

MX at Diamond Light Source

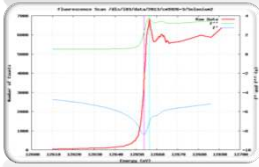


Katherine McAuley
I03 PBS, Diamond

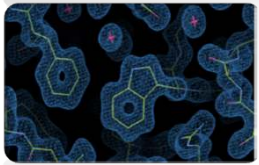
The aim of this talk is to give an introduction to synchrotrons and MX beamlines and how they work



Why do you need to collect MX data at synchrotrons?



Phasing experiments



High resolution



High throughput



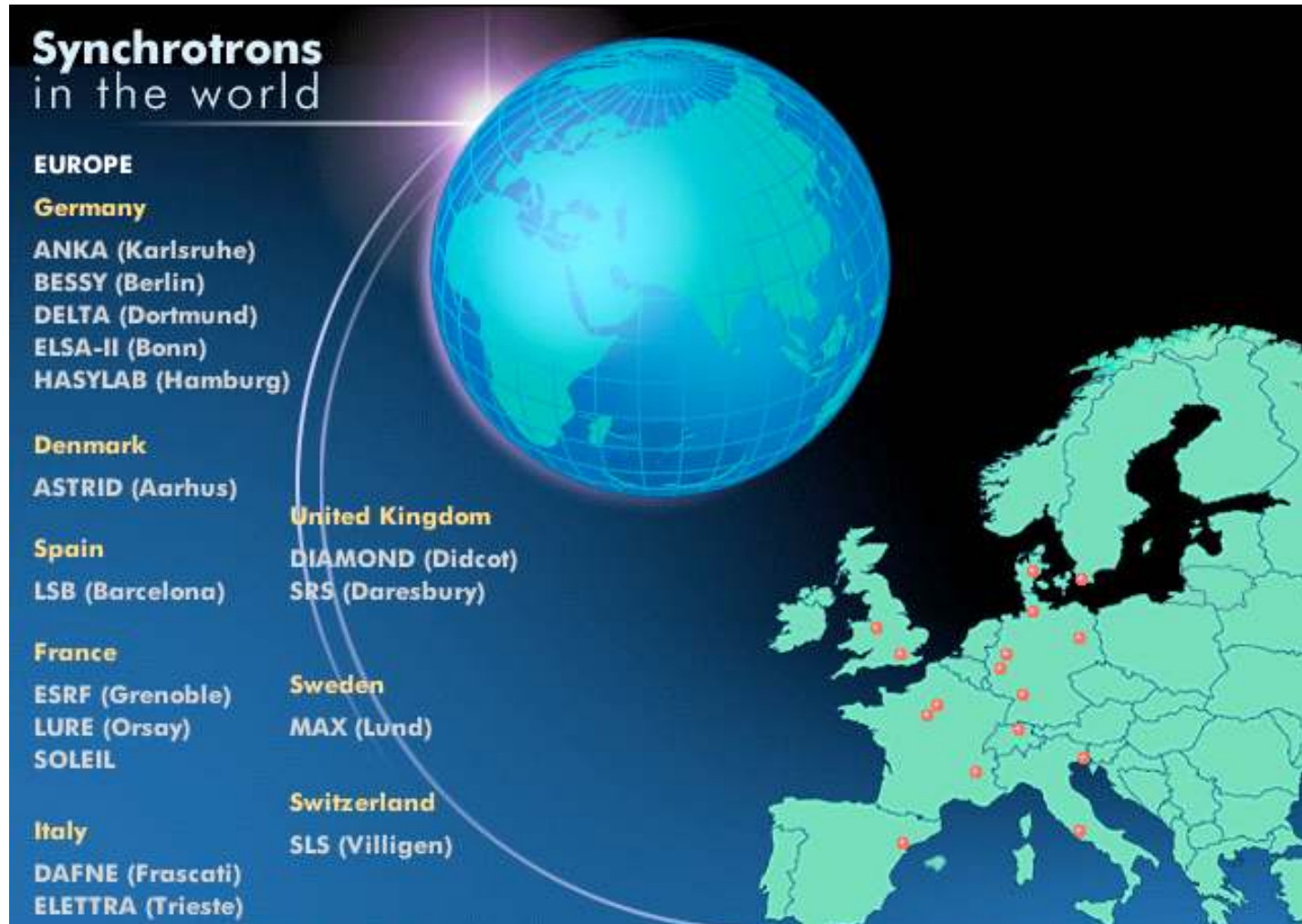
Special Conditions

What is a synchrotron?

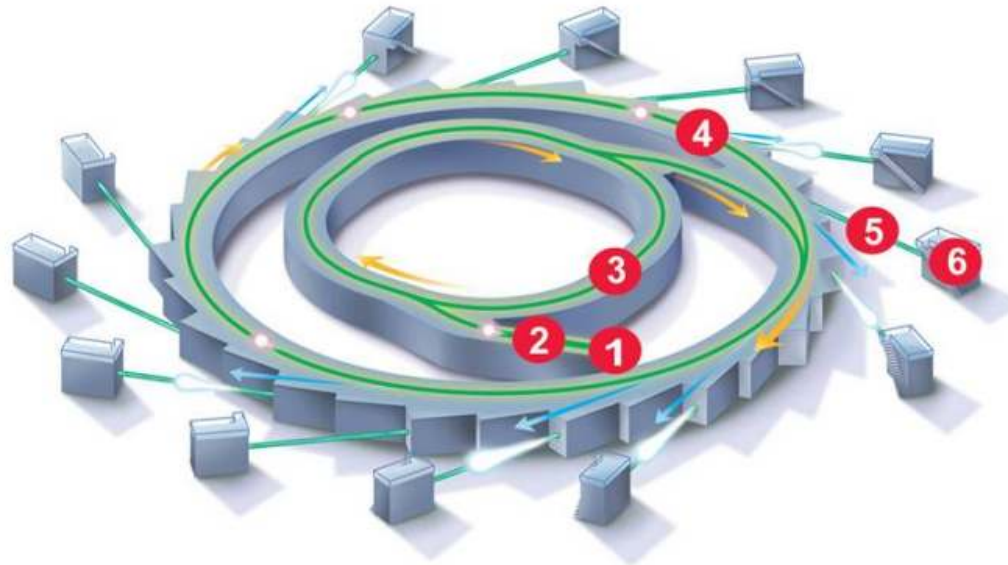
- ▶ A synchrotron is a particle accelerator (typically electrons) which provides a source of extreme intensity light
- ▶ Can be compared to a series of 'super microscopes' or a gigantic x-ray machine;
- ▶ Diamond is the UK's synchrotron light facility that provides intense light ranging from Infrared to hard X-rays



Synchrotrons in Europe



Synchrotron - main components



1 Electron Gun: produces electrons

2 Linear Accelerator (LinAc): accelerates electrons to near the speed of light

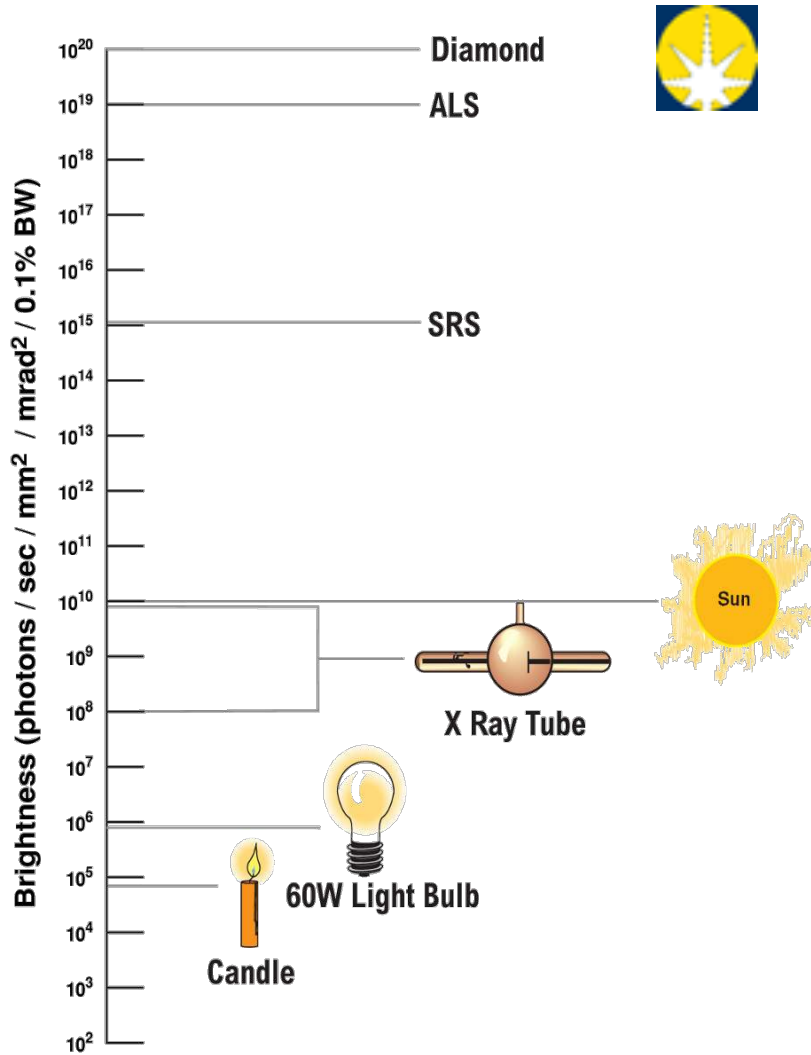
3 Booster Ring: further increases the energy of the electrons

4 Storage Ring: maintains the electron energy and produces radiation

5 Beamline: set up of radiation for experiments

6 Experimental Hutch: User area

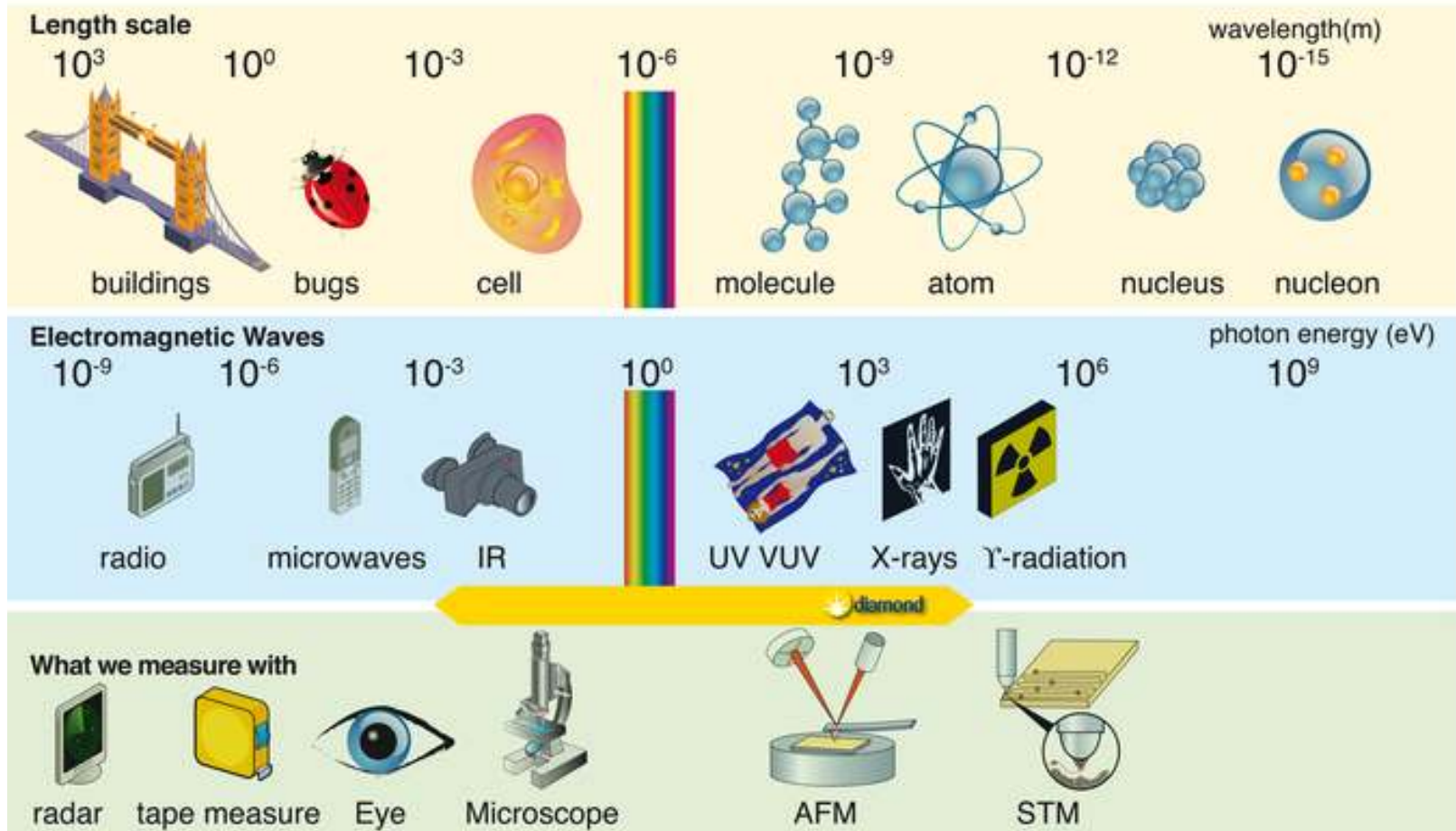
In this business, it's all about brightness !



