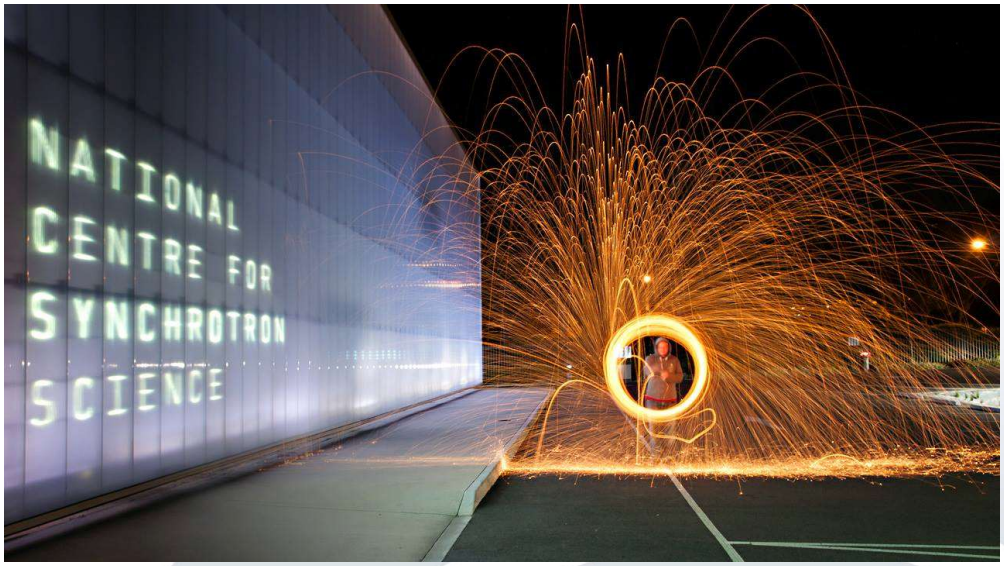
HDR DETECTORS and CHEMICAL CRYSTALLOGRAPHY at the AUSTRALIAN SYNCHRORTON MX BEAMLINES



Dr Jason Price

ANSTO - Melbourne
Beamline Scientist, MX1 and MX2 Beamlines

Science. Ingenuity. Sustainability.

