Australian Government

ANSTO

Australian Synchrotron MX beamlines Update 2019 HDRMX Meeting

Dr Tom Caradoc-Davies

Lead Scientist, - MX3 Beamline

Science. Ingenuity. Sustainability.

Two Single Crystal X-ray Diffraction Beamlines: MX1 and MX2

MX1:

- Energy range: 8 keV – 17.5 keV
- Beam focus size at sample: 180 µm x 150 µm (HxV)
- Flux at the sample: 3.6 $e10^{11}$ ph/s @ 13.000 keV
- Mini kappa geometry available
- Dectris Eiger2 9M, detector (dataset in 18 sec)

MX2:

- Energy range: 8.5 keV – 17.5 keV
- Beam focus size at sample: 22 µm x 12 µm (HxV)
- Positional and intensity stability: 1 µm position stability
- Flux at the sample: 2.4 $e10^{12}$ ph/s @ 13.000 keV
- Micro collimator (7.5, 10, 20 µm)
- Dectris Eiger 16M detector (dataset in 18 sec)

Both MX1 and MX2:

- Robotic loading of samples
- Remote collection available

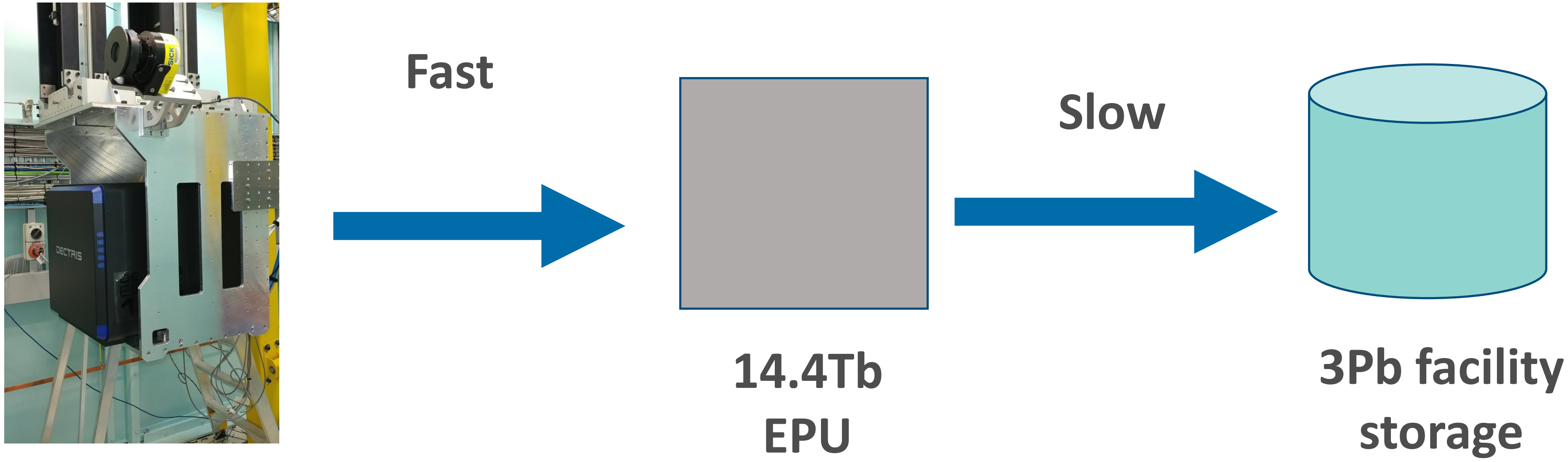
ANSTO

A little bit about infrastructure

Typical monthly use stats

Beamline	Total number of EPNs	Total data size (Tb)	Mean data per EPN	Total Robot mounted pins	Mean mounts per EPN
MX1	23	11.374	0.495	1796	78
MX2	32	33.108	1.035	3363	105

Local vrs central storage

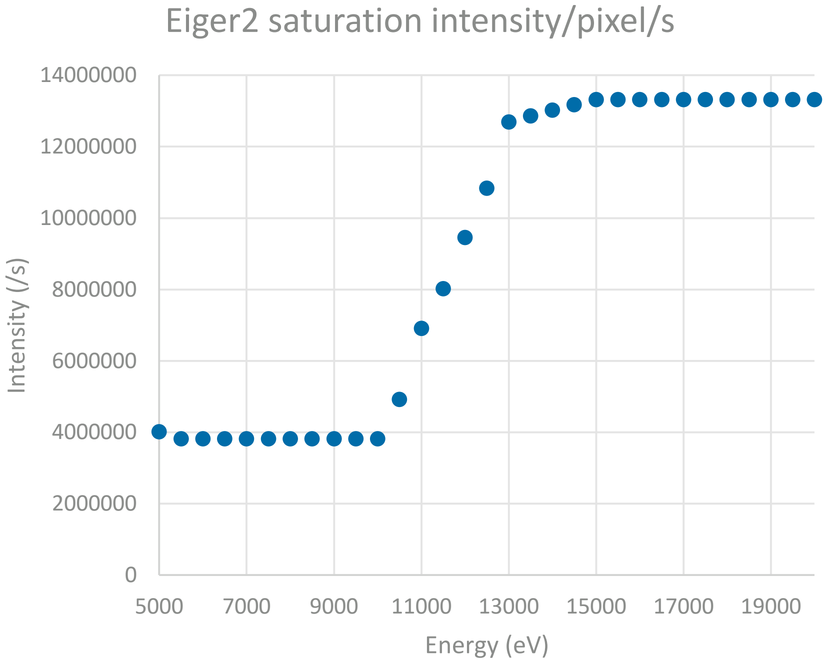


MX3 on the way!

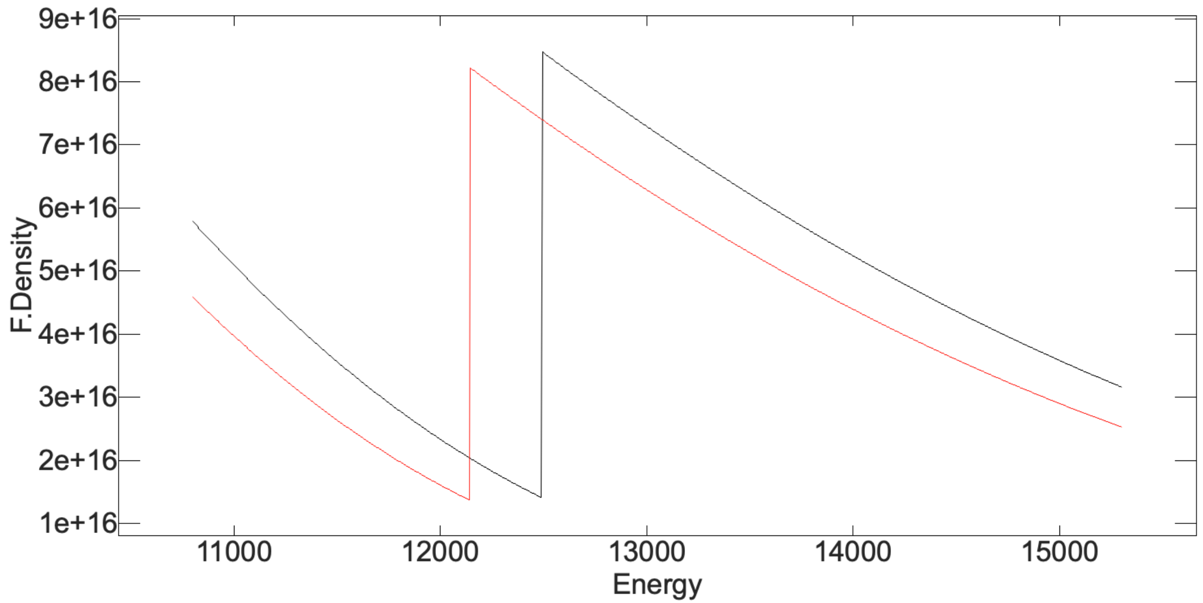
Beamline	Full beam size (μ)	Min collimated beam size (μ)	Full beam flux at sample (ph/s)	Key Capabilities	Science
MX1	180x150	50x50	3.40E+11	High stability, kappa goniometer, low energy collection, high data quality, high throughput, fragment screening, MAD/SAD	CX/MX, weak anomalous data, medium sized crystals
MX2	22x12	10x8	2.40E+12	Hot beam, small crystals, High data quality, high throughput, MAD/SAD	Weak and small CX/MX. General commercial
MX3	8.5x1.5	1.5x1.5	1.10E+14	Microfocus, very hot beam, changeable beam size, sophisticated endstation for micro crystals, Serial crystallography via goniometer, fixed targets and injectors. In-tray screening and collection. fixed-energy	Nano MX. **Weakly scattering small crystals** **Membrane proteins** **Large protein complexes**

Potential count-rate problem with larger, better diffracting samples on MX3

Eiger2 and beamline energy – MX3 design



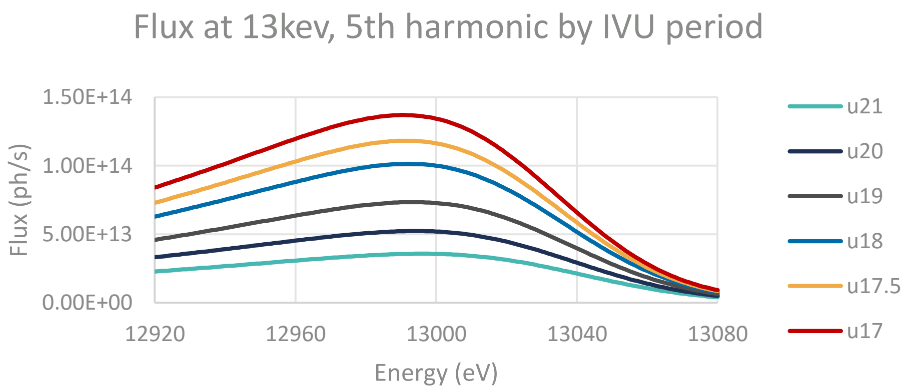
Eiger2 pixel saturation by energy. Data provided by Dr. Cameron Kewish.



Tuning curve for 5th 3m IVUs. U17.5 is shown in black and u18 in red. Calculated at 37m for K range of 0.5-1.41. The bottom of the 5th harmonic is 12.494 keV for u17.5 and 12.146 for u18.

Eiger2 saturation will have a big influence on the ability to handle very high count rates

Calculated for 3m IVU, 20 from source, 1x1mm slit.



