(Automatic) MX Beamlines at the ESRF

Gordon Leonard
ESRF MX Group
31/10/2007
# Acknowledgements

**ESRF**

<table>
<thead>
<tr>
<th>Sean McSweeney</th>
<th>Vicente Rey Bakaikoa</th>
<th>Florent Cipriani</th>
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<td>Gordon Leonard</td>
<td>Darren Spruce</td>
<td>Andrew Thompson</td>
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<td>Xavier Thibault</td>
<td>Antonia Beteva</td>
<td>Raimond Ravelli</td>
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<td>Joanne McCarthy</td>
<td>Jens Meyer</td>
<td>Andrew McCarthy</td>
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<td>Didier Nurizzo</td>
<td>Olof Svensson</td>
<td>David Flot</td>
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<td>David Hall</td>
<td>Romeu Pieritz</td>
<td>Sandor Brockhauser</td>
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<td>Edward Mitchell</td>
<td>Solange Delagenière</td>
<td>Frank Felisaz</td>
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<td>Petra Pernot</td>
<td>Matias Guijarro</td>
<td>Jean-Sebastian Aksoy</td>
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<td>Christoph Müller-Dieckmann</td>
<td>José Gabadinho</td>
<td>Bernard Lavault</td>
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<td>Elspeth Gordon</td>
<td>Instrument Support Group</td>
<td>Arnaud Clere</td>
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<td>Sasha Popov</td>
<td>BLISS</td>
<td>Pierre-Yves Lanquetin</td>
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<td>Stéphanie Monaco</td>
<td>Optics Group</td>
<td>David Annequin</td>
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<td>Matthew Bowler</td>
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<td>Lionel Mallet</td>
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<td>MX Group Post-docs/students</td>
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<td>Jean-Marie Bois</td>
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**EMBL - Grenoble**

<table>
<thead>
<tr>
<th>Trevor Mairs</th>
<th>Ludovic Launer</th>
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<tr>
<td>Pascal Theveneau</td>
<td>Martin Walsh</td>
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<td>John Surr</td>
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<td>Mario Lentini</td>
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<td>Thierry Giraud</td>
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<td>Andrew Bruton</td>
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<td>Fabien Dobias</td>
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**MRC-France, BM14**

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<thead>
<tr>
<th>MRC-France, BM14</th>
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**ESRF Users**: Johan Turkenburg & York BAG, Gerlind Sulzenbacher & Marseilles BAG, Dave Brown, MRC BAG
# ESRF MX Beamline characteristics

<table>
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<tr>
<th>Beamline</th>
<th>ID14-1</th>
<th>ID14-2</th>
<th>ID14-3</th>
<th>ID14-4</th>
<th>ID23-1</th>
<th>ID23-2</th>
<th>ID29</th>
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<tbody>
<tr>
<td>E (keV)</td>
<td>13.270</td>
<td>13.294</td>
<td>9.6-14.5</td>
<td>6-20</td>
<td>14.2</td>
<td>6-20</td>
<td></td>
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<tr>
<td>( \lambda ) (Å)</td>
<td>0.934</td>
<td>0.933</td>
<td>0.9-1.3</td>
<td>0.62-2.07</td>
<td>0.873</td>
<td>0.62-2.07</td>
<td></td>
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<tr>
<td>Beamsize* (µm)</td>
<td>20-200</td>
<td>20-200</td>
<td>20-200</td>
<td>60</td>
<td>7 x 5</td>
<td>60</td>
<td></td>
</tr>
<tr>
<td>Detector</td>
<td>ADSC Q210</td>
<td>ADSC Q4r</td>
<td>ADSC Q315r</td>
<td>ADSC Q315r</td>
<td>Mar 225</td>
<td>ADSC Q315r</td>
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<tr>
<td>Maxm Resolution (Å)</td>
<td>1.0</td>
<td>1.0</td>
<td>0.9</td>
<td>0.6</td>
<td>0.9</td>
<td>0.6</td>
<td></td>
</tr>
<tr>
<td>Flux (phot/s)</td>
<td>5.8x10^{10}</td>
<td>1.3x10^{11}</td>
<td>1.8x10^{12}</td>
<td>1.5x10^{12}</td>
<td>4.0x10^{11}</td>
<td>1.0x10^{13}</td>
<td></td>
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</table>
MX Experiments at the ESRF

- Experiment duration typically 3 shifts (24 hrs), 6 shifts at weekends.

