Australian Government

ANSTO

Beamline Basics

AOFSRR School

August 12th 2024

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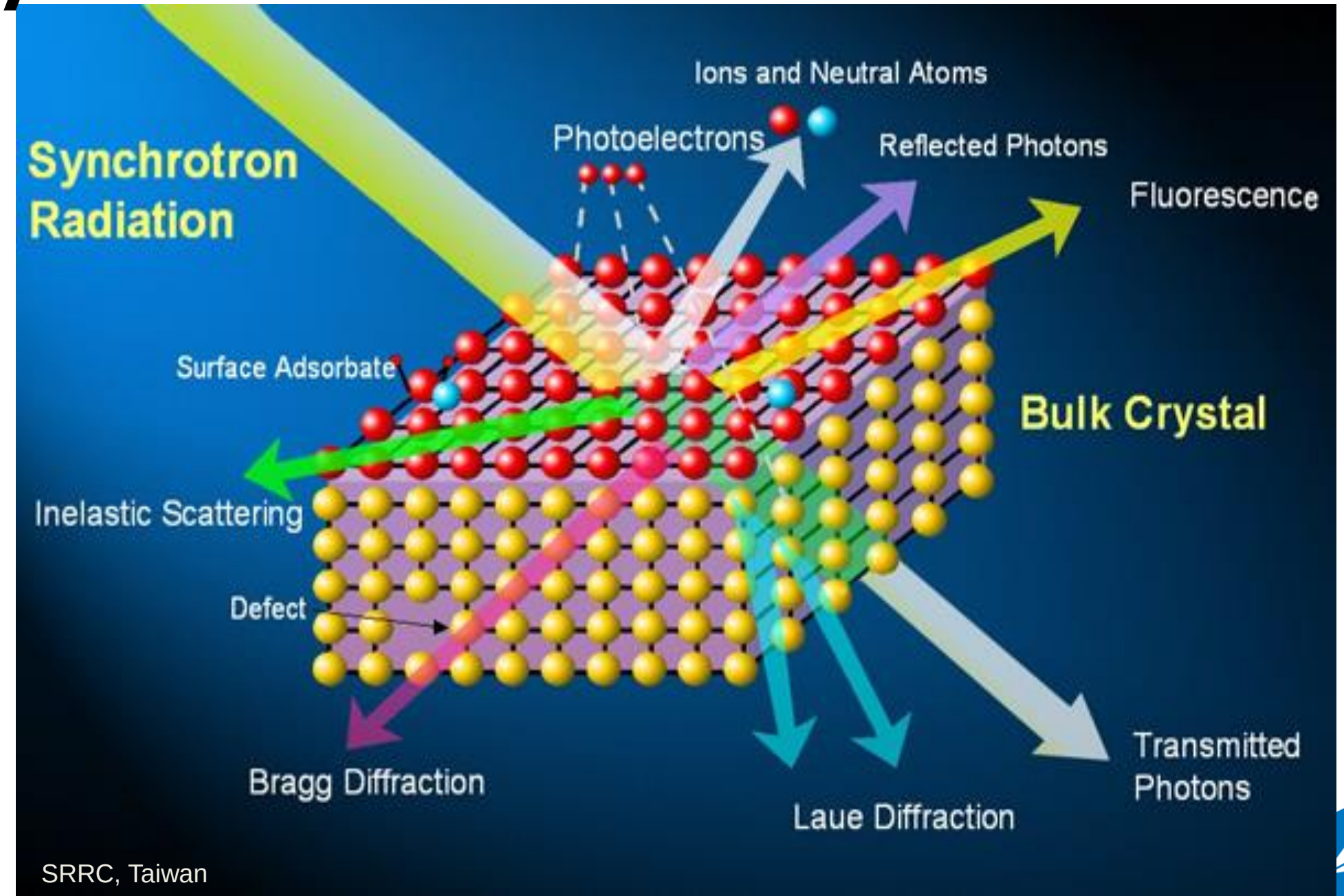
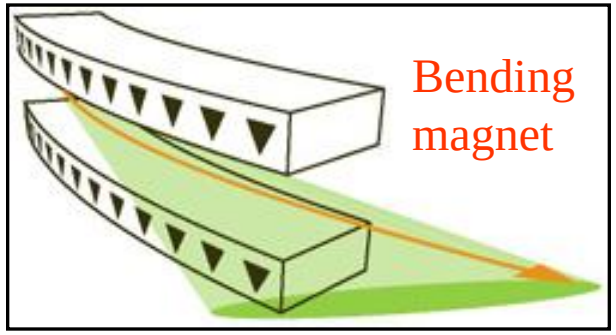
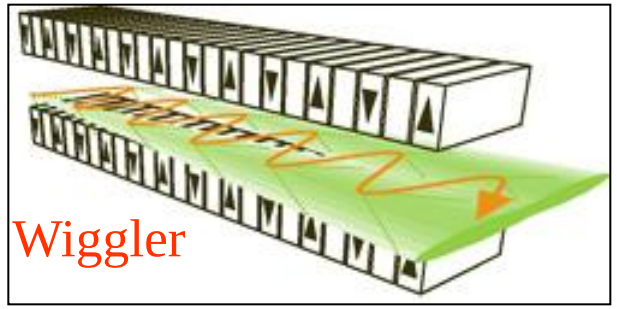
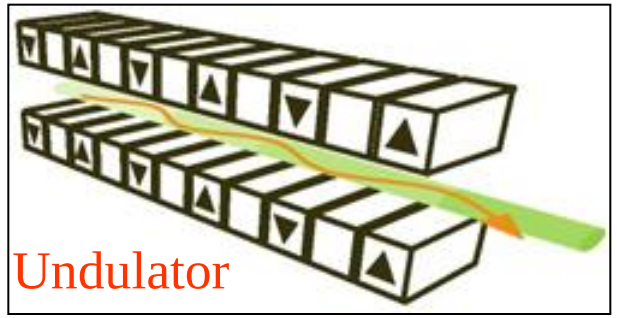
X-ray interactions

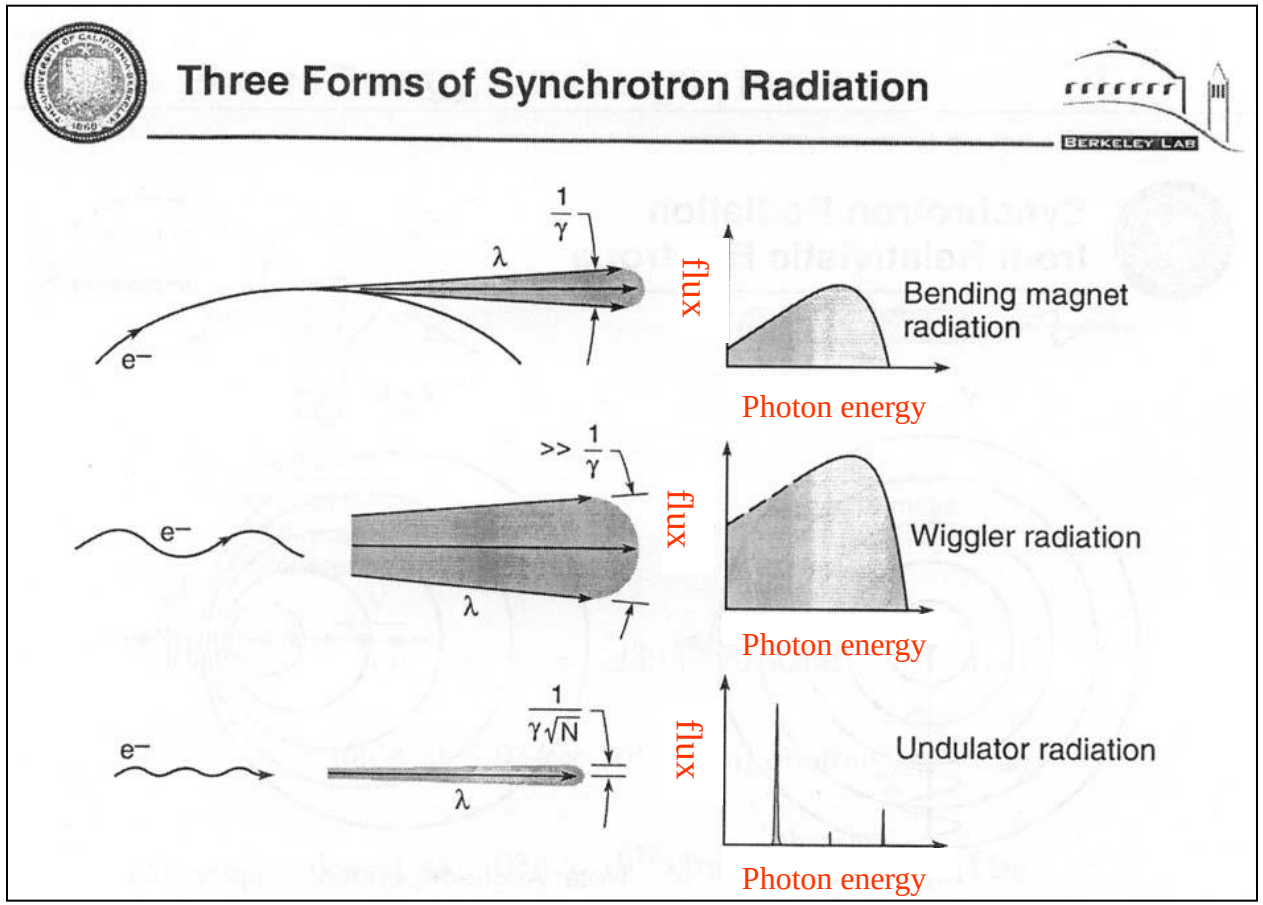


Properties of Sources



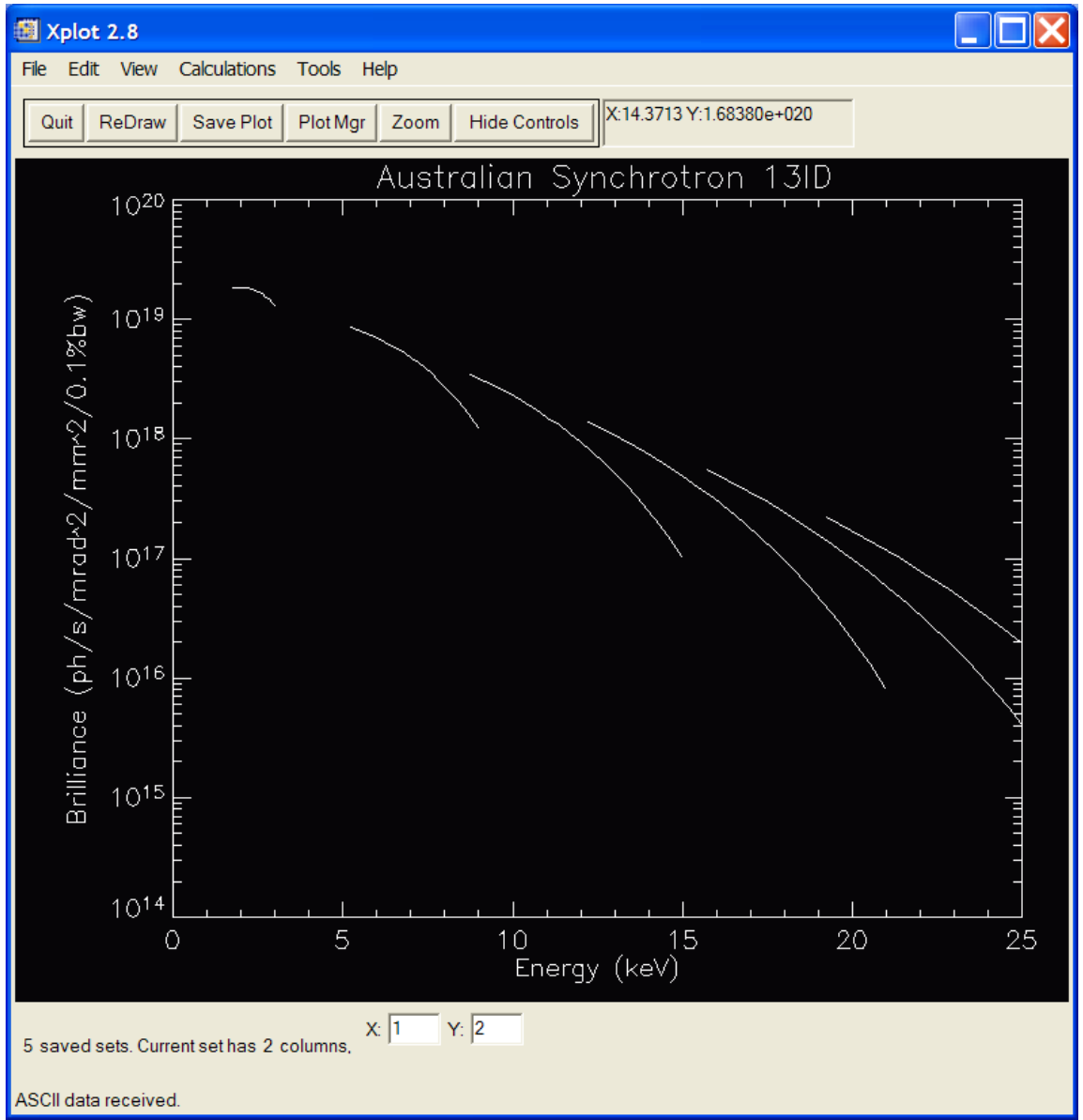






David Attwood: Introduction to Synchrotron Radiation (presentation)

