

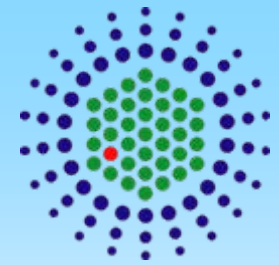
(Automatic) MX Beamlines at the ESRF

Gordon Leonard
ESRF MX Group
31/10/2007





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ESRF

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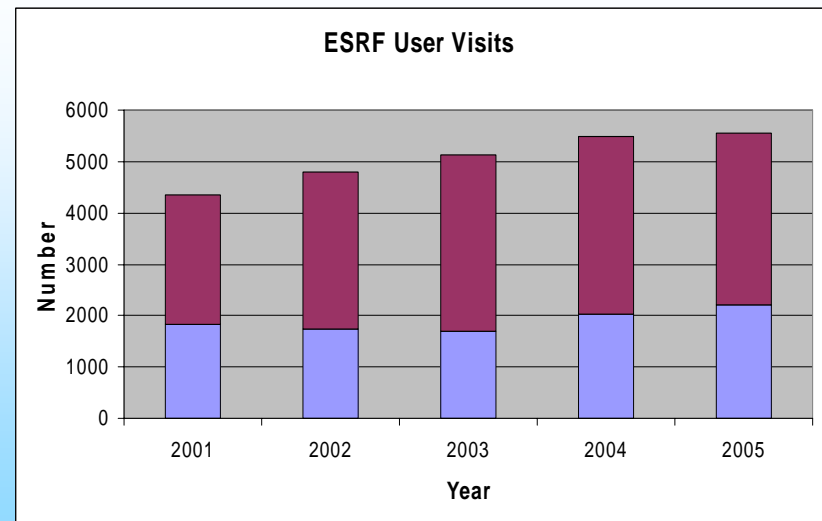
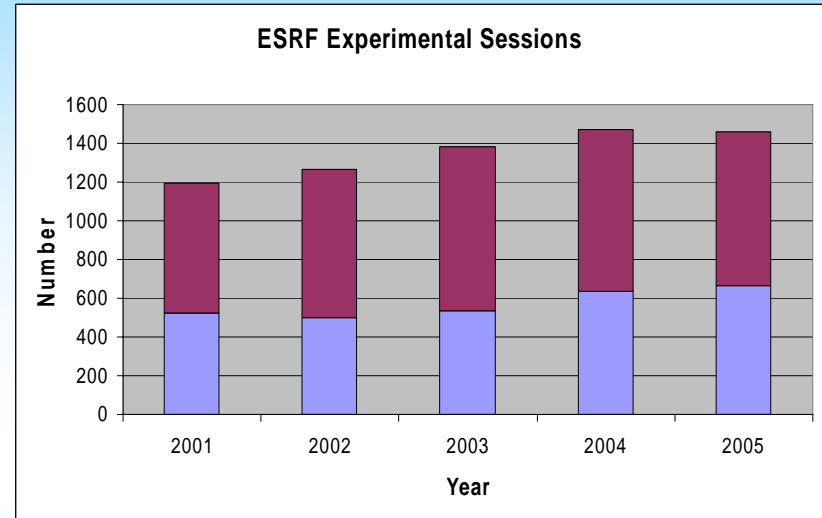
ESRF Users: Johan Turkenburg & York BAG, Gerlind Sulzenbacher & Marseilles BAG, Dave Brown, MRC BAG

ESRF MX Beamline characteristics

Beamline	ID14-1	ID14-2	ID14-3	ID14-4	ID23-1	ID23-2	ID29
E (keV)	13.270	13.294	SAXS	9.6-14.5	6-20	14.2	6-20
λ (Å)	0.934	0.933		0.9-1.3	0.62-2.07	0.873	0.62-2.07
Beamsize* (μm)	20-200	20-200		20-200	60	7 x 5	60
Detector	ADSC Q210	ADSC Q4r		ADSC Q315r	ADSC Q315r	Mar 225	ADSC Q315r
Max ^m Resolution (Å)	1.0	1.0		0.9	0.6	0.9	0.6
Flux (phot/s)	5.8×10^{10}	1.3×10^{11}		1.8×10^{12}	1.5×10^{12}	4.0×10^{11}	1.0×10^{13}

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.



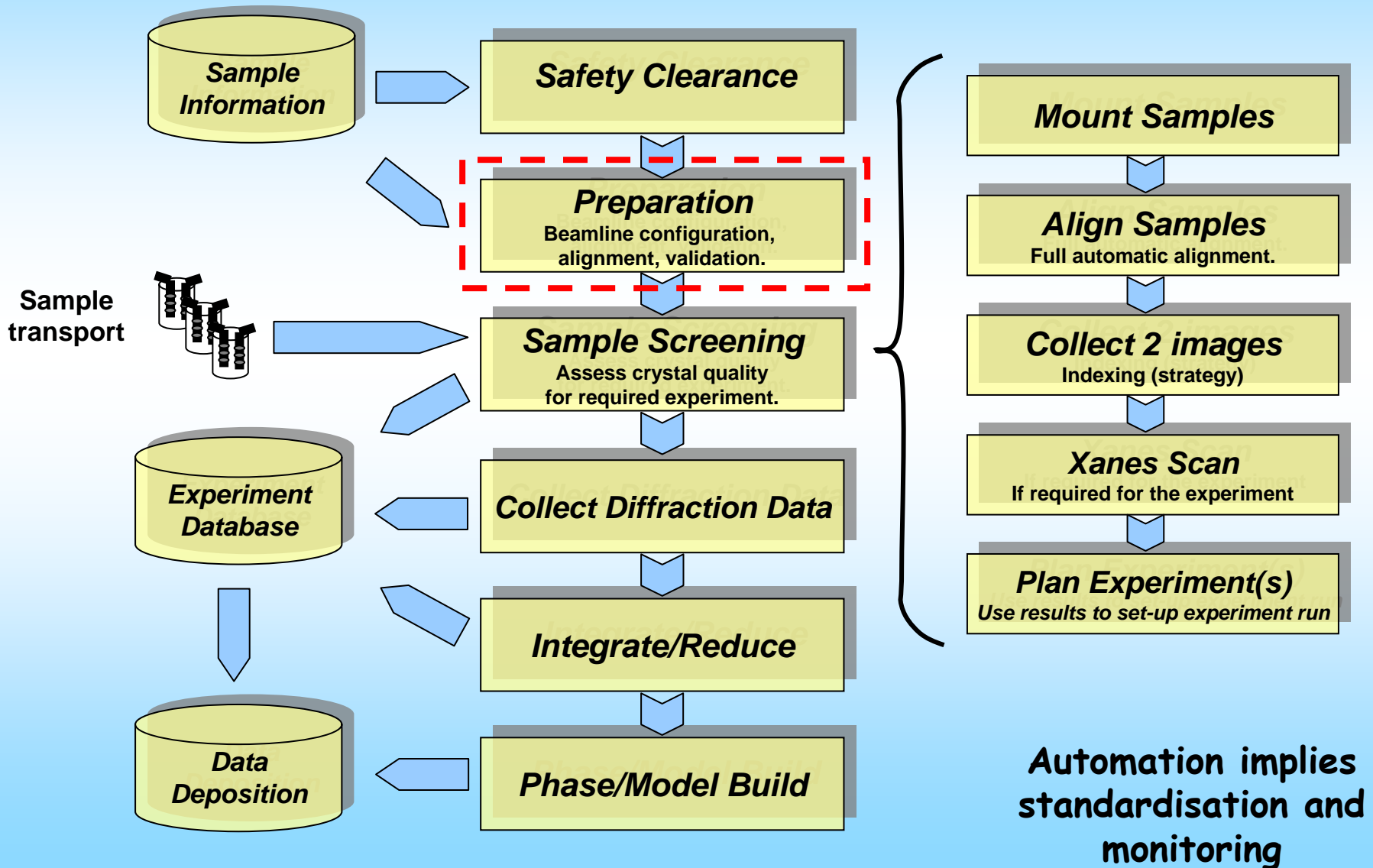
A common BL control GUI (mxCuBE)

The screenshot displays the mxCuBE GUI interface, which is used for controlling the beamline. The interface is organized into several panels:

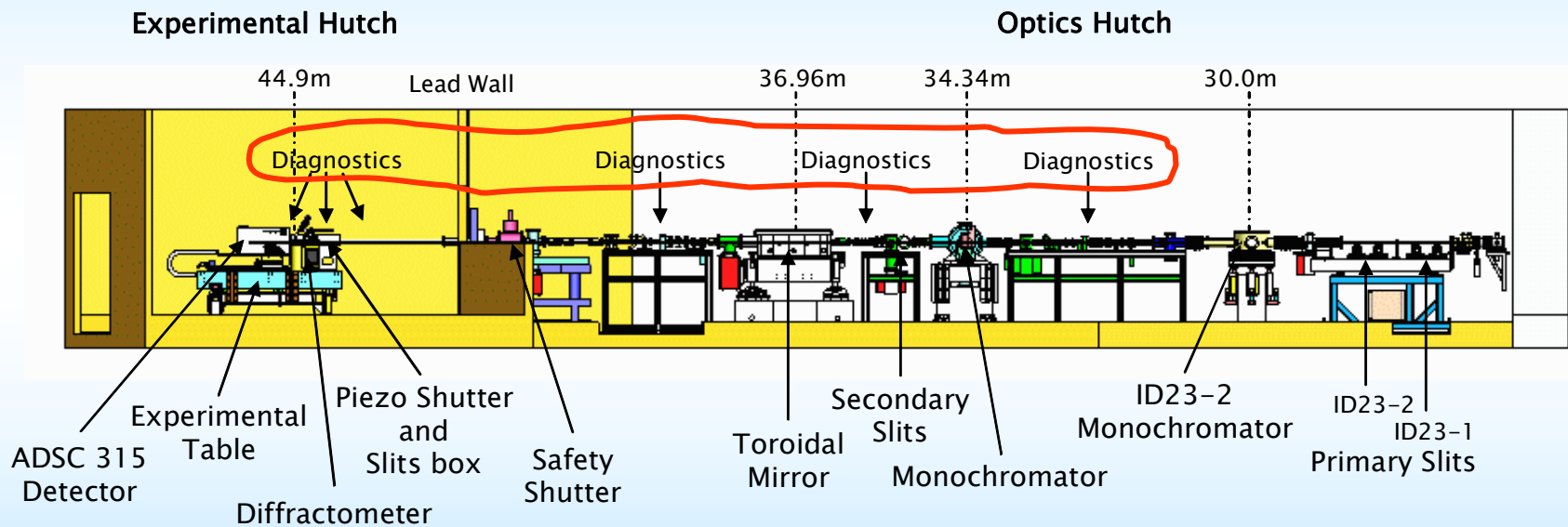
- Top Panel:** Includes a menu bar (File, New, Bookmarks, Desktop, Windows, Help) and a toolbar with icons for Huch, collect, energy scan, file spectrum, and image.
- Left Panel:** Contains control panels for "Available motors" (with sliders for phi and phi vertical), "Energy" (13.275 keV), "Wavelength" (0.934 Å), "Transmission" (100.00%), "Beamstop" (out), "Safety shutter" (disabled), and "Foil shutter" (closed). A "Scan plot" is also present, showing a graph with X and Y axes ranging from 0 to 1000.
- Center Panel:** Features a "Sample centring" section with radio buttons for "Manual 3-click" and "Computer automatic", and buttons for "Centre", "Accept", "Autofocus", and "Snapshot". Below this is a "Sample display" area showing a central image with a red crosshair and various control sliders for "Light" (0.55), "Zoom" (4), and "Focus" (-0.3). A "Beam size" slider is located at the bottom of this panel.
- Right Panel:** Displays the ESRF logo, "Machine current" (0.0 mA), a "Collect stage" list (1. Collect stage, 2. Preparing beamline, 3. Mounting sample, 4. Centring sample, 5. Taking snapshot, 6. Collecting images, 7. Unmounting sample), and "Current users" (local contact).
- Bottom Panel:** Includes an "Information messages" section with a log of system events, a "Submit feedback" button, a "chat (2)" button, and a "spec" button. A system tray at the very bottom shows the Start button, taskbar, and system clock (12:03).