- Over 600 user experiments per year: ~45% of all ESRF exp’tal sessions.

- Over 2000 user visits per year: ~40% of all ESRF user visits.

- Rapid turnover of experiments.

- Simplification/automation/consistency of equipment.

- Above figures for academic users only - MX BLs also host a flourishing industrial activity.
A common BL control GUI (mxCuBE)
The typical MX 'experiment' can be automated

- Safety Clearance
  - Preparations
    - Beamline configuration, alignment, validation.
    - Sample Screening
      - Assess crystal quality for required experiment.
- Collect Diffraction Data
- Integrate/Reduce
- Phase/Model Build
- Mount Samples
- Align Samples
  - Full automatic alignment.
- Collect 2 images
  - Indexing (strategy)
- Xanes Scan
  - If required for the experiment
- Plan Experiment(s)
  - Use results to set-up experiment run

Automation implies standardisation and monitoring
Automating X-ray beam provision and maintaining & monitoring beam integrity on the MX Beam-lines
Automating Alignment of Optics Hutch Elements [ID23-1, ID29]

- Diamond + Camera
- Diode + Carbon foil
- YAG scintillator + Camera
- Diode + Fe/Cu/Pt/Zr/Mo foil
- Kapton Diode
- YAG scintillator + Camera
- Kapton Diode
- Diamond + Camera
- Diode + Carbon foil
Automating X-ray beam provision

E.g. Primary Slits:
- Initialisation using limit switches
- Series of scans allow proper centering around white beam

Analogous protocols for secondary slits, monochromator, mirror. Soon to be combined into 'one button' procedure and 'rolled out' to all MX BLs.
Automatic Calibration of Monochromator

Initialisation using absolute encoder.
Calibration using Pt foil (transmission @ at L_{III} edge)

Validation procedure (if desired) using scans of Fe K, Cu K, Pt L_{III}, Pt L_{II}, Zr K and Mo K absorption edges.
Aligning the Mirror

Mirror Alignment:
- Utilisation of encoders
- Prealignment of the mirror
  - In height
  - In horizontal translation

- Automatic optimisation of bender, tilt, yaw on-going
Automation in the experimental hutch

X-ray BPM

2 diodes:
• i_0 after the slits
• i_1 after the shutter

X-ray Camera

Minidiffractometer (cryostream, fluorescence detector)
SC3, YAG Screen

Detected

Slit Box

Attenuators

Safety Beam Shutter

Slits: alignment in same way as PS etc..

YAG screen: allows automatic alignment of X-ray beam to goniometer rotation axis.

X-ray camera: allows automatic (and dynamic) focussing of X-ray beam (ongoing)
Automating the MX ‘experiment’

Sample Information

Safety Clearance

Preparation
Beamline configuration, alignment, validation.

Sample Screening
Assess crystal quality for required experiment.

