free email/phone ahead of your experiment.
Local contacts are usually one of

- Robin Owen
- Danny Axford
- Sam Horrell
- Ali Ebrahim
- Pierre Aller
- Jitka Waterman (industrial users)

Come and work with us..

PDRA position available

- Closing data 23rd Jan
- See Diamond website to apply or get in touch to find out more