A small dialog box titled "mxCuBE - z-" is overlaid on the center panel, displaying the message "Successfully connected to the server application." with an "OK" button.

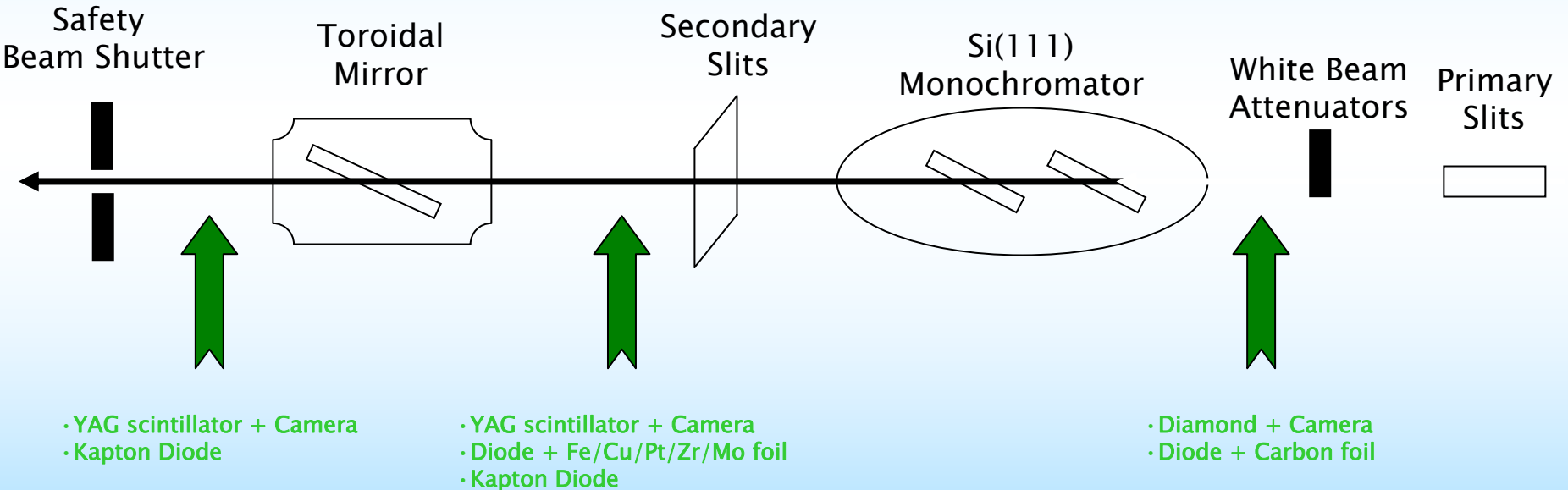
The typical MX 'experiment' can be automated



Automating X-ray beam provision and maintaining & monitoring beam integrity on the MX Beam-lines



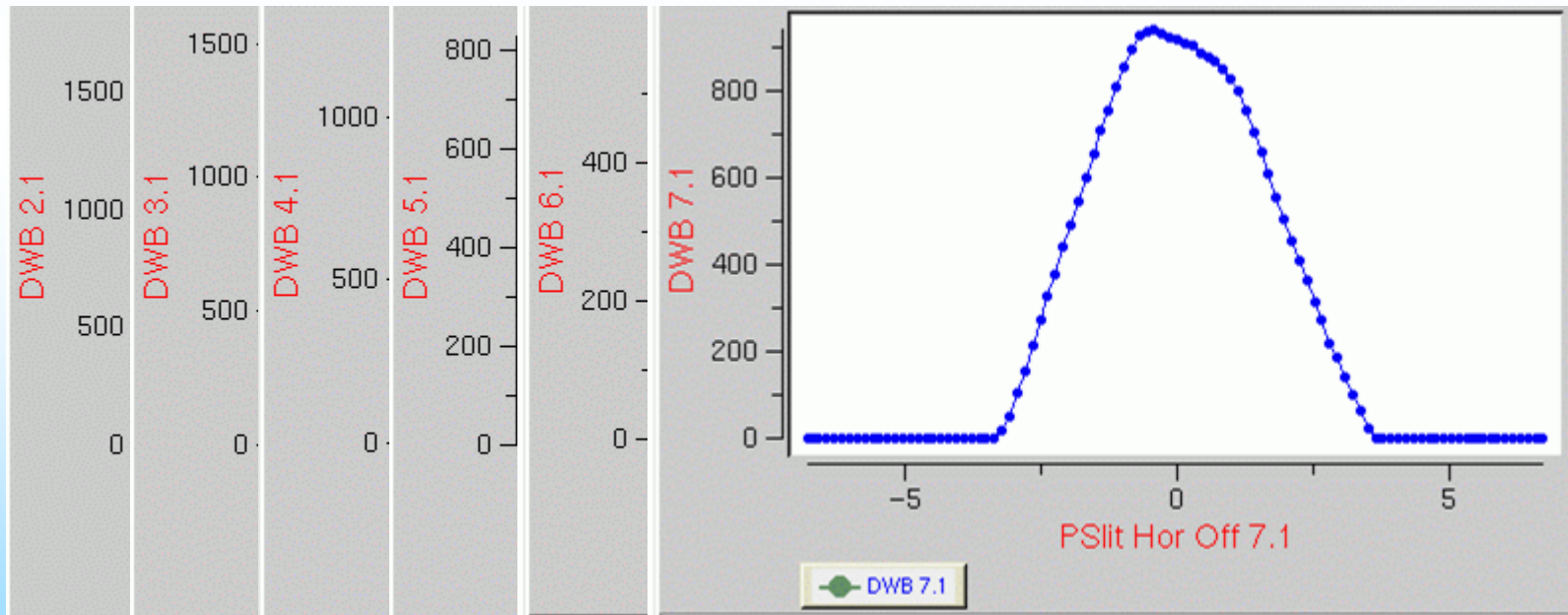
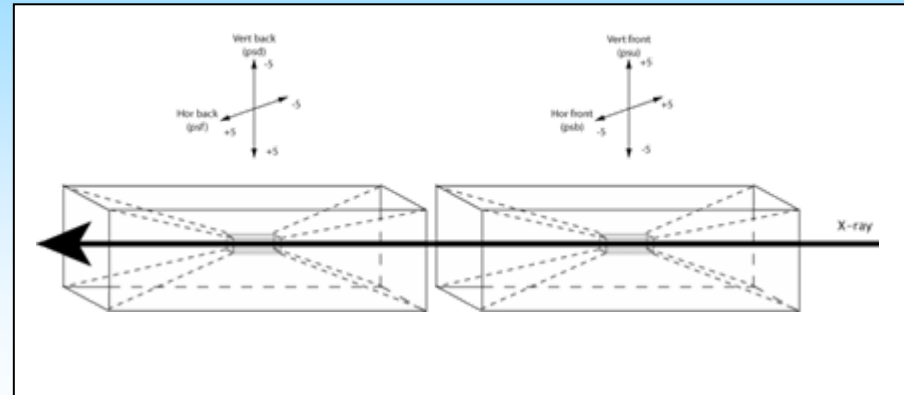
Automating Alignment of Optics Hutch Elements [ID23-1, ID29]



Automating X-ray beam provision

e.g. Primary Slits :

- Initialisation using limit switches
- Series of scans allow proper centering around white beam

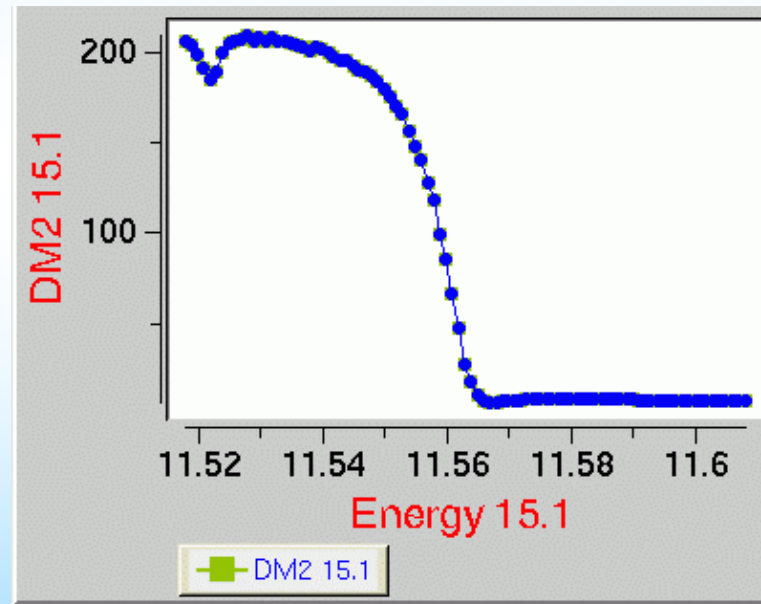


Analogous protocols for secondary slits, monochromator, mirror. Soon to be combined into 'one button' procedure and 'rolled out' to all MX BLs

Automatic Calibration of Monochromator

Initialisation using absolute encoder.