Undulator Tuning C



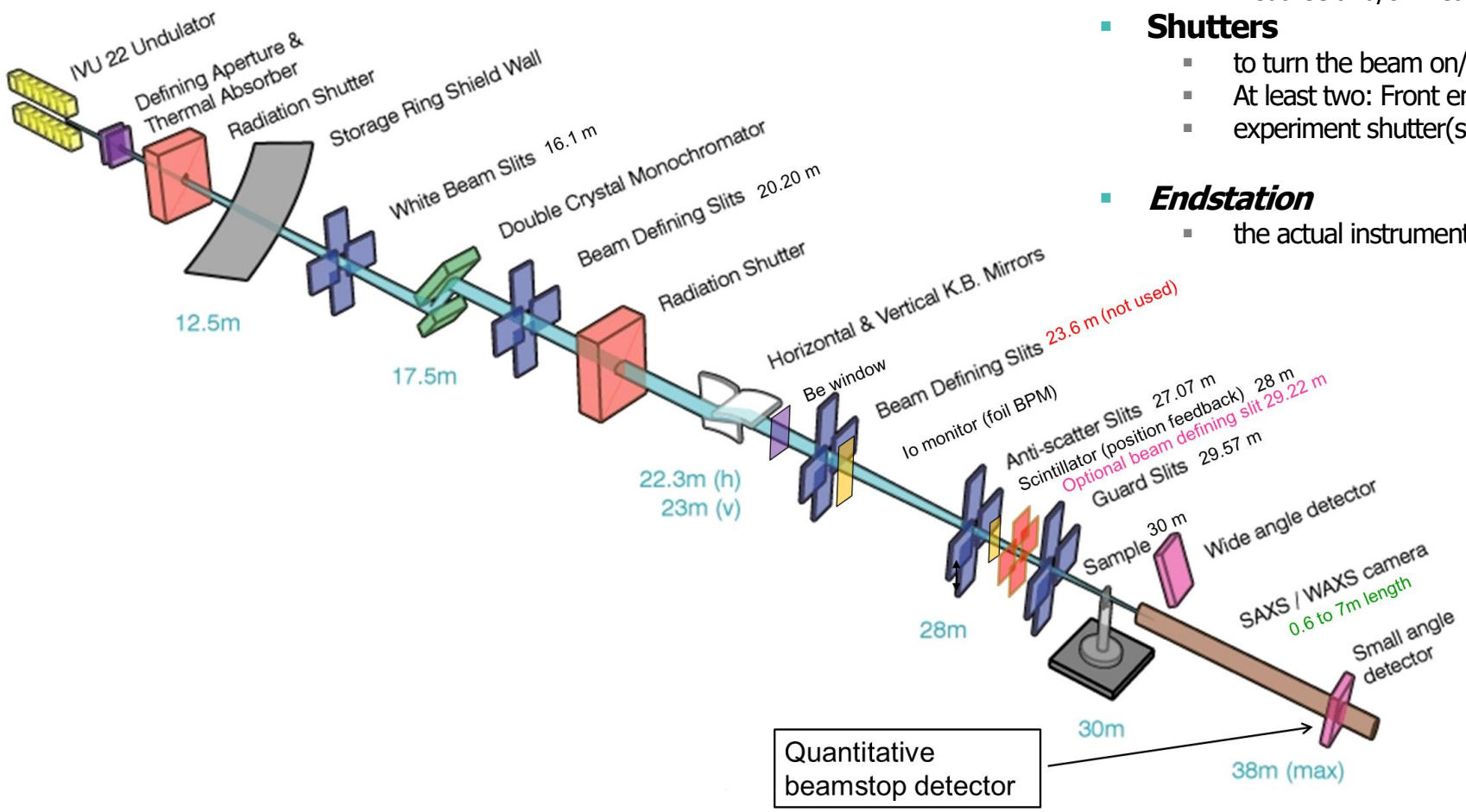
What's good about synchrotron light?

- Source properties
  - High BRIGHTNESS
  - Small SOURCE SIZE
  - Low angular DIVERGENCE (beam emitted over low angle beam)
  - Energy
    - Hard x-ray, soft x-ray, UV, visible and IR wavelengths
    - broadband or narrow band
  - POLARISATION
  - Partial coherence
- Beamline Optics
  - to process beam and supply it to experiment
    - Flux, beamsize, divergence, energy, bandpass, polarisation
    - Stability (flux, beam position, quality)
    - Versatility, speed, reliability
- Endstation
  - Handle samples
  - Take User data (detectors)

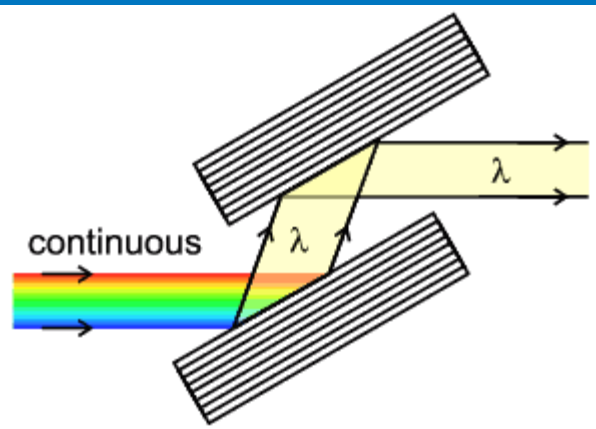
Beamline Components

- **Monochromator**
  - selects the wavelength for the experiment (unless white/pink beam used)
    - Crystal, multilayer or grating
- **Focusing device(s)**
  - To collimate and/or focus beams
  - Reflective (mirrors), refractive(lenses). One or two focusing dimensions
  - May steer the beam
  - May provide harmonic rejection
- **Diagnostics**
  - to help control optics
  - Visualise and/or measure beam intensity, position
- **Shutters**
  - to turn the beam on/of
  - At least two: Front end, mono shutter
  - experiment shutter(s)

- ***Endstation***
  - the actual instrument used to do the experiment and take most of the data



Monochromator Diffraction



Crystal
(most common)

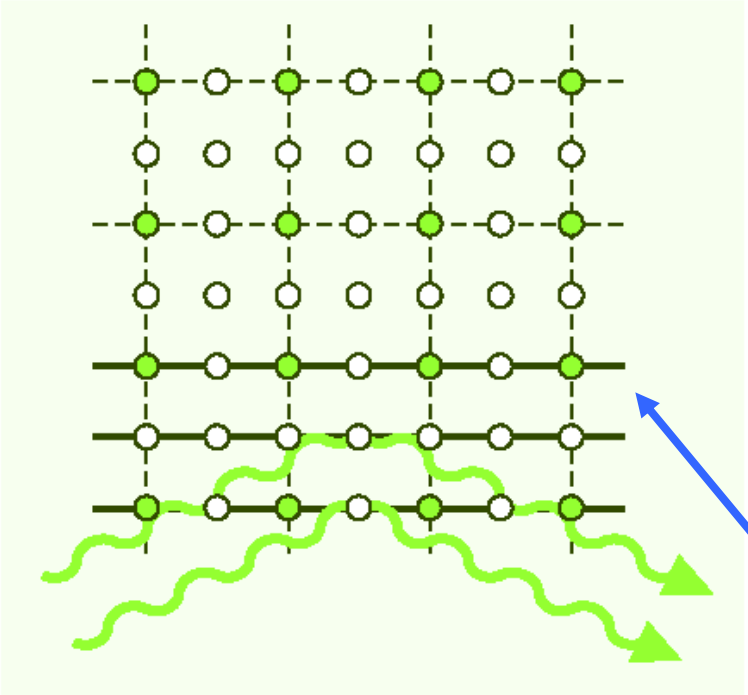
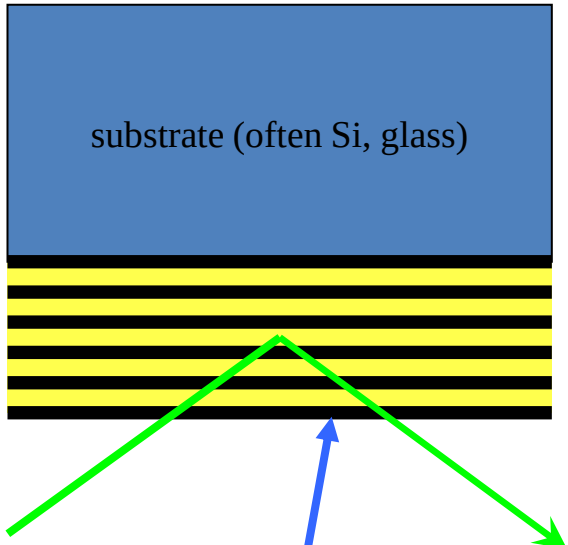


Figure 2.7. The x-ray beam scattered by the electrons associated with the atoms in the crystal structure. Because of the regular array the scattered x-rays reinforce each other to form a diffracted ray, whose angle from the incident ray provides a measure of the distance between the planes of atoms.

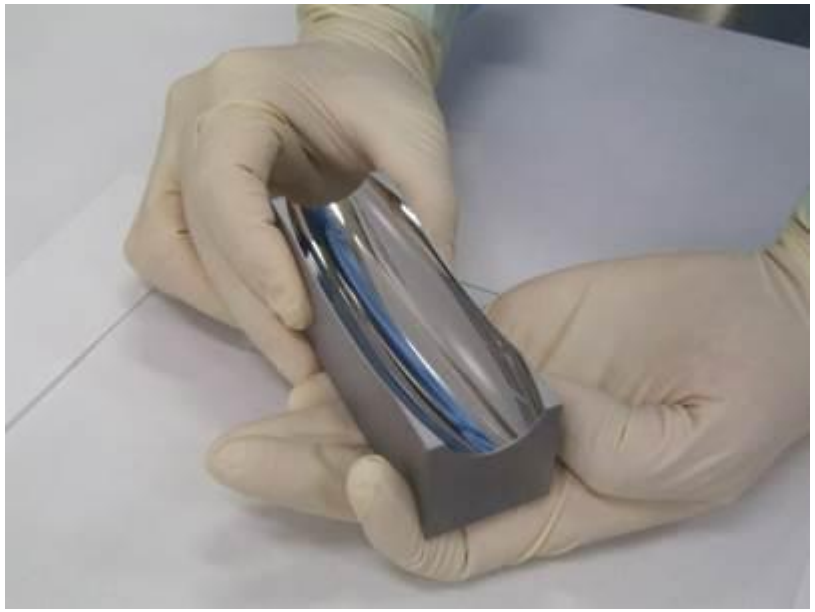
Multilayer

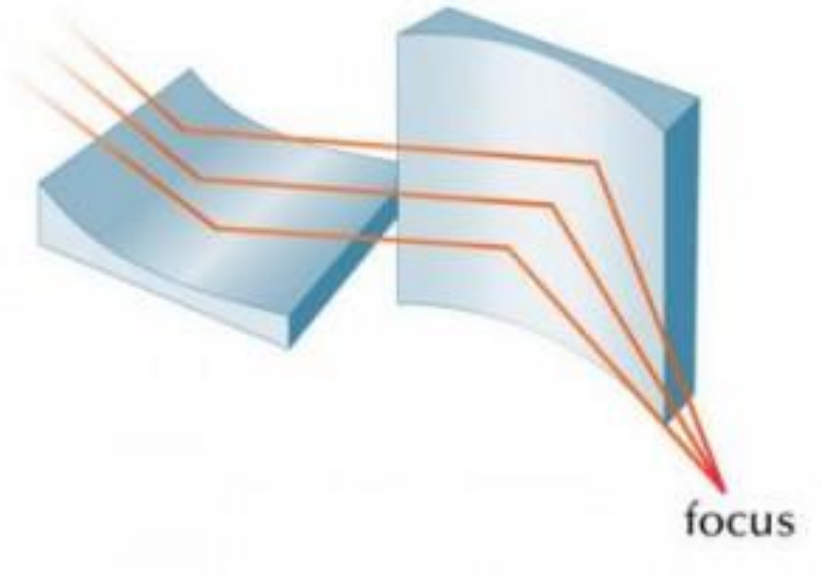


multilayer coating (high/low electron density)