- ▶ X-rays from Diamond are 100 billion times brighter (10^{11}) than from an X-ray tube
- ▶ Or 10 billion times brighter (10^{10}) than the Sun
- ▶ $>10^5$ times brighter than the previous UK Synchrotron, the SRS !

And Broad Continuous Spectrum!

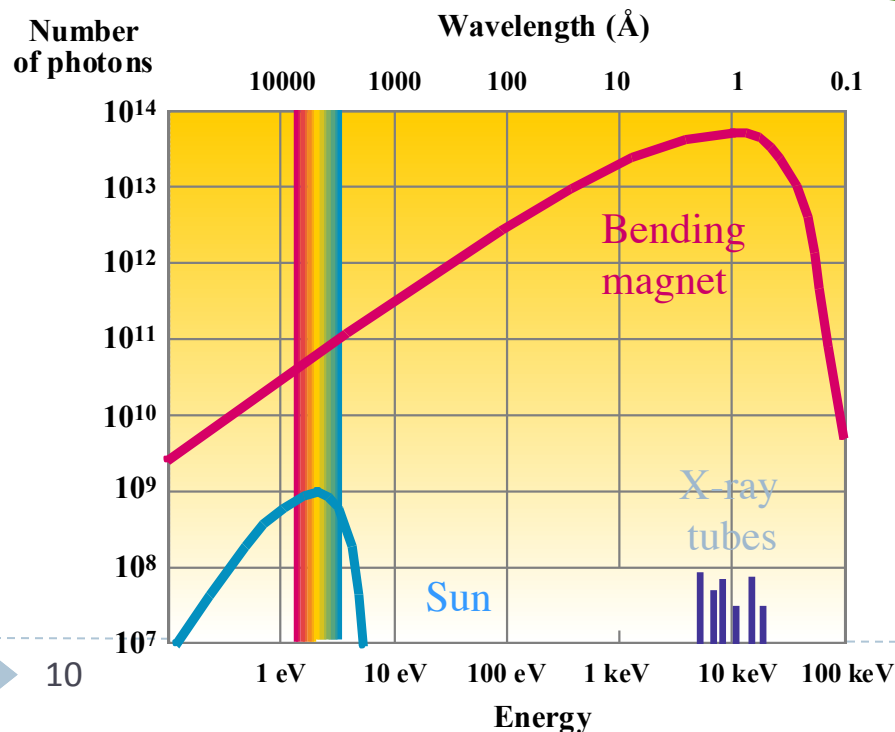
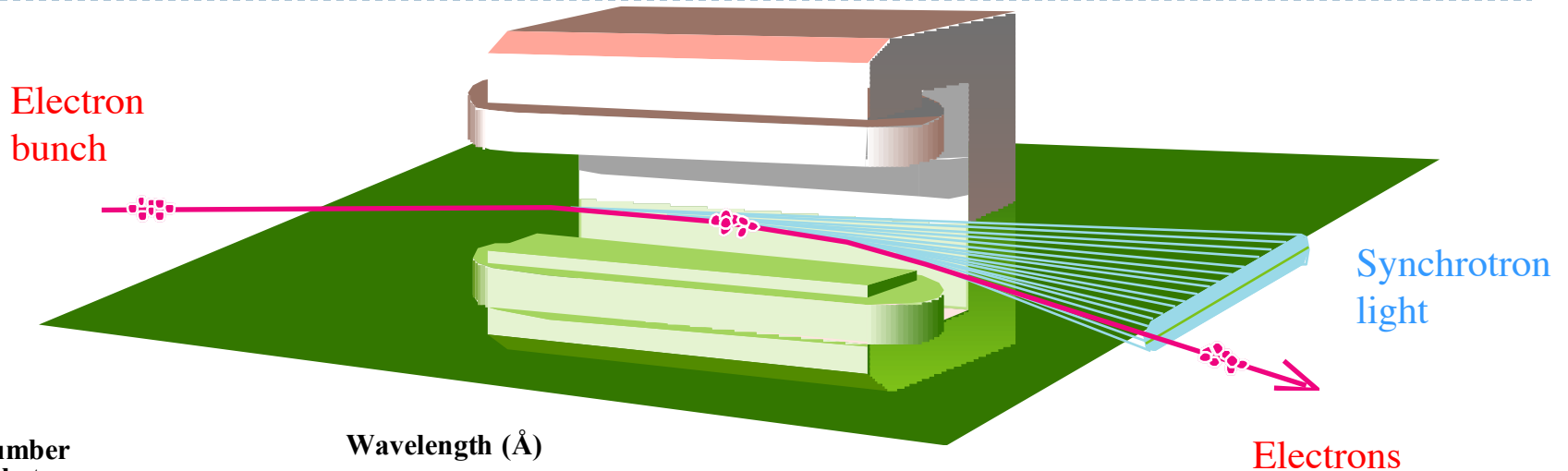
The many colours of light



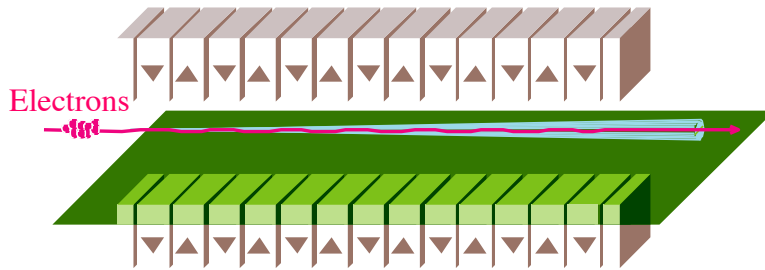
Diamond is a 3rd generation light source

- ▶ 1st Generation sources
 - ▶ parasitic
- ▶ 2nd Generation sources
 - ▶ dedicated machines to synchrotron radiation
 - ▶ emittance typically high (150 nmrad)
- ▶ 3rd Generation sources
 - ▶ low beam emittance
 - ▶ use of insertion devices (wigglers and undulators)
 - ▶ emittance on order of 3-20 nmrad

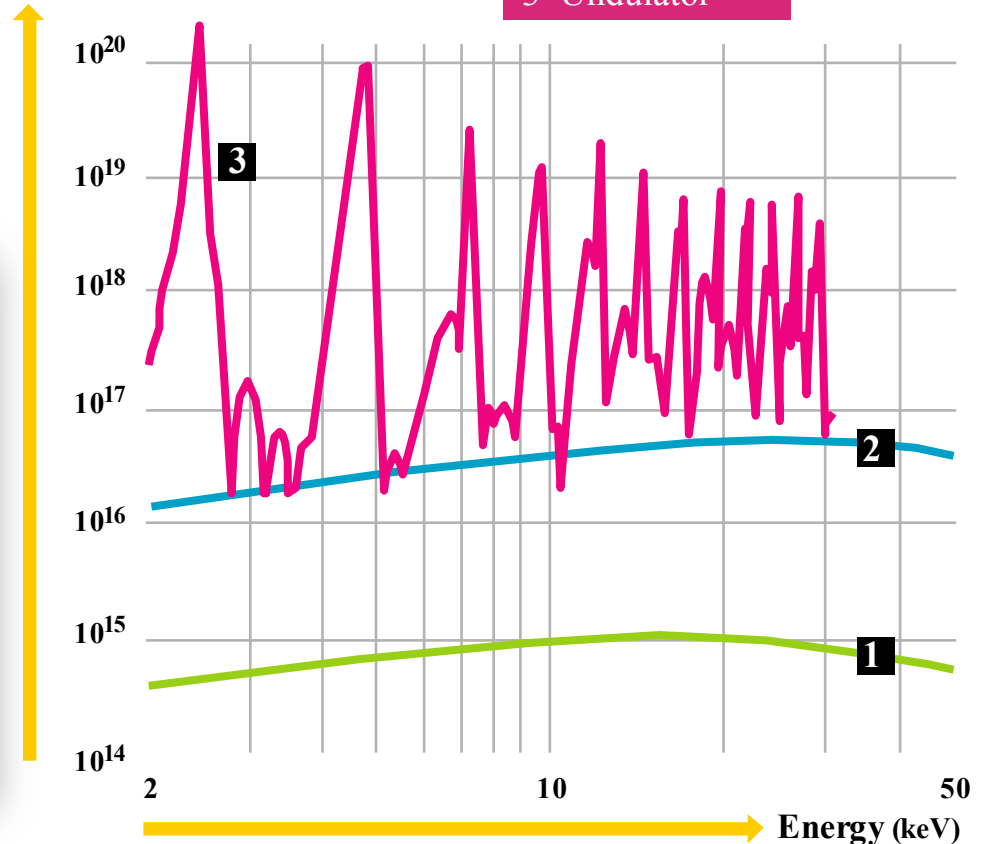
Bending Magnets curve the electron beam between adjacent straight sections and generate synchrotron light



The MX beamlines at Diamond use insertion devices called undulators



Brilliance
(photons/s/mm²/mrad²/0.1% BW)