Calibration using Pt foil (transmission @ at L_{III} edge)

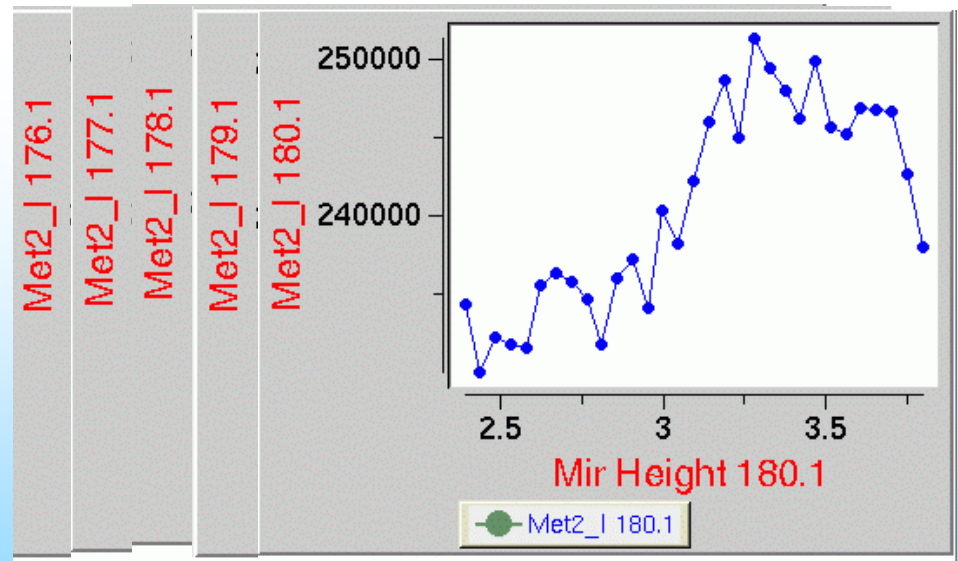
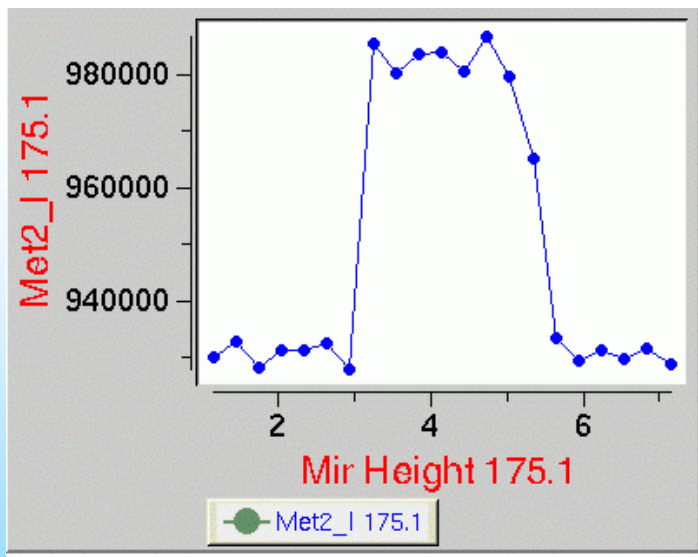


Validation procedure (if desired) using scans of Fe K, Cu K, Pt L_{III} , Pt L_{II} , Zr K and Mo K absorption edges.

Aligning the Mirror

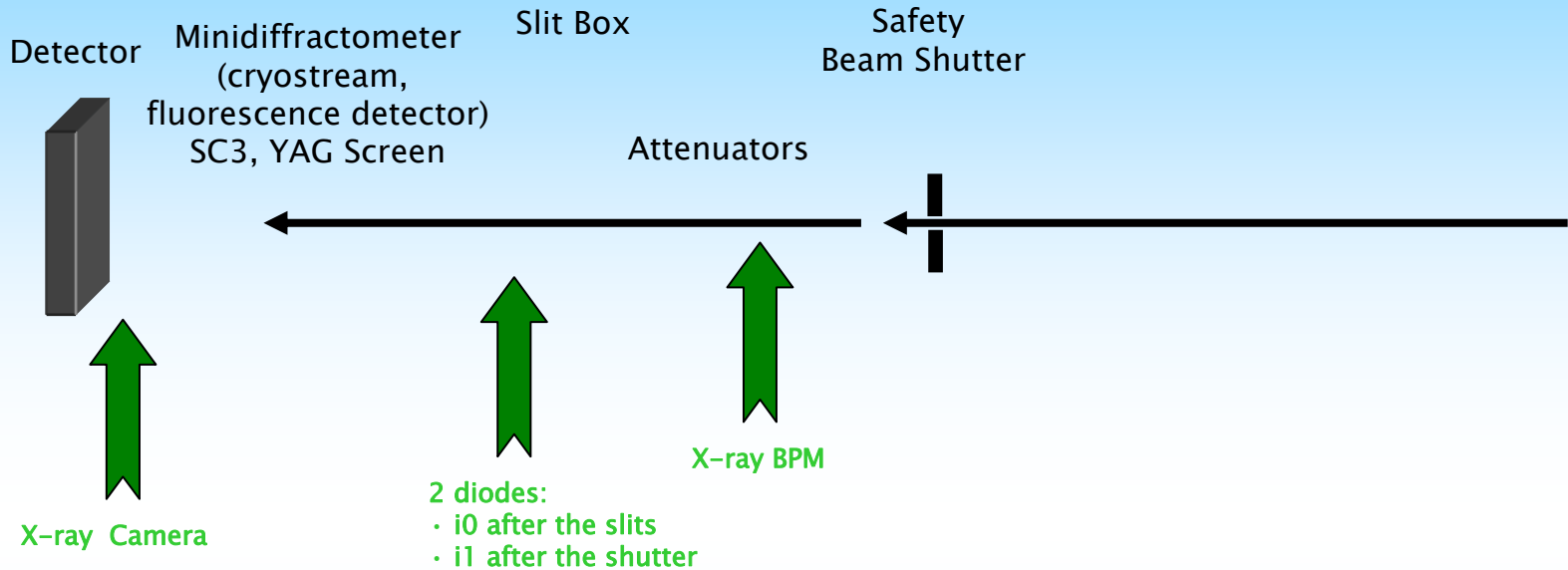
Mirror Alignment :

- Utilisation of encoders
- Prealignment of the mirror
 - In height
 - In horizontal translation



- Automatic optimisation of bender, tilt, yaw on-going

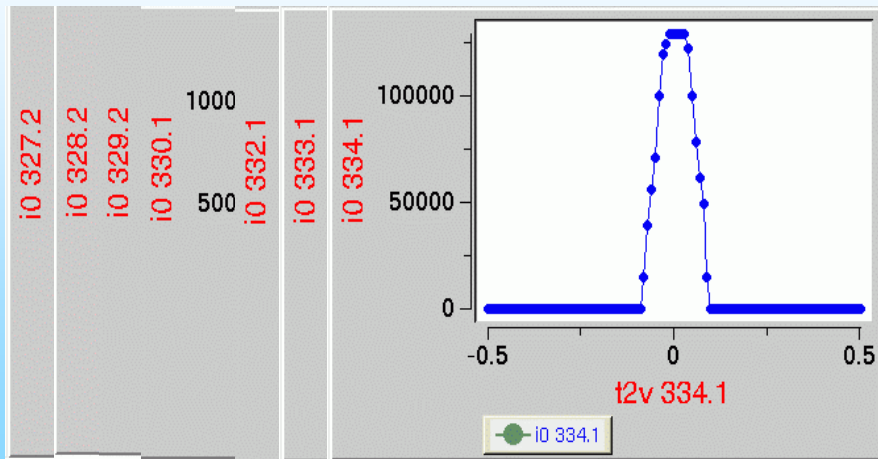
Automation in the experimental hutch



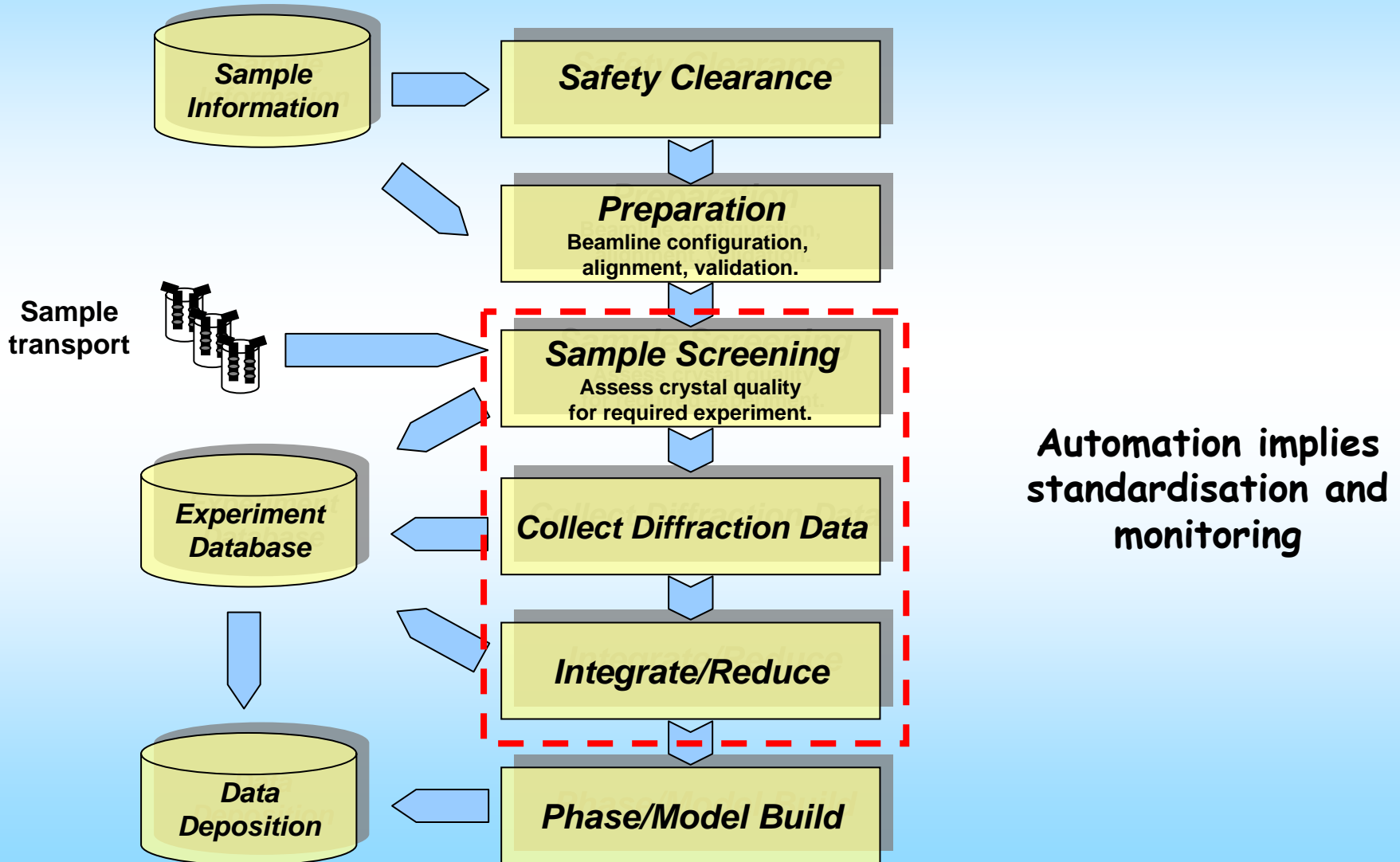
Slits: alignment in same way as PS etc..