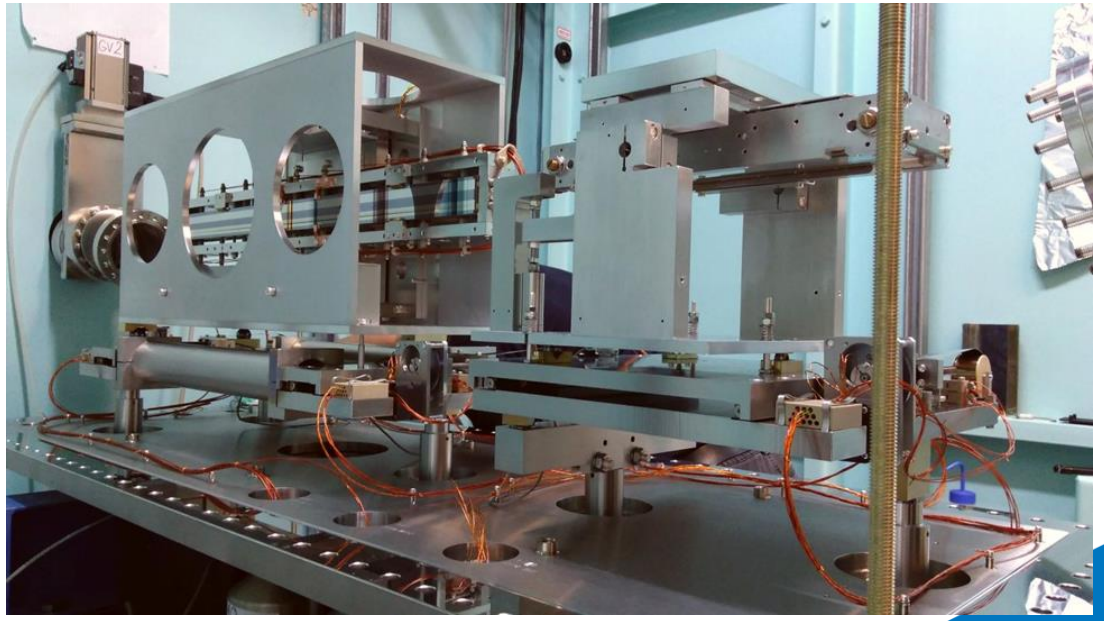
$$n\lambda = 2d \sin\theta$$

Mirrors

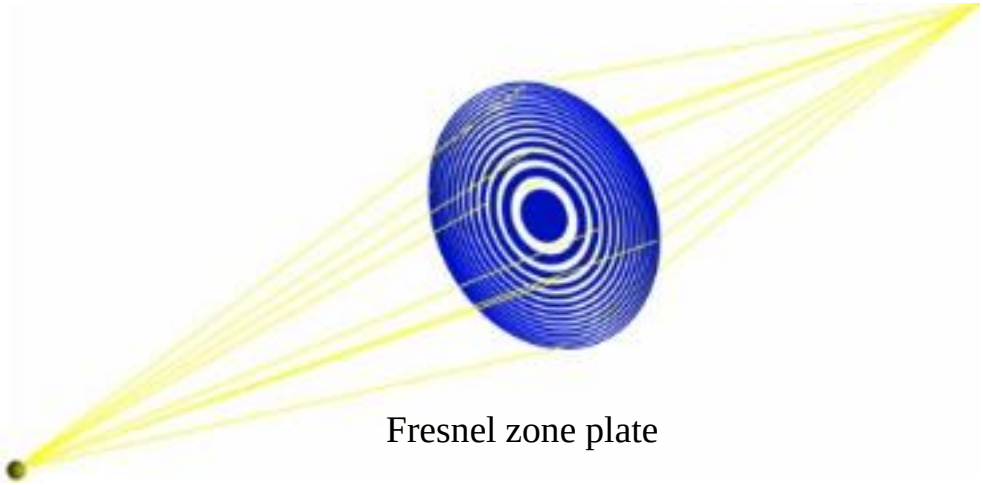




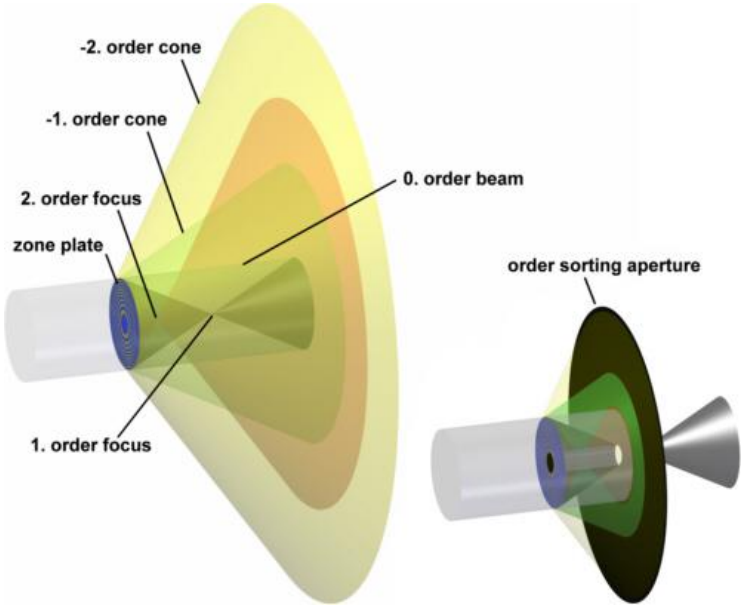
Kirkpatrick-Baez (KB) mirrors

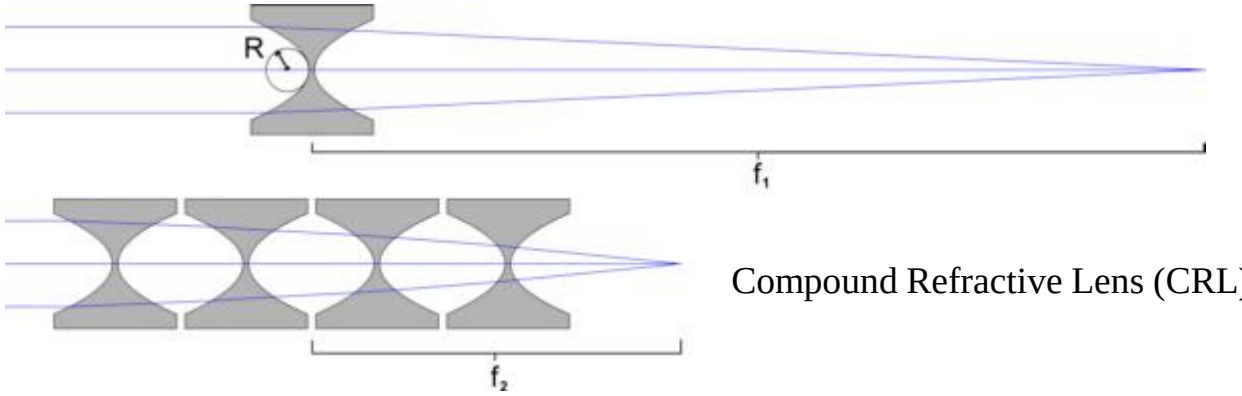


Other focusing elements



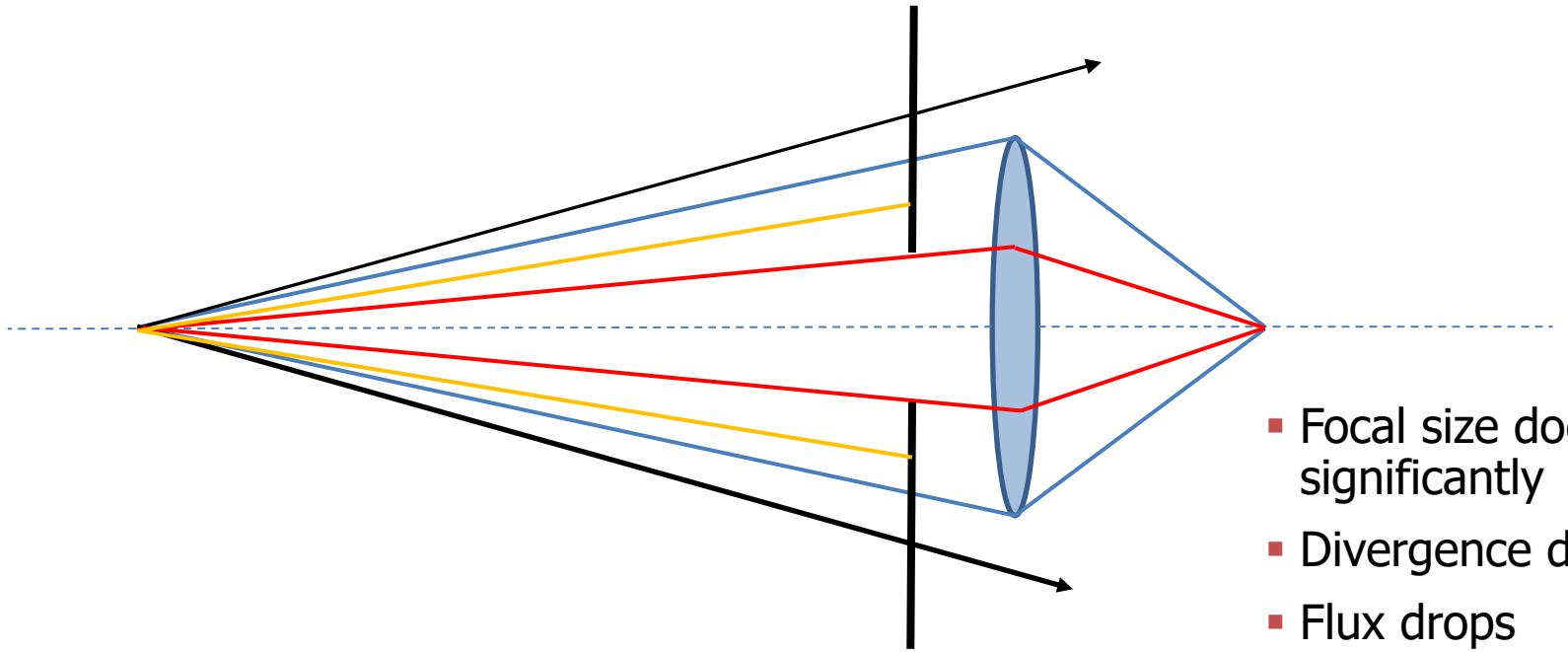
Fresnel zone plate





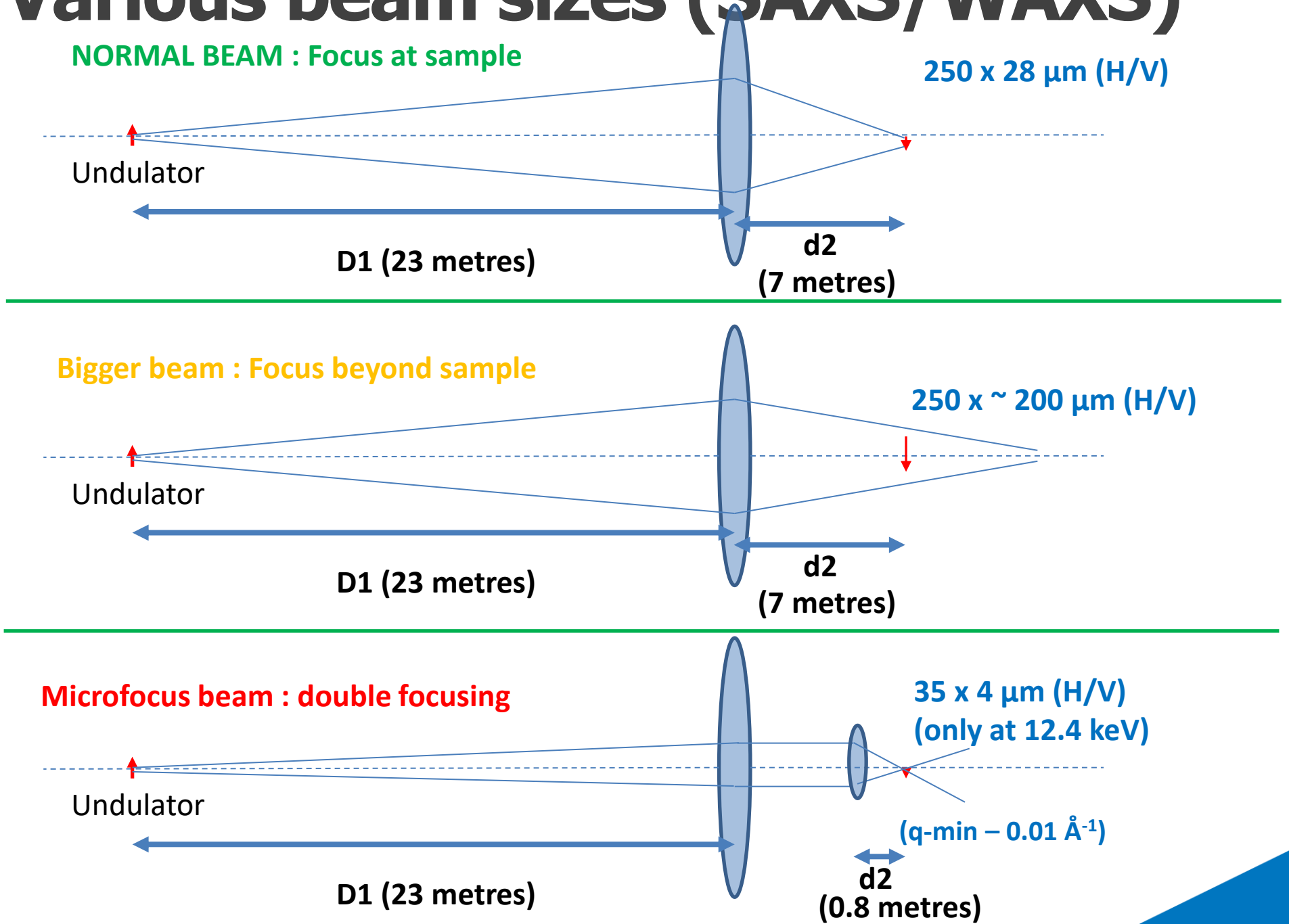
Compound Refractive Lens (CRL)

Acceptance / Divergence



- Focal size does not change significantly
- Divergence drops
- Flux drops
  - User adjustable

Various beam sizes (SAXS/WAXS)



Why are low energy beamlines different from high energy beamlines?