Challenges and opportunities

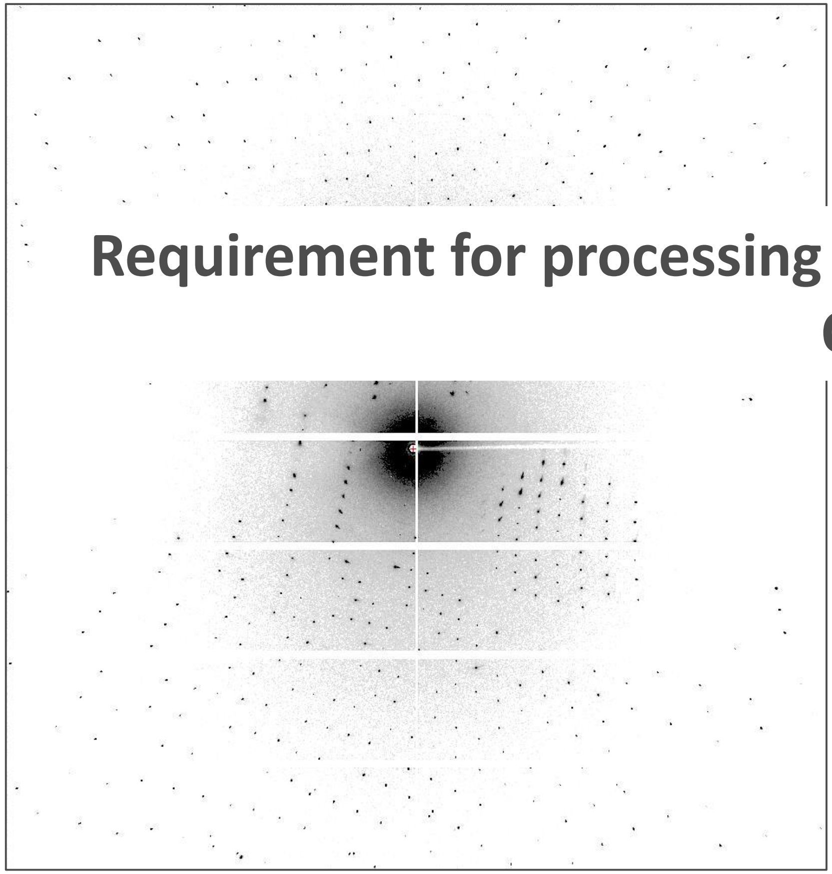
CX can have issues on MX lines!

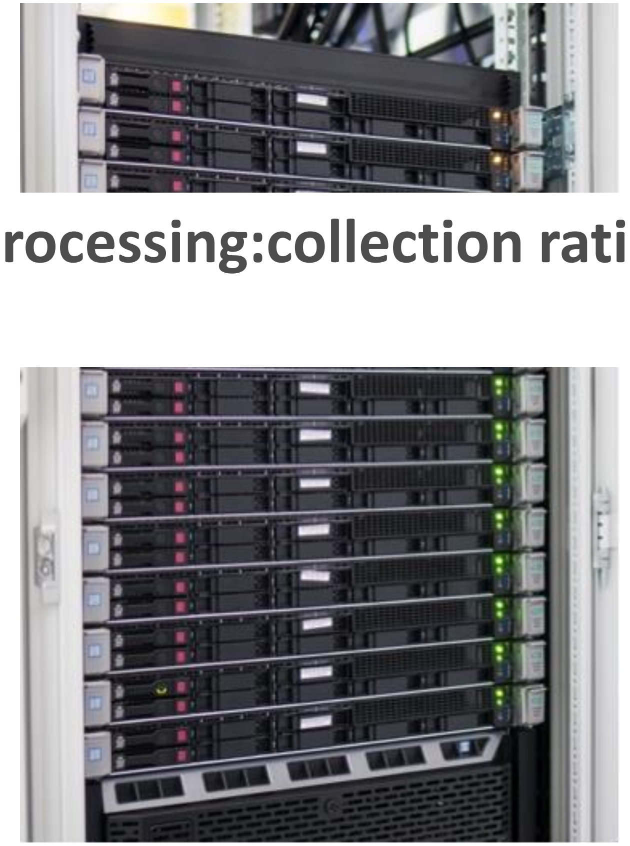
Keeping up with collection – Real time processing

Standard collection 360° in 36 sec, 100Hz (3600 frames)

Previously 15 min 1° collected over 1 sec. 360 frames.

Eiger - Dedicated computing nodes
MX2 - **14 nodes installed**
MX1 - **16 nodes installed**
1920 cores, 3840 Gb RAM





Requirement for processing is min 4:1 processing:collection ratio. Often 2:1.

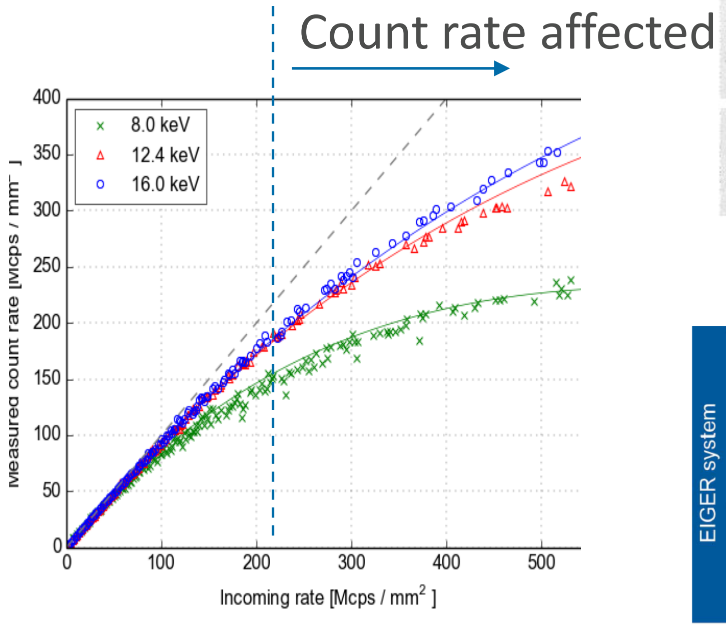
ANSTO

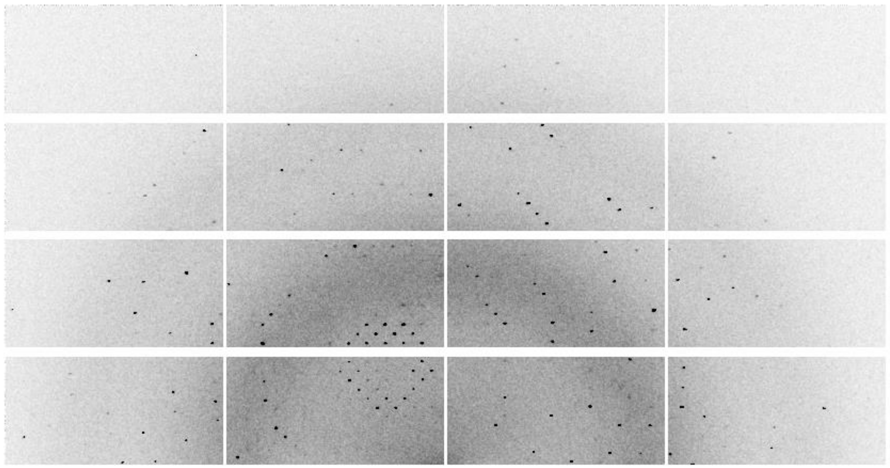
Count rate and CX

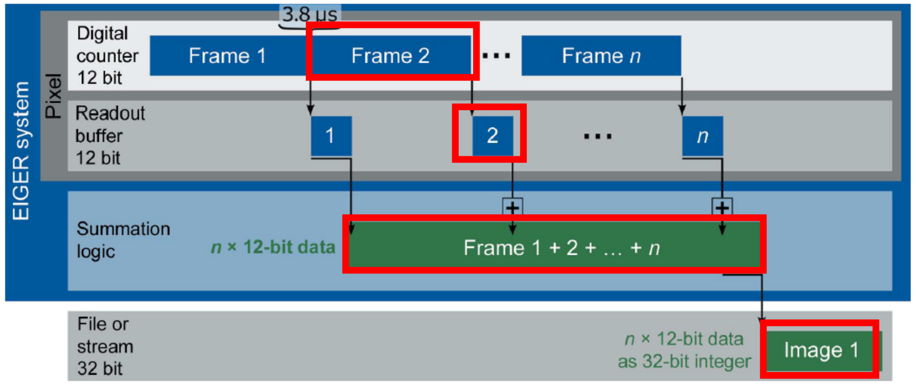
CX has more issues with count rate

- Well-ordered materials necessary for high resolution structures
- Small mosaicity, so diffraction peaks are sharper
- Stronger scatterers can cause stronger diffraction
- Small unit cell means fewer diffraction spots, so each bad pixel has a proportionally larger impact