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 (on-going)



Automating the MX 'experiment'



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



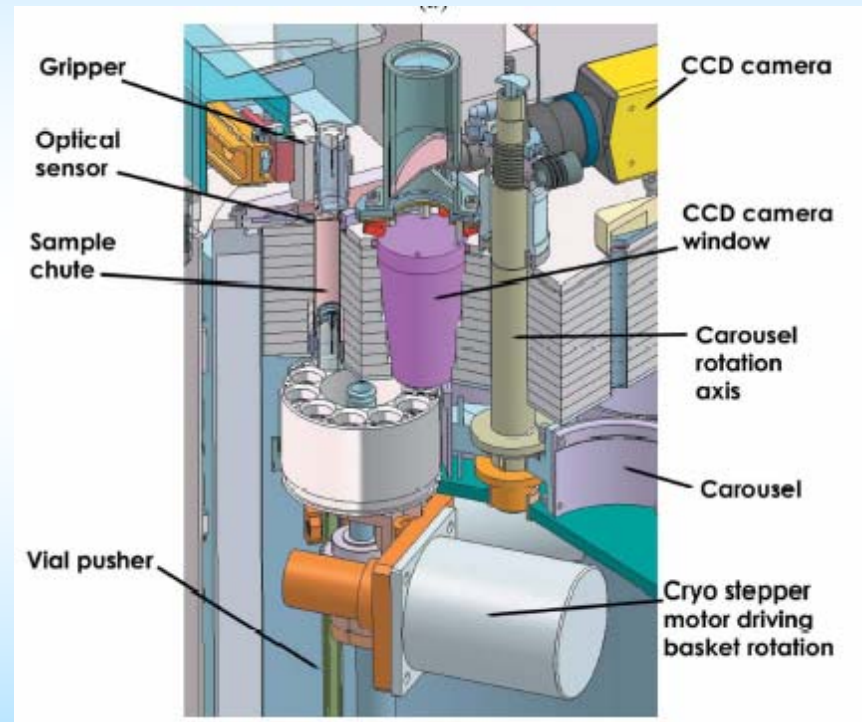
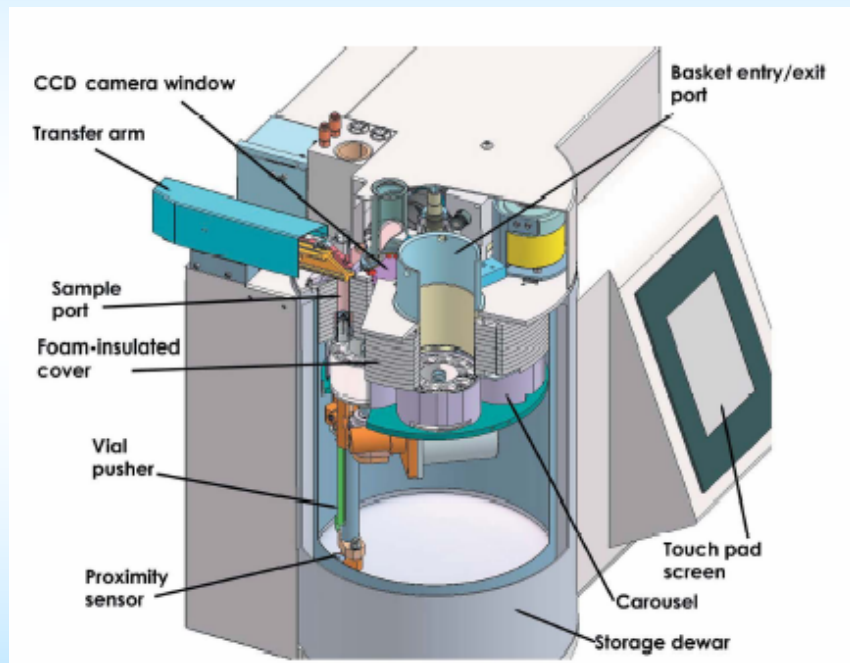
Microdiffractometer specs

- Handle crystals down to 10 μm size
- High Precision air bearing PHI axis (2 μm)
- On beam axis viewing video microscope
- Integrated beam shaping
- Assisted and automatic crystal alignment
- and more...

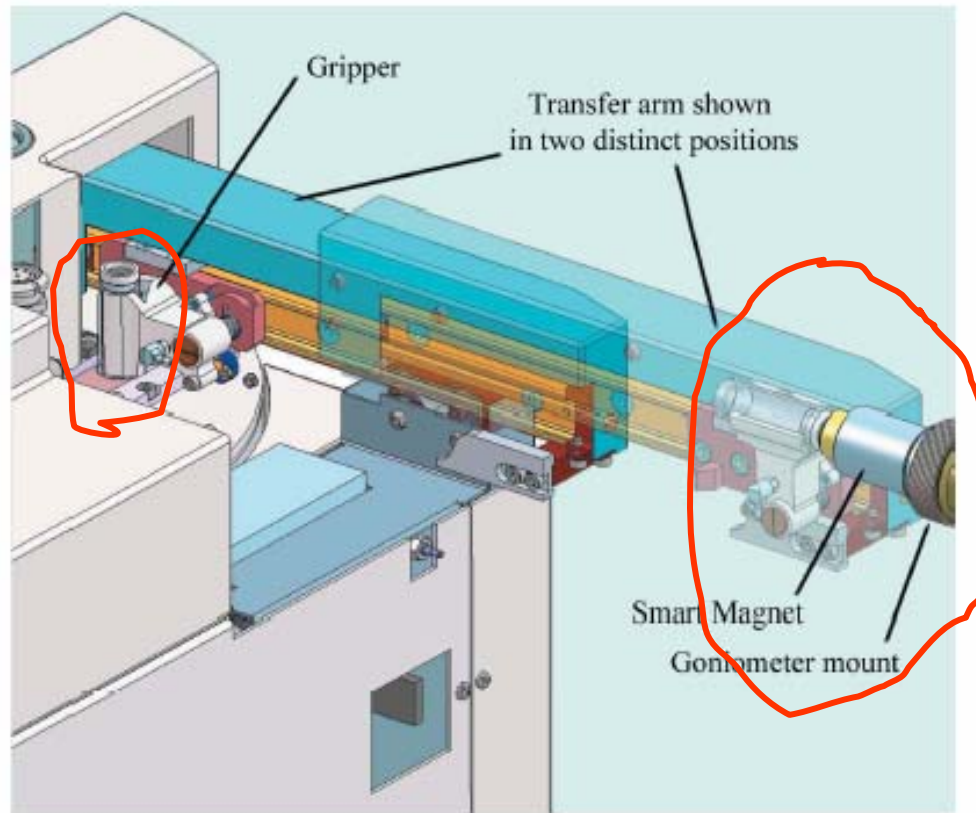
Why Do We Need Robotic Sample Changers?



Inside the SC3



How does the SC3 load/unload samples?