Bending magnet

2 Wiggler

3 Undulator



MX Village consists of 5 operational beamlines and several under development

■ Operational

■ Phase I

- Tuneable I02, I03, I04

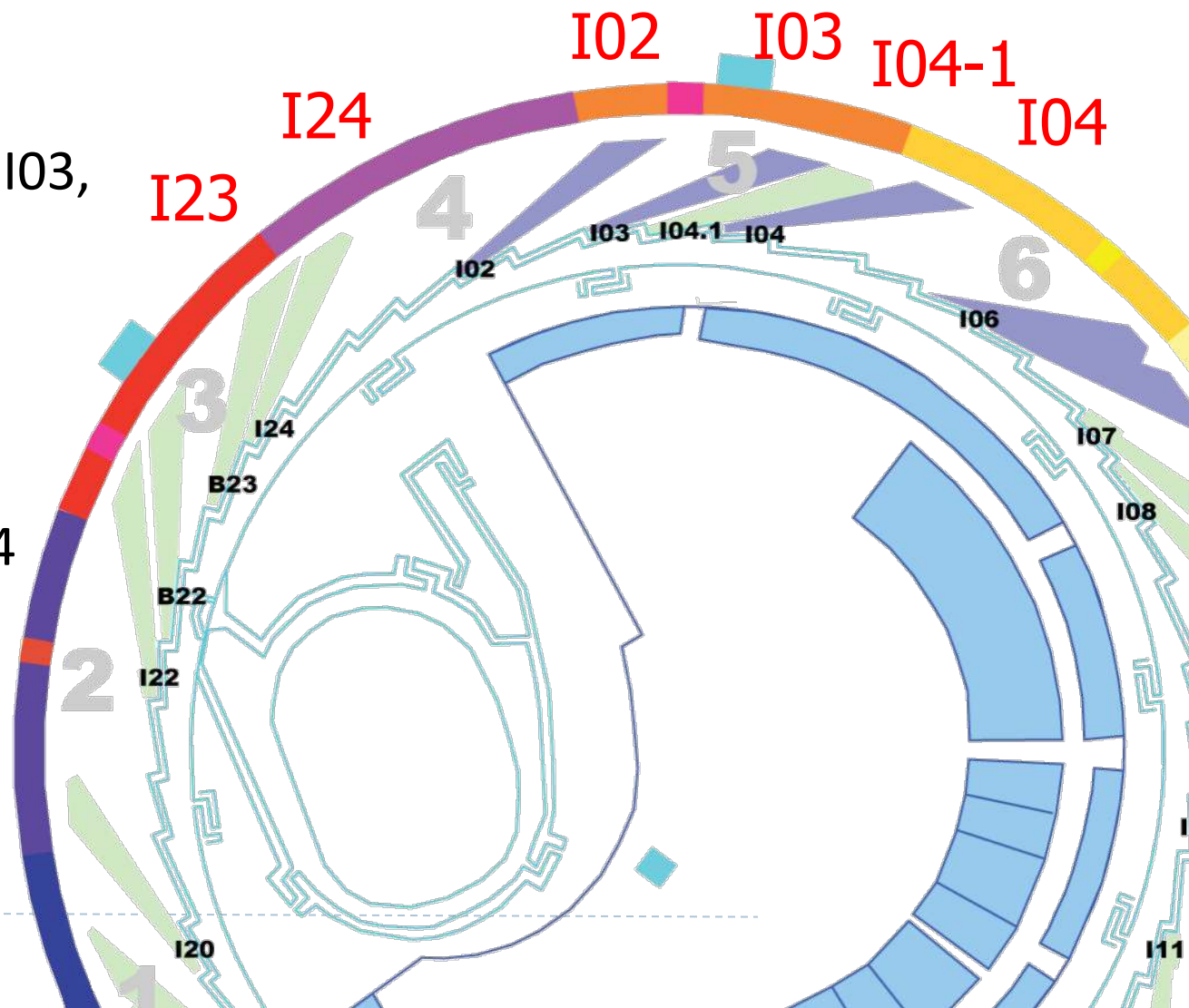
■ Phase II

- Fixed λ I04-1
- Tuneable microfocus I24

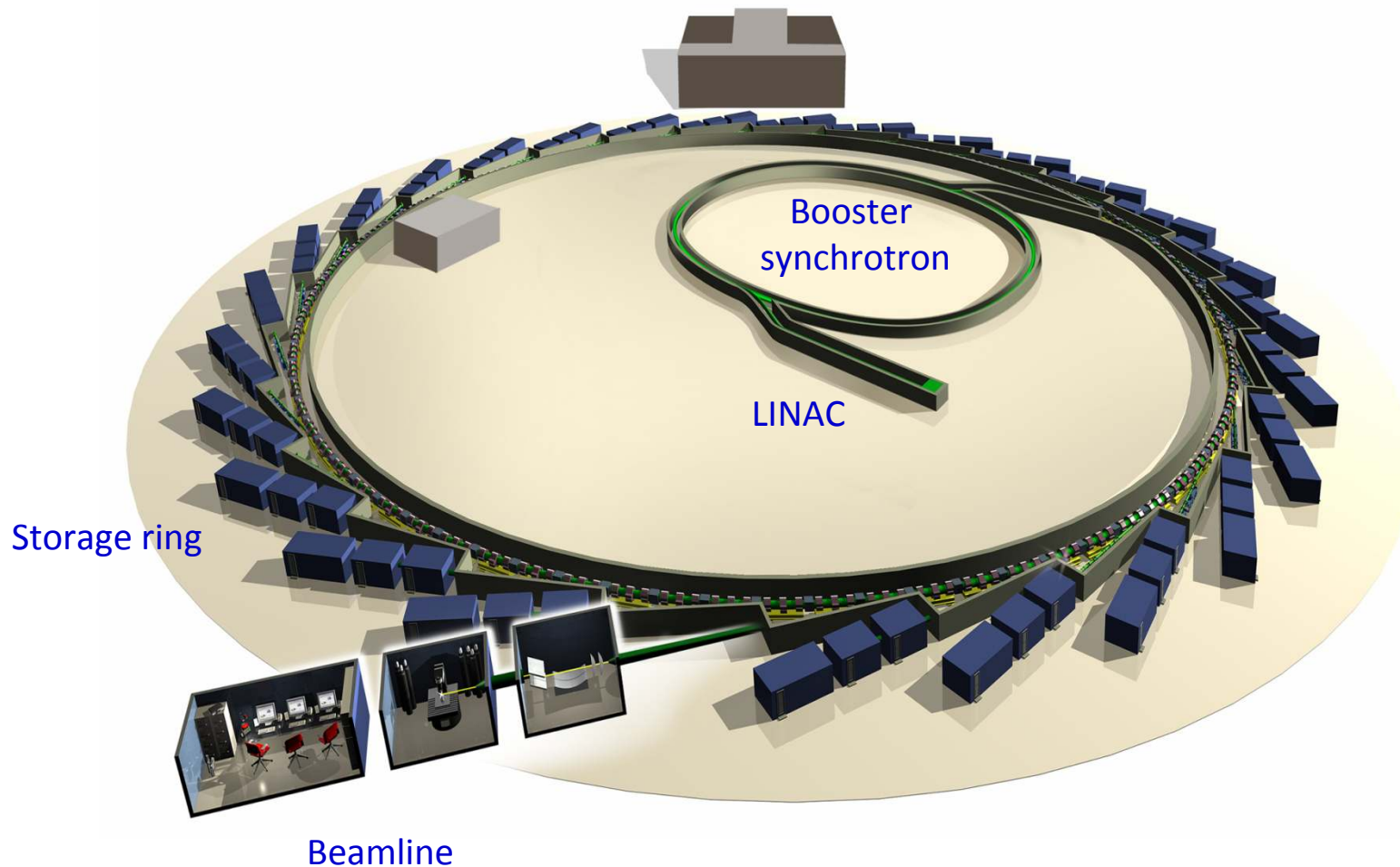
■ In Development

■ Phase III

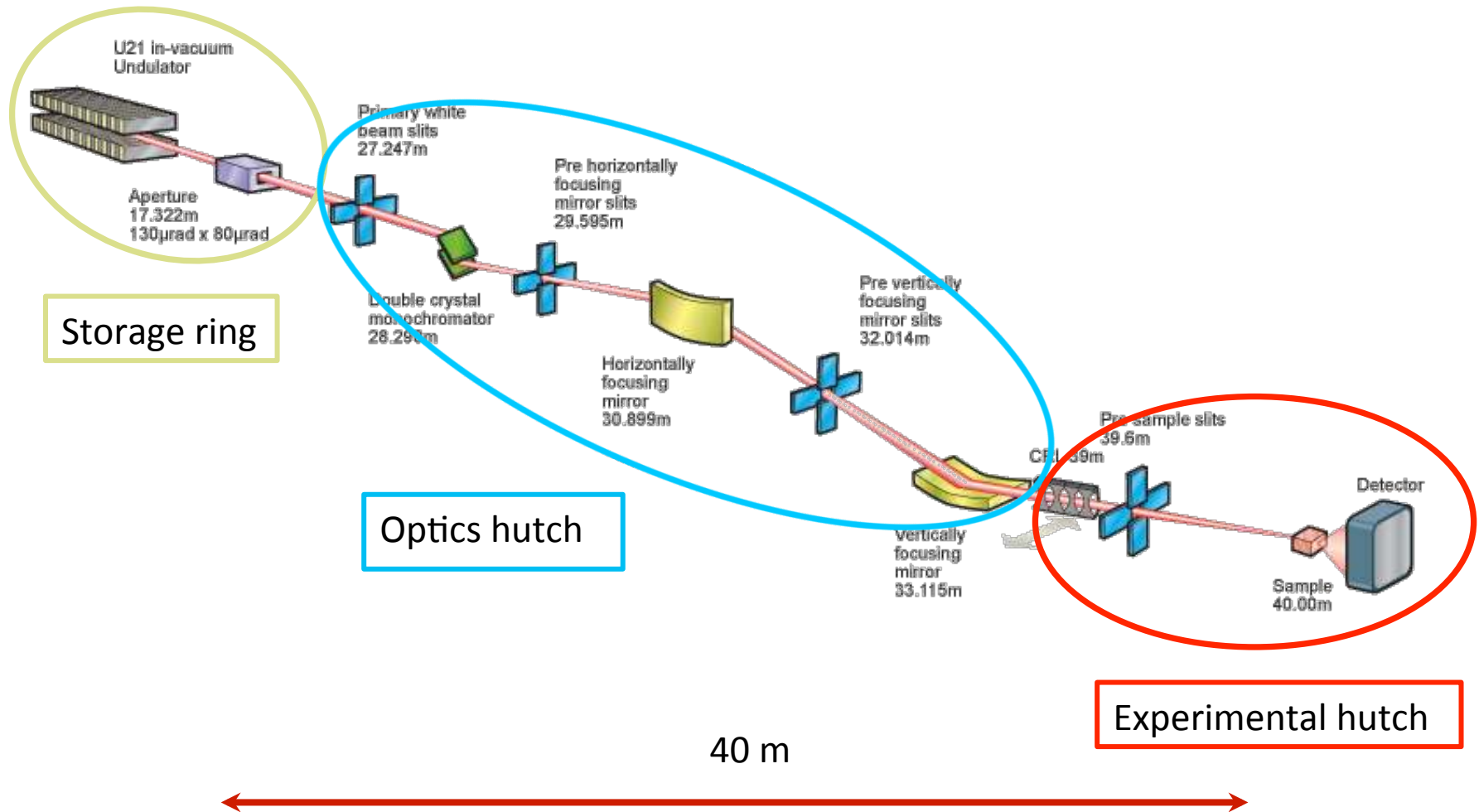
- Long λ I23
- VMX *m* and *i*



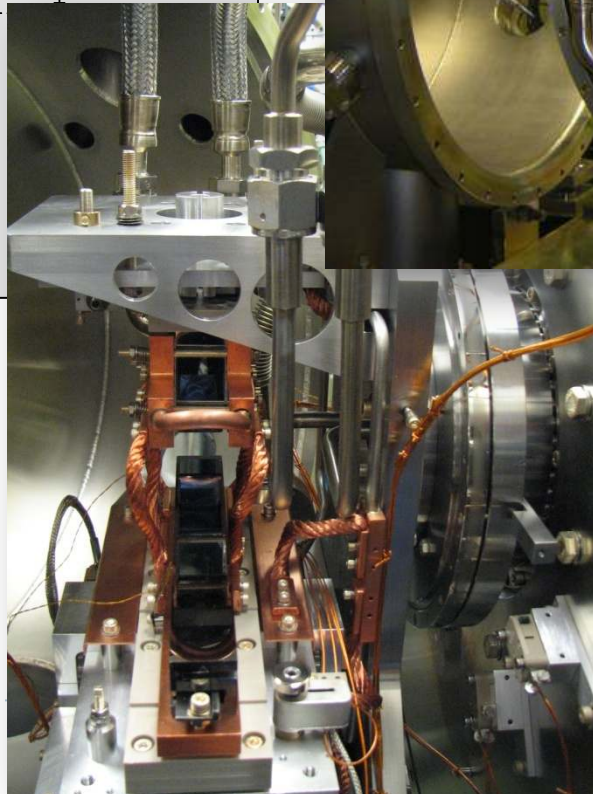
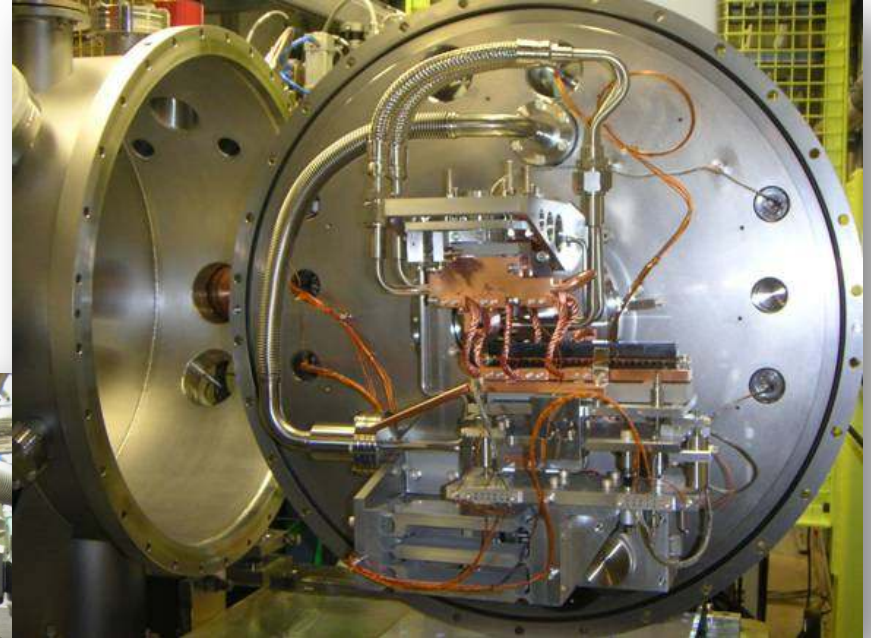
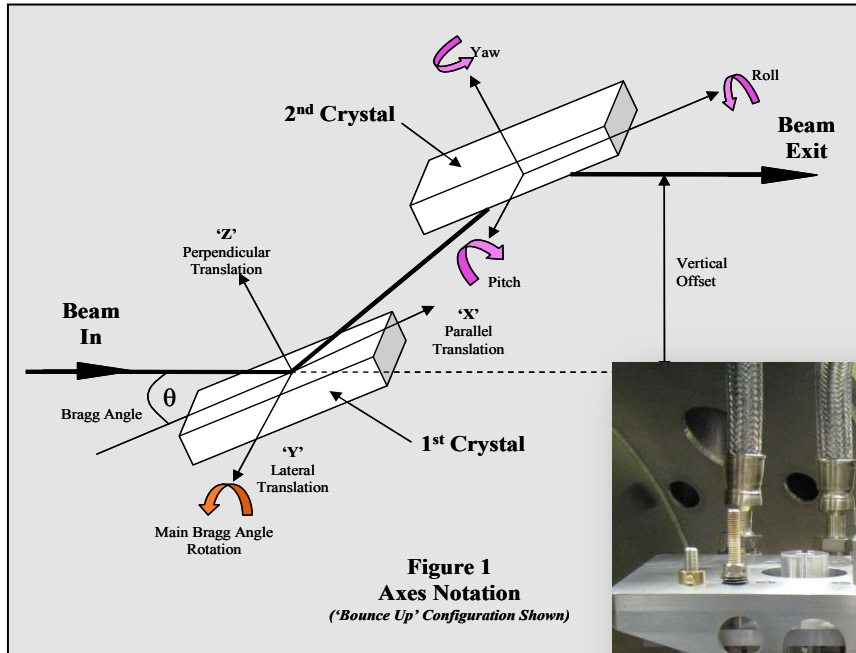
Beamlines consist of an optics hutch, experimental hutch and a control area