On one level they are no different - a beamline is just a mechanism for transferring light from the source to the sample and then often into a detector.

We cannot make any more light than the source outputs but we can try to change some characteristics of the light.

What characteristics? Monochromation, beam size at the sample or beam size at the detector! Flux density

For Low Energies we use diffraction gratings as our means of monchromating the photon energies in the beam. For high >2KeV energies we use X-ray diffraction from crystals.

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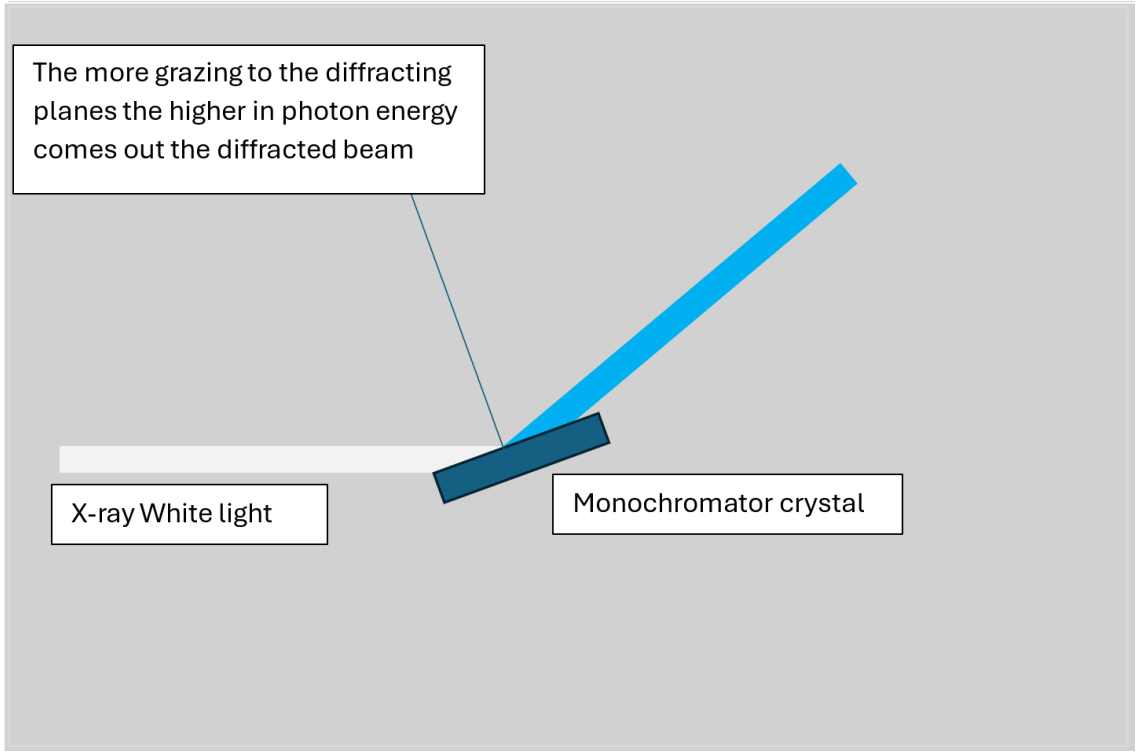
Just to orientate you and to reinforce the previous speakers remarks

The vast majority of X-ray crystals in monochromators act as if they were mirrors that only "reflected" a very small range of X-ray energies



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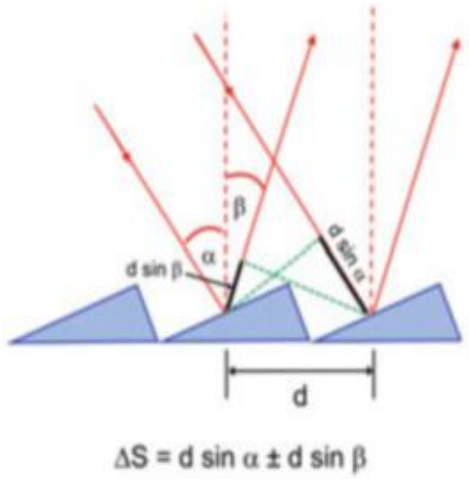
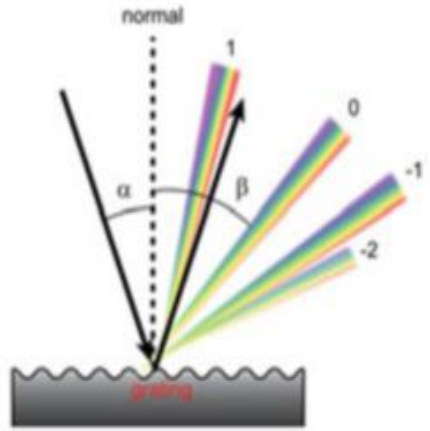
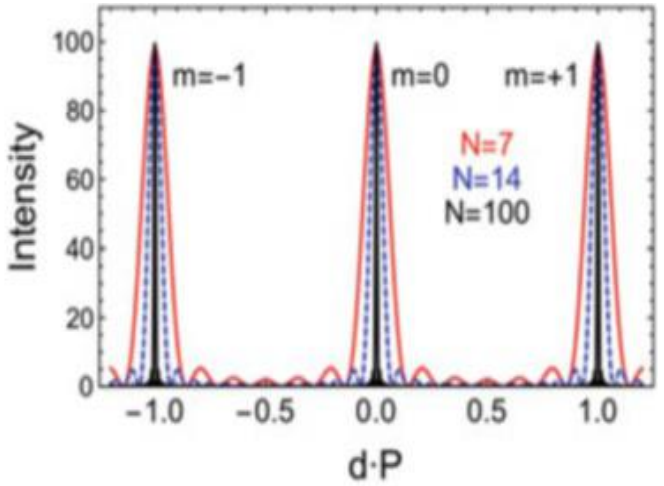


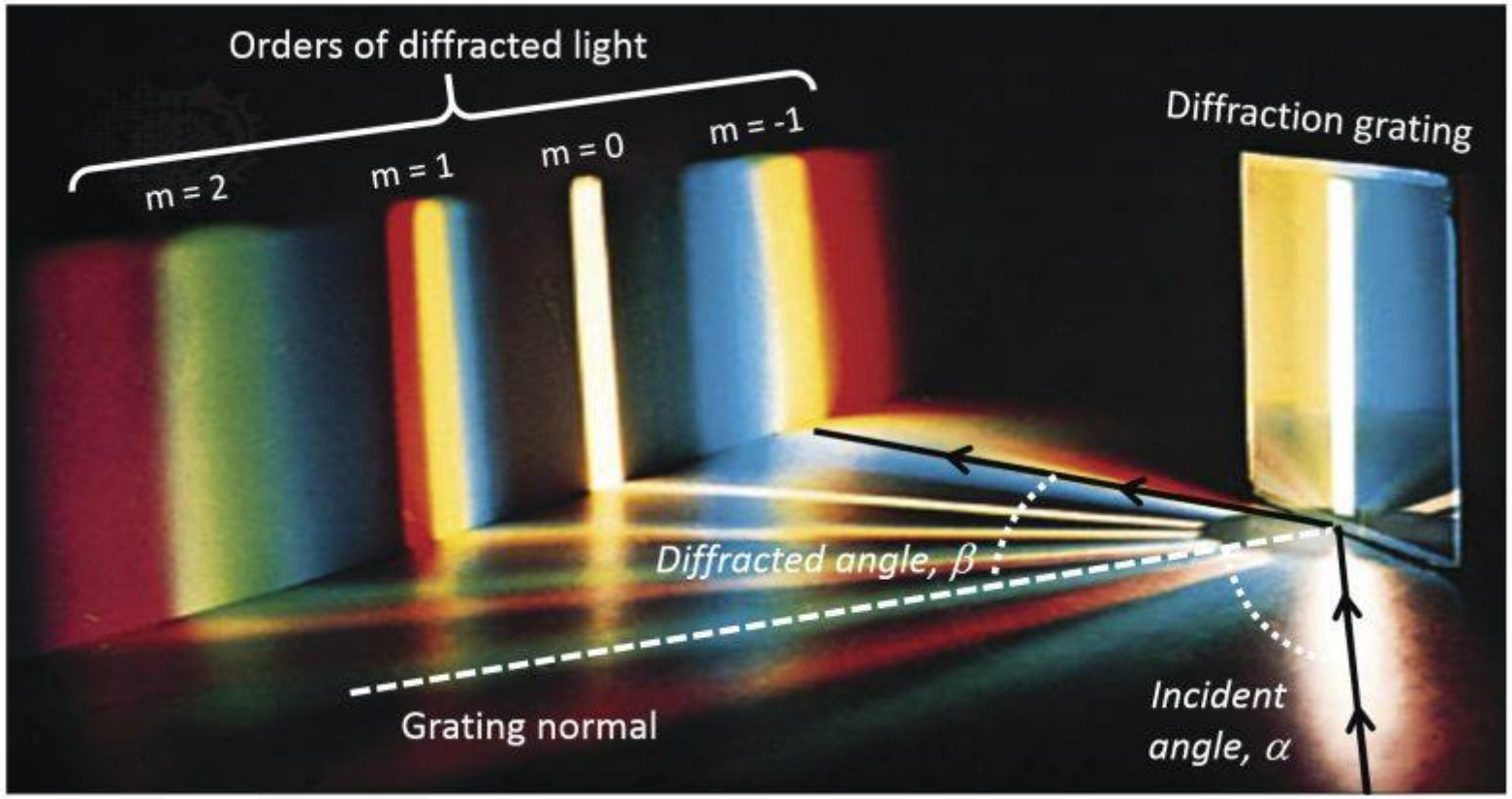




At its simplest we use a stepped structure the light "bounces" off the flat part and if light from the adjacent terraces are integer multiples of the wavelength, then in that direction the light interferes constructively, and we have a diffraction maximum.

As there are multiple ways that can happen we get light split into different orders of diffraction. The M0 is just reflection

Using Visible light



Here is a visible light diffraction grating where you can see how the range of wavelengths are split into different "orders"

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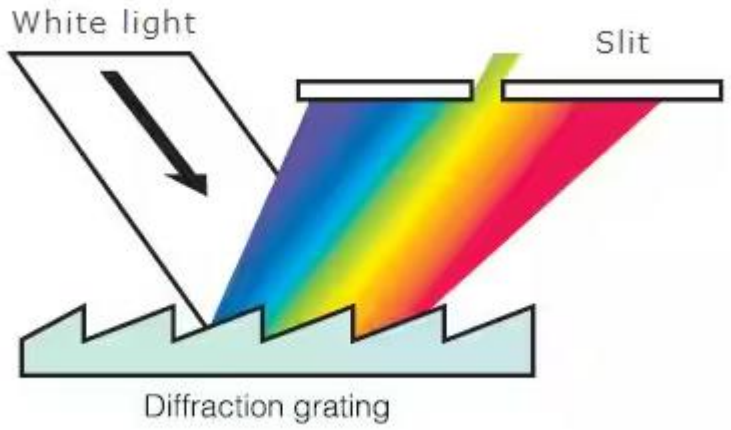

It's easy you just rotate the grating the wavelength you want to select then passes out of a fixed slit.