Collect Diffraction Data

Integrate/Reduce

Phase/Model Build

Automation implies standardisation and monitoring
MX Beamline Sample Environment

- High precision mini-diffractometer
- Fast CCD Detector
- Backlight & Beamstop/Collimator
- Sample Changer Robot
- Oxford Cryostream
- Fluorescence Detector
- Evacuated Slitbox & Attenuators
- On-axis Camera
Micro- and Mini-diffractometers

- Handle crystals down to 10 μm size
- High Precision air bearing PHI axis (2μm)
- On beam axis viewing video microscope
- Integrated beam shaping
- Assisted and automatic crystal alignment
- and more…
Why Do We Need Robotic Sample Changers?
Inside the SC3
How does the SC3 load/unload samples?
European SPINE standard
Full specifications at http://www.spineurope.org, protocols menu

- Design based on Hampton Magnetic Crystal Cap™ and Vial System

- 22 mm fixed sample holder length (18mm pins)

- 10 Characters identification code:
  - DataMatrix on the base of Caps
  - Clear code near the DatatMatrix
Automatic sample loading/unloading & centering in X-ray beam
Automatic absorption edge scans - I
Automatic absorption edge scans -II
Metal ions in crystals: fluorescence analysis
Automatic sample screening & characterisation

DNA (automated collection of data), Collaborative project involving the ESRF, CCLRC Daresbury Laboratory, MRC-Cambridge, Diamond Light Source; EMBL (both Grenoble an Hamburg outstations); Global Phasing Ltd, MRC - France (BM14) and SOLEIL.
Automatic data collection, reduction & scaling
ISpyB (http://ispyb.esrf.fr)

Communication of information concerning individual samples (description, bar-code, location, known unit cell dimensions, space group, ‘diffraction plans’) from crystallographers in the home lab to those performing the experiments at the ESRF. Real time monitoring of data collections: diffraction images, crystal snapshots, harvesting of output from DNA.
The Data Collection Pipeline (DCP) strings automated procedures together.

- **Sample Information**
- **Safety Clearance**
- **Preparation**
  - Beamline configuration, alignment, validation.
- **Sample Screening**
  - Assess crystal quality for required experiment.
- **Collect Diffraction Data**
- **Integrate/Reduce**
- **Phase/Model Build**

- **Mount Samples**
- **Align Samples**
  - Full automatic alignment.
- **Xanes Scan**
  - If required for the experiment
- **Collect 2 images**
  - Indexing (strategy)
- **Plan Experiment(s)**
  - Use results to set-up experiment run
What the DCP does

- Scans of the contents of SC3 robot, compares these to ISpyB LIMS, displays the data matrices, associated sample information, position of the sample in the SC3. Then for each sample selected
  - Mount the sample
  - Centres the sample in the X-ray beam.
- Takes two diffraction images at 90° in phi to each other for use by DNA
- Carries out the DNA characterisation of the crystal mounted
  - Writes the results to IspyB.
- Attempts to rank the crystal and suggest an order for data collection for crystals of the same type
  - Dismounts the crystals from the goniometer head.
MxCuBE & DNA together

Available samples

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<thead>
<tr>
<th>Name</th>
<th>Acronym</th>
<th>Barcode</th>
<th>Location</th>
<th>Space group</th>
<th>a</th>
<th>b</th>
<th>c</th>
<th>Alpha</th>
<th>Beta</th>
<th>Gamma</th>
<th>MinRes.</th>
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Directory: /tmp/xbadino/external/mx15/20070820/tryp/Trypsin07
Prefix: Tryp-Trypsin07
Run number: 1

1st step: Take reference images
Template: /tmp/xbadino/external/mx15/20070820/tryp/Trypsin07/ref_Trypsin07_1_###_img
Oscillation range (deg): 1.0
Exposure time (s): 5.0
Default resolution (Å): 3.0

2nd step: Index reference images
Template: 
Space group: Unknown

3rd step: Calculate strategy
Space group: Unknown

4th step: Collect and integrate
Template: 
Number of images: 
Oscillation range (deg): 
Exposure time (s): 
Resolution (Å): 

Execute steps: Status: not logged

Current results
Results file: Go to Results tab
Space group: 

alpha: 
beta: 
gamma: 

MxCuBE & DNA together

MxCuBE & DNA together
EMBL/ESRF Mini-Kappa
Thaumatin - tetragonal
(ESRF; ID23-1)

Omega  Kappa  Phi
2.4    60.6    -74.7