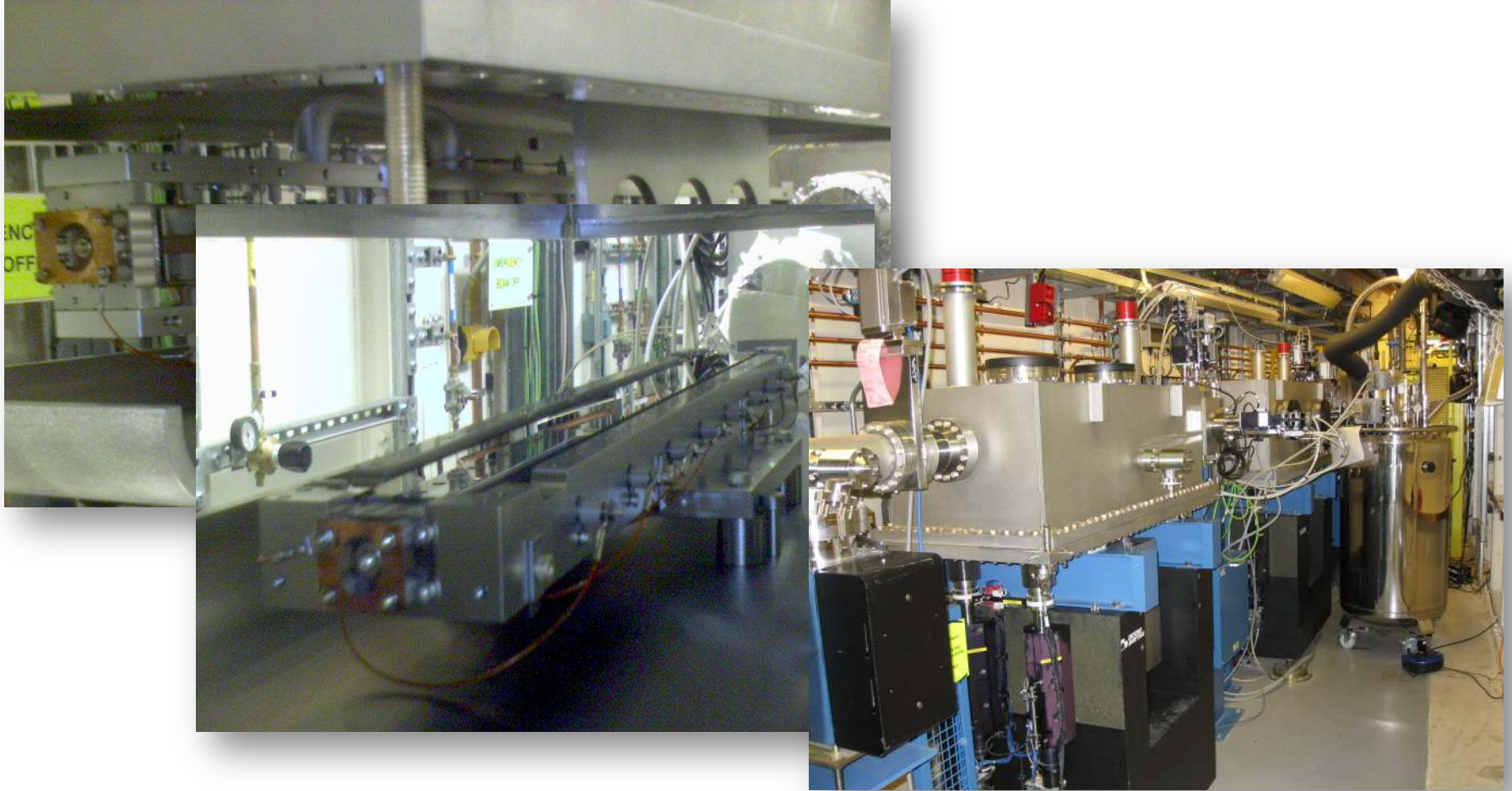
The components in the optics hutch are used to select the X-ray energy and adjust the beam size



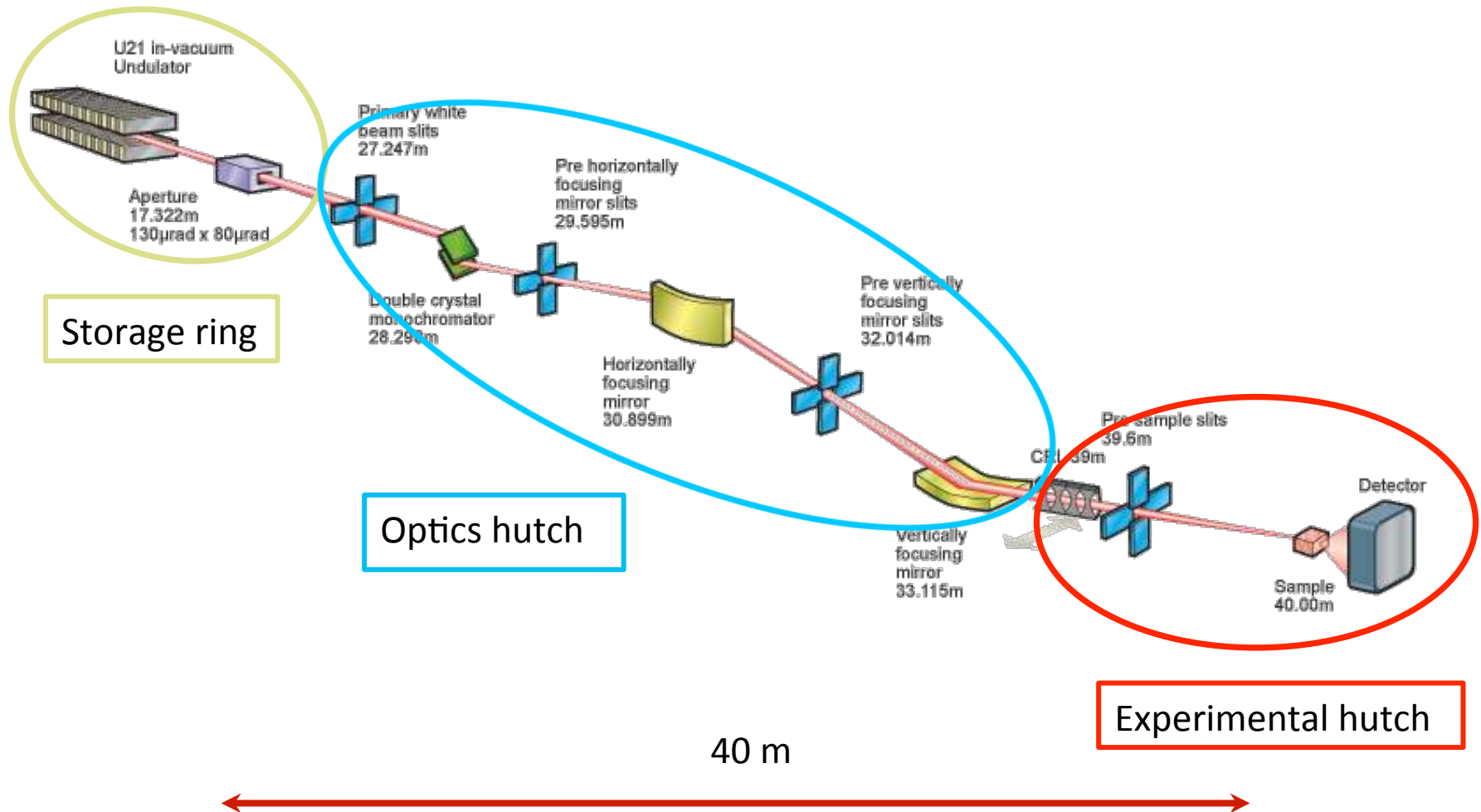
A double crystal monochromator is used to select the X-ray energy (wavelength)



One mirror is used to focus the X-ray beam horizontally and another to focus vertically

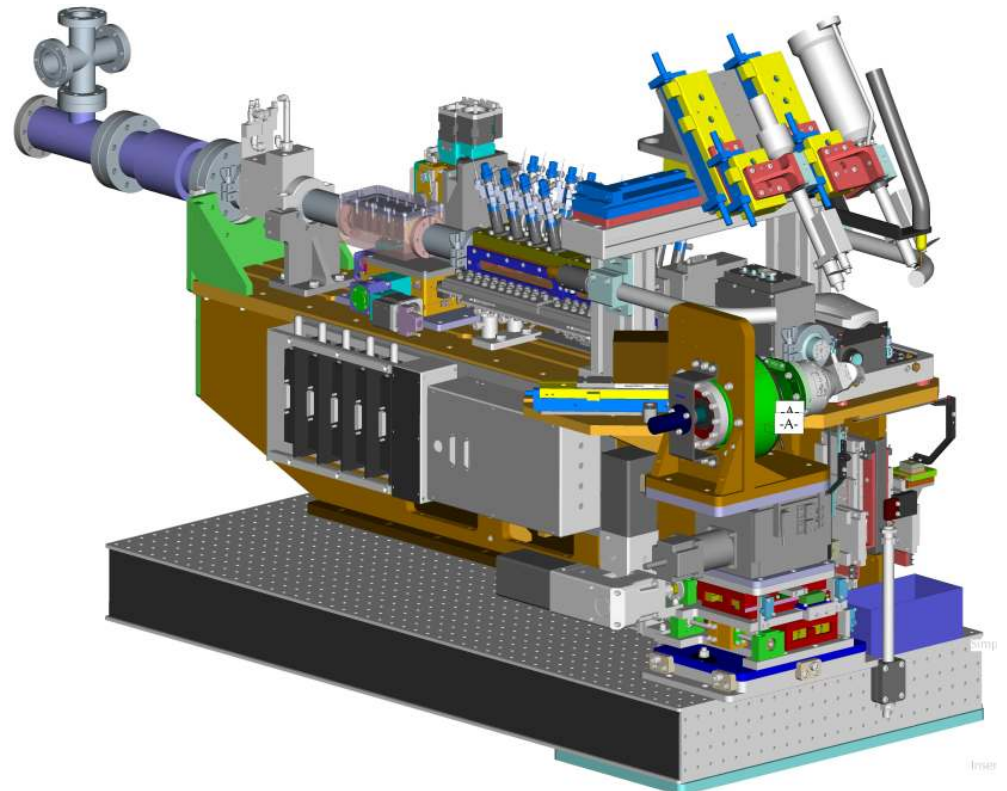


The experiment and final beam conditioning happens in the experiment hutch



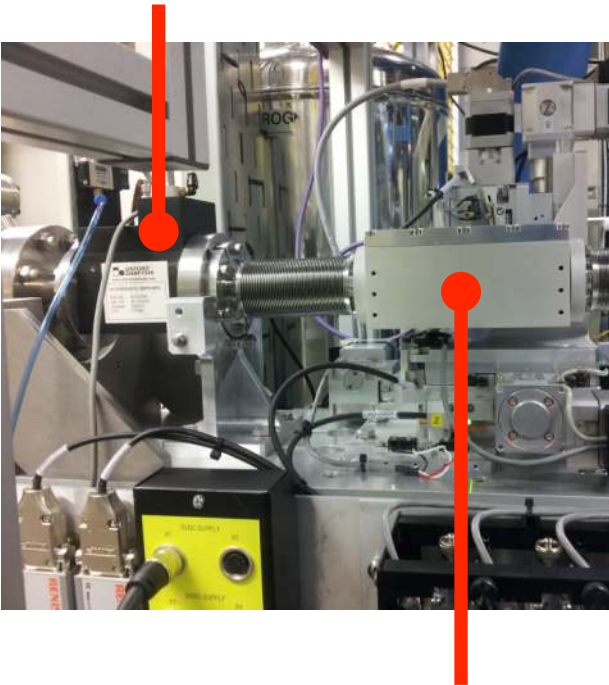
The end-station table supports:

- ▶ CRLs
- ▶ Attenuators
- ▶ Beam diagnostics
- ▶ Slits
- ▶ On-axis viewing system
- ▶ Sample environment

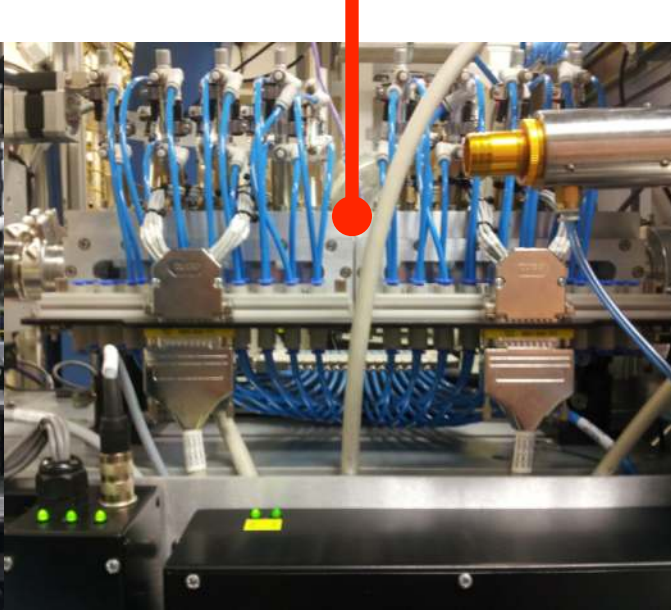


Beam conditioning

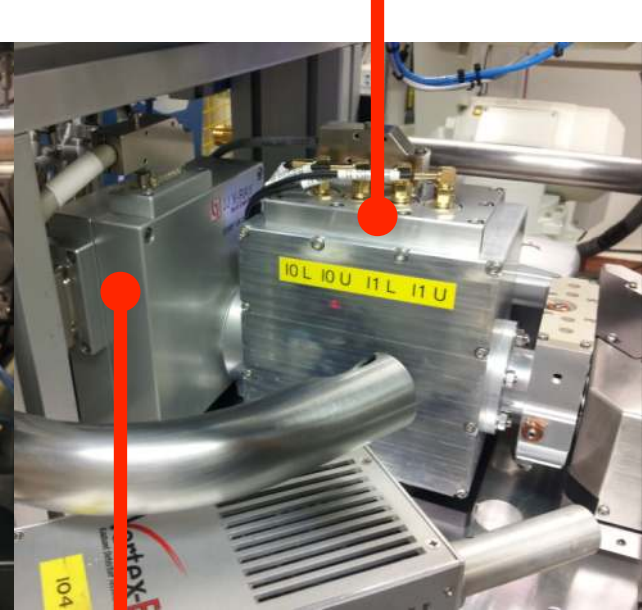
QBPM



16 Foil Attenuators



Diode – Shutter - Diode



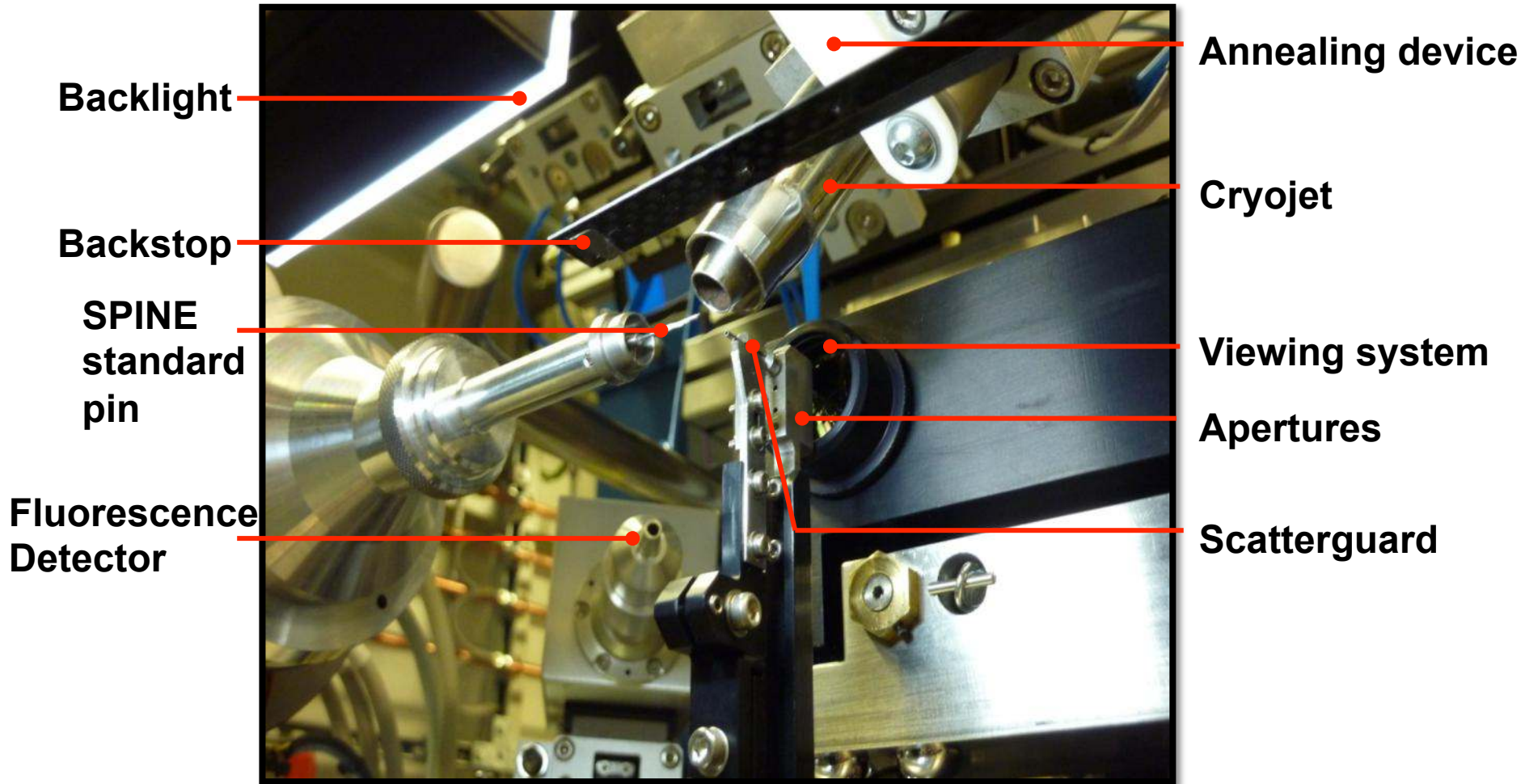
Compound
Refractive
Lenses

Slits

X Rays

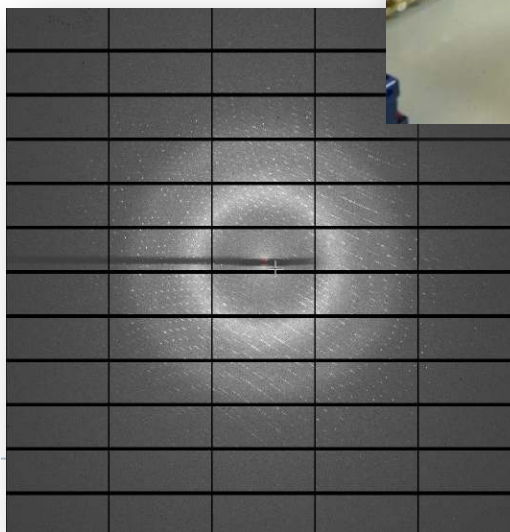


The sample environment



Fast pixel array detectors record the diffraction images

- Advantages
 - S/N much better than CCD
 - Higher throughput
 - Fine phi slicing – better data
 - Beating radiation damage – go even faster with high dose can collect more data at room temp
 - Key for CL3, protein dynamics, etc
- On all beamlines since April 2013
- 100Hz on I03 and I24

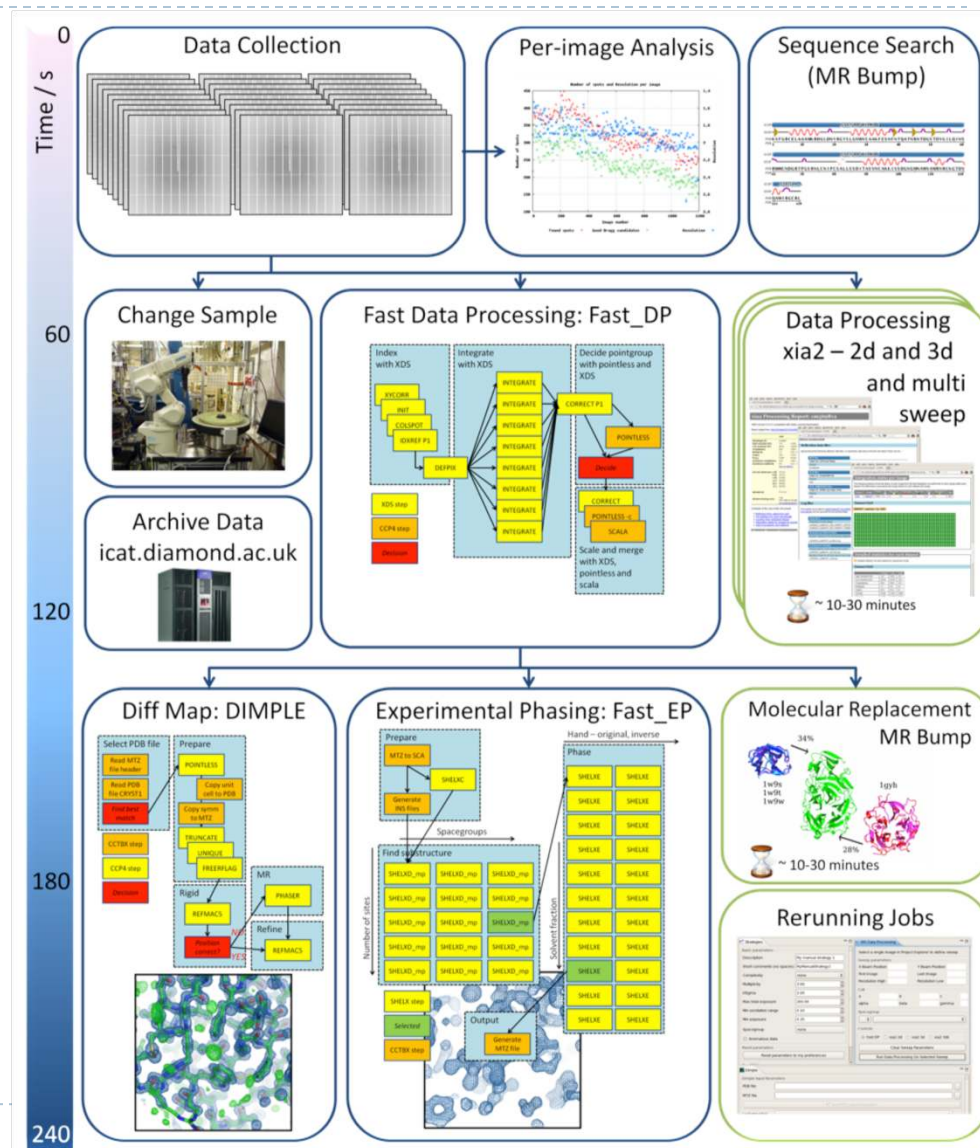


At the end of each data collection, the data are automatically processed

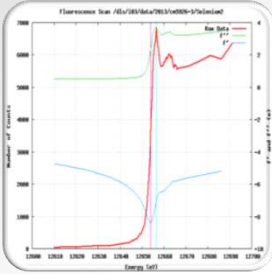
Data collection and experiment monitoring

Automated data reduction

Automated structure solution



Choose which beamline is appropriate to your experiment requirements



Energy/wavelength?

- Fixed energy – suitable?
- Tuneable



Microfocus?

- Use of apertures?
- Dedicated microfocus beamline



Special Apparatus?

- Dehydration equipment
- Spectroscopy
- Biocontainment
- *In-situ* experiments

Use the web pages to compare the MX beamlines at Diamond

I02 MX

High throughput and highly automated beamline for optimised MAD and SAD experiments.

[More information...](#)

Beamsize: 90 x 20

Wavelength: 0.5Å - 2.5Å

Detector: Pilatus 6M-F



I03 MX

High throughput and highly automated beamline for optimised MAD and SAD experiments. Capable of accepting CL3 type experiments on crystals of pathogens.