- New tools being developed





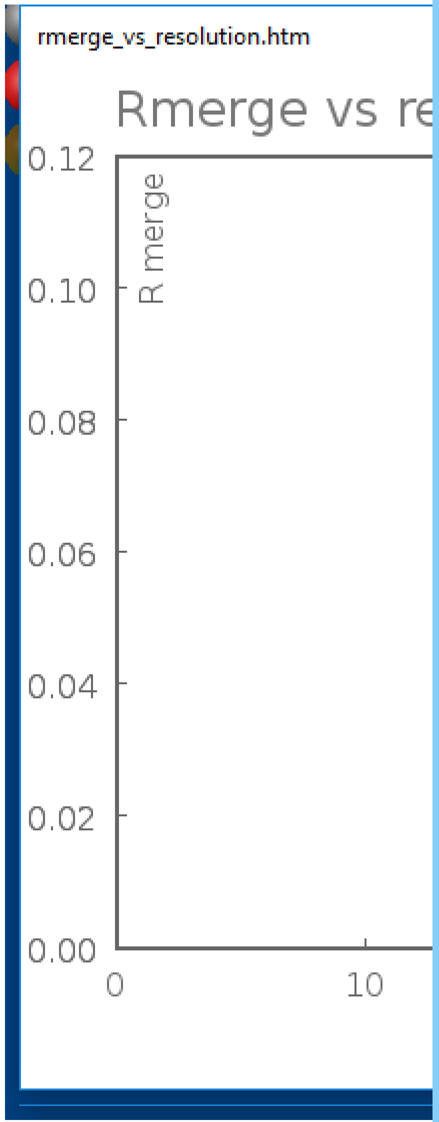


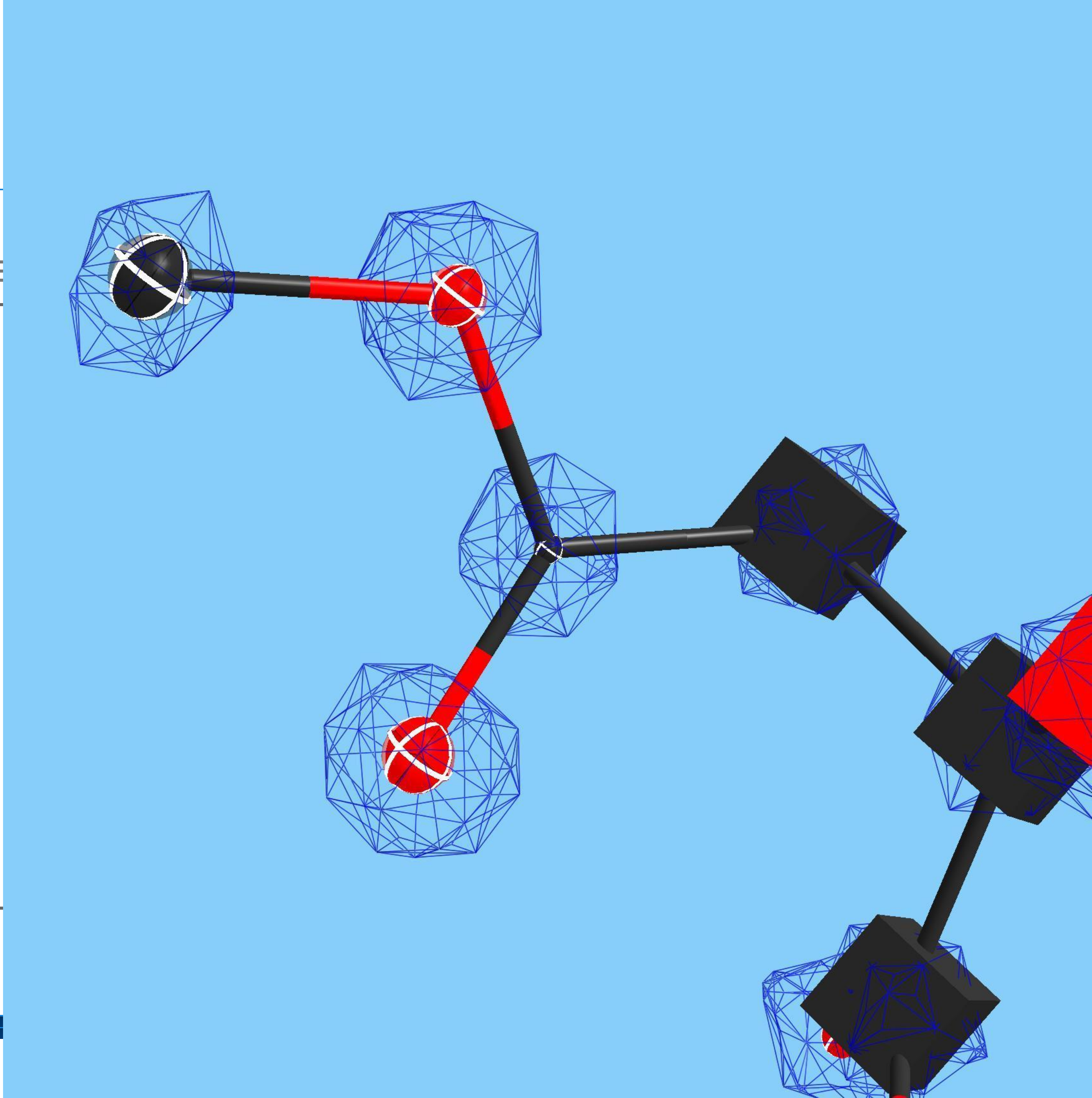
If any subframe is overexposed, the entire frame (a single pixel) is marked as overexposed. (Source: Dectris, Ltd.)

Count rate and CX

CX has more issues with count rate

- Issues with 100% transmission datasets and decent crystals
- New tools being developed to flag problem datasets and warn users.





Eiger2 use-cases

Chemical Crystallography (CX)

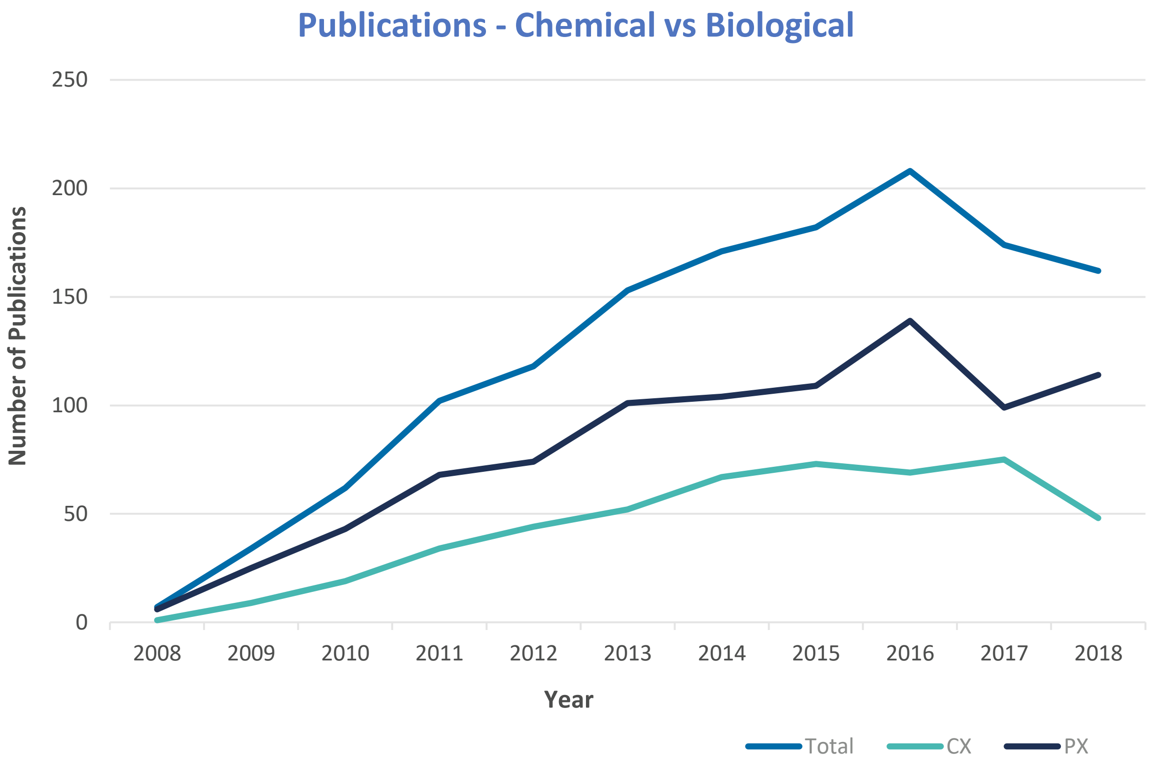
A broad user base

MX1 and MX2 cater to both the CX and PX communities

162 publications (to date) in 2018

1373 papers from the MX beamlines so far.....

~ 30% of publications are from CX community



	2008	2009	2010	2011	2012	2013	2014	2015	2016	2017	2018	Total
Total	7	34	62	102	118	153	171	182	208	174	162	1373
CX	1	9	19	34	44	52	67	73	69	75	48	491
PX	6	25	43	68	74	101	104	109	139	99	114	882
% CX	14.3%	26.5%	30.6%	33.3%	37.3%	34.0%	39.2%	40.1%	33.2%	43.1%	29.6%	35.8%

ANSTO

A broad CX user base

Diversity of sample and experiment types in CX

Research Areas

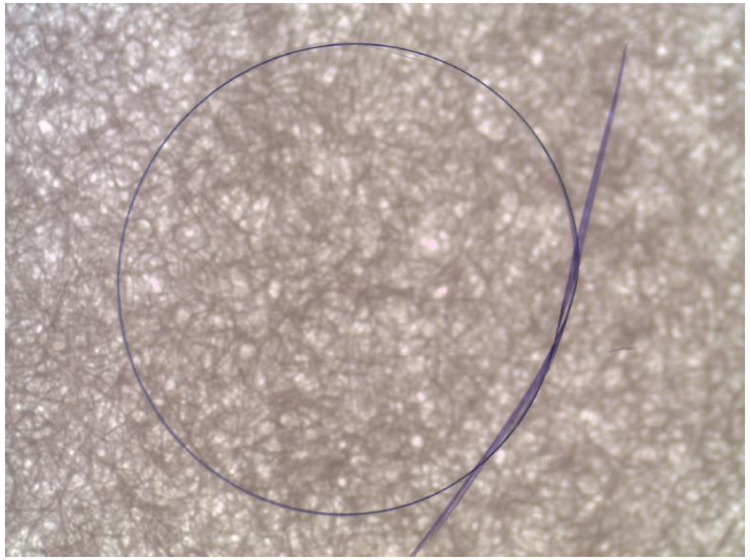
Supramolecular assemblies of light elements.
MOFS – Metal Organic Frameworks (Coordination polymers)
Minerals
Inorganic Materials
Supramolecular Chemistry
Spin cross over phase transitions
Actinides
Structure Determination for Magnetic properties
(Lanthanide SMM)
SCXRD – overlap with PD
Solid State Photochemistry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

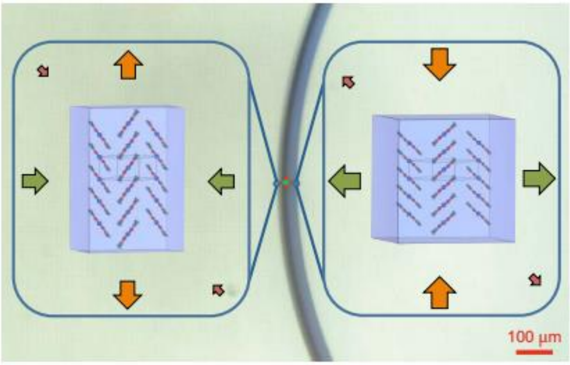
FIG. 1. Intergrown crystals of camerolaite from Padern. Field of view 2 mm across. Georges Favreau specimen, Pierre Clolus photograph.