At its heart that's what all grating beamlines do, but not with visible light (500 nm 2.5 eV) but with X-rays 100 nm to 0.6 nm (12 eV to 2000 eV)

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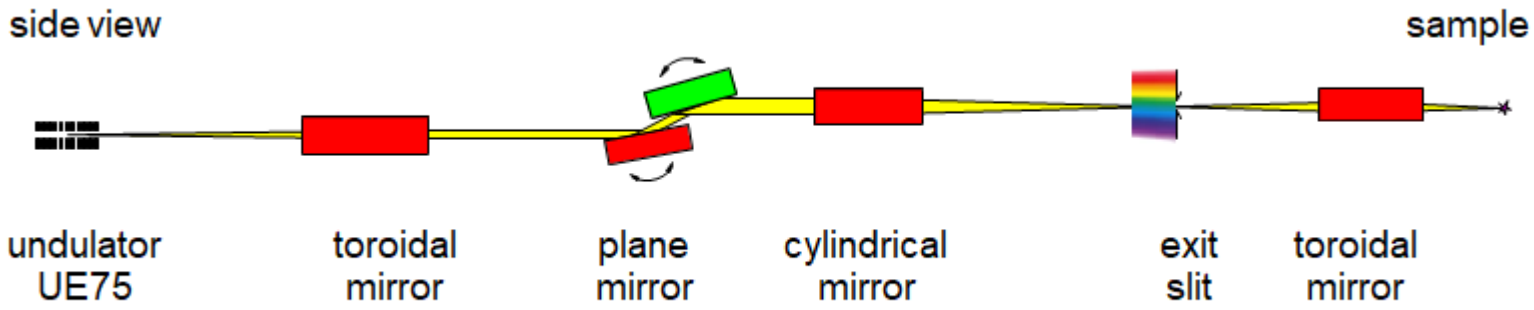
To effectively use the rainbow of X-rays

You have to make the sharpest "rainbow" at the slits you use to select the photon energies that you want to monochromate



The image of the source is transferred to the exit slit where only a small part of the Dispersed "rainbow" then passes through a 20-micron wide slit

DO NOT call it a rainbow the "professionals" call it a Dispersion

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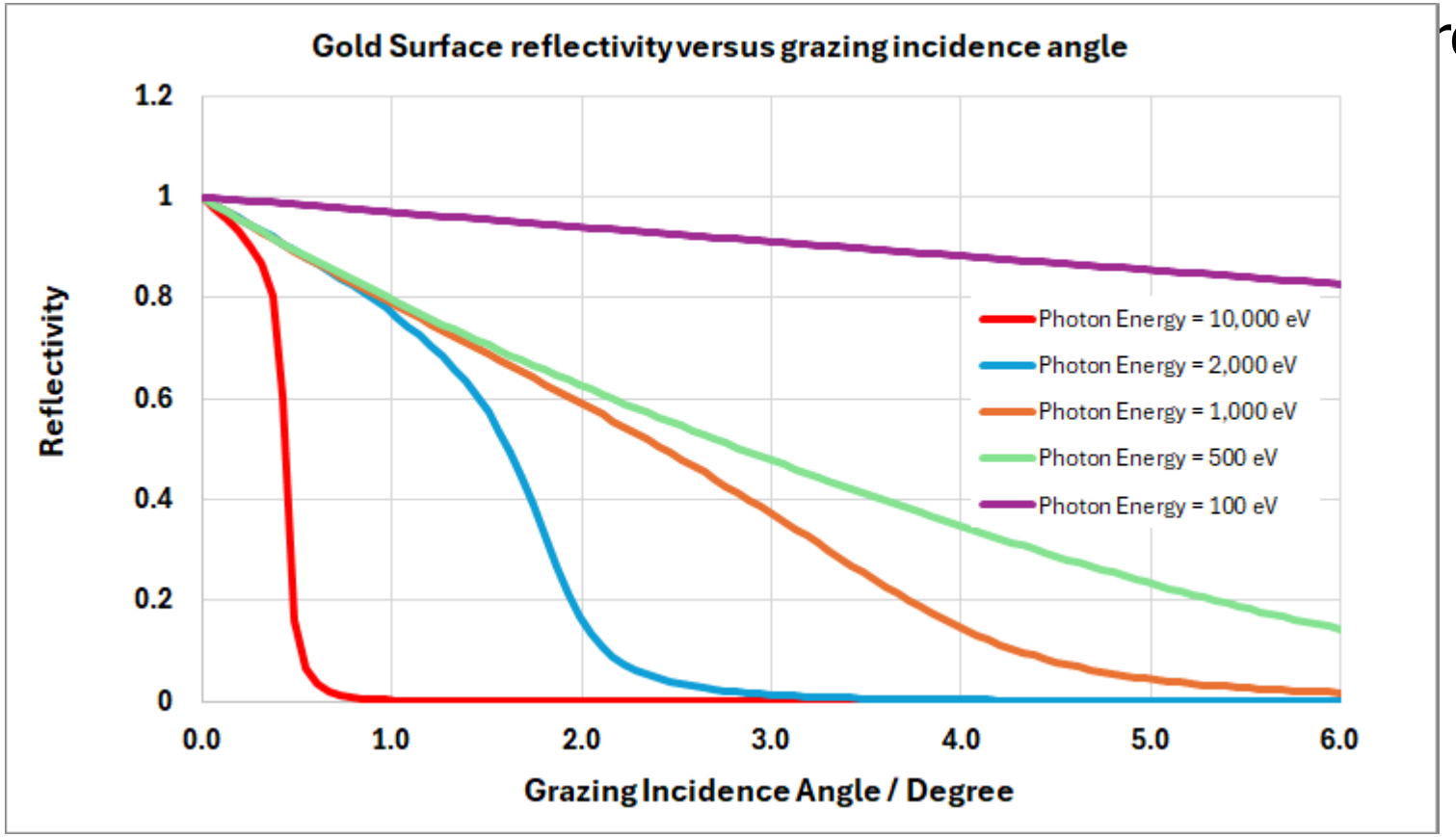
Grating Advantages

You can play some games with the parameters of the grating

- More lines per mm more spreading of the rainbow (Dispersion) higher photon resolution less flux
- Less lines per mm more flux into the exit slit but less resolution
- Major Disadvantage Efficiency of monochromation is a maximum of 25%. Typically for higher energies 1 to 3% is considered good
- The first mirror removes a lot of high energy X-rays beyond 2 KeV so typically the gratings can be cooled with water

Some Slight Issues with using X-rays

Visible light - each of the grating surfaces reflects the light very well, over 98%. There is plenty of



For X-rays they have a tendency to pass into the surface and not reflect off it.

WHAT DO OUR GRATINGS PHYSICALLY LOOK LIKE

The grating is made from a single piece silicon very highly polished and then in this case they mechanically etched lines 1200 lines /mm onto the surface then coated it with 30 nm of gold. The grating is 150 mm long hence there are a total 180,000 lines on this surface! 50K Aus$ (2006)

Our gratings have to work between 1.3 to 10 degrees grazing but the number of lines per mm is 1200 l/mm this will work reasonably well for visible light

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Good News In comparison to high photon energy beamlines We can use mirrors with higher incidence angles

For beamlines using Mirrors to optically focus the beam typical angles are 0.3 to 0.5 degrees Mirror lengths can easily be 1 to 2 metres long

For low energy beamlines typical incident angles for mirrors are more like 1 to 2 degrees compared to the 0.3 to 0.5 degrees for these mirrors are a lot smaller. Easier to make, easier to support, vacuum vessels to hold them are smaller

Larger angles also allow subsequent X-ray beams to be deflected horizontally. You can share all the expensive beamline components more easily with different final end stations. For high energy beamlines they have to pass through one end chamber to another.

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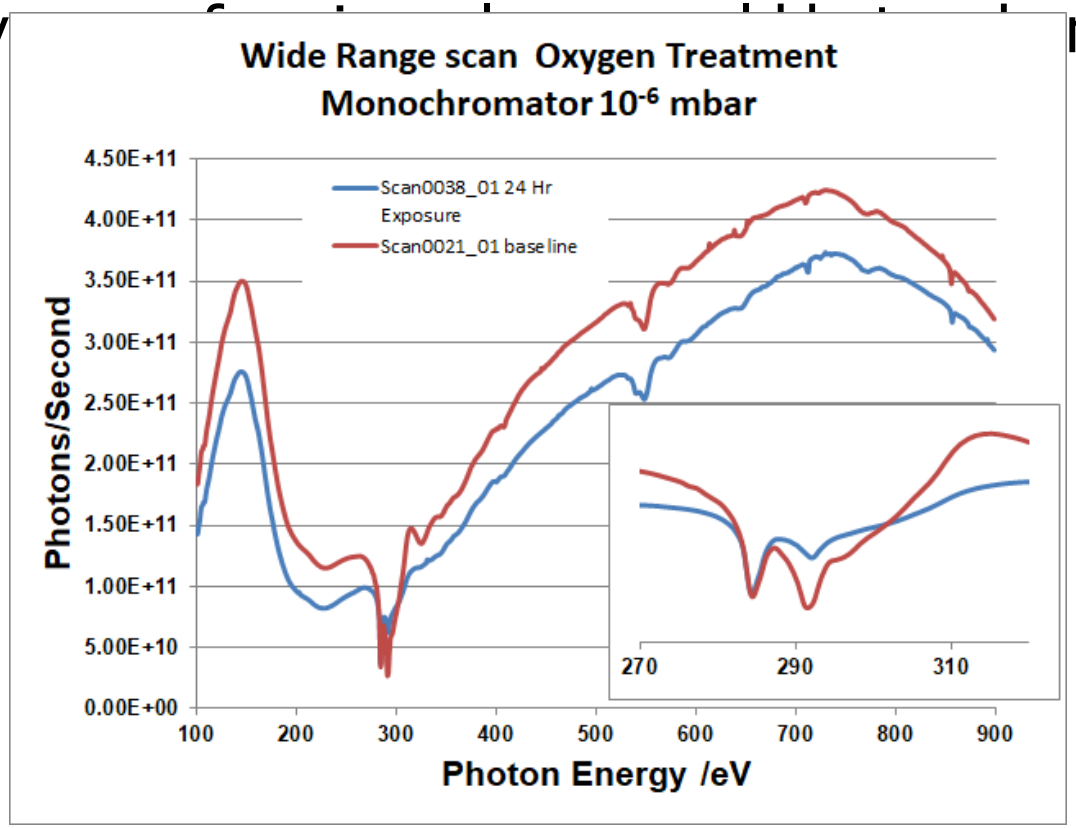
Reflection only from the surface

The X-rays break down molecules in the vacuum and they can build up on the surfaces after a while y n.



All reflective surfaces have to be maintained in Ultra High Vacuum conditions to slow down contamination

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Strong X-ray absorption below 2 KeV

Not only the beamline has to be in vacuum often the sample and all the detectors have to also be in vacuum

The vacuum environment does allow you to measure the electrons given off when atoms absorb X-rays rather than any X-ray photons – This will be described more fully in the talk of the spectroscopy group tomorrow

For more high energy beamlines they almost exclusive measure X-rays emitted, transmitted or scattered by the sample

Beamlines Below 2KeV

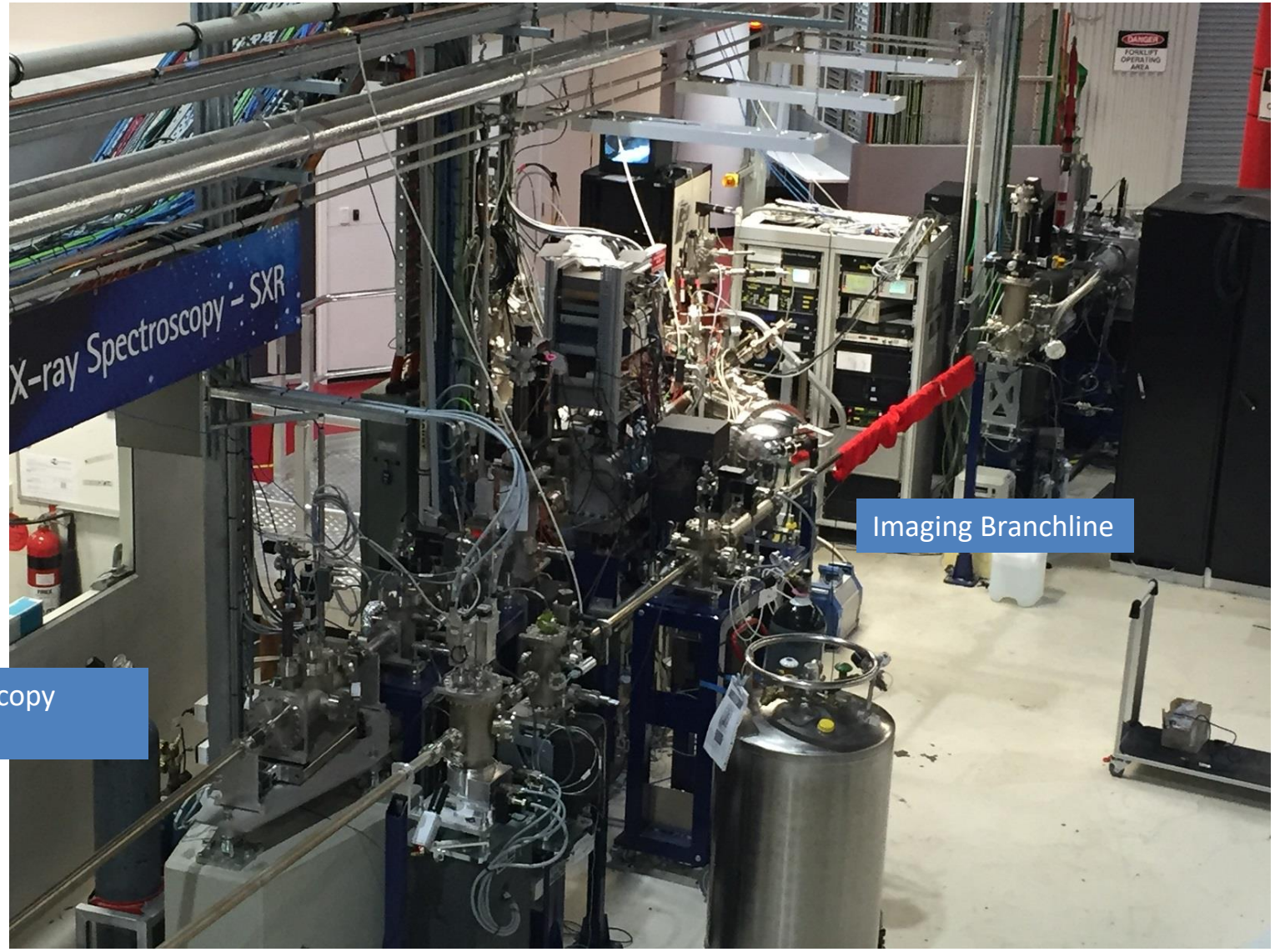
Final Advantage Radiation Safety!