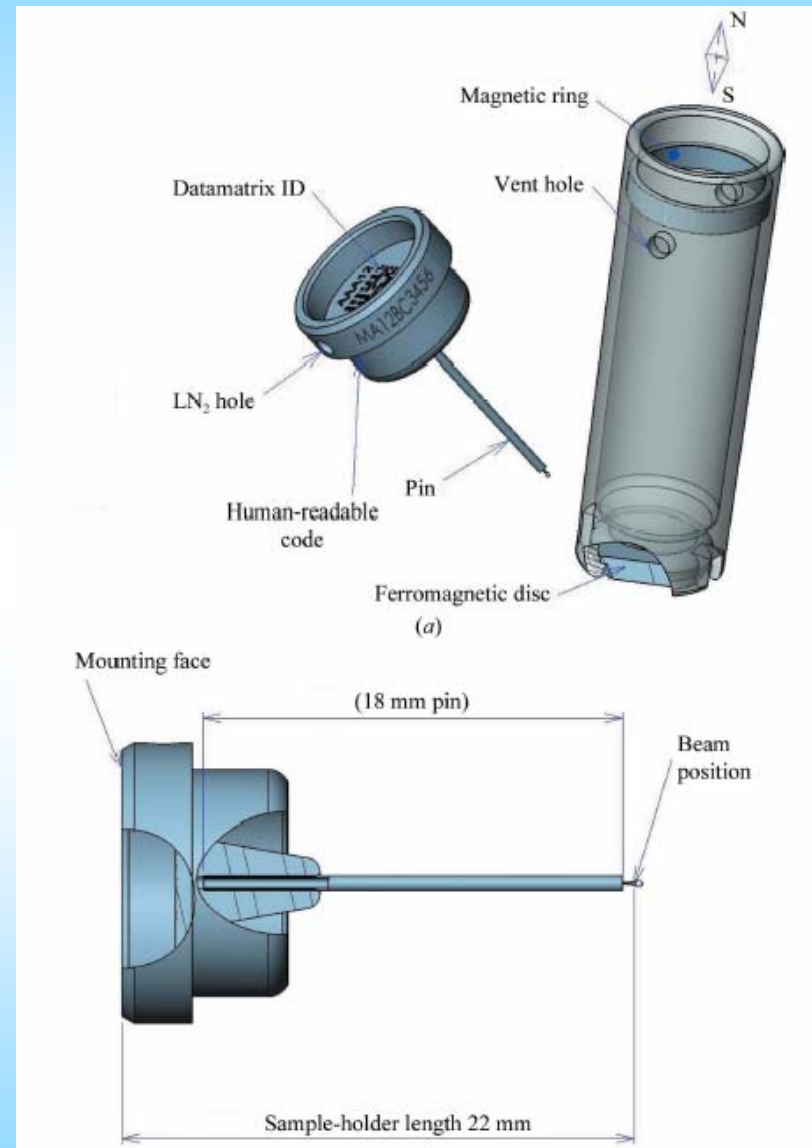


SPINE Standard Sample Holders & Vials

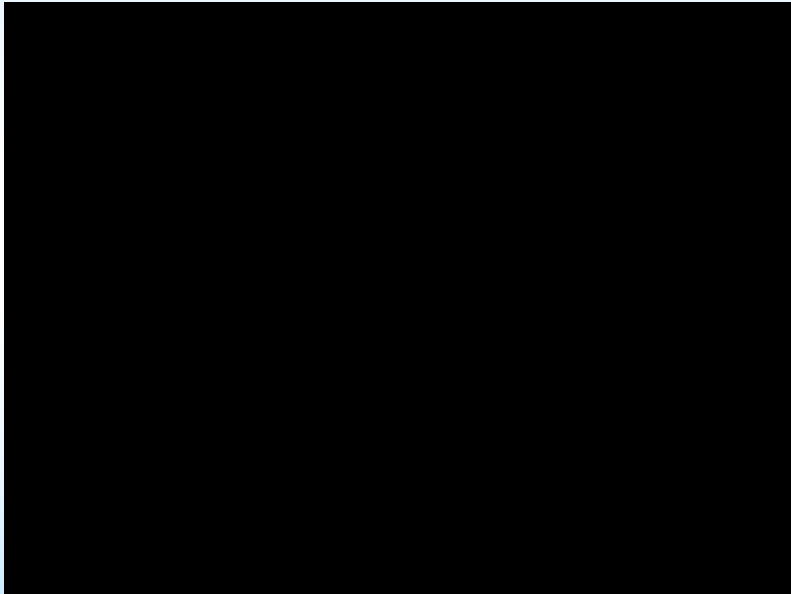
European SPINE standard

Full specifications at <http://www.spineurope.org>, protocols menu

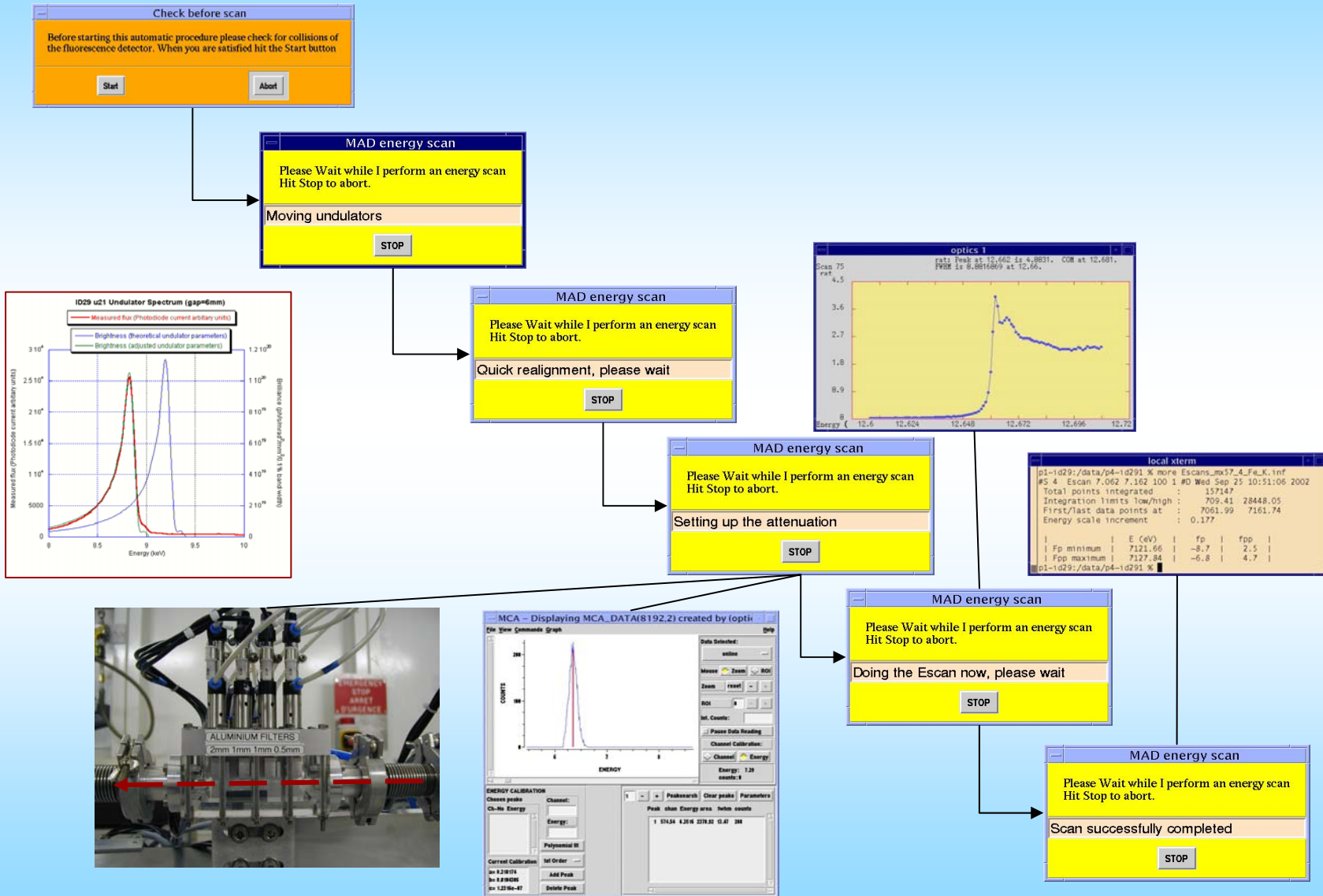
- Design based on Hampton Magnetic Crystal Cap™ and Vial System
- 22 mm fixed sample holder length (18mm pins)
- 10 Characters identification code:
 - DataMatrix on the base of Caps
 - Clear code near the DatatMatrix



Automatic sample loading/unloading & centering in X-ray beam



Automatic absorption edge scans - I



Automatic absorption edge scans -II

mxCuBE (opid-231)

File Instrumentation Help Expert mode

Hutch Collect Energy scan Xls spectrum

Available elements

Se - K (34,selenium)

Scan plot

monoscan (X: 12700.172414, Y: -210.526316)

MCA counts

energy - scan

Parameters

Prefix: opid231 Directory: /data/d23eh1/inhouse/opid231/20070727

Energy scan

Se - K

Start scan

Peak: 12.6520 (keV) Inflection: 12.6496 (keV) Remote: 12.7020 (keV) 2nd Remote: (keV)

Accept Reset

Y

X

- spline - fp

Machine current

-0.0 mA

Empty

Cryo

100.0 K

51%

Dry: unknown

Superdry: unknown

Icing: unknown

Collect stage

1. Preparing beamline
2. Mounting sample
3. Centring sample
4. Taking snapshot
5. Collecting images
6. Unmounting sample

Jul 23 08:50 Shutdown; End of RUN :Shutdown until 16/08

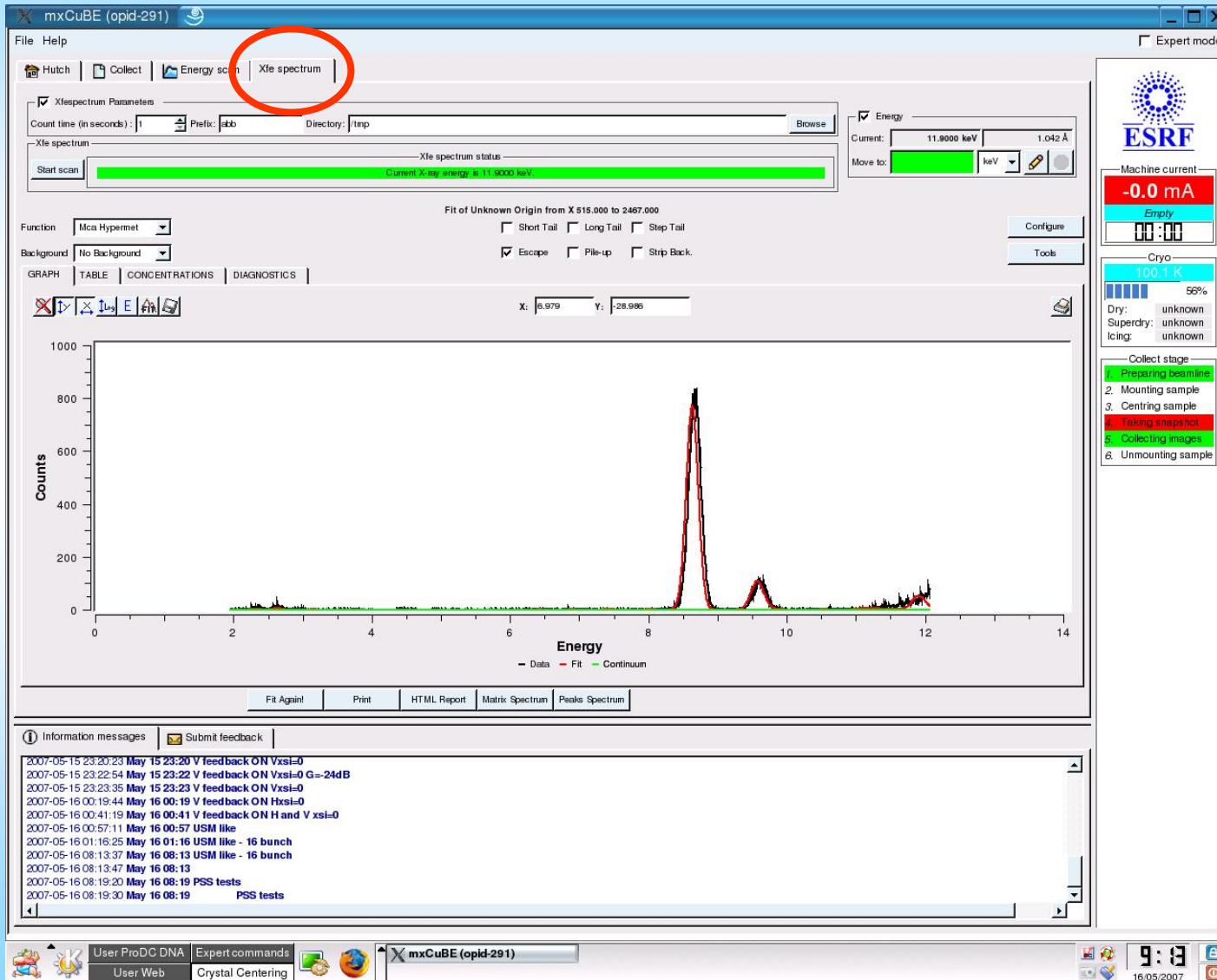
spec: eh

mxCuBE ISPyB mxCuBE (opid-231) Vacuum Viewer 1.6

12:47 27/07/2007



Metal ions in crystals: fluorescence analysis



Metal ions in crystals: fluorescence analysis

mxCuBE (opid-291)