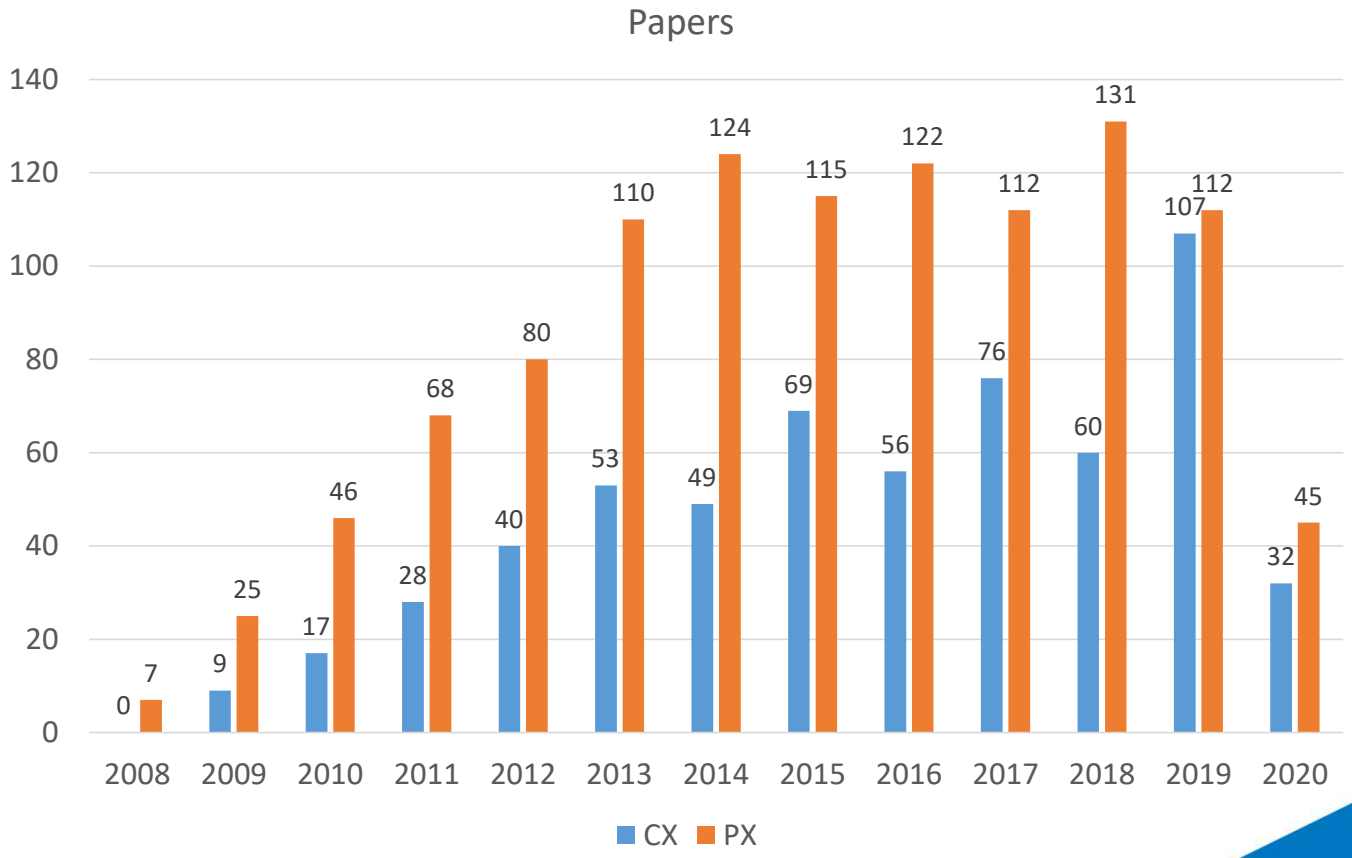
CX and PX Journal Publications

MX1 and MX2 cater to both the CX and PX communities

1693 publications

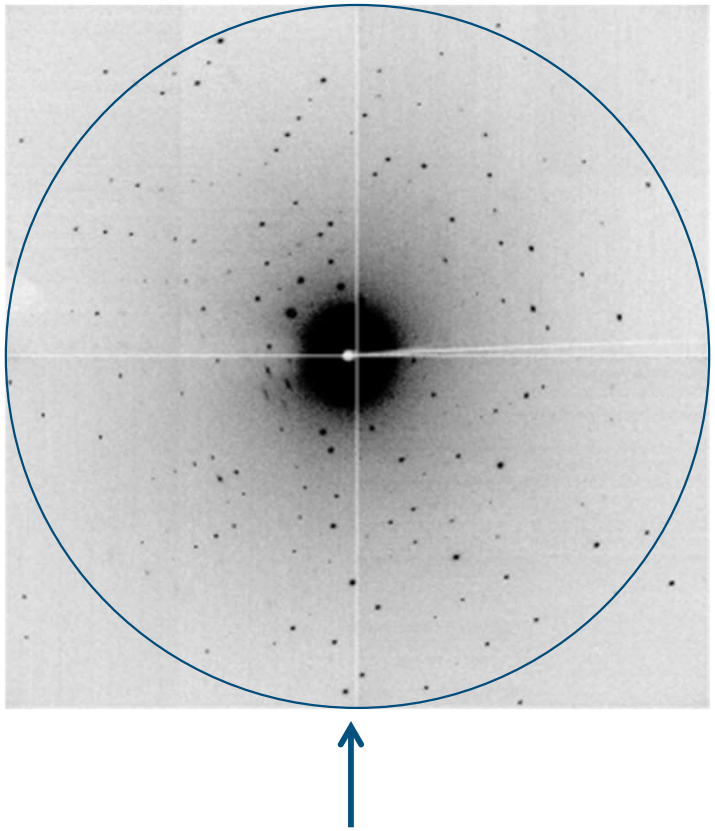
596 papers from CX
1097 paper from PX

~ 35% of publications are from CX community

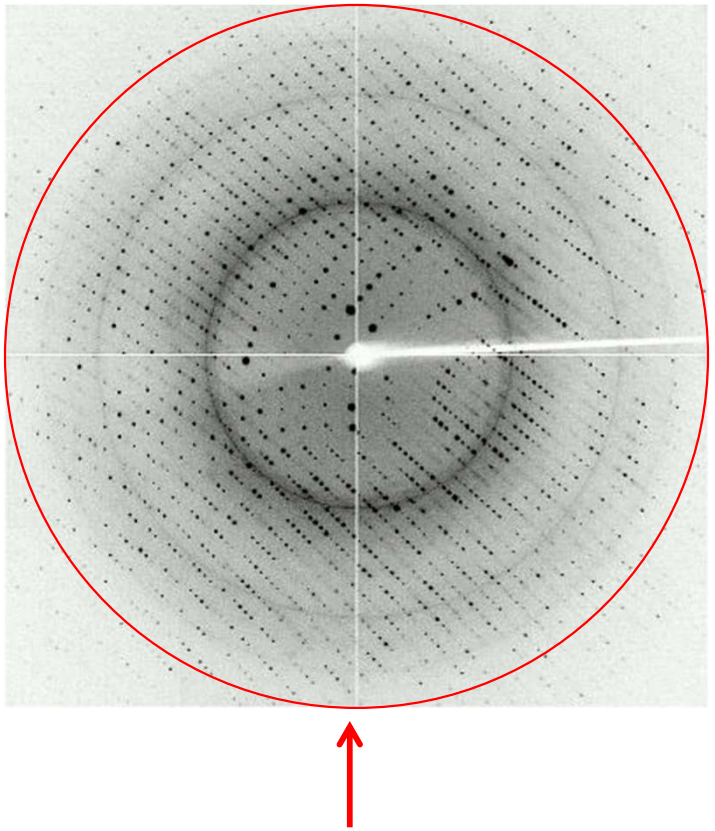


ANSTO

Resolution CX vs. PX



0.76 Å (17.444 keV, 72 mm)



1.76 Å (13.0 keV, 171 mm)

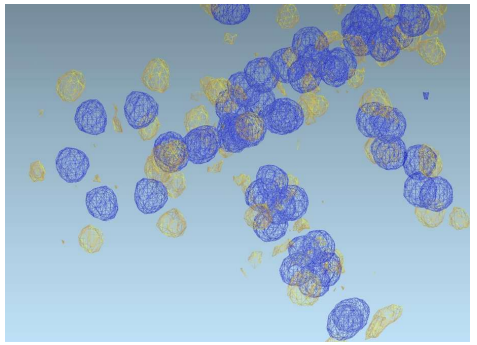
ANSTO

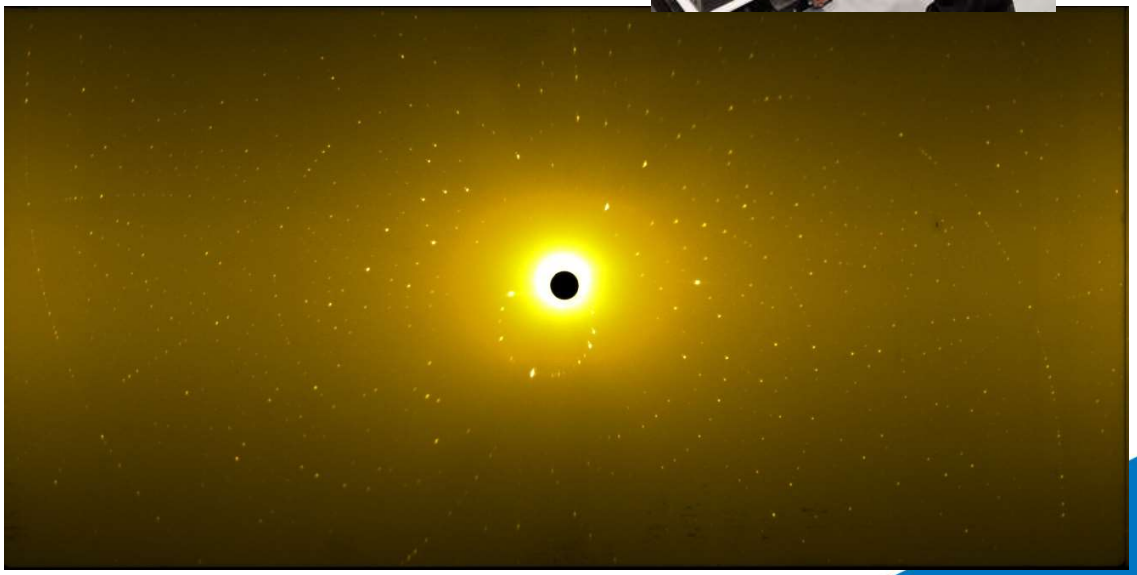
Detector type preferred by CX

Different requirements from Structural biology

Smaller unit cells give, more cells for a crystal volume.