ANSTO

Bendable crystals

- Assoc. Prof. Jack Clegg (UQ)
- 2018 Malcolm McIntosh Prize for Physical Scientist of the year
- Crystals of copper(II) acetylacetonate
- Repeatably, reversibly bent
- Full data collection on MX1
- Mapping studies on MX2 using the 7.5 micron micro collimator



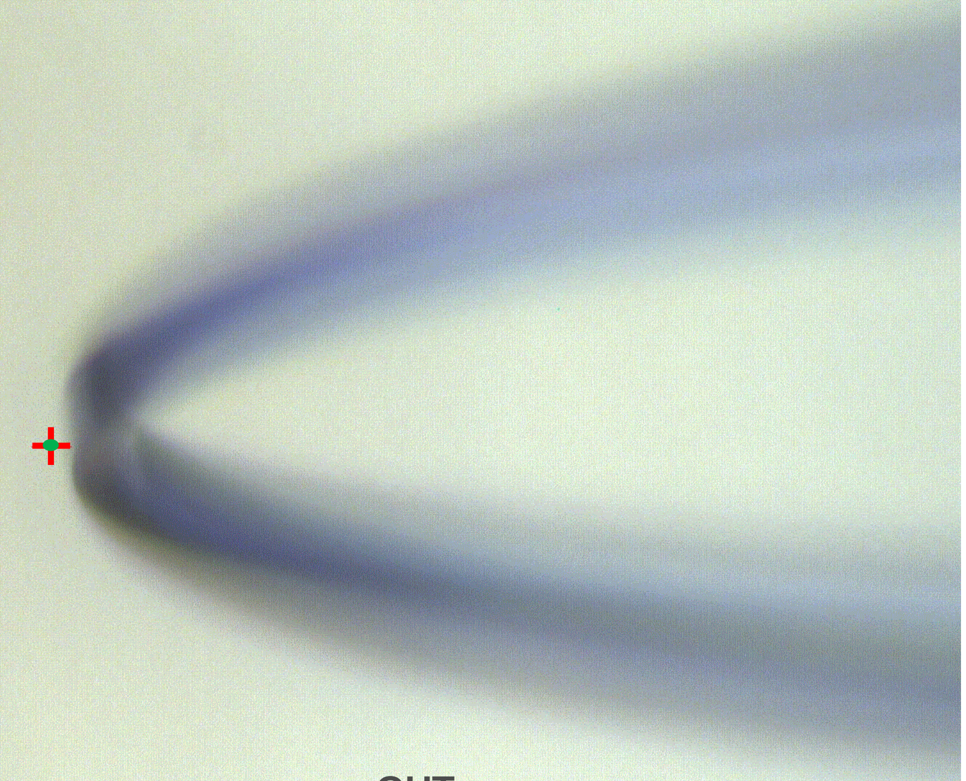


Worthy et al. 2018. *Nature Chemistry* 10 (1), 65-69

ANSTO

MX2 - Structural mapping using micro X-ray diffraction

- X-ray beam with a FWHM of 7.5 x 11.25 µm (green dot)



University of Queensland

- Dr Arnaud Grosjean
- Dr Jack Clegg
- Dr Michael Pfrunder

QUT

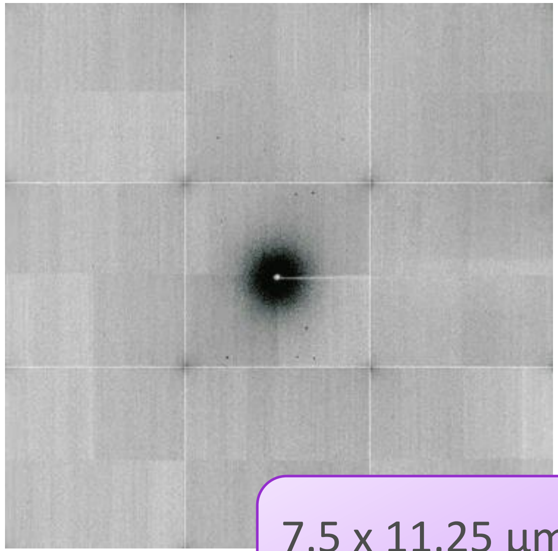
A/Prof. John McMurtrie

Anna Worthy

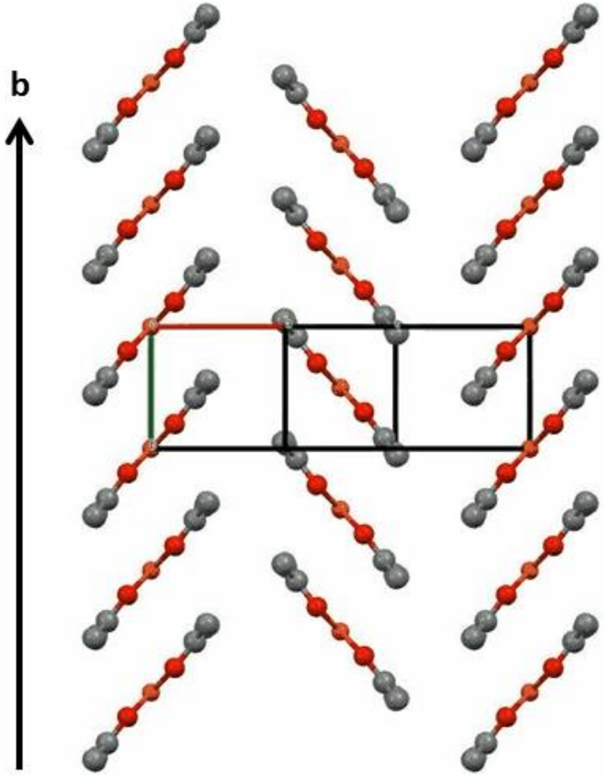
ANSTO

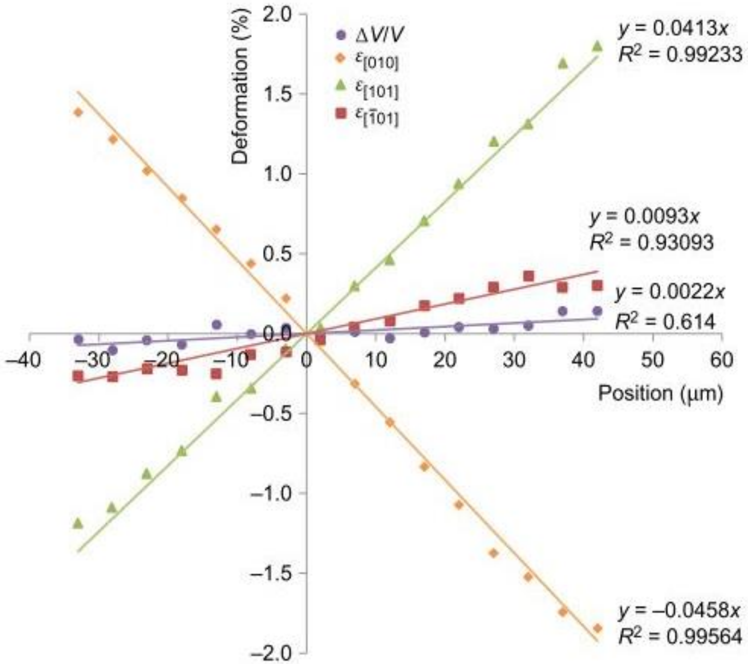
Structural Changes Across a Bent Crystal Using MX2 Micro-Focused Synchrotron Radiation

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.