File Help Expert mode

Hutch Collect Energy scan Xfe spectrum

Xfespectrum Parameters

Count time (in seconds): 1 Prefix: abh Directory: /tmp Browse

Xfe spectrum Xfe spectrum status

Start scan: Current X-ray energy is 11.9000 keV

Energy: Current: 11.9000 keV 1.042 Å Move to: keV

Function: Mca Hypermet Background: No Background

Fit of Unknown Origin from X 515.000 to 2467.000

Short Tail Long Tail Step Tail Escape Pile-up Strip Back

Configure Tools

GRAPH TABLE CONCENTRATIONS DIAGNOSTICS

	Element	Group	Fit Area	Sigma	Energy	Ratio	FWHM	Chi square
1	S	K	1.4734e+02	1.86e+01				
2		KL3	1.3523e+02	1.71e+01	2.307	0.91784	0.130	3.40
3		KM3	1.2105e+01	1.53e+00	2.464	0.08216	0.132	3.41
4		KL3 Si_KM3esc	5.8041e-02	7.33e-03	0.471	0.00043	0.099	0.00
5		KL3 Si_KL3esc	2.0060e+00	2.53e-01	0.568	0.01483	0.101	0.00
6		KM3 Si_KM3esc	4.9766e-03	6.29e-04	0.628	0.00041	0.102	0.00
7		KM3 Si_KL3esc	1.7128e-01	2.16e-02	0.724	0.01415	0.104	0.00
8	Cl	K	2.5926e+02	2.22e+01				
9		KL3	2.3222e+02	1.99e+01	2.622	0.89574	0.134	3.14
10		KM3	2.7031e+01	2.32e+00	2.816	0.10426	0.137	3.38
11		KL3 Si_KM3esc	9.0967e-02	7.80e-03	0.786	0.00039	0.105	0.00
12		KL3 Si_KL3esc	3.1177e+00	2.67e-01	0.882	0.01343	0.107	0.00
13		KM3 Si_KM3esc	9.9190e-03	8.50e-04	0.980	0.00037	0.109	0.00
14		KM3 Si_KL3esc	3.3786e-01	2.90e-02	1.076	0.01250	0.110	0.00
15	K	K	1.5254e+01	9.99e+00				
16		KL3	1.3296e+01	8.71e+00	3.313	0.87168	0.144	0.93
17		KM3	1.9573e+00	1.28e+00	3.590	0.12832	0.147	0.77
18		KL3 Si_KM3esc	4.1266e-03	2.70e-03	1.477	0.00031	0.117	0.00
19		KL3 Si_KL3esc	1.3864e-01	9.08e-02	1.573	0.01043	0.119	0.00
20		KM3 Si_KM3esc	5.5140e-04	3.61e-04	1.754	0.00028	0.121	0.00
21		KM3 Si_KL3esc	1.8394e-02	1.21e-02	1.850	0.00040	0.123	1.49
22	Ca	K	6.0047e+01	9.13e+00				
23		KL3	5.1967e+01	7.90e+00	3.690	0.86543	0.149	0.86
24		KM3	8.0805e+00	1.23e+00	4.013	0.13457	0.153	0.50
25		KL3 Si_KM3esc	1.4097e-02	2.14e-03	1.855	0.00027	0.123	1.53
26		KL3 Si_KL3esc	4.6901e-01	7.13e-02	1.951	0.00903	0.124	1.40
27		KM3 Si_KM3esc	1.9276e-03	2.93e-04	2.177	0.00024	0.128	1.87
28		KM3 Si_KL3esc	6.3599e-02	9.66e-03	2.273	0.00787	0.129	3.23
29	Mn	K	5.5875e+00	1.70e+01				

Fit Again! Print HTML Report Matrix Spectrum Peaks Spectrum

Machine current: **-0.0 mA**
Empty
00:00

Cryo: 100.2 K
56%


Dry: unknown
Superdry: unknown
Icing: unknown

Collect stage

1. Preparing beamline
2. Mounting sample
3. Centring sample
4. Taking snapshot
5. Collecting images
6. Unmounting sample

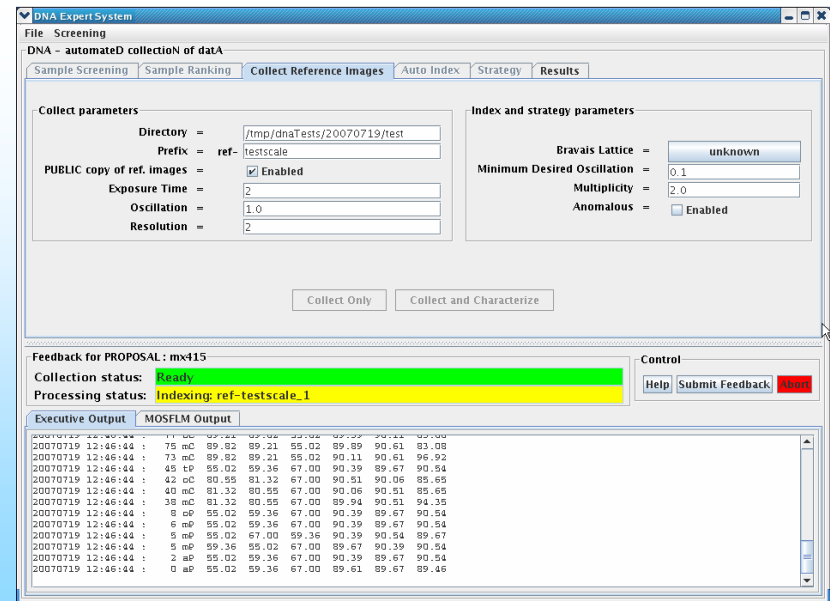
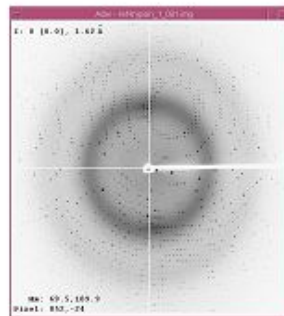
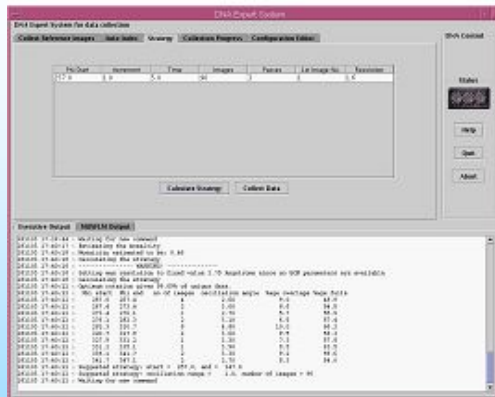
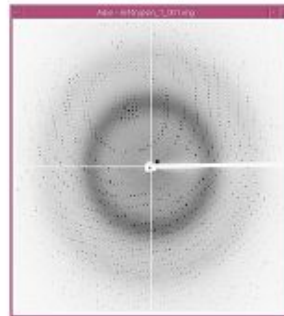
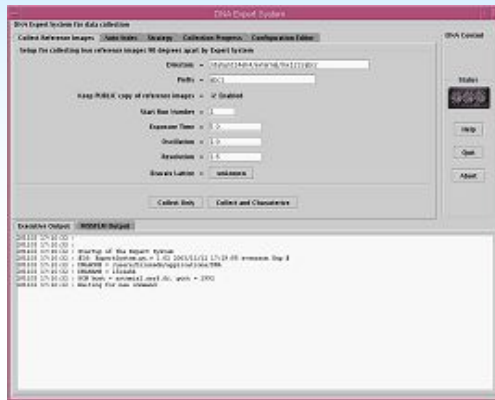
User ProDC DNA Expert commands User Web Crystal Centering mxCuBE (opid-291) KSnapshot

9:14 16/05/2007



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 and Hamburg outstations); Global Phasing Ltd, MRC - France (BM14) and SOLEIL.



Automatic data collection, reduction & scaling

The screenshot shows the 'DNA Expert System' interface with the 'Auto Index' tab selected. The 'Source Reference Images' section contains the following fields:

- Directory: /tmp/dnaTests/20070719/test
- Prefix: testscale
- Start Run Number: 1

A 'Start Auto Index and Integrate' button is visible. A warning dialog box is open, displaying the following text:

Warning!
The program BEST has calculated the strategy to resolution: 1.98 Å
This is the resolution that will be used if you choose to start a data collection.

BEST has calculated that it should be possible to collect data to 1.26 Å from this sample.
If you want to calculate a new strategy for data collection to this resolution you must first recollect reference images at this resolution ("Collect Reference Images" tab).

The 'Feedback for PROPOSAL' section shows 'Collection status: Ready' and 'Processing status: Ready'. The 'Executive Output' window displays the following log data:

```
20070719 12:47:10 : .
20070719 12:47:10 : Strategy results:
20070719 12:47:10 : Completeness: 100.00
20070719 12:47:10 : Multiplicity: 3.45
20070719 12:47:10 : Phi start: 118.00
20070719 12:47:10 : Phi end: 203.50
20070719 12:47:10 : Phi width: 2.25
20070719 12:47:10 : Exposure time: 0.10
20070719 12:47:10 : I/sigma: 2.70
20070719 12:47:10 : R merge: 12.20
20070719 12:47:10 : Ranking resol: 1.26
20070719 12:47:10 : .
```

The screenshot shows the 'DNA Expert System' interface with the 'Strategy' tab selected. The 'DNA Reference Strategy' and 'Data Collection Strategy' tables are displayed:

Phi Start	Increment	Time	Images	1st Imag...	Resolution
118.0	2.25	0.1	38	1	1.98

Phi Start	Increment	Time	Images	1st Imag...	Resolution
118.0	2.25	0.1	38	1	1.98

Buttons for 'Calculate Strategy', 'Collect Data', 'Collect & Integrate Data', and 'Quick Scaling Statistics' are visible. The 'Feedback for PROPOSAL' section shows 'Collection status: Ready' and 'Processing status: Ready'. The 'Executive Output' window displays the following log data:

```
20070719 12:47:10 : Resolution returned by strategy: 1.26
20070719 12:47:16 : Ranking resolution returned by strategy: 1.26
20070719 12:47:16 : .
20070719 12:47:16 : =====
20070719 12:47:16 : The program BEST has calculated the strategy to resolution: 1.98 Å
20070719 12:47:16 : This is the resolution that will be used if you choose to start a data collection.
20070719 12:47:16 : .
20070719 12:47:16 : BEST has calculated that it should be possible to collect data to 1.26 Å from this sample.
20070719 12:47:16 : If you want to calculate a new strategy for data collection to this resolution you
20070719 12:47:16 : must first recollect reference images at this resolution ("Collect Reference Images" tab).
20070719 12:47:16 : .
20070719 12:47:16 : Waiting for new command
```

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

ISPyB
 Home Lab Data Collection

Shipment | Samples | Prepare experiment | Data collection | Feedback | Help

View sessions | Search data collections

Session Comments

Session Operator:
 Session Start:

Export as PDF

Run Prefix	Run No	Start Time	#images	Wavelength	Exposure Time	Phi start	Phi range	Detector Resolution	Run Status	Ship	Comments
test1	1	2009-10-12 15:01:14.0	10	0.876	0.1	100.05	0.95	2	DataCollect: collection successful		
test1	1	2009-10-12 15:00:00.0	3	0.876	0.1	90	0.95	2	DataCollect: collection successful		
test1.test1	1	2009-10-12 15:00:25.0	3	0.876	0.1	100	0.95	2	DataCollect: collection successful		
test1.test1	1	2009-10-12 14:50:00.0	2	0.876	0.5	0	1	2	DataCollect: collection successful		
test1.test1	9	2009-10-11 22:40:18.0	9	0.876	1	0	1	3	DataCollect: collection successful		ACT
test1.test1	9	2009-10-11 22:41:02.0	2	0.876	1	0	1	3	DataCollect: collection successful		SDCA

ISPyB
 Interaction System for Home and Synchrotron Beamlines

Samples | View | All samples | /sort.do

Shipment | Samples | Prepare experiment | Data collection | Feedback | Help

Create
 New sample
 New crystal form

View
 Proteins and crystal forms
 Unassigned Samples
 All samples

Search
 Protein
 Sample

Update Database

Shipments

Name: BAG meeting Outfit
 Shipping Date: 2009-09-21
 Back to this Shipment

Export as PDF (sort by acronym/sample name)

Protein	Sample name	Sm code	Dewar	Container	Loc. in cont.	Space Group	Cell a	Cell b	Cell c	Cell alpha	Cell beta	Cell gamma	Crystal	Already obs.	Minimal resol.	Sample comments	Edit sample	Sample status	Data Collections
THAU	xtal81	H400A0402	dewar1	A4010A	1	P41212	67.3	67.3	148.8	90.0	90.0	90.0	0	0			Edit	TEST_2009-10-02	
TLN	xtal80	H400A0399	dewar1	A4010A	10	P6122	83.0	83.0	130.2	90.0	90.0	120.0	0	2.5			Edit		
THAU	xtal82	H400A0411	dewar1	A4010A	2	P41212	67.3	67.3	148.8	90.0	90.0	90.0	0	0			Edit		
THAU	xtal83	H400A0701	dewar1	A4010A	3	P41212	67.3	67.3	148.8	90.0	90.0	90.0	0	0			Edit	TEST_2009-10-02	
THAU	xtal84	H400A0700	dewar1	A4010A	4	P41212	67.3	67.3	148.8	90.0	90.0	90.0	0	0			Edit	TEST_2009-10-02	
Prot_K	xtal85	H400A0700	dewar1	A4010A	5	P6212	88.2	88.2	108.3	90.0	90.0	90.0	0	0			Edit		
Prot_K	xtal86	H400A0651	dewar1	A4010A	6	P4212	88.2	88.2	108.3	90.0	90.0	90.0	0	0			Edit		
Prot_K	xtal87	H400A0654	dewar1	A4010A	7	P4212	88.2	88.2	108.3	90.0	90.0	90.0	0	0			Edit	TEST_2009-10-02	
TLN	xtal88	H400A0657	dewar1	A4010A	8	P6122	83.0	83.0	130.2	90.0	90.0	120.0	0	2.5			Edit		

View sessions | Search data collections

Session Comments

Start Date: 11-10-2006
 Session Name: 029

Export as PDF

Session Parameters

Exp. prefix	xxx1
Run no.	1
Start Time	11-10-2006 19:03:40
End Time	11-10-2006 19:04:16
Wavelength	1.277 Angstrom
Detector Distance	333.69 mm
Exposure Time	3.0 sec
Phi start	0.0 deg
Phi range	3.0 deg
Xbeam	155.76 mm
Ybeam	161.37 mm
Detector Resolution	3.0 Angstrom

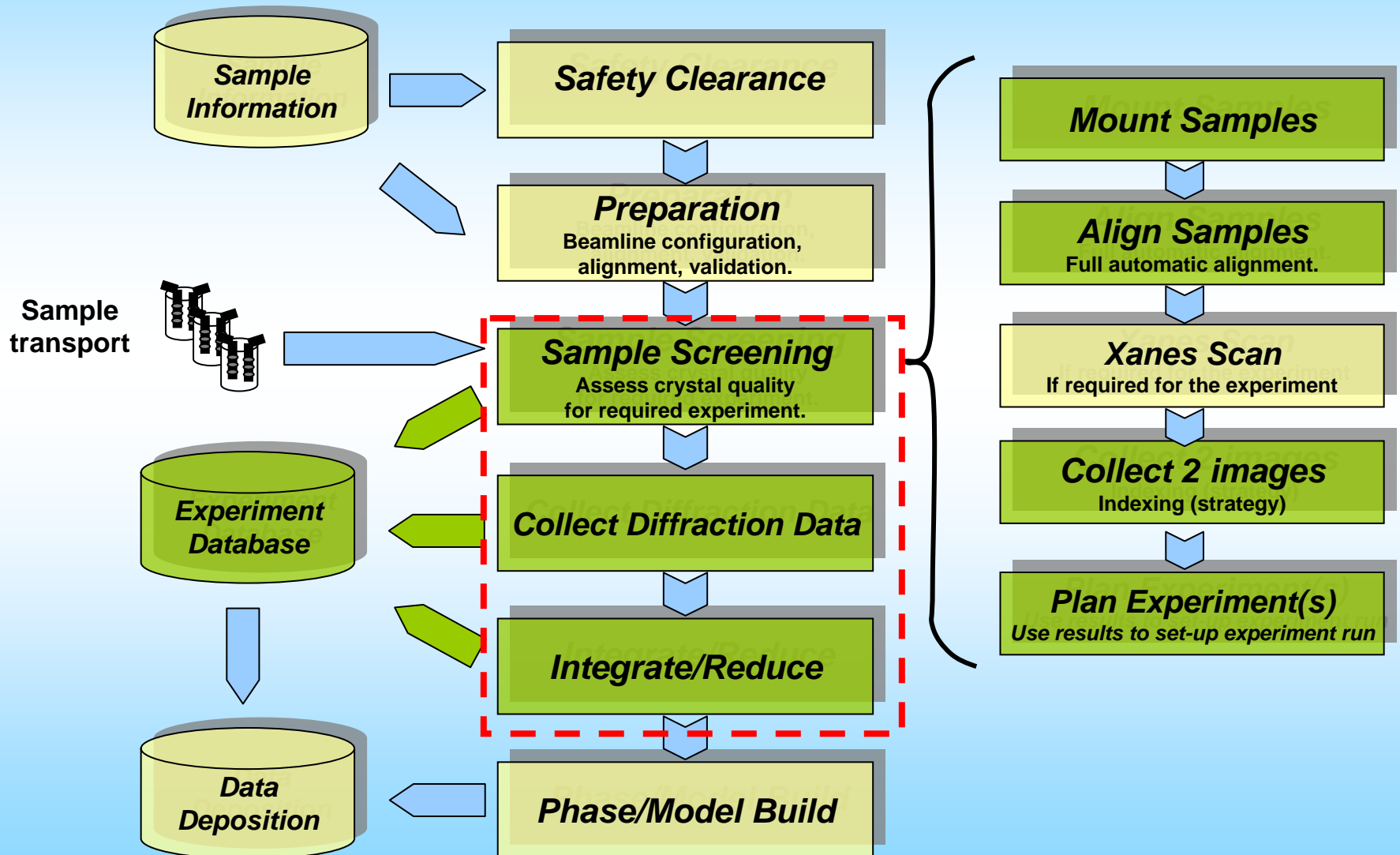
Crystal Snapshot

Expected Snapshot Location: /data/ED2/archive/mx412/20061031/ARX/xx1_1_1_4-image.tif

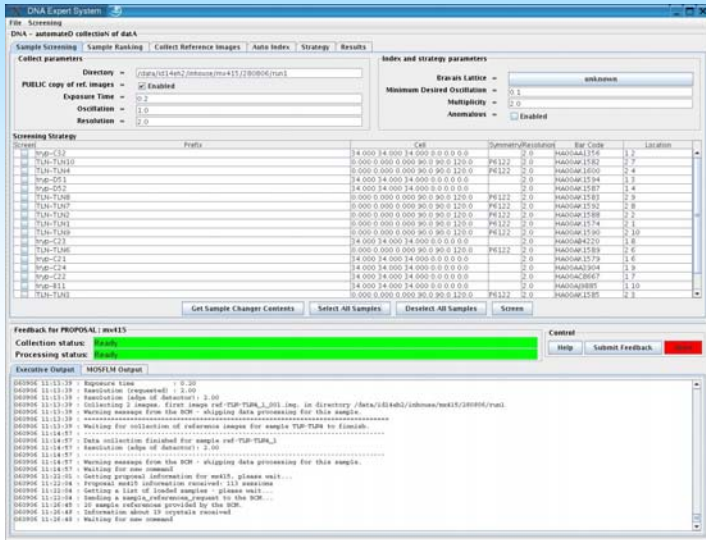
Images collected

Image	Image name	Image Location

The Data Collection Pipeline (DCP) strings automated procedures together



What the DCP does



- Scans 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.

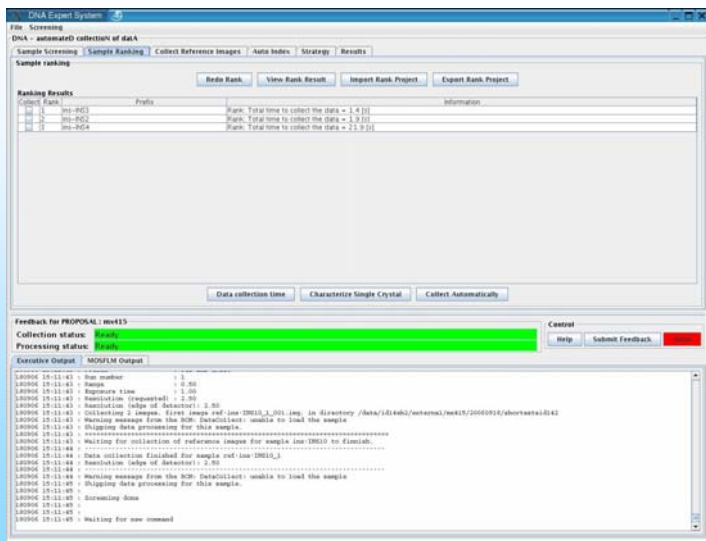
- Takes two diffraction images at 90° in phi to each other for use by DNA

- 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

Process
Results

Available samples

Name	Acronym	Barcode	Location	Space group	a b c α β γ	Min.res.	Basket
Trypsin01	tryp	HA00AI8492		Undefined	34.0 34.0 34.0 ???		
Trypsin02	tryp	HA00AI8493		Undefined	34.0 34.0 34.0 ???		
Trypsin03	tryp	HA00AI8494		Undefined	34.0 34.0 34.0 ???		
Trypsin04	tryp	HA00AI8495		Undefined	34.0 34.0 34.0 ???		
Trypsin05	tryp	HA00AI8496		Undefined	34.0 34.0 34.0 ???		
Trypsin06	tryp	HA00AI8497		Undefined	34.0 34.0 34.0 ???		
Trypsin07	tryp	HA00AI8498		Undefined	34.0 34.0 34.0 ???		
Trypsin08	tryp	HA00AI8499		Undefined	34.0 34.0 34.0 ???		
Trypsin09	tryp	HA00AI8500		Undefined	34.0 34.0 34.0 ???		
Trypsin10	tryp	HA00AI8501		Undefined	34.0 34.0 34.0 ???		