The initial X-rays that come out of the storage ring have a range of X-rays from the low to high energies We often call this the White beam.

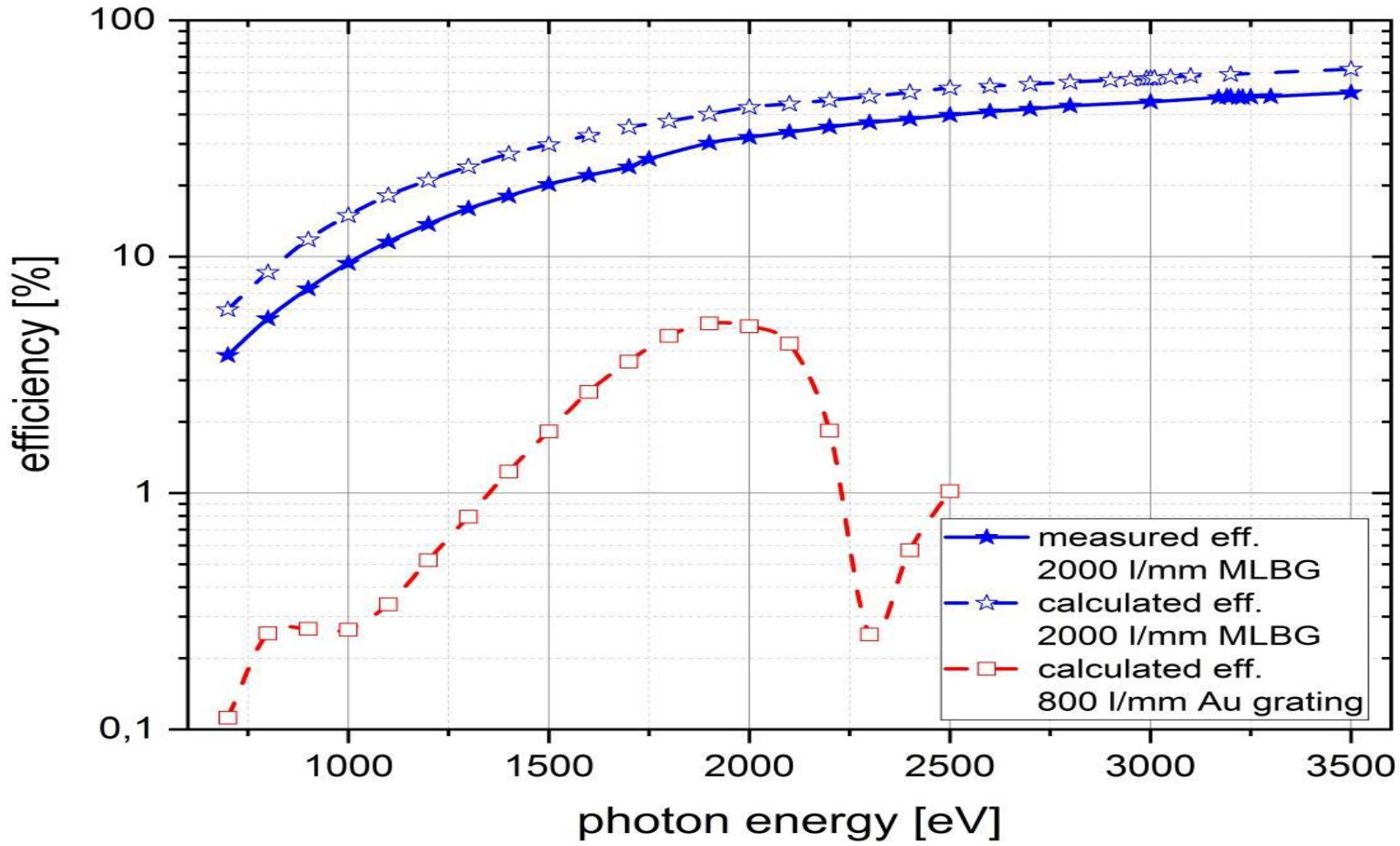
For grating beamlines we can only use the energies from 3 KeV and below. If we arrange the first mirror angle correctly, we can bounce out all the low energy X-rays and absorb all the dangerous radiation.

The rest of the beamline has to work in a very good vacuum and the stainless steel and glass viewports now act as a containment for all the dangerous radiation i.e. you can stand beside the vessels even when the X-ray beam is on.

- The soft x-ray beamline consists of 2 branches: The Spectroscopy Mainline and the Imaging branchline
- Only one branch can run at a time



Gratings with Multilayers



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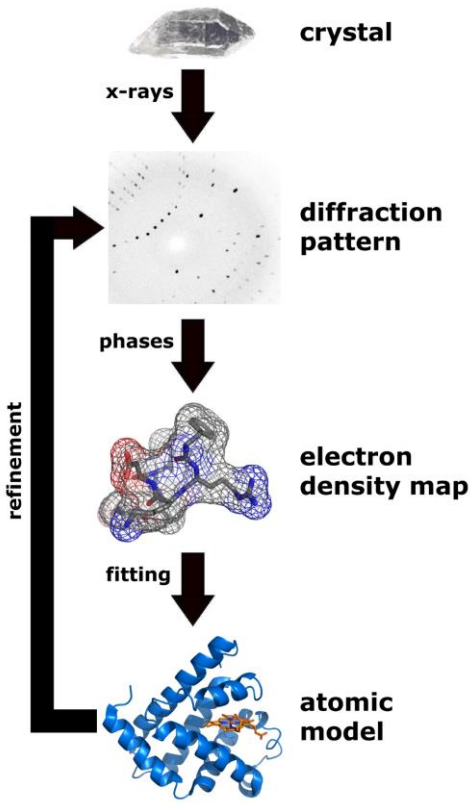
Intro to MX

In order to talk about MX3 design some concepts in MX and what is needed for good data:



$$\rho(xyz) = \frac{1}{V} \sum_{hkl}^{+\infty}_{-\infty} |F(hkl)| \, . \, e^{-2\pi i[hx+ky+lz-\phi(hkl)]}$$

Amplitudes

Phases?

Dauter, Z. Data-collection strategies (1999). Acta Cryst. D55, 1703-1717.

Dauter Z. Collection of X-Ray Diffraction Data from Macromolecular Crystals. *Methods Mol Biol*. 2017; 1607:165-184