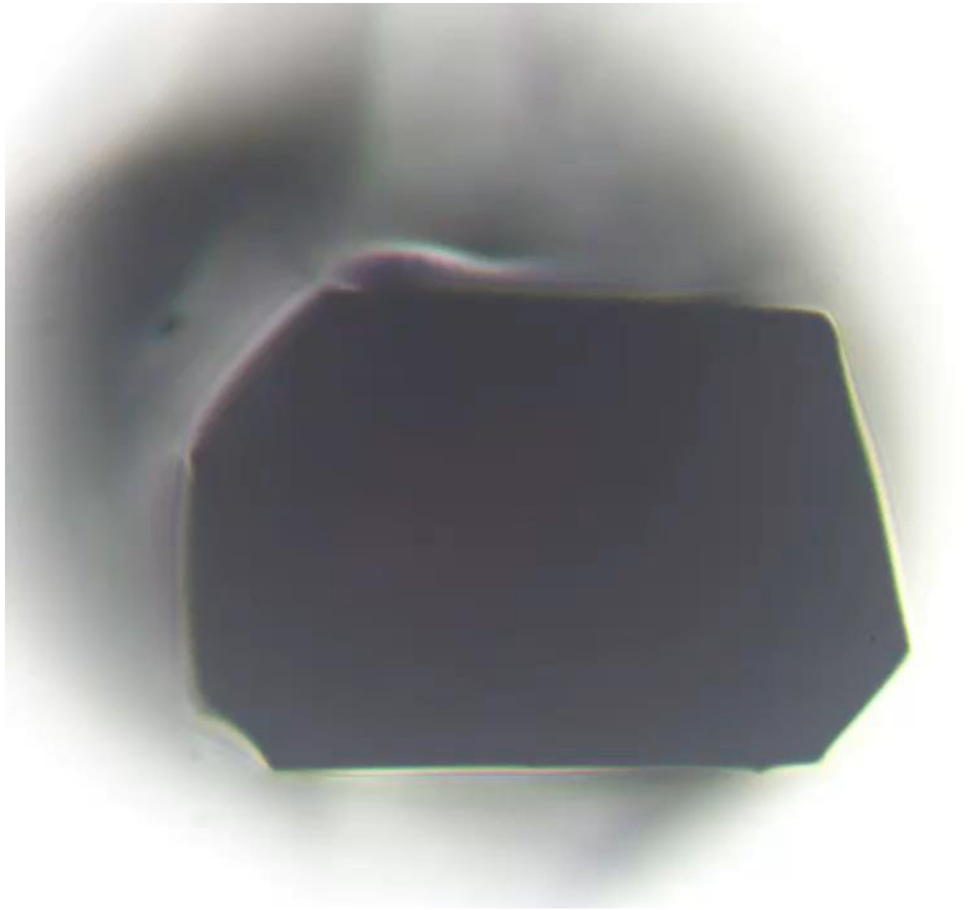
Automation Upgrades

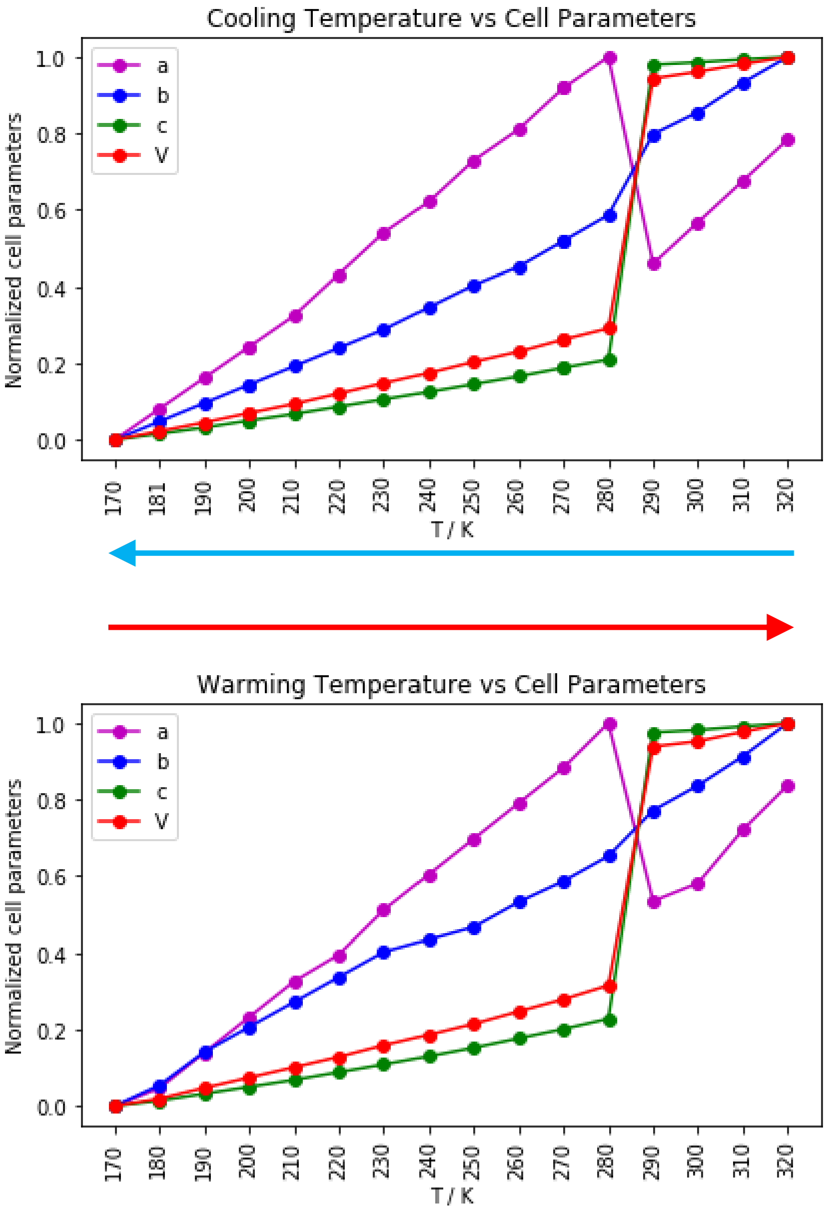
- Each experiment requires 20-40+ datasets
- One full experiment on the upgraded Dectris Eiger X 16M Detector provides > 20 GB raw data in < 1 hour
- Automated rastering for collection and programmed analysis greatly increases throughput and efficiency for these types of experiments

Unbent Reference Structure (manually refined) → Automated Collection of Bent Crystal → Automated Analysis of Mechanism → Finalised CIF Files and Deformation Graphs

- Utilises unbent structure and unchanging symmetry to automatically refine all structures

Investigation of an abrupt phase transition





Uses all of the tools developed.

- Monoclinic system
- Strategy
- Temperature ramp
    - Change temperature, equilibrate and collect
    - Ramp temperature and trigger collect at temperature.
- Heat 5K for every minutes
- Collect every 2 mins (10K), 30 full data collections in 60 minutes
- 240° sweep, 12 s, 200Hz

Alexander Angeloski, *et. al.*, UTS

ANSTO

MX – Australian Synchrotron

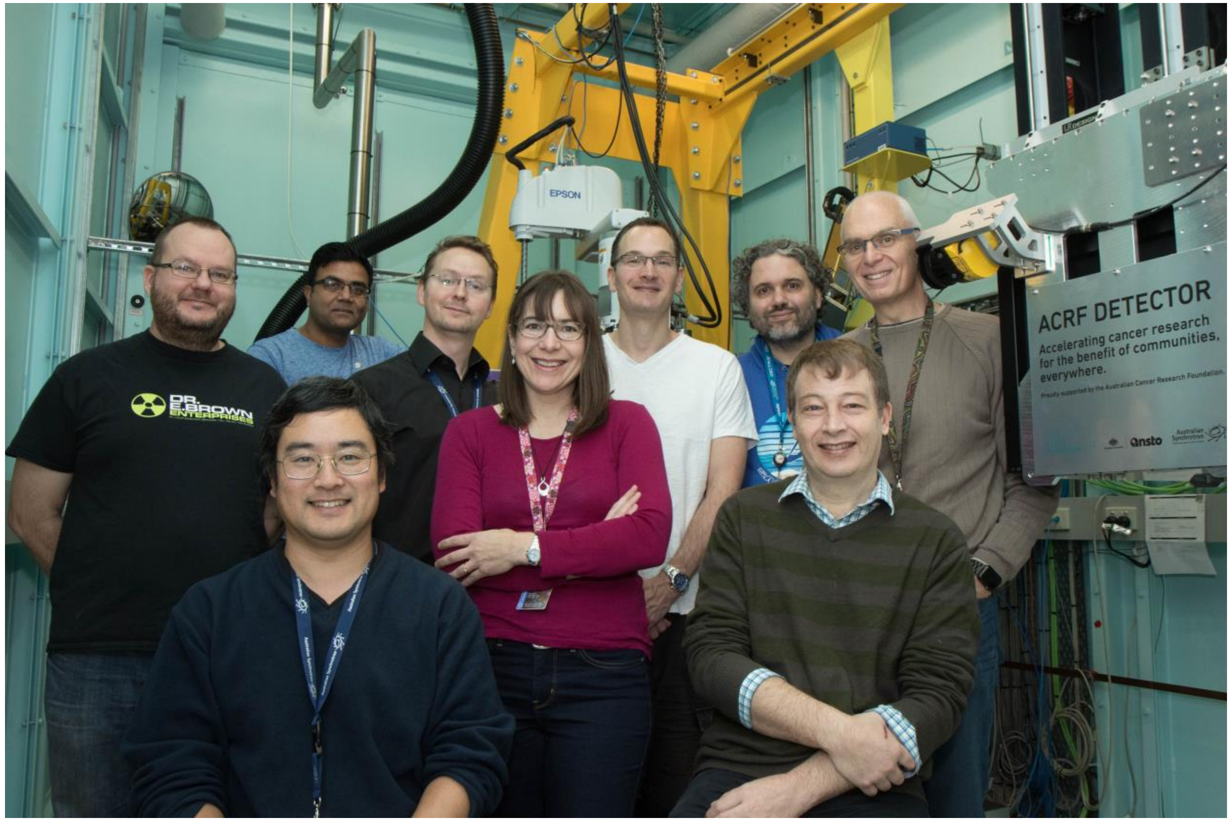
The Australian Synchrotron MX Team

MX3 design
Tom Caradoc-Davies

MX 1 & 2 Team-leaders
Alan Riboldi-Tunnicliffe (Structural Biology)
Rachel Williamson (Chemical Crystallography)

MX Team
Daniel Eriksson (Structural Biology)
Stephen Harrop (Structural Biology)
Santosh Panjikar (Structural Biology)
Jason Price (Chemical Crystallography)
Kate Smith (Structural Biology)

Mark Clift (Controls Engineering)



ANSTO

Thank you

Questions?

Australian Government

ANSTO

Dynamic Range – Progressive Phase Transition

J. Am. Chem. Soc. **1992**, *114*, 5197–5203 5197

Crystal and Molecular Structures of $[Ru(bpy)_3](PF_6)_3$ and $[Ru(bpy)_3](PF_6)_2$ at 105 K

M. Biner,[1a] H.-B. Bürgi,*[,1b] A. Ludi,*[,1a] and C. Röhr[1b]

Contribution from the Institut für Anorganische Chemie, Universität Bern, CH 3000 Bern 9, Switzerland, and Laboratorium für chemische und mineralogische Kristallographie, Universität Bern, Freiestrasse 3, CH 3012 Bern, Switzerland. *Received November 21, 1991*

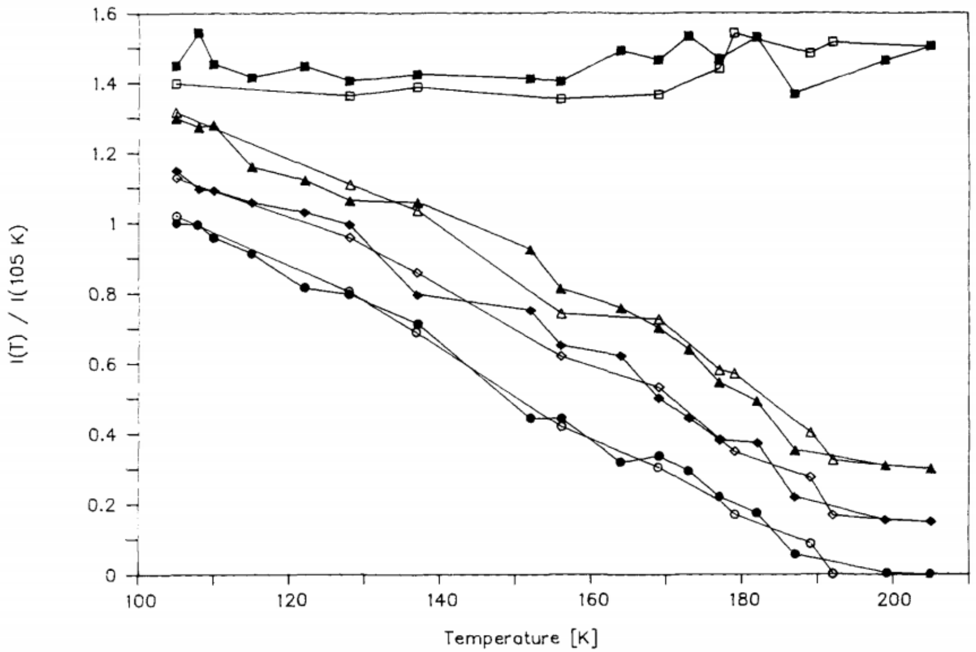


Figure 1. Temperature dependence of the normalized intensities of one main and three superstructure reflections during heating (filled symbols) and cooling (empty symbols). For clarity three of the four curves are shown shifted vertically ("symbol (*hkl*), shift" are given): ■, □ (−8 7 2), 0.45; ▲, △ (2 3 3), 0.3; ◆, ◇ (0 5 6), 0.15; ●, ○ (3 4 −7), 0.