- Stronger signal
- Radiation damage - fewer defects for total diffraction of crystal





https://www.rigaku.com/products/smc/synergys?index=1

NEUTRON SCATTERING INSTRUMENTS

Koala - Laue Diffractometer

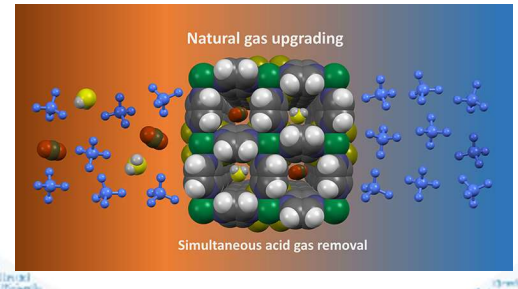
ANSTO

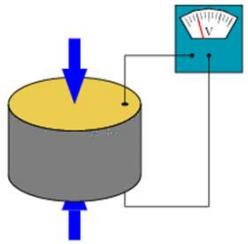
A broad CX user ba

Diversity of sample and experiment types in CX

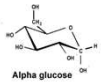
Research Areas

Supramolecular assemblies of light elements.
MOFS – Metal Organic Frameworks (Coordination polymers)
Minerals
Inorganic Materials
Supramolecular Chemistry
Spin cross over phase transitions
Actinides
Structure Determination for Magnetic properties
(Lanthanide SMM)
SCXRD – overlap with PD
Solid State Photochemistry
High pressure cells
Absolute Structure Determination





Piezoelectric crystals



polymers

Solar cells

Two Single Crystal X-ray Diffraction Beamlines

MX1:

- Energy range: 8 keV – 17.5 keV
- Beam focus size at sample: **180 µm x 160 µm** (HxV)
- Flux at the sample: 3.6 e10^{11} ph/s @ 13 keV
- Mini kappa geometry available
- Dectris Eiger 2 9M detector

MX2:

- Energy range: 8.5 keV – 17.5 keV
- Beam focus size at sample: **22 µm x 13 µm** (HxV)
- Positional and intensity stability: 1 µm position stability
- Flux at the sample: 1.2 e10^{12} ph/s @ 13 keV
- Micro collimator (7.5, 10, 20 µm)
- Dectris Eiger 16M detector (ACRF and user community funded)

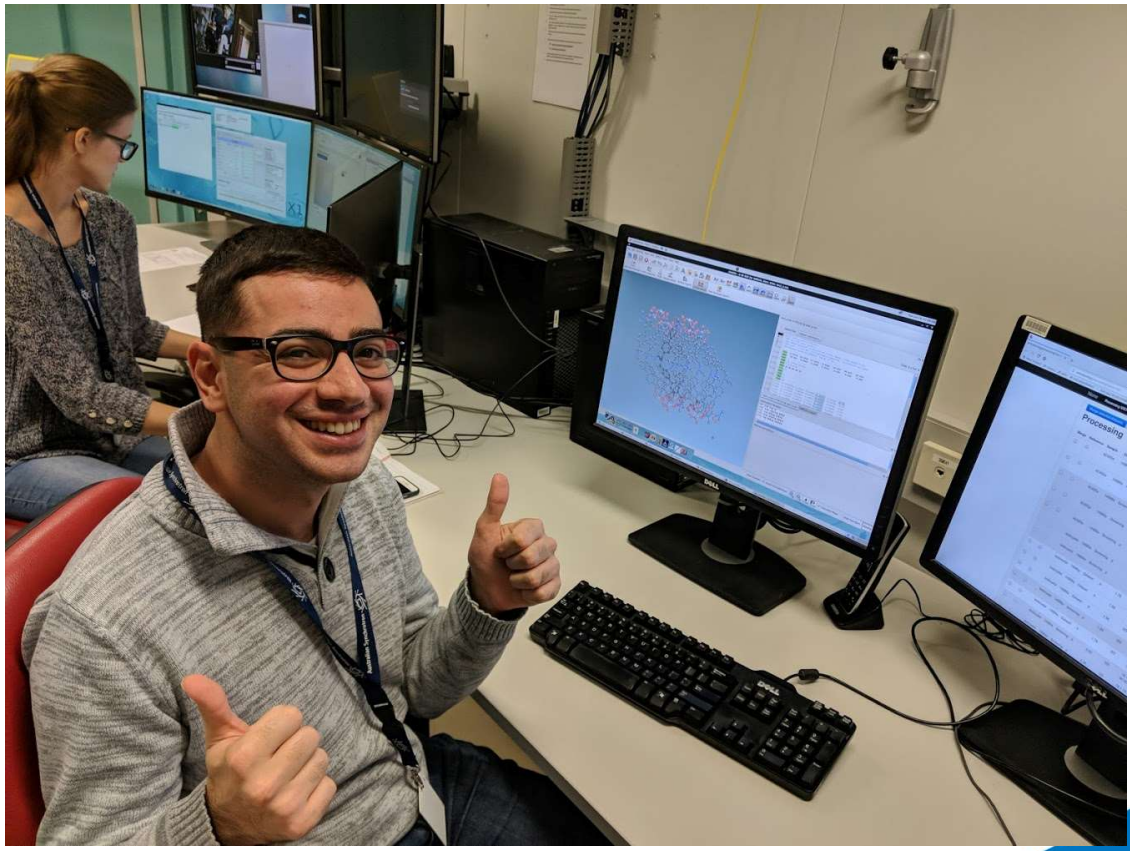
Both MX1 and MX2:

- Robotic loading of samples
- Remote collection available

ANSTO

Implementation Challenges

- Angle of Incidence correction (CCD)
- Unfamiliar Detectors
- Unfamiliar Software
- Data reprosseing
- Beam Center
- Fall off at high angle
- Different intensities at same resolution
- Count Rate Correction
  - Very low mosaic spread



