HDR DETECTORS and CHEMICAL CRYSTALLOGRAPHY at the AUSTRALIAN SYNCHROTON MX BEAMLINES

Dr Jason Price
ANSTO - Melbourne
Beamline Scientist, MX1 and MX2 Beamlines
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
Resolution CX vs. PX

0.76 Å (17.444 keV, 72 mm)

1.76 Å (13.0 keV, 171 mm)
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
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
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
Implementation Challenges

- Angle of Incidence correction (CCD)
- Unfamiliar Detectors
- Unfamiliar Software
- Data reprocessing
- 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
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$_3$O$_{45}$

Prof. Chris Ling, Chemistry, The University of Sydney

<table>
<thead>
<tr>
<th>Element</th>
<th>Percent by Weight</th>
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<tbody>
<tr>
<td>O</td>
<td>12.29%</td>
</tr>
<tr>
<td>Cr</td>
<td>0.89%</td>
</tr>
<tr>
<td>Nb</td>
<td>4.76%</td>
</tr>
<tr>
<td>Bi</td>
<td>82.06%</td>
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<table>
<thead>
<tr>
<th>Space group</th>
<th>Fm-3m</th>
<th>F-43m</th>
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<tbody>
<tr>
<td>a, b, c (Å)</td>
<td>~5.5</td>
<td>~16.57</td>
</tr>
<tr>
<td>V (Å$^3$)</td>
<td>1426.87(7)</td>
<td>5707.7(3)</td>
</tr>
</tbody>
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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

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

- 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

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

- **Sample**: [Ru(II)(2,2'-bipyridine)$_3$(PF$_6$)$_2$
- **Crystal System**: Trigonal
- **Space Group**: P-3c1
- **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
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
FAIR and FACT, compliance issues

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

**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)
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- Christine Gee (Structural Biology) -> Berkley USA
- Santosh Panjikar (Structural Biology)
- Jason Price (Chemical Crystallography)
- Kate Smith (Structural Biology) -> PSI CH