http://en.wikipedia.org/wiki/X-ray_crystallography

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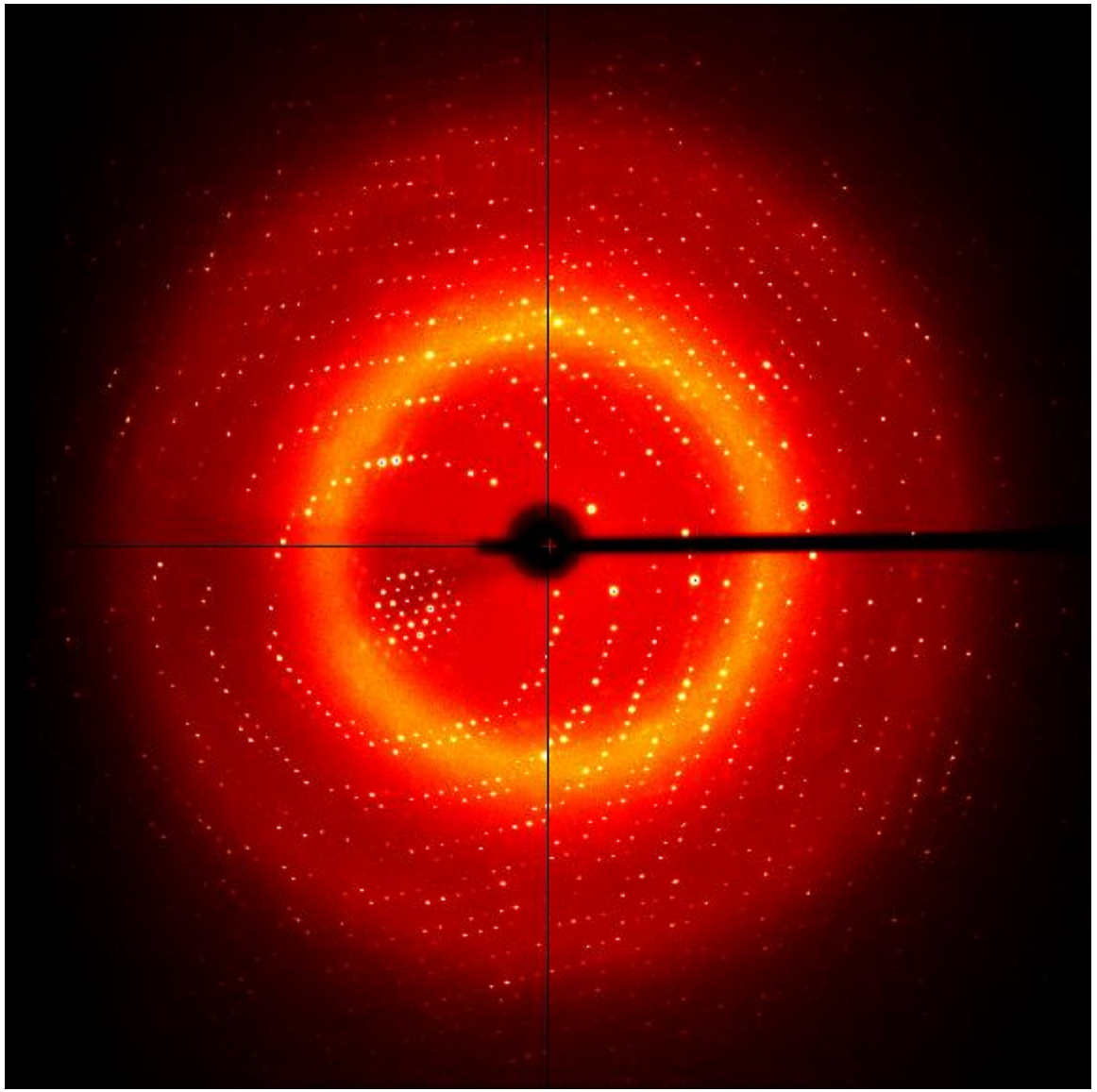
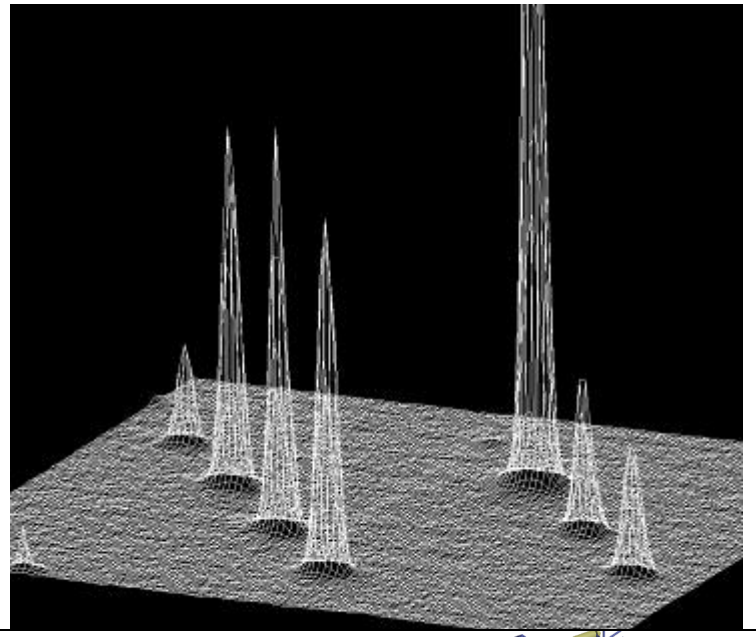
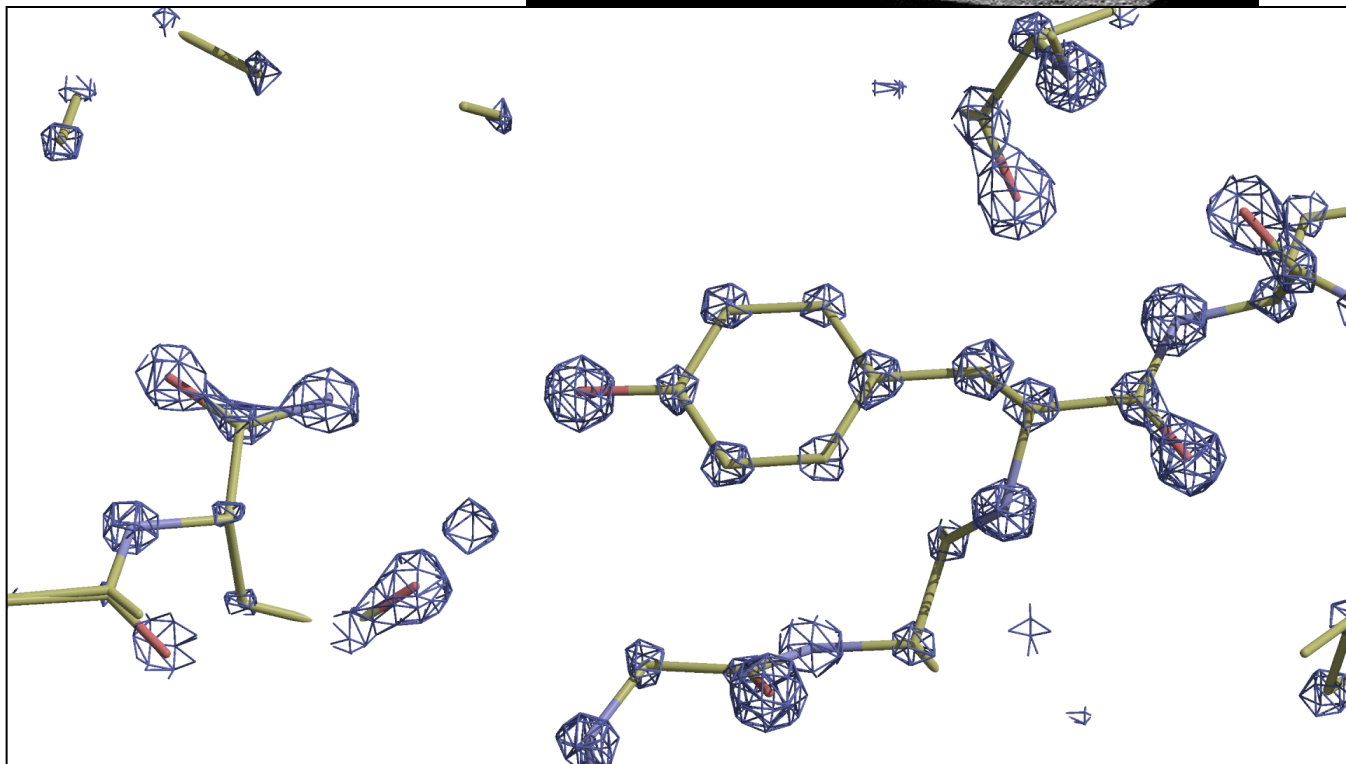
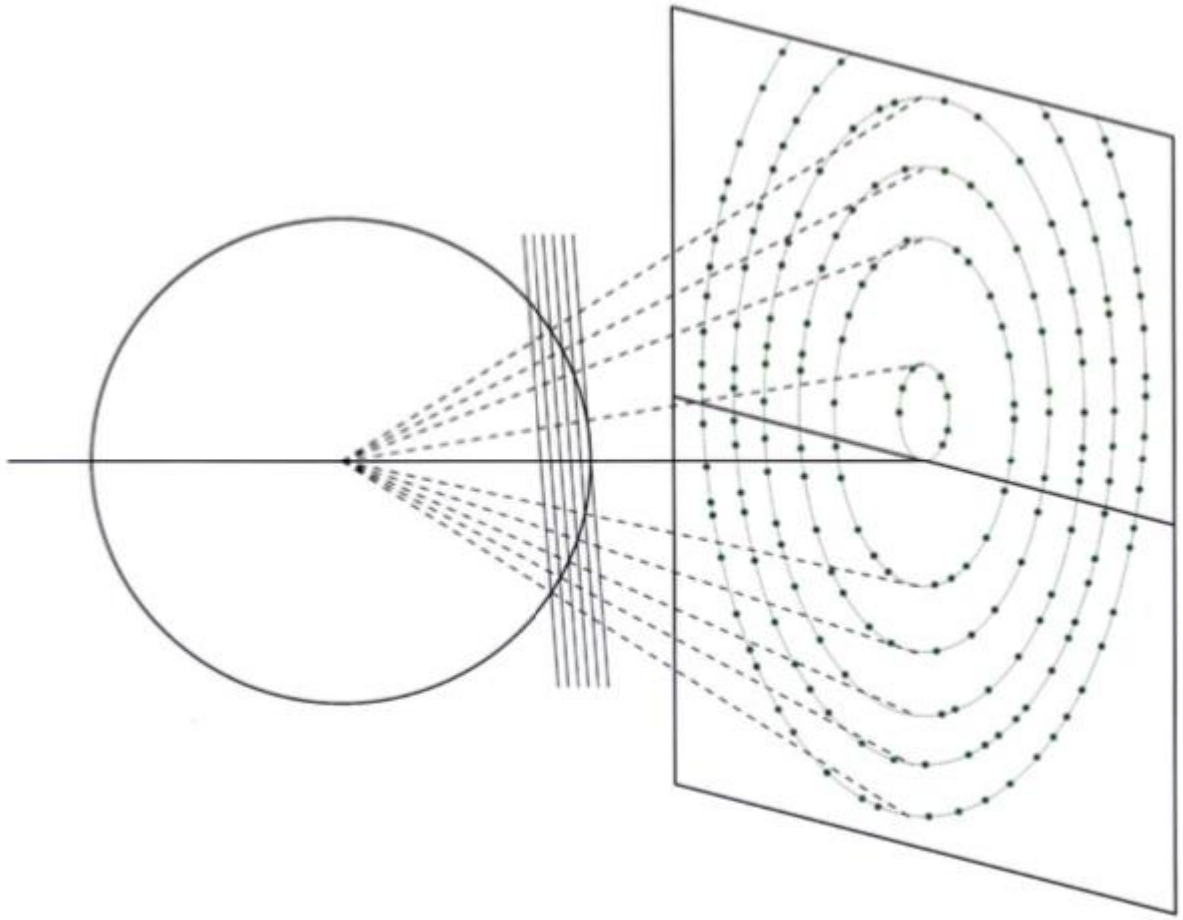
Intro to MX