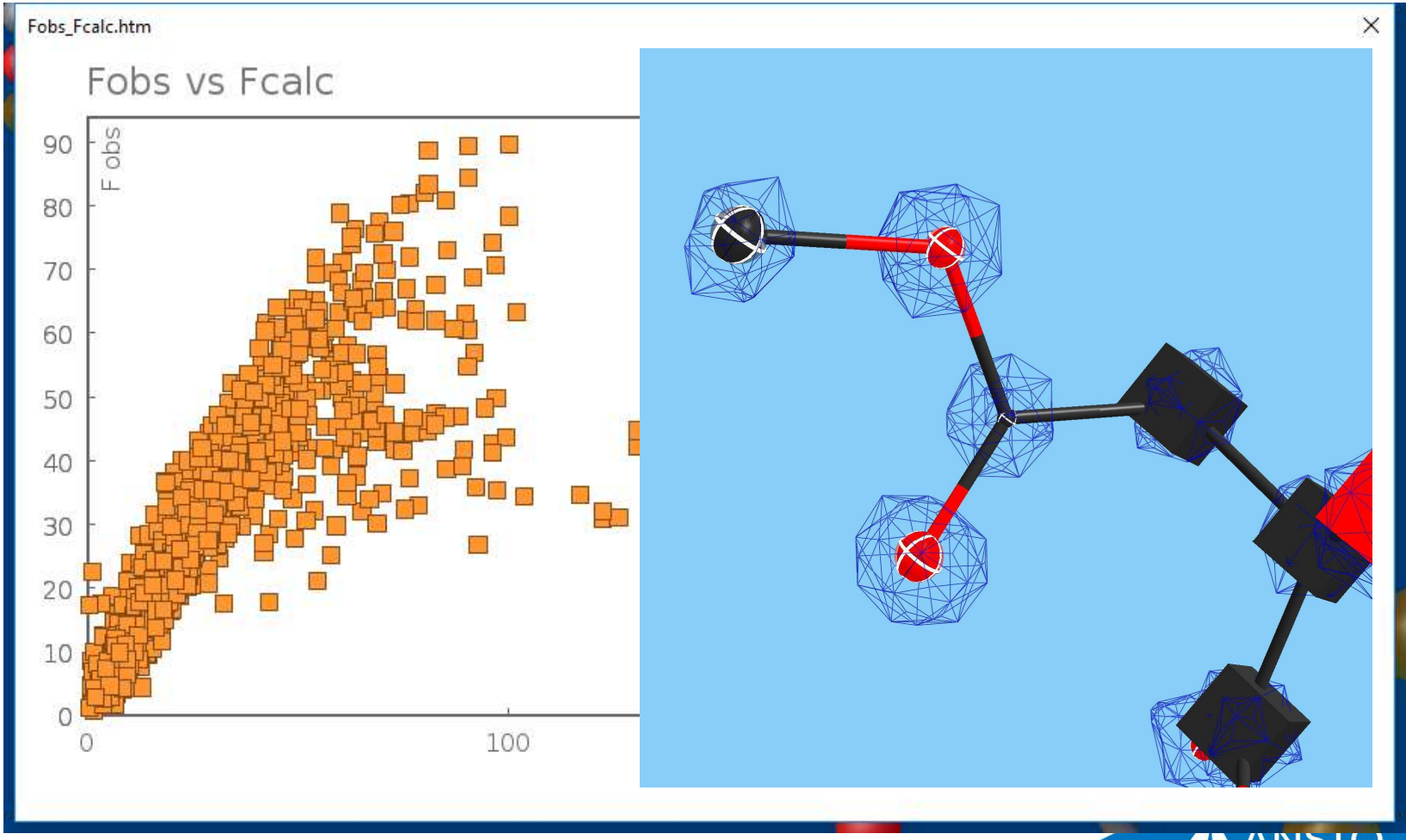
Ali Chahine – Monash Chemistry
First new CX structure collected on Eiger 2 9M at MX1, 18/06/2019

ANSTO

Count rate and CX

CX has more issues with count rate

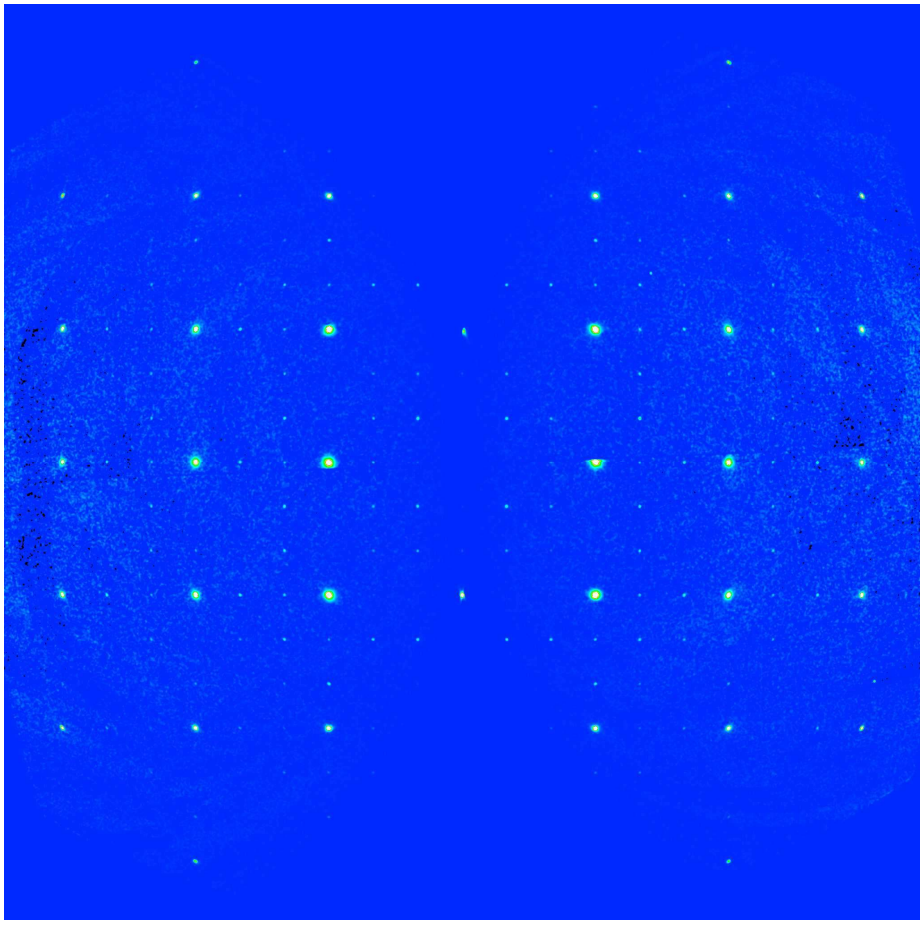
- What is the best attenuation.
- A few overloads On a CCD is not so bad.
- On Hybrid Photon Counting (HPC) indicates well above count rate.