[More information...](#)

Beamsize: variable

Wavelength: 0.5Å - 2.5Å

Detector: Pilatus3 6M



I24 Microfocus MX

High throughput variable microfocus beamline for optimised MAD and SAD experiments on crystals down a few microns in size.

[More information...](#)

Beamsize: 5x5 - 50x40 (variable aspect ratio)

Wavelength: 0.62Å - 1.77Å

Detector: Pilatus3 6M



I04 MX

High throughput and highly automated beamline for optimised MAD and SAD experiments.

[More information...](#)

Beamsize: 90 x 40

Wavelength: 0.62Å - 2.48Å

Detector: Pilatus 6M-F



I04-1 Fixed wavelength MX

High throughput and highly automated fixed wavelength SAD beamline for macromolecular crystallography.

[More information...](#)

Beamsize: 60 x 50

Wavelength: 0.92Å (fixed)

Detector: Pilatus 2M



Microfocus



Sample Humidity Control



Mini Kappa



In situ Data Collection



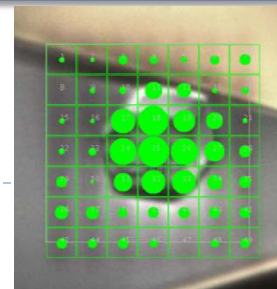
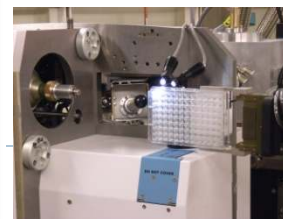
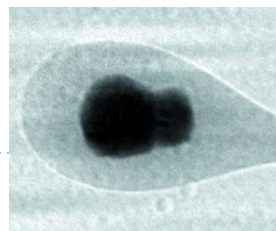
Spectroscopy



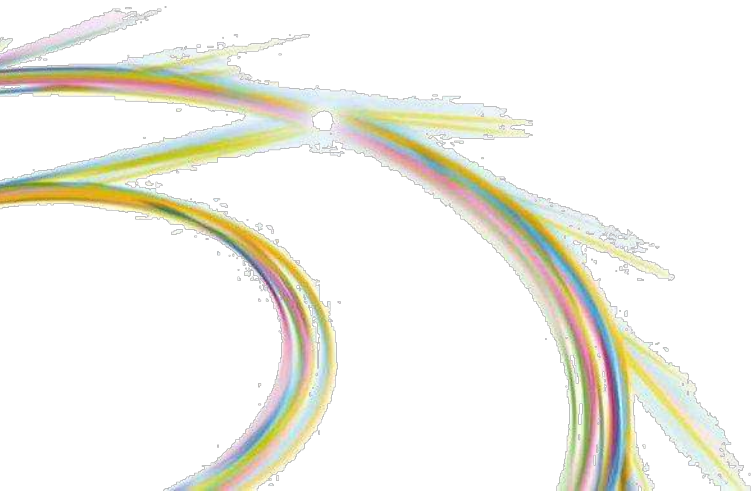
Biocontainment



Beamline	I02	I03	I04	I04-1	I24	I23	VMXi	VMXm
Type	Tuneable			Optimised fixed	Tuneable μ -focus	Tuneable long λ	Tuneable	Tuneable μ -focus
Beamsize [μm]	70 x 20 (mirrors) down to 8 x 3 (CRLs)			40 x 60	5 – 60	120 x 120	5 x 5	4 x 0.5
Flux [ph/s]	$> 10^{12}$			10^{11}	$> 10^{12}$	$> 10^{12}$	$> 10^{12}$ (DCM) $> 10^{14}$ (DMM)	$> 10^{13}$
Pilatus Detector	P6M (25 Hz)	P3-6M (100 Hz)	P6M (25 Hz)	P2M (30 Hz)	P3-6M (100 Hz)	Custom P12M	TBD	TBD
Sample changer	Rigaku ACTOR (< 35 s)			Irelec CATS (< 45 s)		Custom made	TBD	TBD
Containment	CL 1	CL 3	CL 1	CL 1	CL 2	CL 2	CL 2	CL 2
Humidity Controller	On request					No	TBD	No
Multiaxis goniometry			Mini-kappa	Mini-kappa		Custom kappa	No	No
Minibeam apertures	Yes				Development			
In situ	Soon	Yes		Yes	Yes	No	Yes	TBD
Crystal Washer and Annealer	Yes	Yes	Yes	Yes	Soon	No	No	No
Remote Access	Yes					No	Yes	TBD

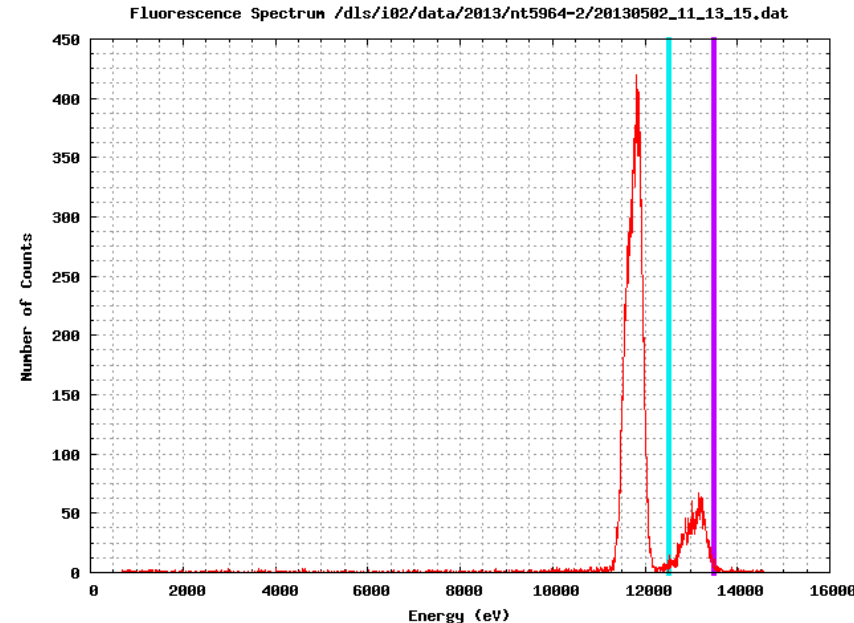


Techniques available on the MX beamlines



Using fluorescence spectra to identify metals in your sample

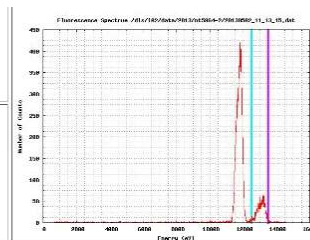
- ▶ All beamlines have fluorescence detectors
- ▶ Take a fluorescence spectrum of your sample in seconds
- ▶ Automatic identification of your sample with summary in webpages:



Thu May 2, 2013 - 11:13:54
[20130502_11_13_15.dat](#)

Fluorescence Spectrum

Beamline Energy: 13488.99eV Omega: 0.0°
Acq Time: 1.0s Trans: 0.05%
Sample Position: (-329,-530,276)



Automated PyMca results

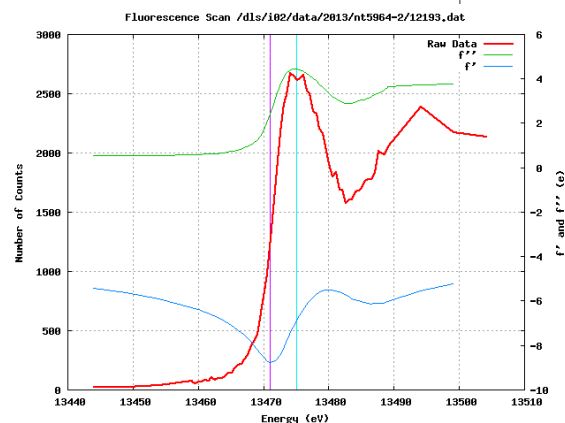
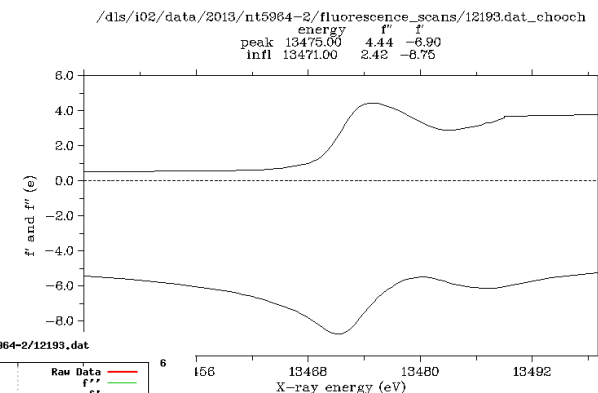
Element	Counts	Percentage
Br-K	16689.7	70.5
Se-K	68.0	0.3
W-L	40.2	0.2
Os-L	39.8	0.2
I-L	19.7	0.1
As-K	17.9	0.1

Counts (total): 23690.0 (background): 861.0

[Guide to AutoPyMCA](#) (pdf)

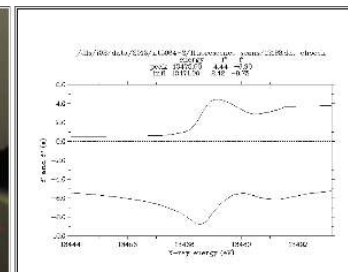
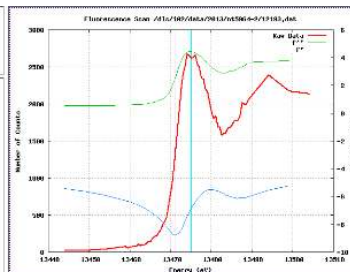
If there is a metal present:

- ▶ On the tuneable beamlines (i.e. not I04-1) collect fluorescence scan around the edge
- ▶ e.g. Signal from Br soaked lysozyme
- ▶ Results stored in webpages/ISPyB:

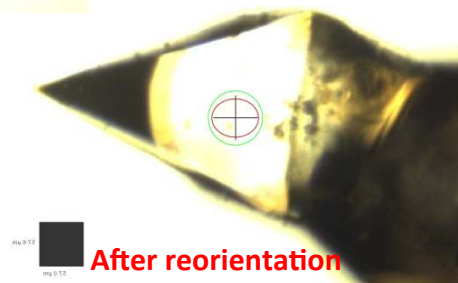
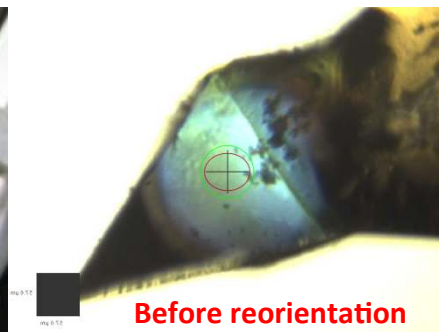
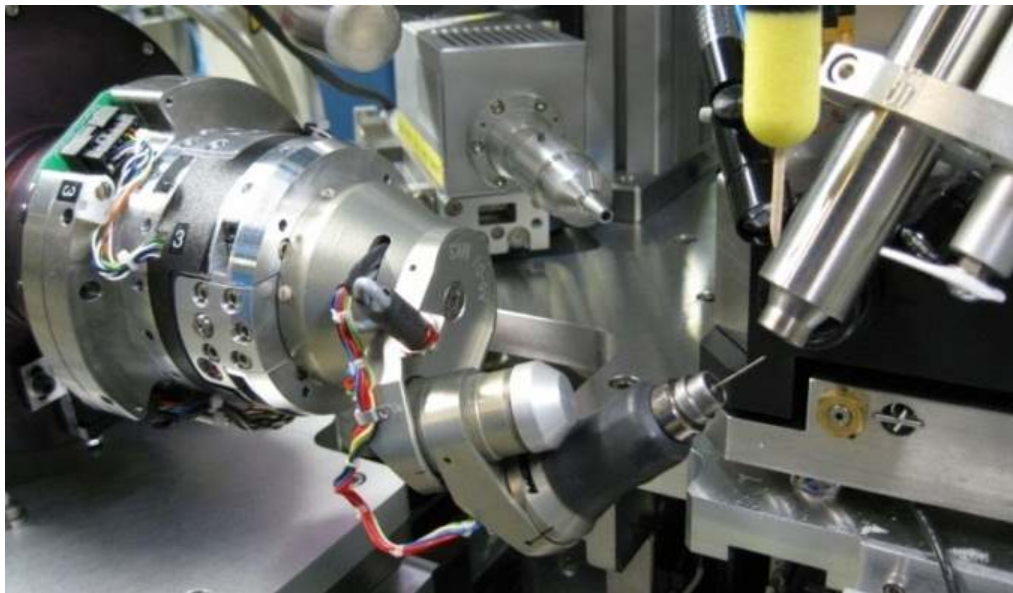