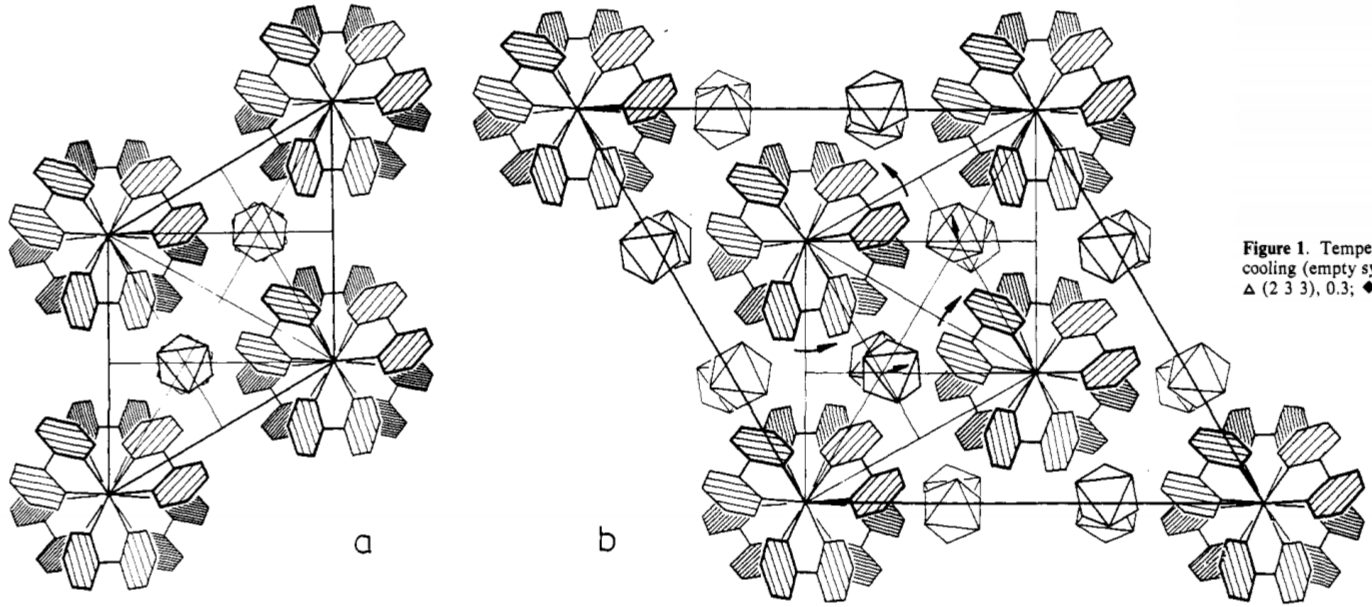
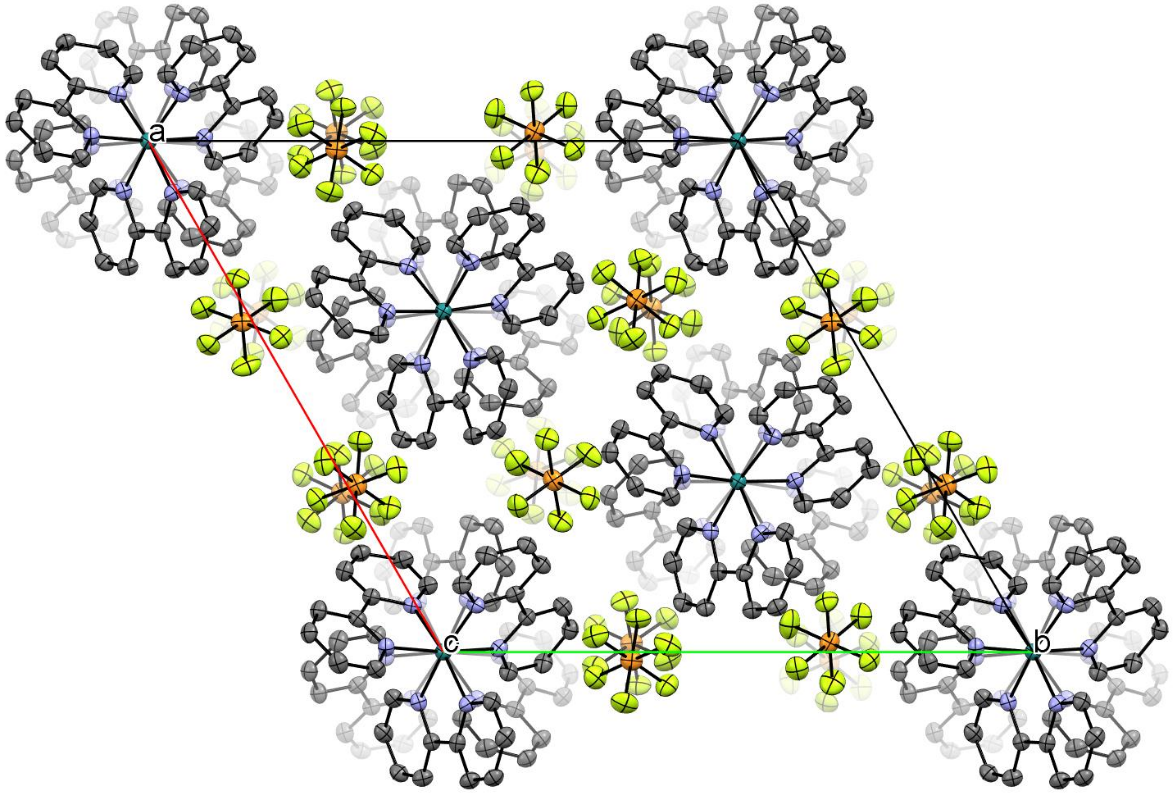
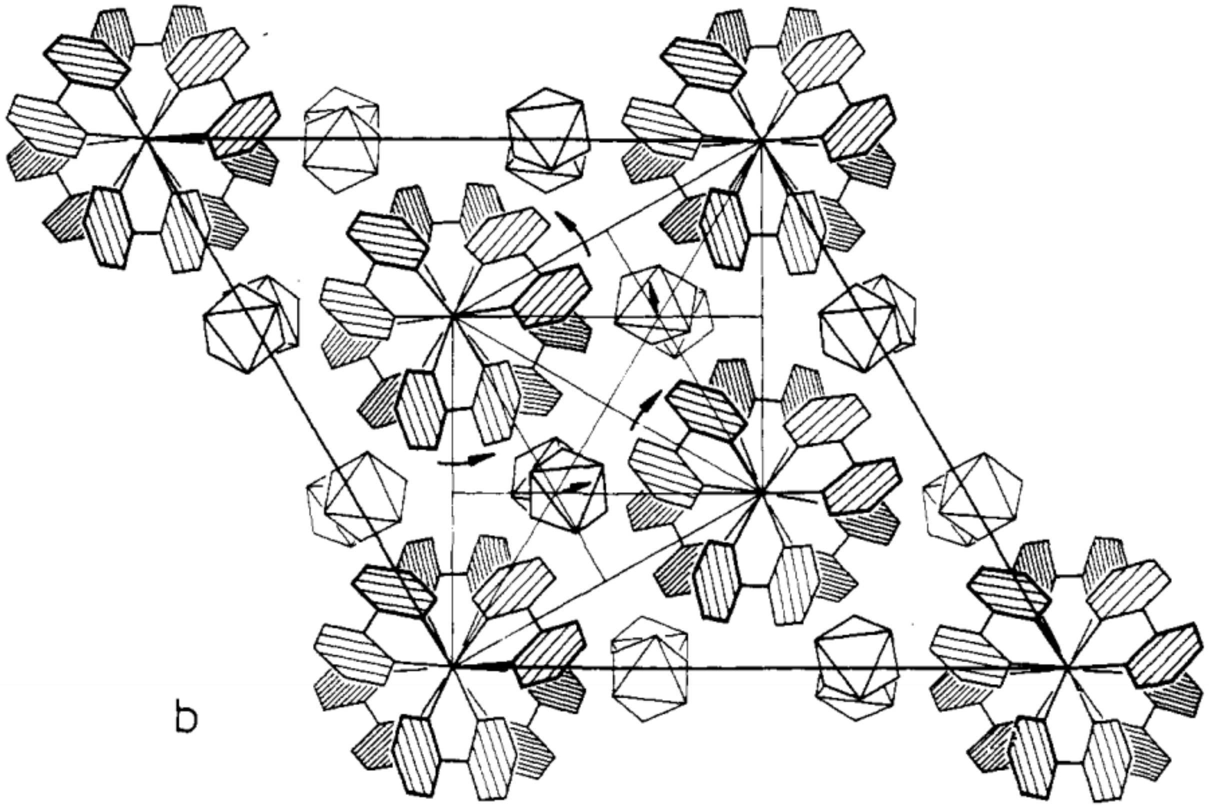


Figure 5. Comparison of the unit cells in the high (a) and low temperature (b) structure of $[Ru(bpy)_3](PF_6)_2$ (arrows in b show direction of displacements relative to the room temperature structure).

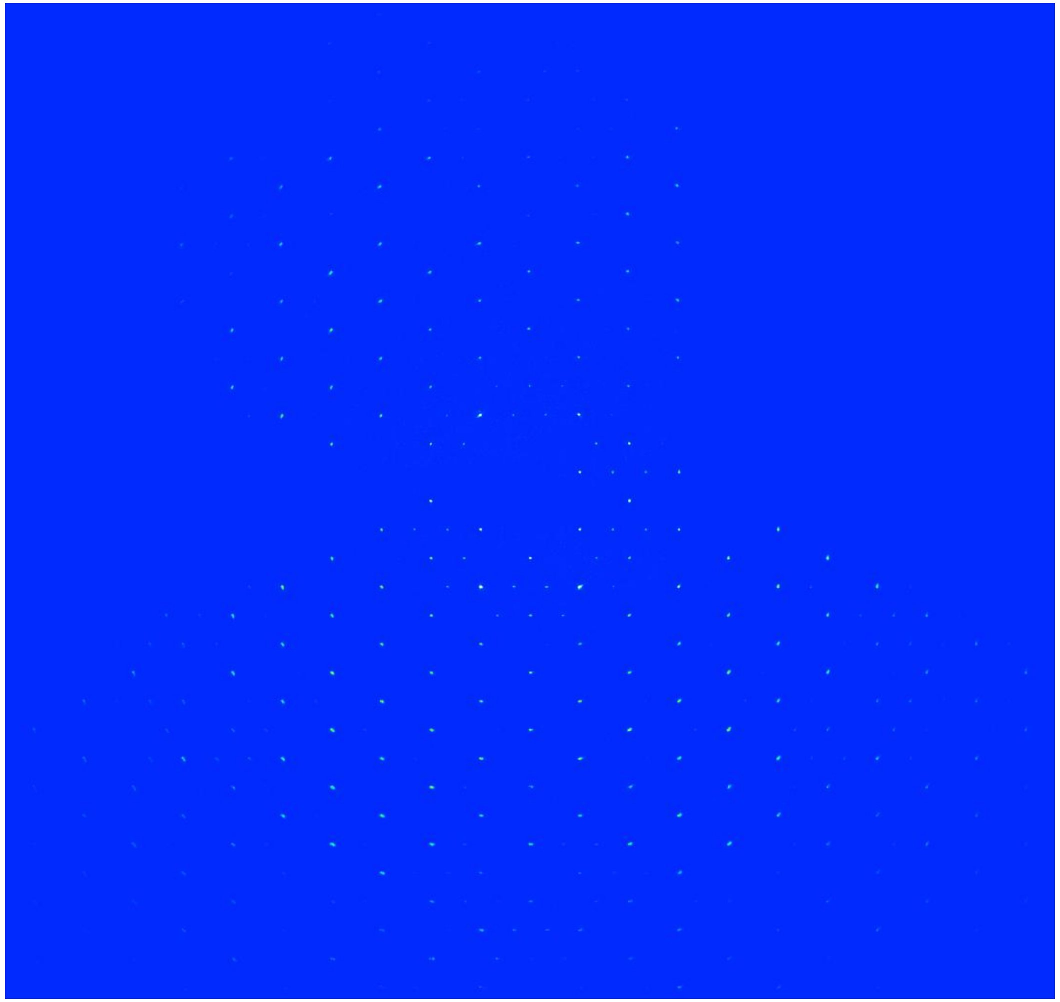
Dynamic Range – Progressive Phase Transition