Count rate and CX – Intensity Differences

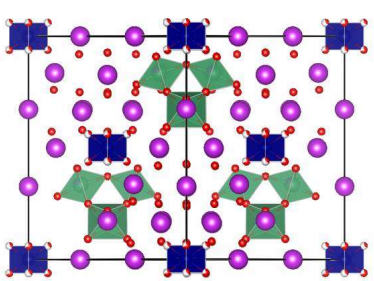
How bad could it really be? Modulation in $Bi_{23}CrNb_3O_{45}$

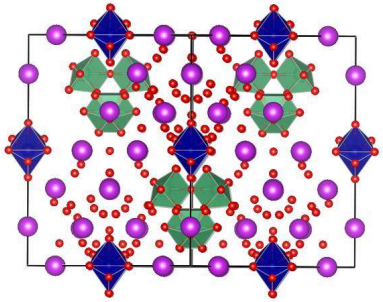
Prof. Chris Ling, Chemistry, The University of Sydney



Element	Percent by Weight
O	12.29%
Cr	0.89%
Nb	4.76%
Bi	82.06%

Space group	Fm-3m	F-43m
a, b, c (Å)	~5.5	~16.57
V (Å^3)	1426.87(7)	5707.7(3)

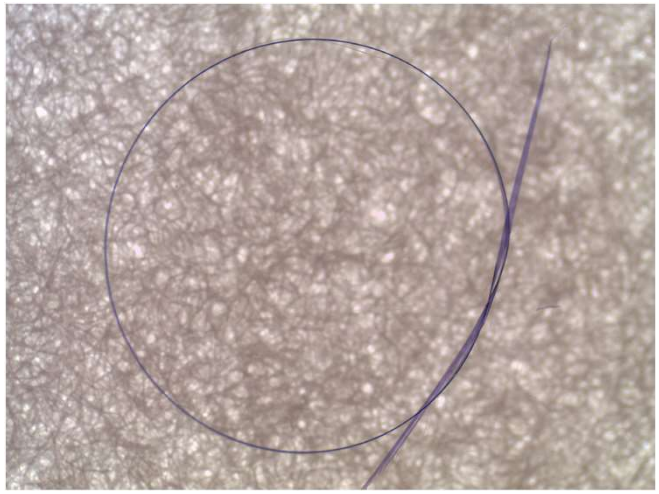


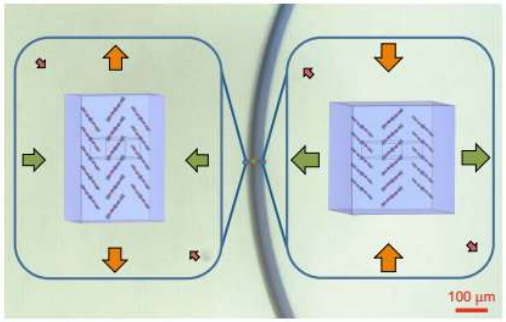


ANSTO

Bendable crystals

- Crystals of copper(II) acetylacetonate
- Repeatably, reversibly bent
- Full data collection on MX1
- Mapping studies on MX2 using the 7.5 micron micro collimator

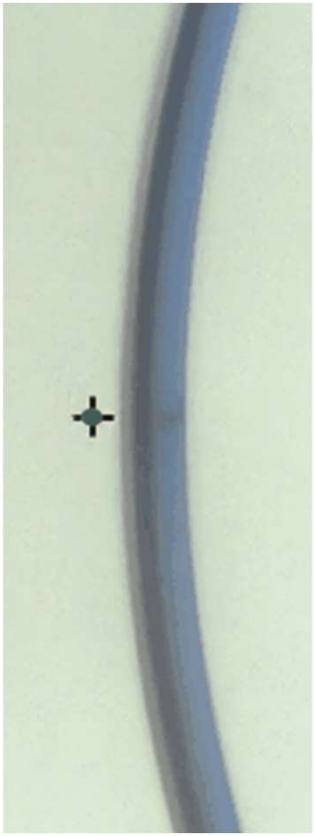




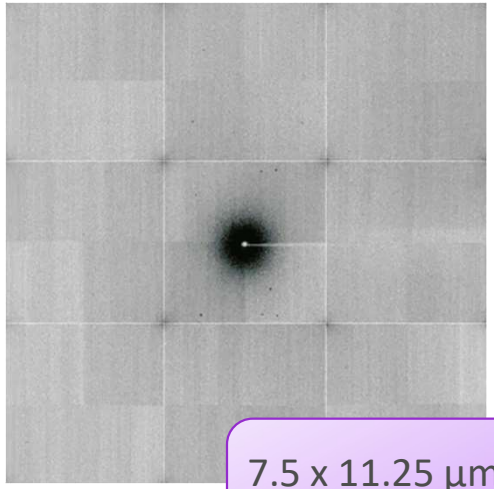
Worthy et al. 2018. *Nature Chemistry* 10 (1), 65-69

ANSTO

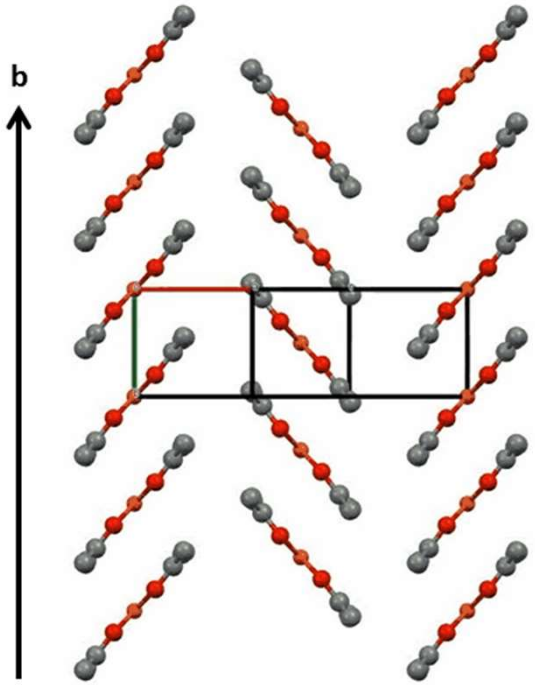
Structural Changes Across a Bent Crystal Using MX2 Micro-Focused Synchrotron Radiation