Show only the samples inside the sample changer (109 samples) Group by: no grouping

Directory: /tmp/gabadinho/external/mx415/20070820/tryp/Trypsin07 Browse Prefix: Run number: 1

1st step: Take reference images

Template:

Oscillation range (deg): Exposure time (s): Default resolution (Å):

2nd step: Index reference images

Template:

Space group: ▶

3rd step: Calculate strategy

Space group: ▶ Min. oscillation (deg): Multiplicity: Anomalous:

4th step: Collect and integrate

Template:

First image #: Oscillation start (deg): Exposure time (s):

Number of images: Oscillation range (deg): Resolution (Å):

Execute steps

Status: not logged!

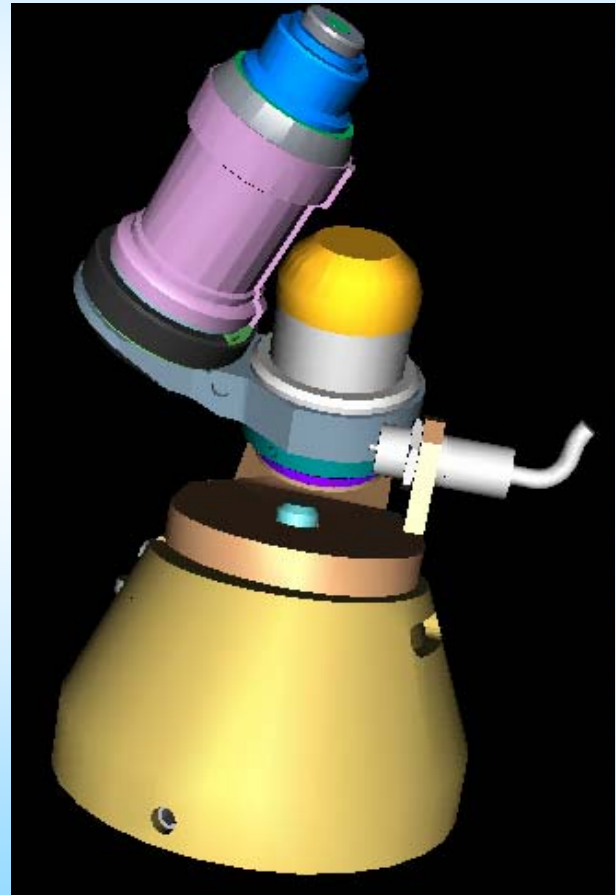
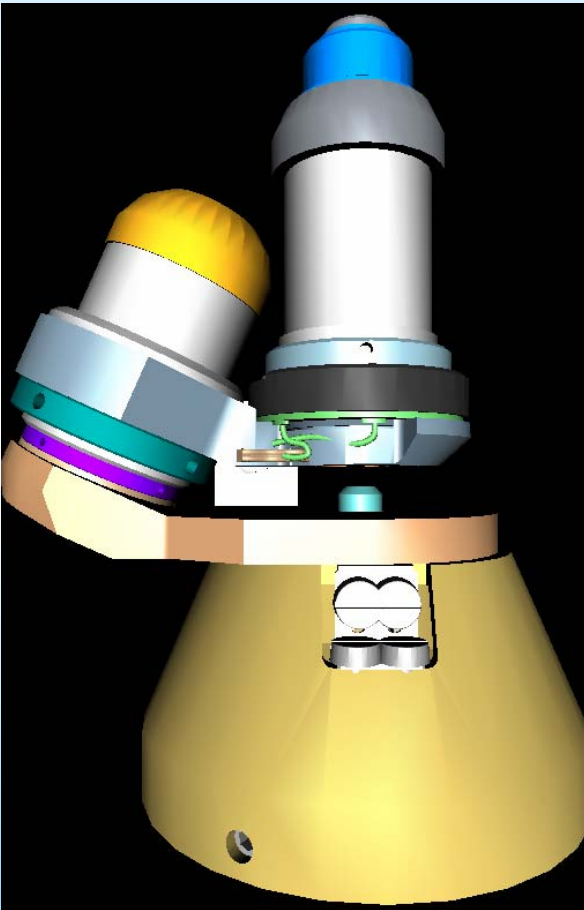
Abort

Current results

Results file: Go to Results tab a: b: c:

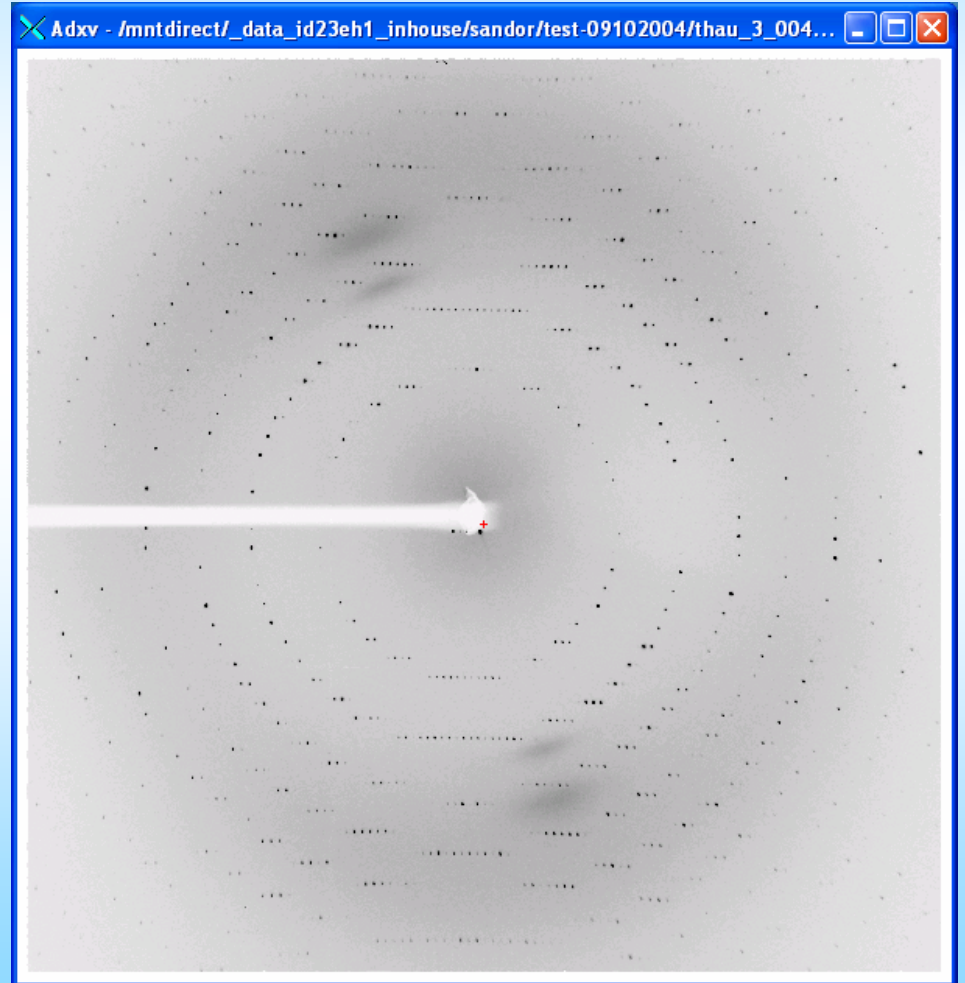
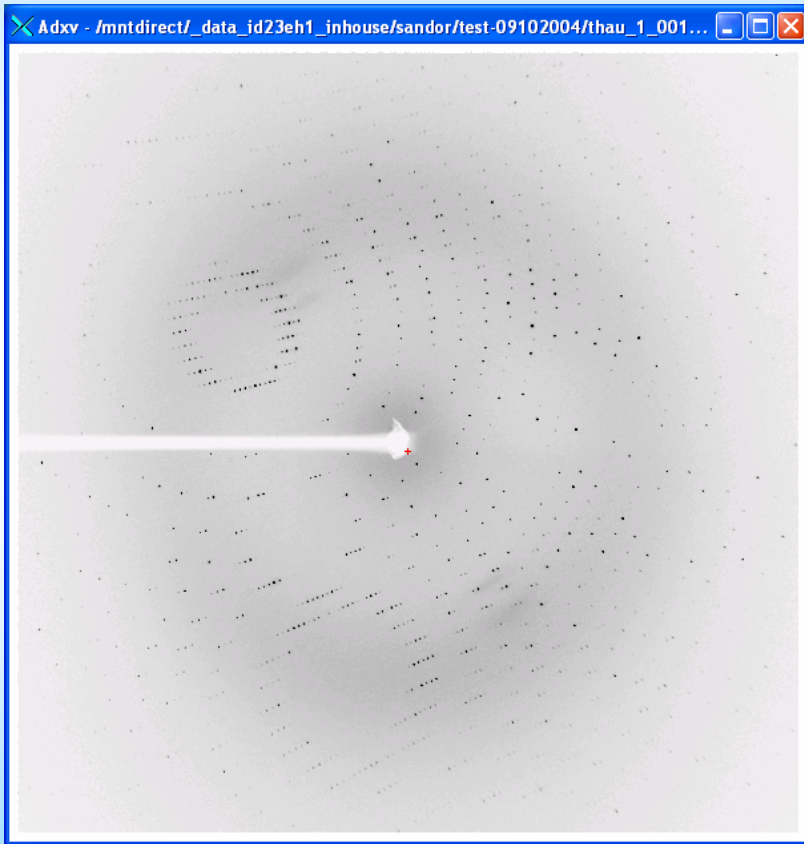
Space group: alpha: beta: gamma:

EMBL/ESRF Mini-Kappa



Thaumatococcus - tetragonal

(ESRF; ID23-1)



Omega	Kappa	Phi
2.4	60.6	-74.7