Thu May 2, 2013 - 11:02:29
[12193.dat](#)

Fluorescence Scan			
Element:	Br	Edge:	K
Omega:	0.0°	Transmission:	0.0125%
Sample Position:	(-329,-530,276)		
	Energy	f''	f'
Peak	13475.0eV	4.44	-6.9
Inflection	13471.0eV	2.42	-8.75

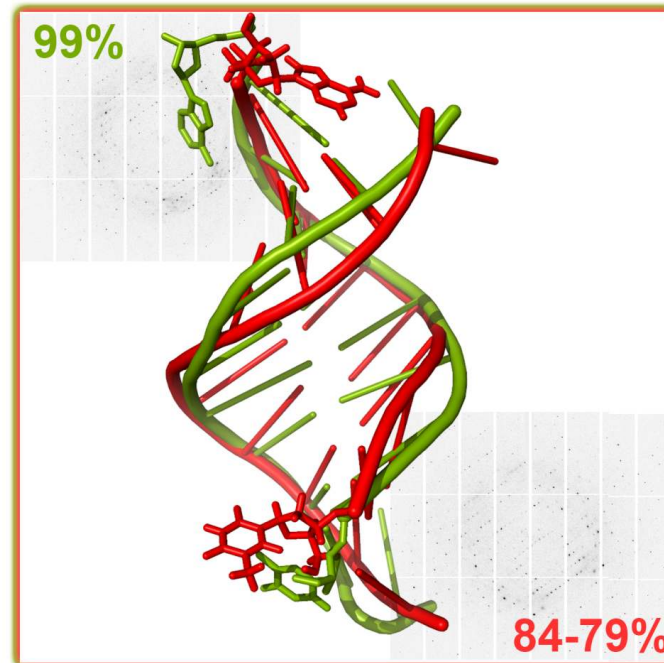
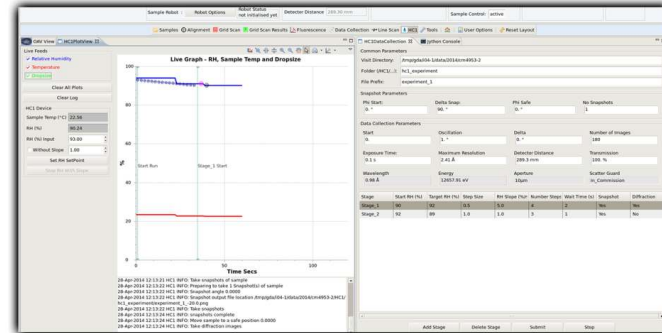
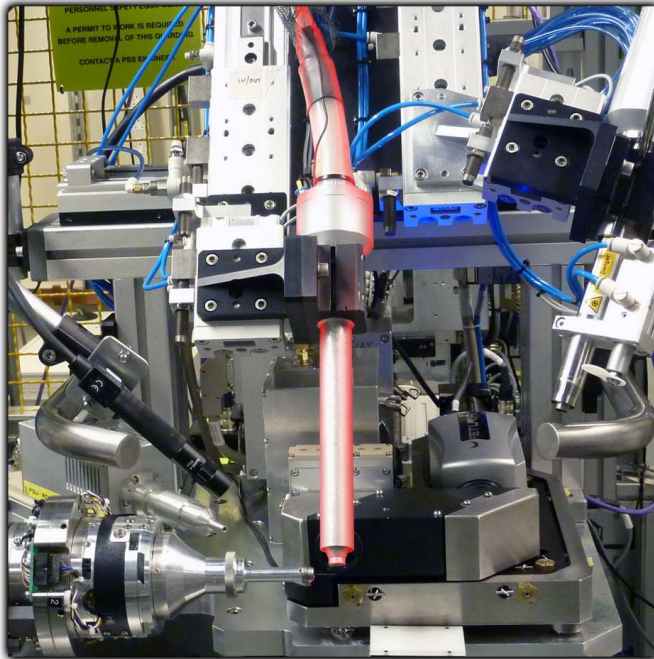


The Mini Kappa can be used to reorientate crystals for data collection

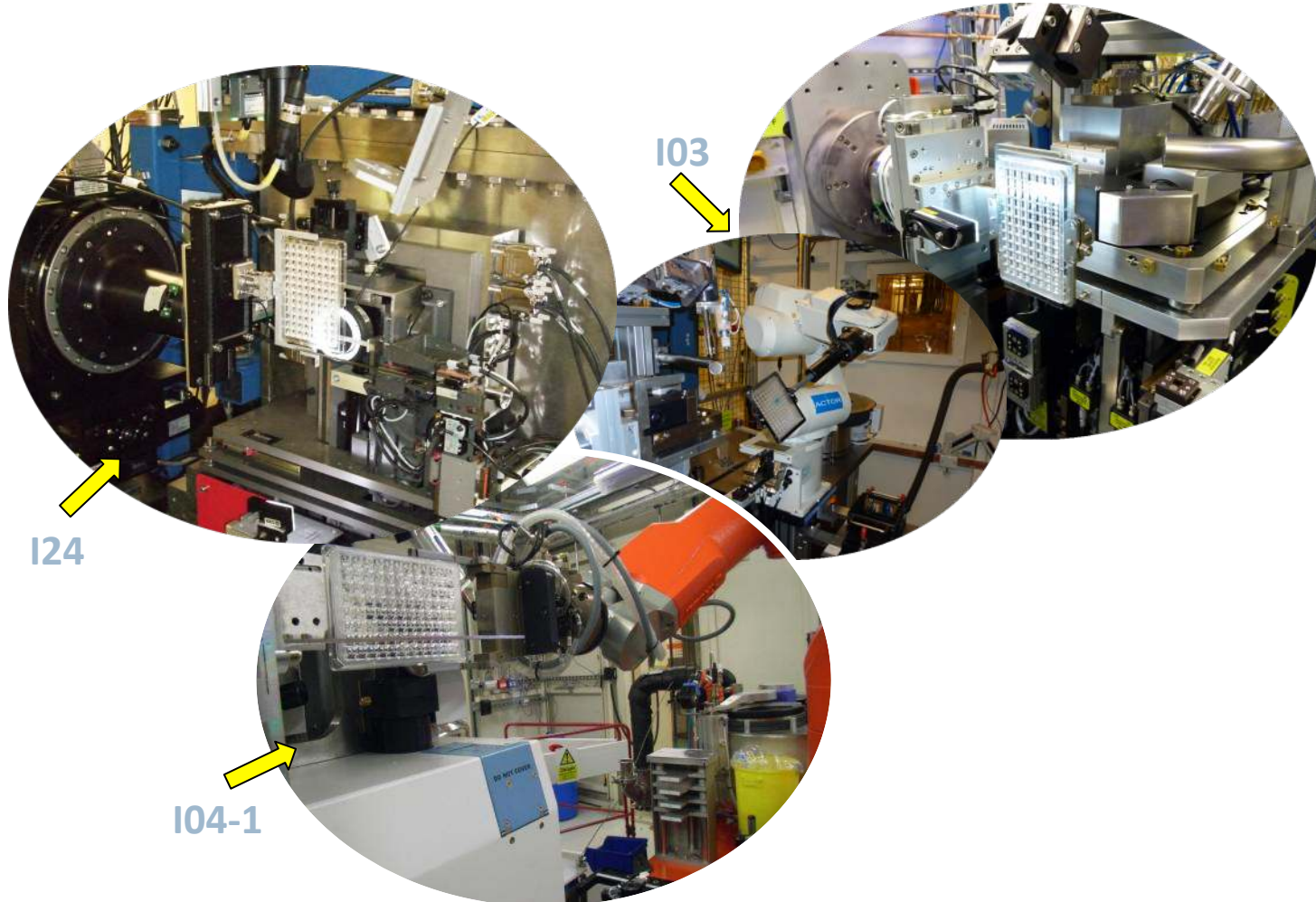


- ▶ Optimised MAD data collection; Bijvoet pairs on the same frame
- ▶ Improved high-multiplicity SAD data collection protocols
- ▶ Smart data collection strategies, better completeness of data, especially P1
- ▶ Reducing/avoiding spot overlap, better spot separation (long unit cell axes)
- ▶ Multi-crystal data collection: collect missing data
- ▶ Help in point group determination
- ▶ Comparing crystals in the same orientation

The HC1 device controls sample humidity and can improve crystal diffraction

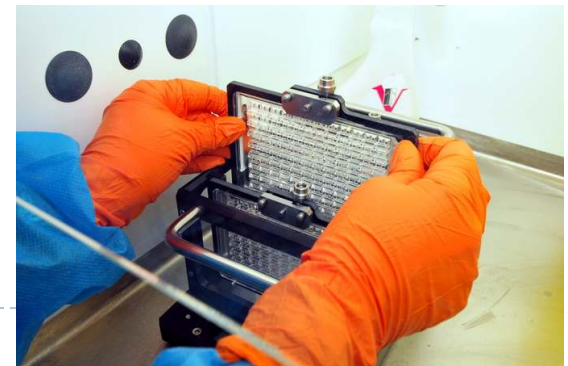
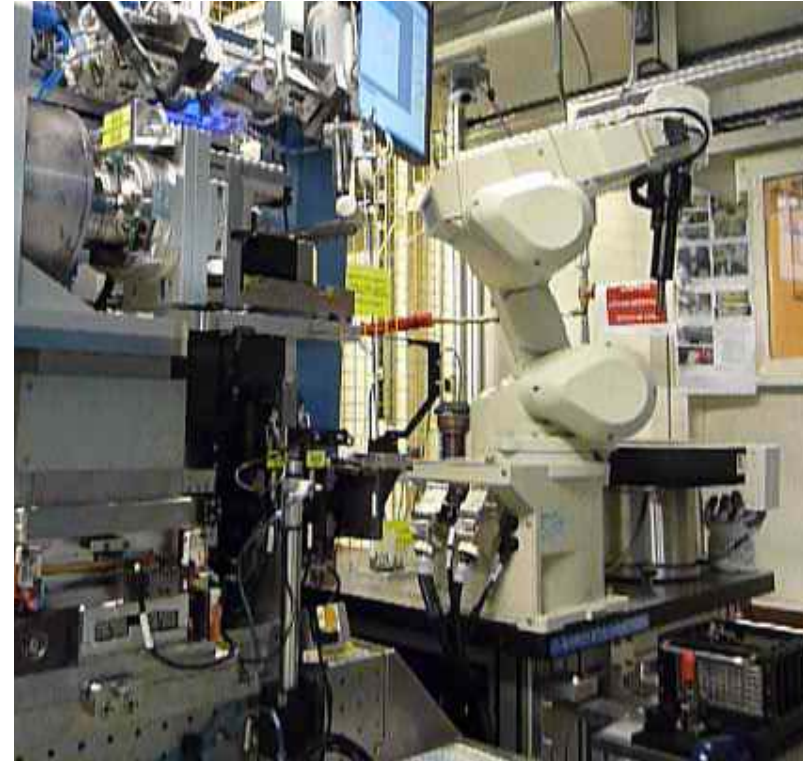


***In-situ* crystallography: data collection from crystals in crystallisation plates**



Pathogenic samples can be studied on I03 and I24

- ▶ All beamlines level 1
- ▶ I03 and I24 up to level 2 biological containment
 - ▶ Cryo and *in-situ* samples
- ▶ I03 containment level 3 compliant
 - ▶ *In-situ* samples only
- ▶ Request via standard beamtime application process



Coming soon to the MX Village (new developments)



Performance: Crystal preparation

500 crystals in 2-day experiment

now

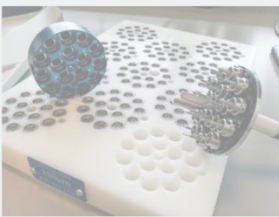
future

500 crystals

Echo + fragment libraries



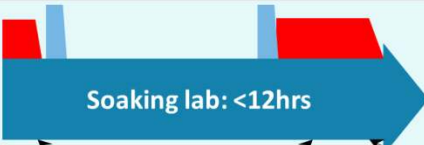
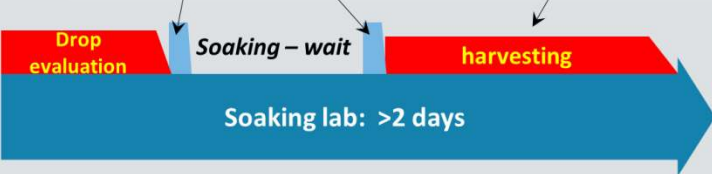
Mitogen loops, trays, wand