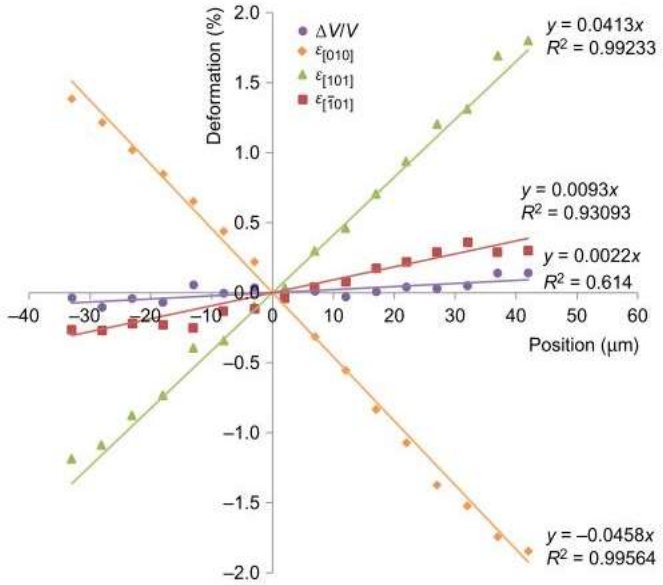


10° wedge every 5 µm



7.5 x 11.25 µm beam



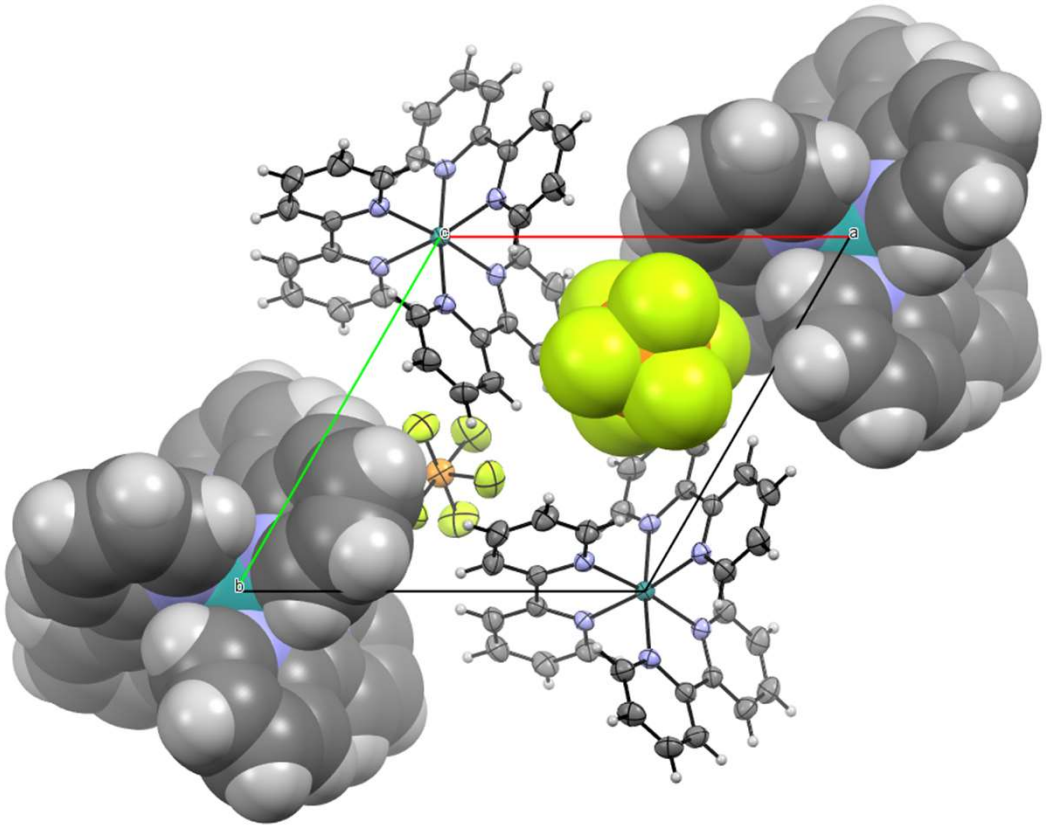


- Atomic-resolution of cell deformation
- Rotation of molecules facilitates bending

But this study was done by manually moving the beam and refining each structure individually

Worthy, A.; Grosjean, A.; Pfrunder, M. C.; Xu, Y.; Yan, C.; Edwards, G.; Clegg, J. K.; McMurtrie, J. C., *Nat Chem* **2018,** *10* (1), 65-69.

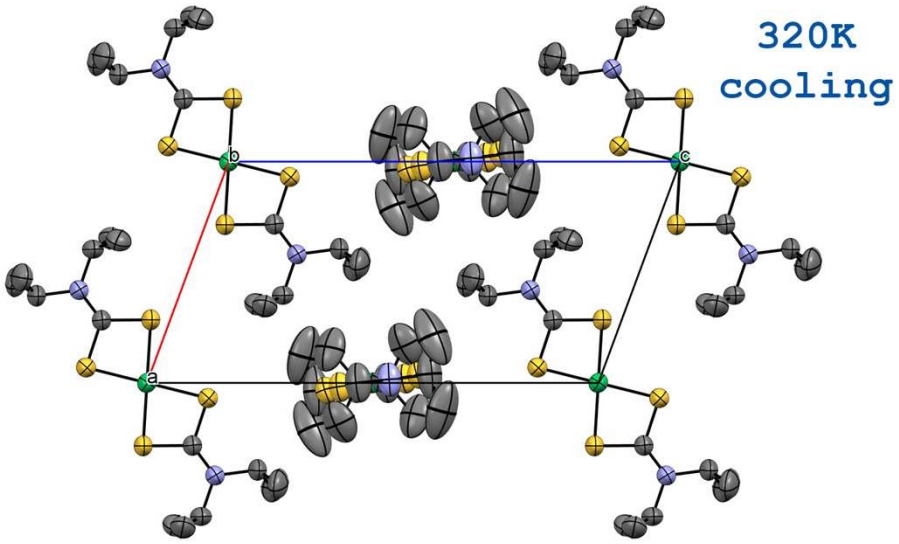
1 Second Data Collection the MX1 Dectris Eiger 2, 9M