Space group	***P*31*c*** **(101K)**	***P*-3*c*1** (191K)
a, b (Å)	**10.6705(15)**	18.377(3)
c (Å)	**16.344(3)**	16.237(3)
V (Å^3)	**1611.6**	4748.82

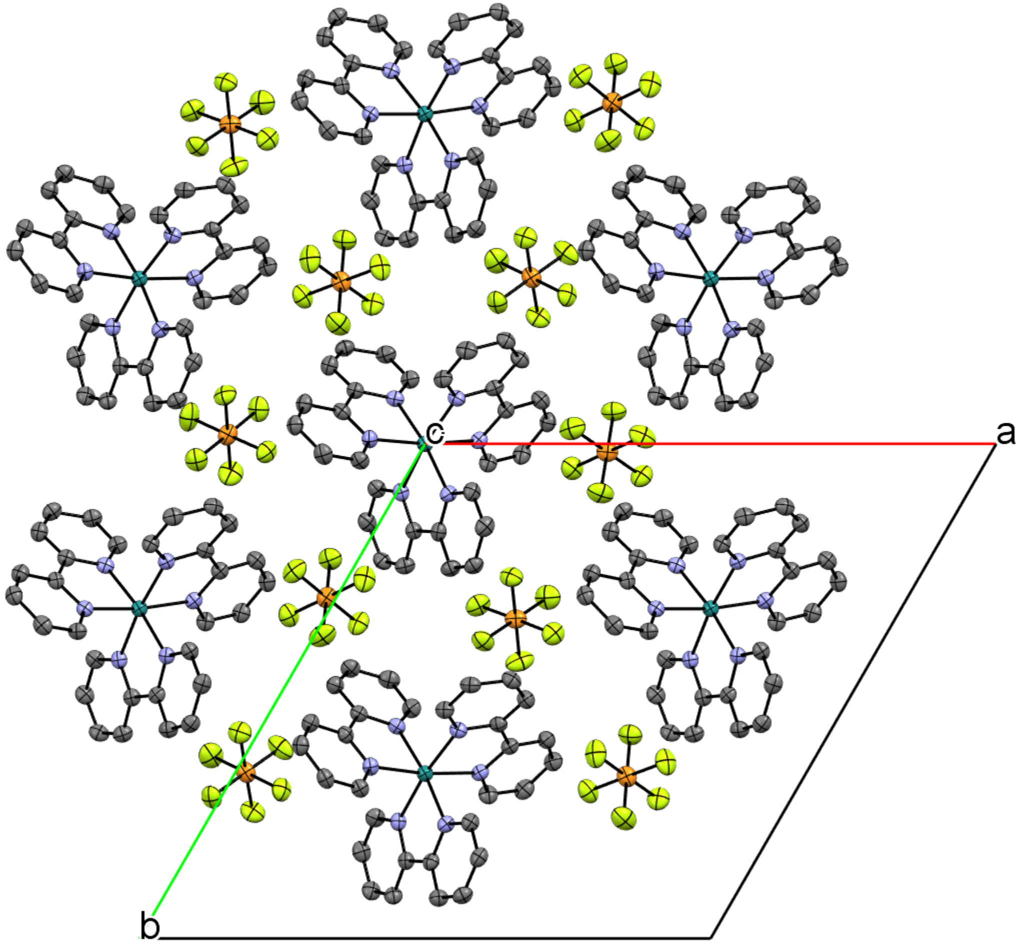




Dynamic Range – Progressive Phase Transition

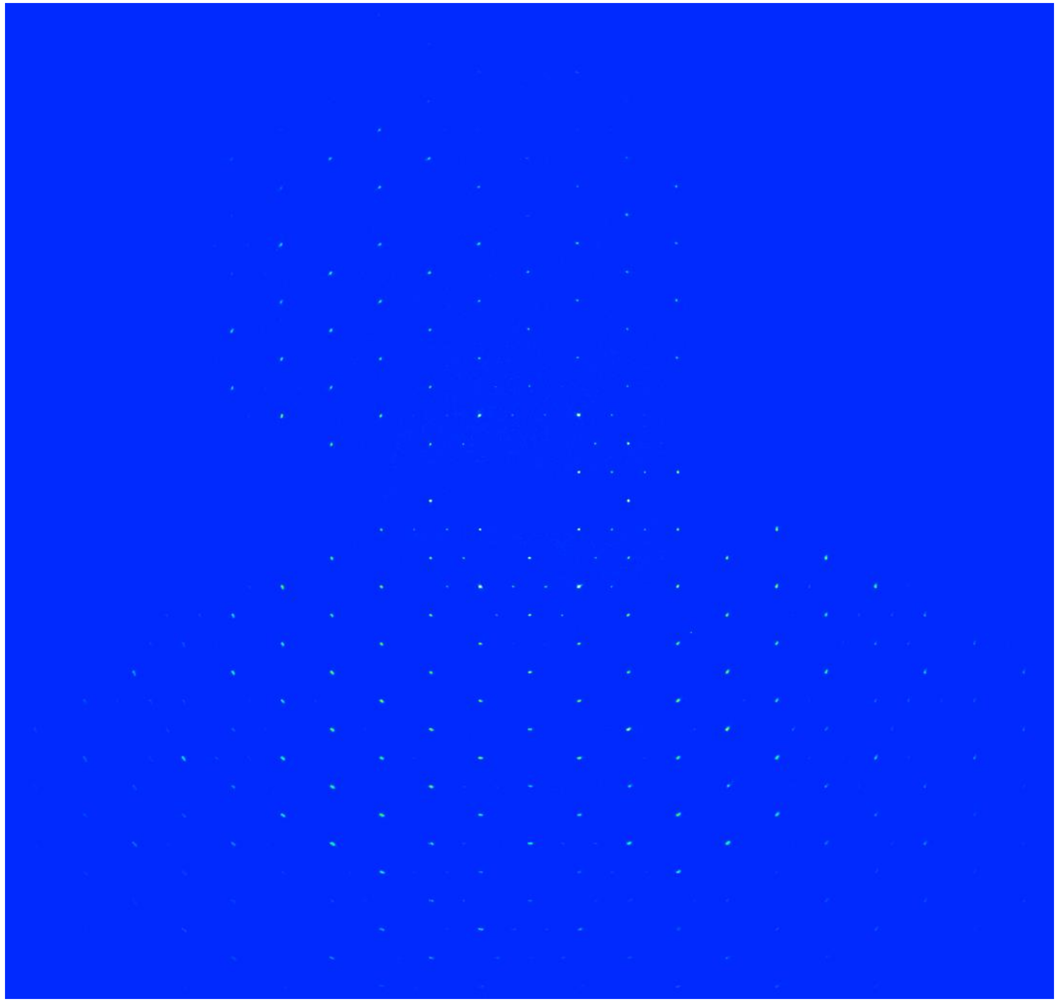


100K

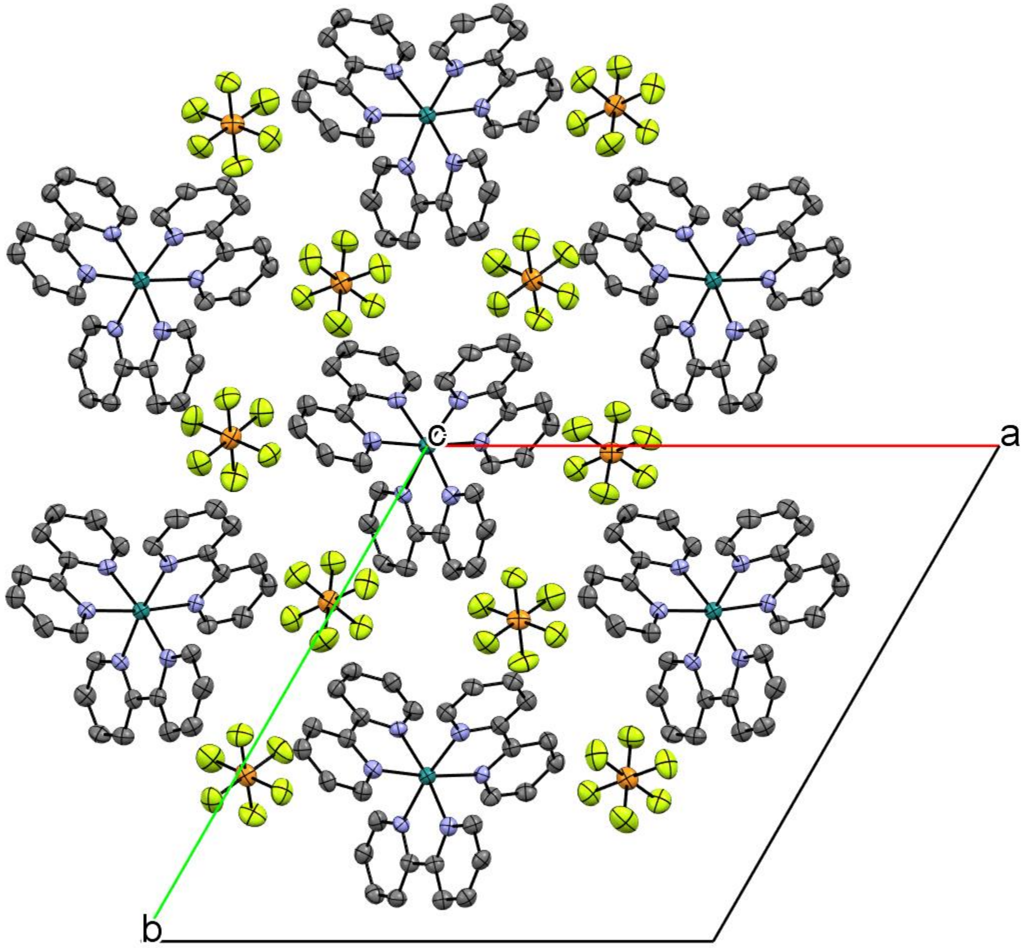