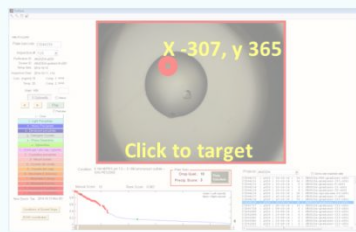
IRELEC robot, loop autocentering



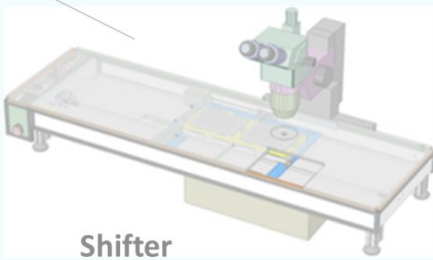
Add fragments
Add cryoprotectant



Robot-assisted harvesting

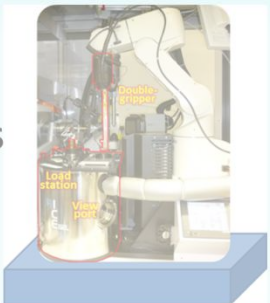


TeXRank



Shifter

HPDS



Recent experiment:

Preparation
200 crystals in 6 hours

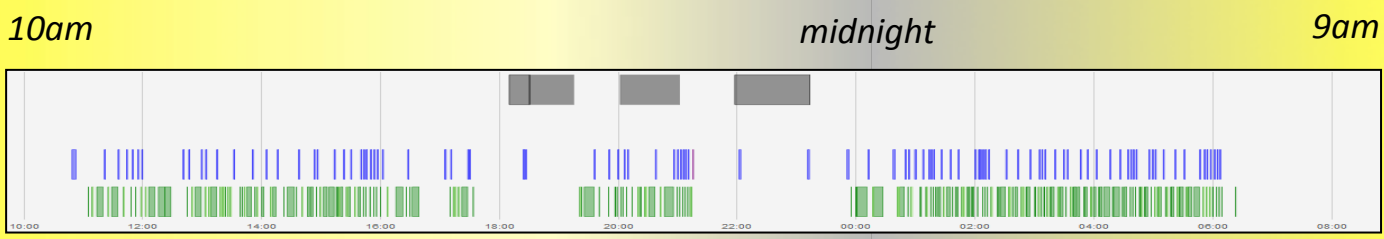
Collection
Overnight

(Full analysis: 2-7 days)

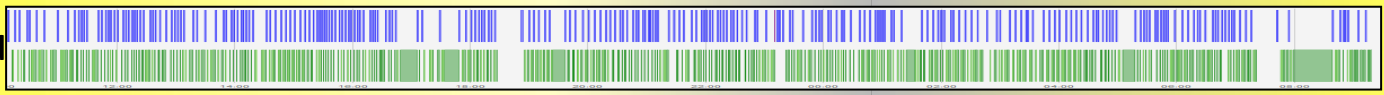
Unattended data collection queues

Sample auto-centring: >97%

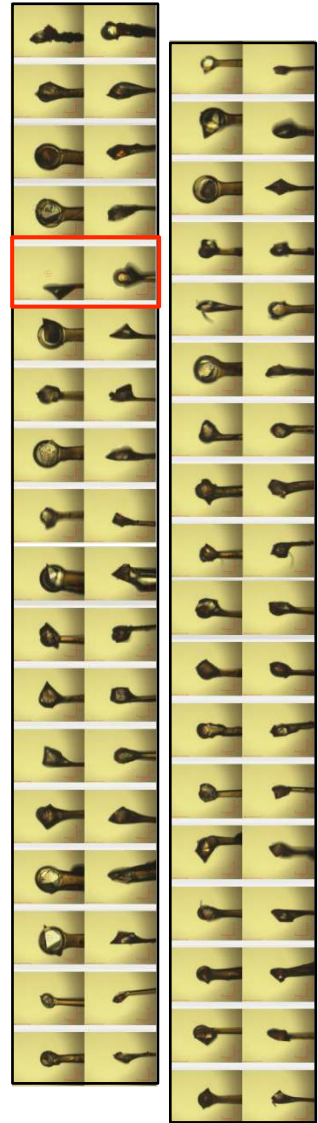
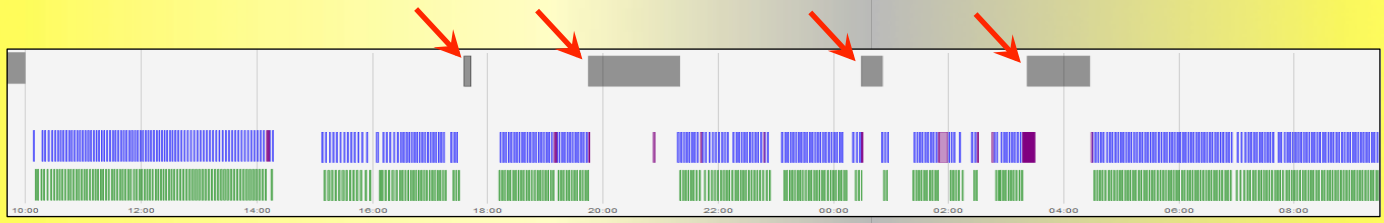
Typical



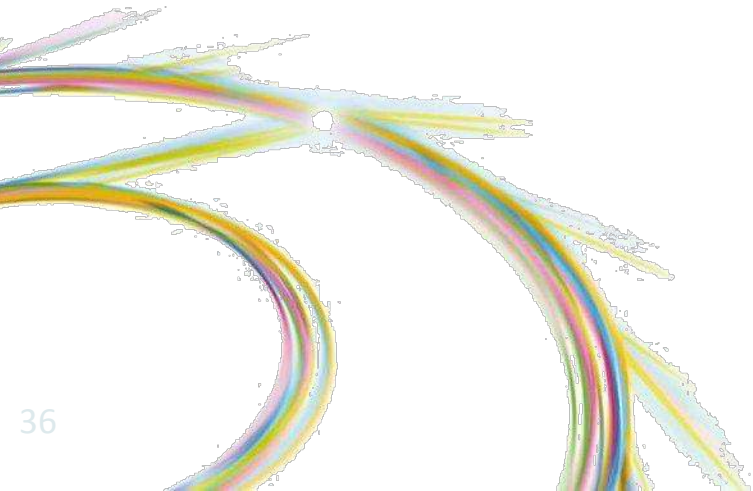
Experienced



Unattended
>350 crystals



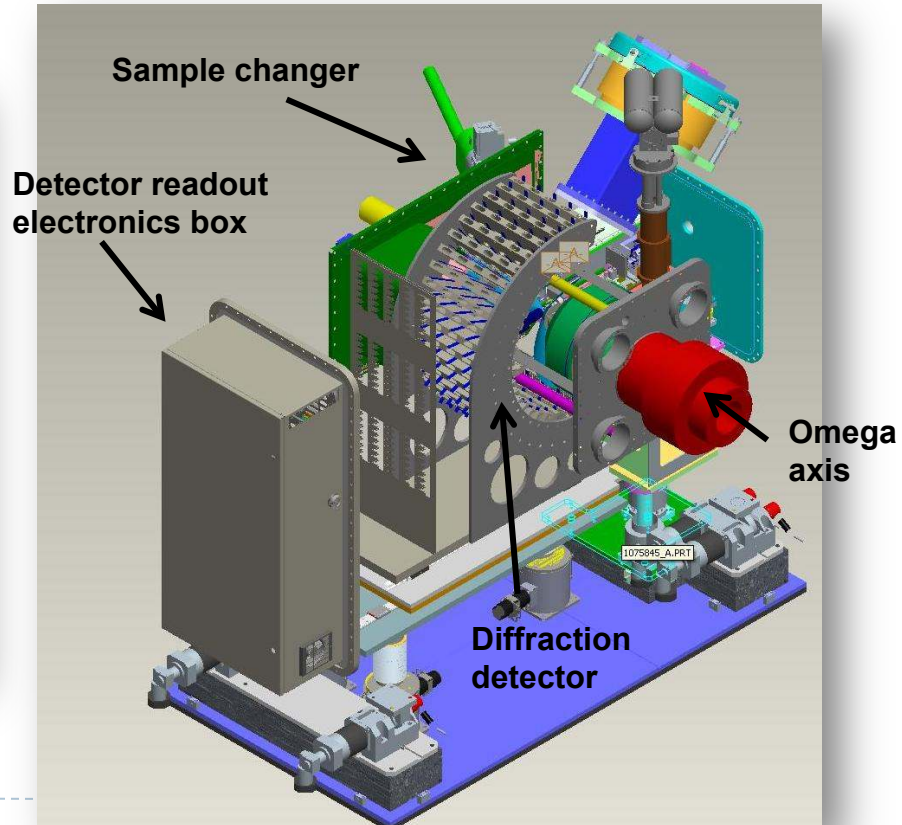
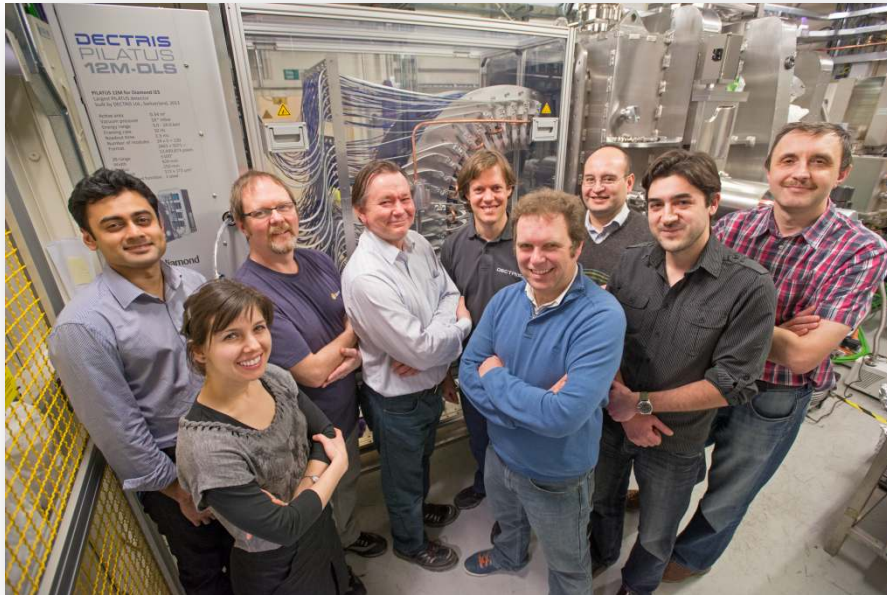
Coming soon to the MX Village (new beamlines)



I23 - Long wavelength Macromolecular Crystallography Beamline

I23 will be the first MX beamline optimized for the long-wavelength region (1.5 – 4 Å).

It will provide a unique tool to fully exploit the potential of experimental phasing from native protein and DNA/RNA crystals.



VMX_{micron} Sub micron variable focus

Aims

- ▶ 0.5 x 0.5 μm – 5 x 5 μm beam
 - ▶ variable aspect ratio
- ▶ rapid beam size change
- ▶ 5 – 30 keV (dependent on ID choice)
- ▶ fast sample exchange/delivery
- ▶ fast crystal location

Challenges

- ▶ sample delivery
- ▶ sample visualization
- ▶ DCM
 - ▶ 13nrad pitch stability
- ▶ optics
 - ▶ facilitating fast beamsizes changes is the real challenge

2014

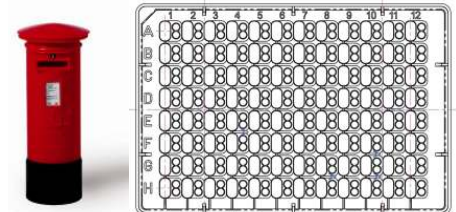
- Technical Design Report
- Prototyping

Team

- Gwyndaf Evans
- Jose Trincão

VMX_i - dedicated to *in-situ* (plate) crystallography

- ▶ This beamline will provide
 - ▶ Plate storage facility
 - ▶ Automated transfer between storage and beamline
 - ▶ Advanced laser imaging of drops (on-line and off-line)
 - ▶ X-ray data collection and screening (automated or interactive)
 - ▶ Tuneable microfocus beam (5x5 micron – 10-25 keV)
 - ▶ Narrow or broad bandpass beam (2eV – 100eV)
 - ▶ Diffraction data available less than 48 hours after shipping plate to Diamond - during run time...



MX Village Support Team

I02

Thomas
Sorensen

Juan Sanchez-
Weatherby

James Sandy

Carina Lobley

Marco
Mazzorana

I03

Katherine
McAuley

Stuart Fisher

Mark Williams

I04

Dave Hall

Ralf Flaig

Pierpaolo
Romano

I04-1

Frank von Delft

Jose Brandao

Alice
Douangamath

Petra Lukacik

I23

Armin Wagner

Vitaliy
Mykhaylyk

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