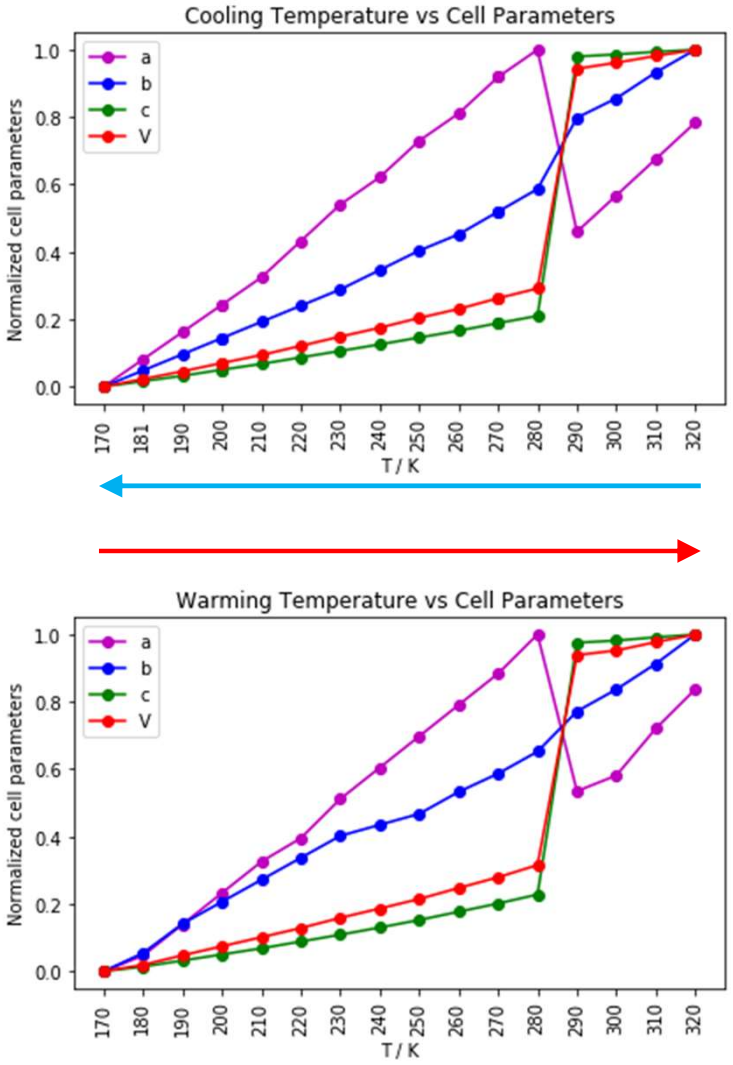


Sample	$[Ru(II)(2,2'\text{-bipyridine})_3](PF_6)_2$
Crystal System	Trigonal
Space Group	*P*-3*c*1
Strategy	**180° sweep, 1 sec, 180 Hz**
a, b (Å)	10.6825 (10)
c (Å)	16.356 (3)
V (Å^3)	1616.4 (6)
Crystal Size (mm)	0.040 x 0.030 x 0.025
Attenuation	0%
Wavelength(Å) / Energy (keV)	0.71073 / 17.444
Temperature	220 (2) K
Data completeness	**0.996**
θ (max) °	25.982
R_1 (reflections)	**0.0303 (918)**
wR_2 (reflections)	0.0850 (1056)
Bond Precision C – C (Å)	0.0040

ANSTO

Investigation of an abrupt phase transition





Uses all of the tools developed.

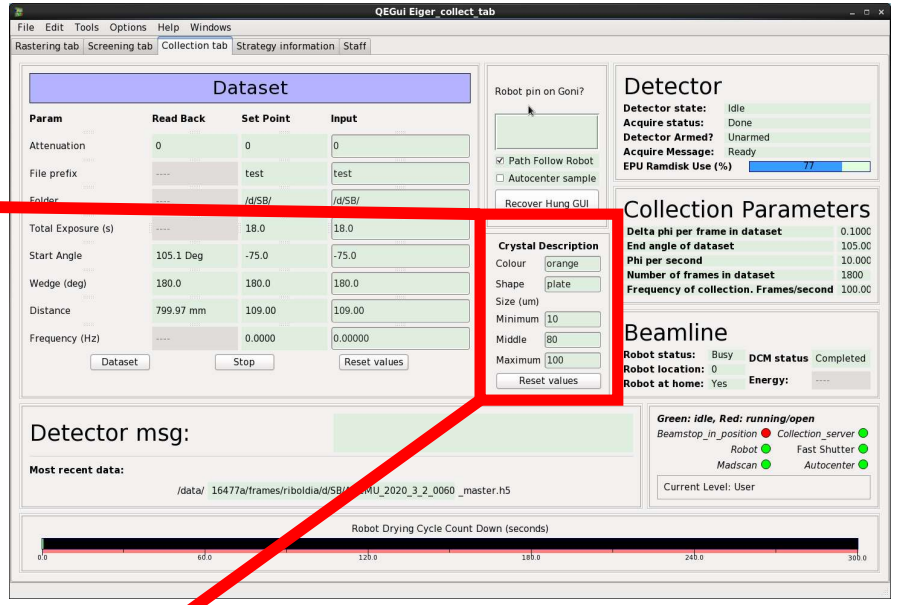
- Monoclinic system
- Temperature ramp
  - Ramp temperature and trigger data collection at temperature.
- Heat 5K for every minute
- Collect every 2 mins (10K), **32 full data collections in 60 minutes**
- 240° sweep, 12 s, 200Hz