ANSTO

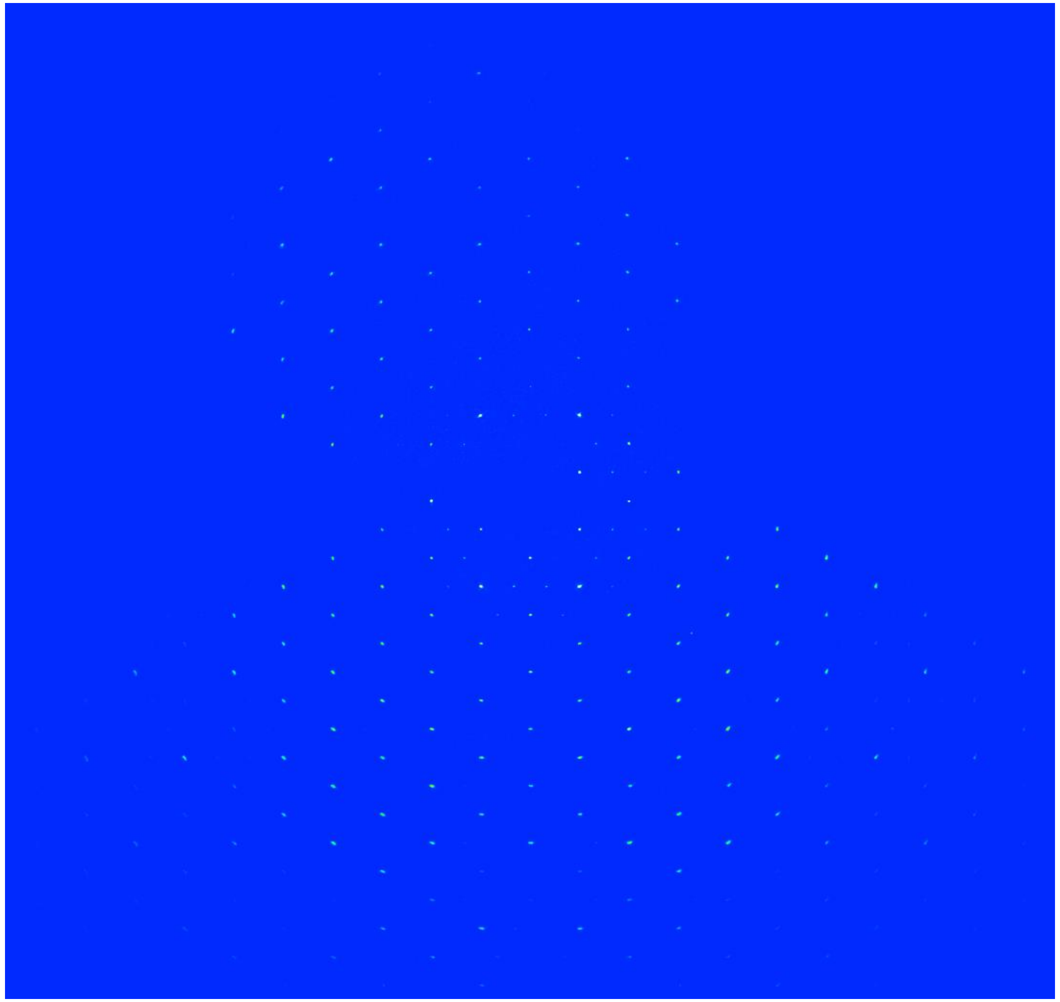
Dynamic Range – Progressive Phase Transition



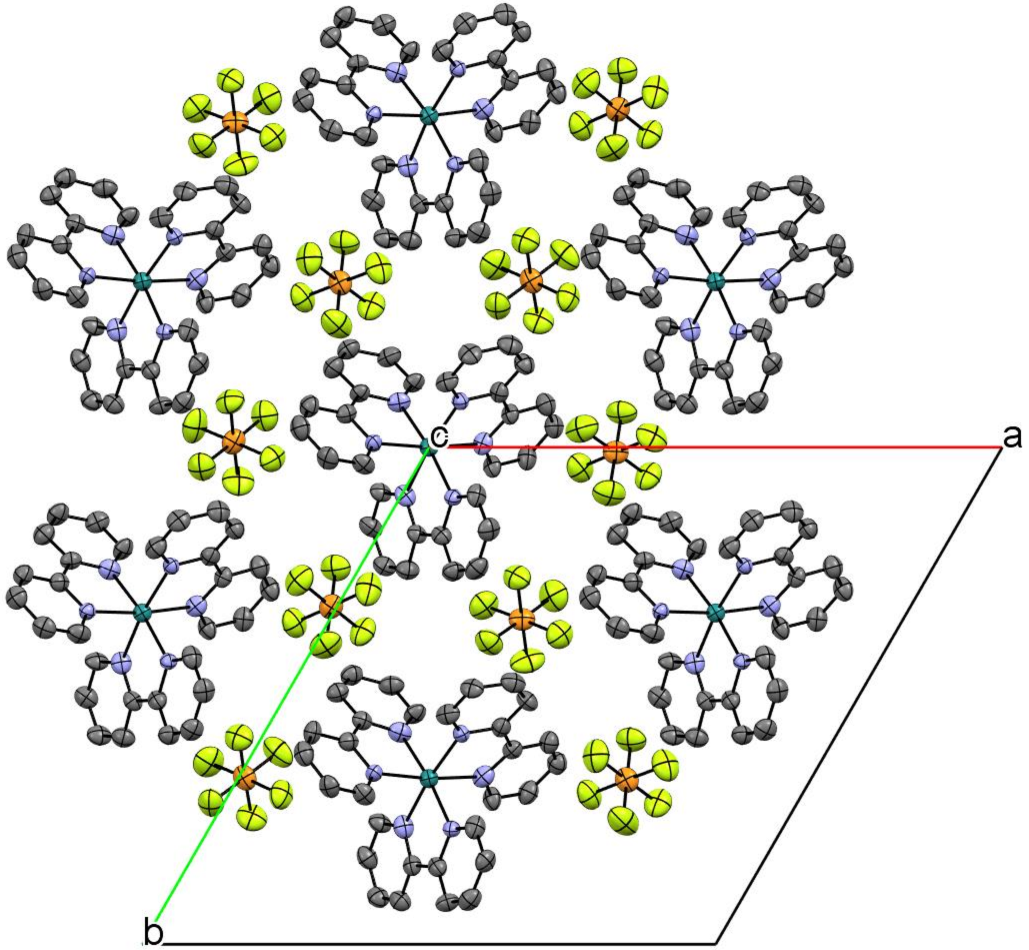
130K



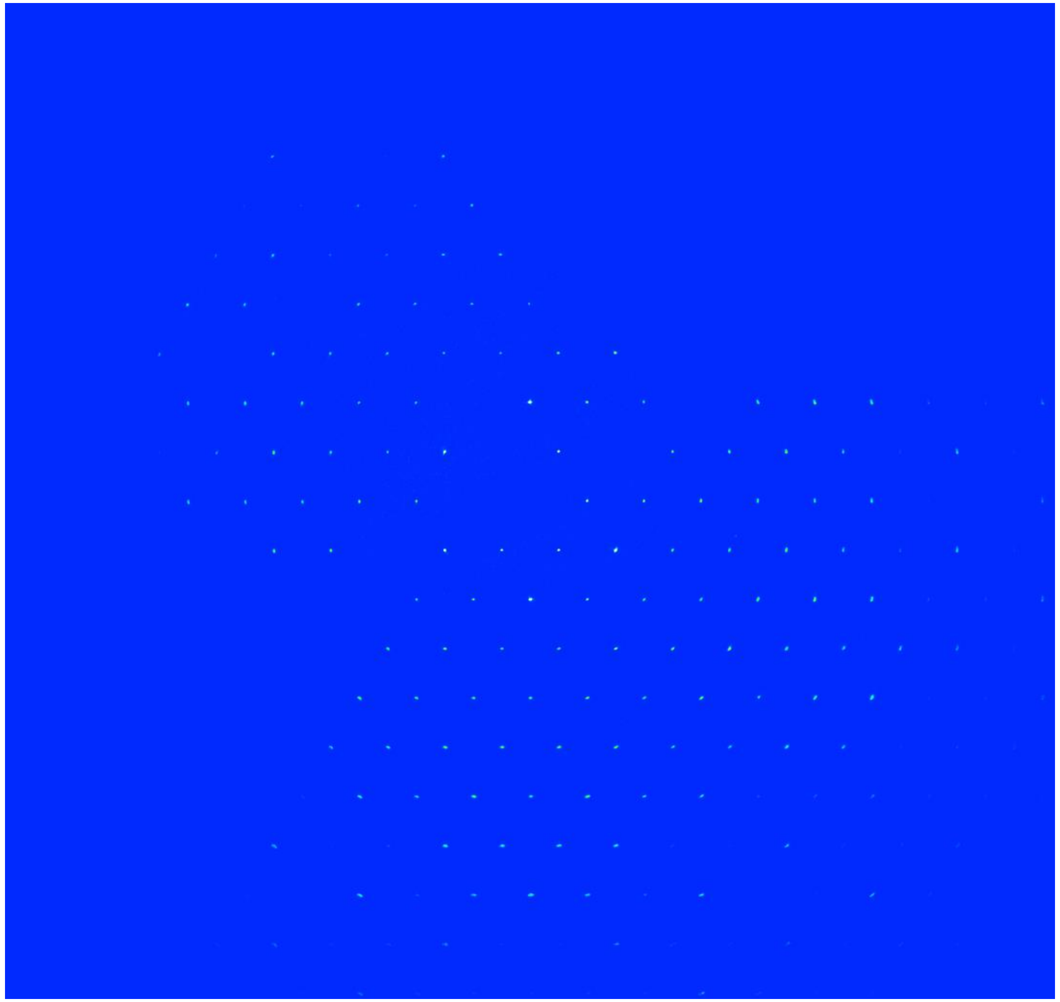
Dynamic Range – Progressive Phase Transition



160K



Dynamic Range – Progressive Phase Transition



190K