Alexander Angeloski, *et. al*., UTS

ANSTO

FAIR and FACT, compliance issues

CIF key terms	CIF key values
_cell_measurement_temperature _cell_measurement_reflns_used _cell_measurement_theta_min _cell_measurement_theta_max	"Temperature from device" "Value from IDXREF.LP" "Value calculated from IDXREF.LP and SPOT.XDS" "Value calculated from IDXREF.LP and SPOT.XDS"
_exptl_crystal_description _exptl_crystal_colour _exptl_crystal_size_max _exptl_crystal_size_mid _exptl_crystal_size_min	User input auto-populated from collection database (MongoDB) if included in GUI. Or "user input"
_exptl_absorpt_correction_type _exptl_absorpt_correction_T_min _exptl_absorpt_correction_T_max _exptl_absorpt_process_details	"Multi-Scan" (true for XDS and sadabs "Value not reported by XDS" or "will be present in autoprocess_sadab "Value not reported by XDS" or "will be present in autoprocess_sadab "XDS (Kabsch, 2010)" or "sadabs (Bruker, 2001)"
_diffrn_ambient_temperature _diffrn_radiation_wavelength _diffrn_radiation_type _diffrn_radiation_source _diffrn_measurement_device_type _diffrn_radiation_monochromator _diffrn_measurement_device_type _diffrn_measurement_method _diffrn_detector_area_resol_mean	"Temperature from device" "Wavelength from beamline" "Synchrotron" "MX1 Beamline Australian Synchrotro "MX2 Beamline Australian Synchrotro "MX1 (ref)" or "MX2 (ref)" "Silicon Double Crystal" "Dectris Eiger2 9M" or "Dectris Eiger 16M" "Omega Scan" 13.3 (Dectris Eiger detectors)
_computing_data_collection _computing_cell_refinement _computing_data_reduction	'AS QEGUI' 'XDS (Kabsch, 2010)' 'XDS (Kabsch, 2010)'



For the NX sample base_class, these values are not included.

```
_exptl_crystal_description          prism
_exptl_crystal_colour               colourless
_exptl_crystal_size_max             0.32
_exptl_crystal_size_mid             0.27
_exptl_crystal_size_min             0.10
```

ANSTO

Acknowledgements

The Australian Synchrotron MX Team

MX 1 & 2 Team-leaders

Alan Riboldi-Tunnicliffe (Structural Biology)

Rachel Williamson (Chemical Crystallography)

MX3 design

Tom Caradoc-Davies – Seconded to Lead MX3 Design and Build

MX Team

~~Jun Aishima (PX Computing Post-Doc)~~ -> NSLS II USA

~~David Aragao (Structural Biology)~~ -> DLS I04 UK

Stephanie Boer (Chemical Crystallography)

Eleanor Campbell (Structural Biology)

~~Nathan Cowieson (Structural Biology)~~ -> DLS BioSAXS UK

Daniel Eriksson (Structural Biology) -> MX3 (September)

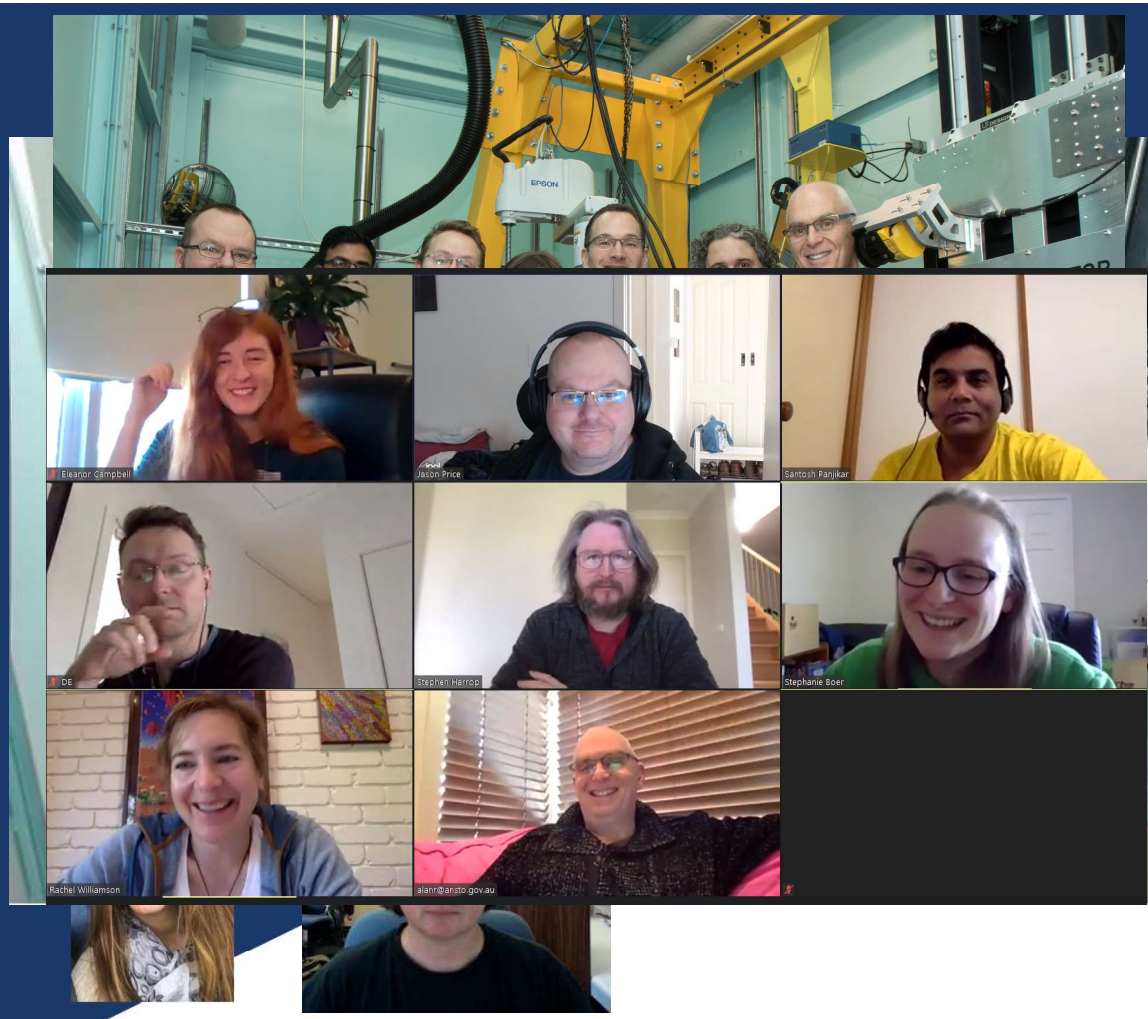
Stephen Harrop (Structural Biology)

~~Christine Gee (Structural Biology)~~ -> Berkley USA

Santosh Panjikar (Structural Biology)

Jason Price (Chemical Crystallography)

~~Kate Smith (Structural Biology)~~ -> PSI CH



Australian Government

ANSTO