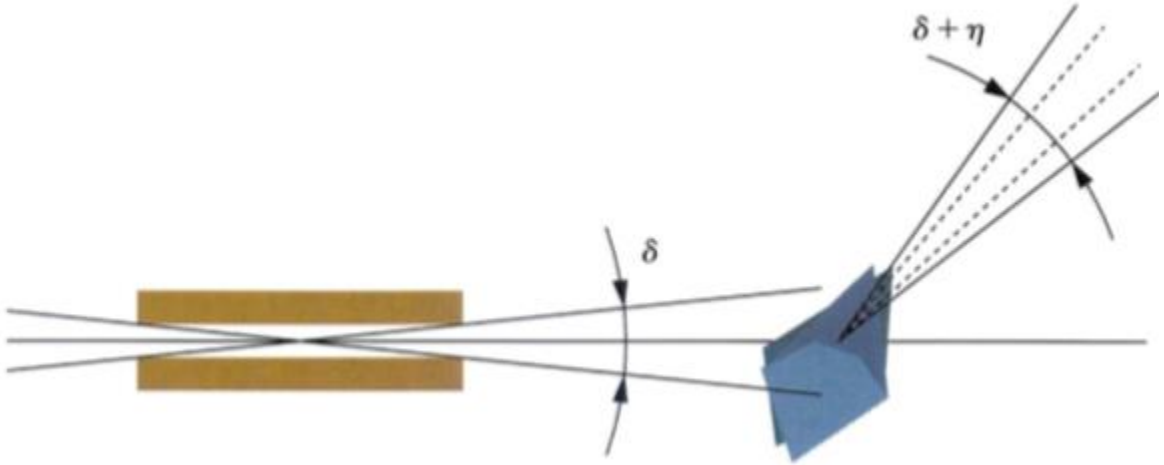


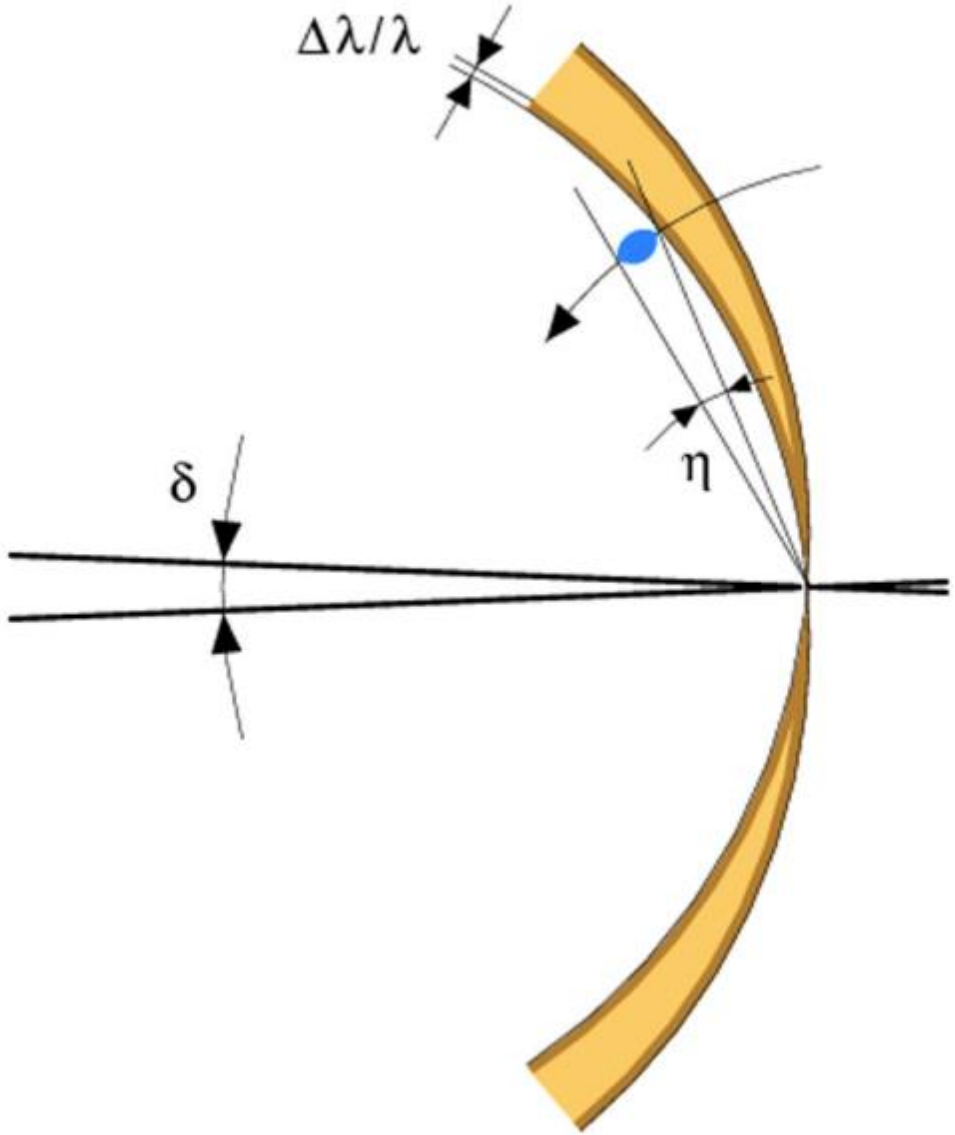


Intro to MX



Intro to MX





δ = divergence at sample

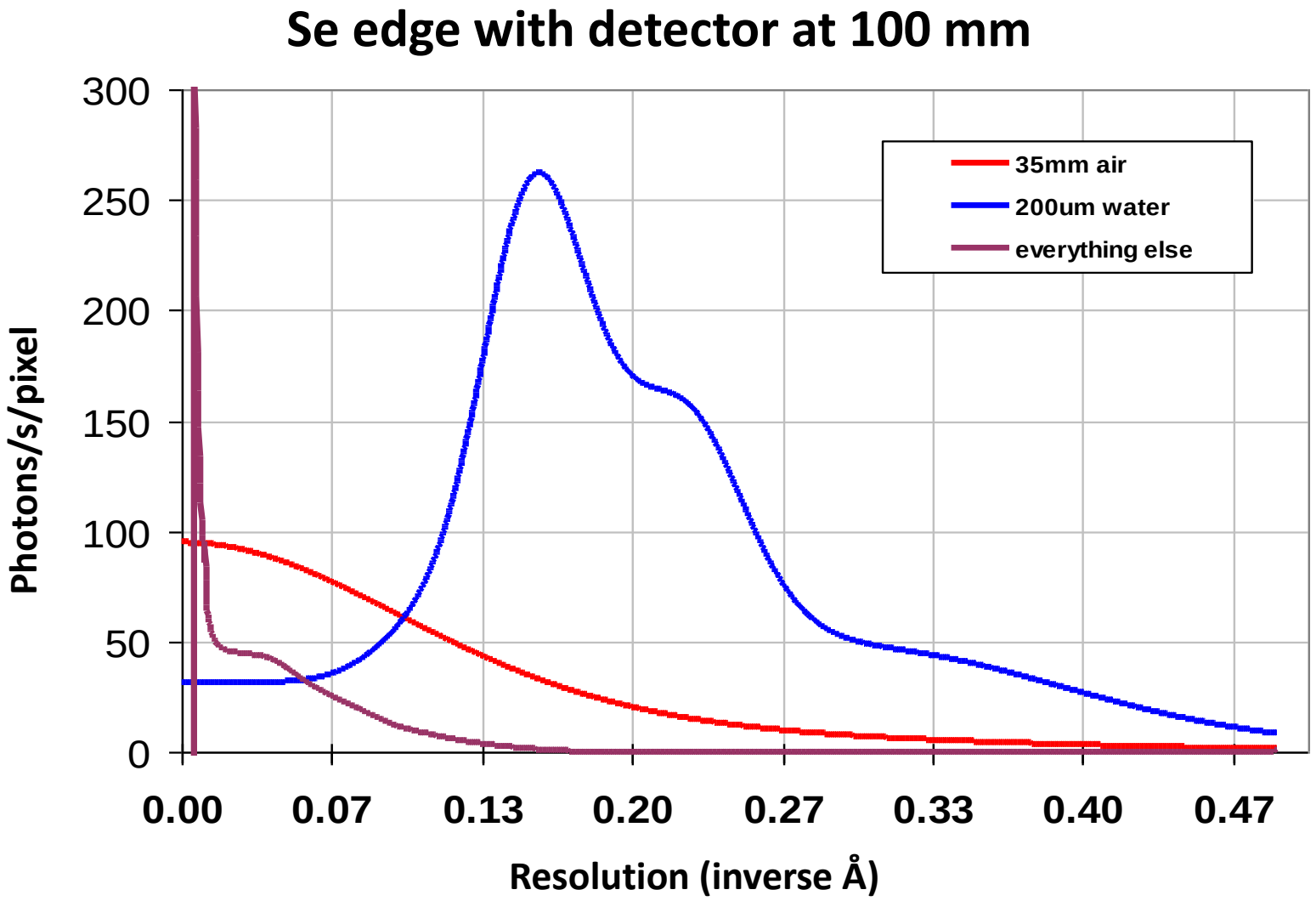
η = mosaicity of the crystal

***Divergence and mosaicity are at 90 degrees to each other on the detector surface!**

Dauter, Z. Data-collection strategies (1999). Acta Cryst. D55, 1703-1717.

Dauter Z. Collection of X-Ray Diffraction Data from Macromolecular Crystals. *Methods Mol Biol*. 2017; 1607:165-184

Scattering background in the experiment



Data from James Holton and data from 8.3.1 at the ALS

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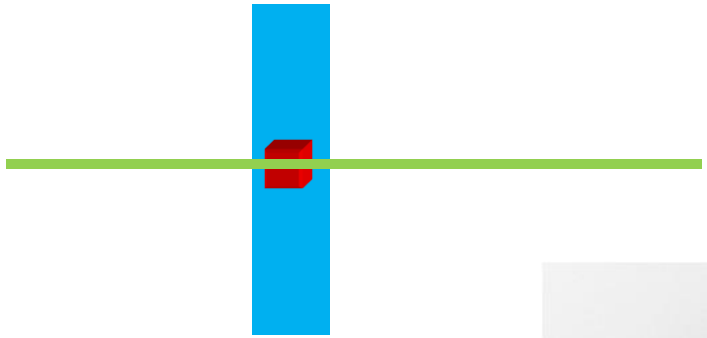
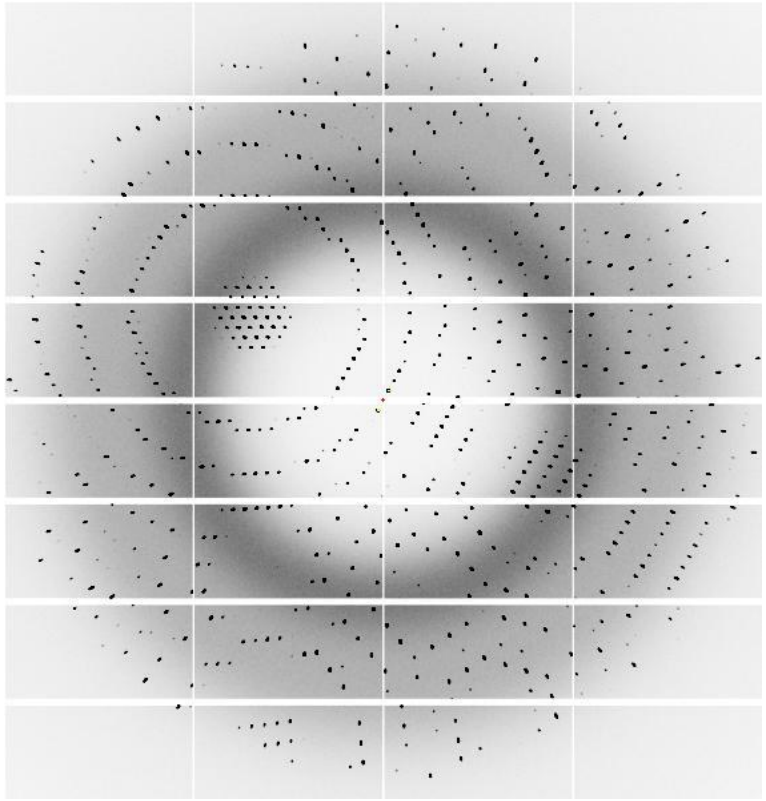
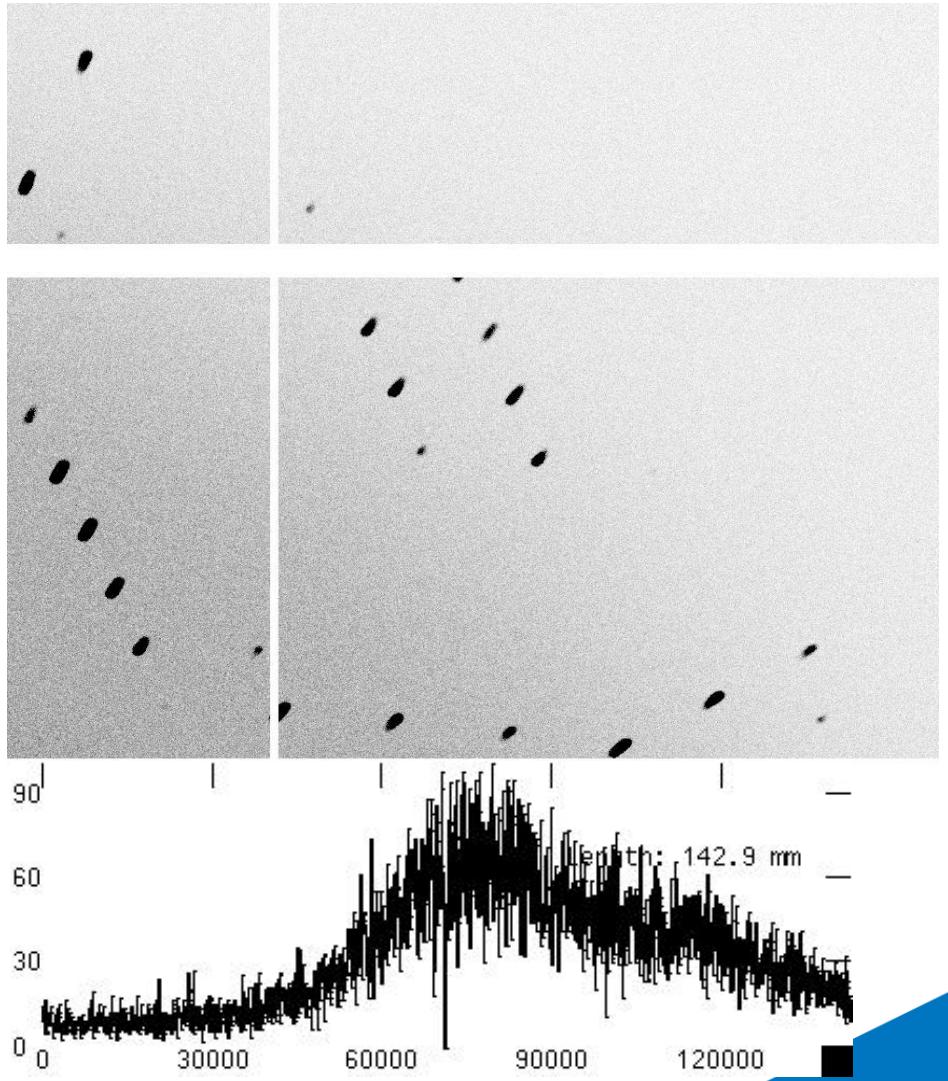
Impact of material in the beam path



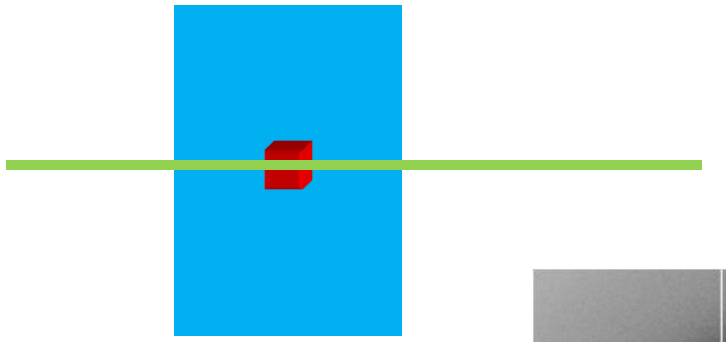
10 micron crystal

10 micron water



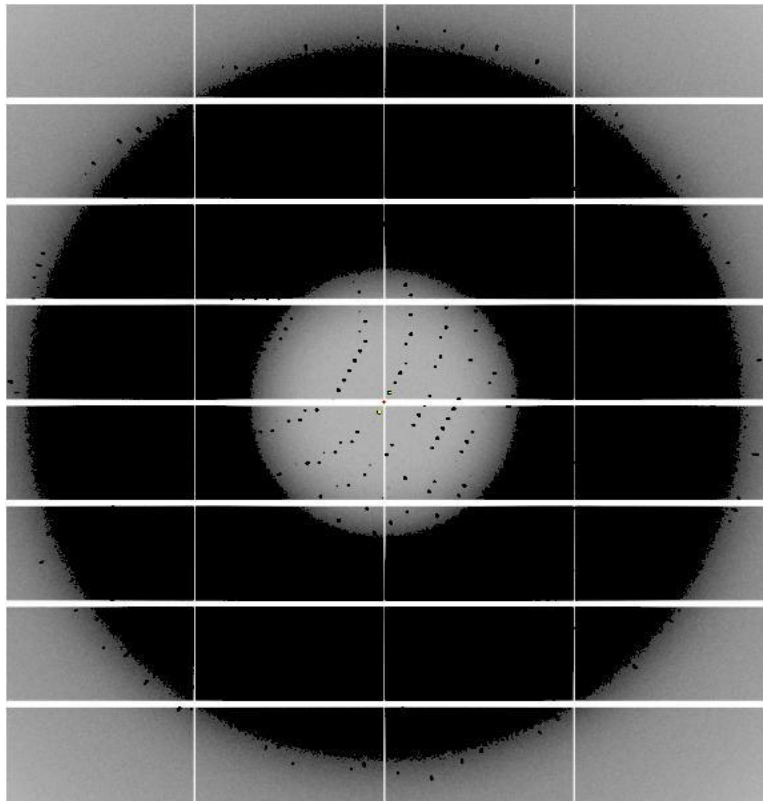


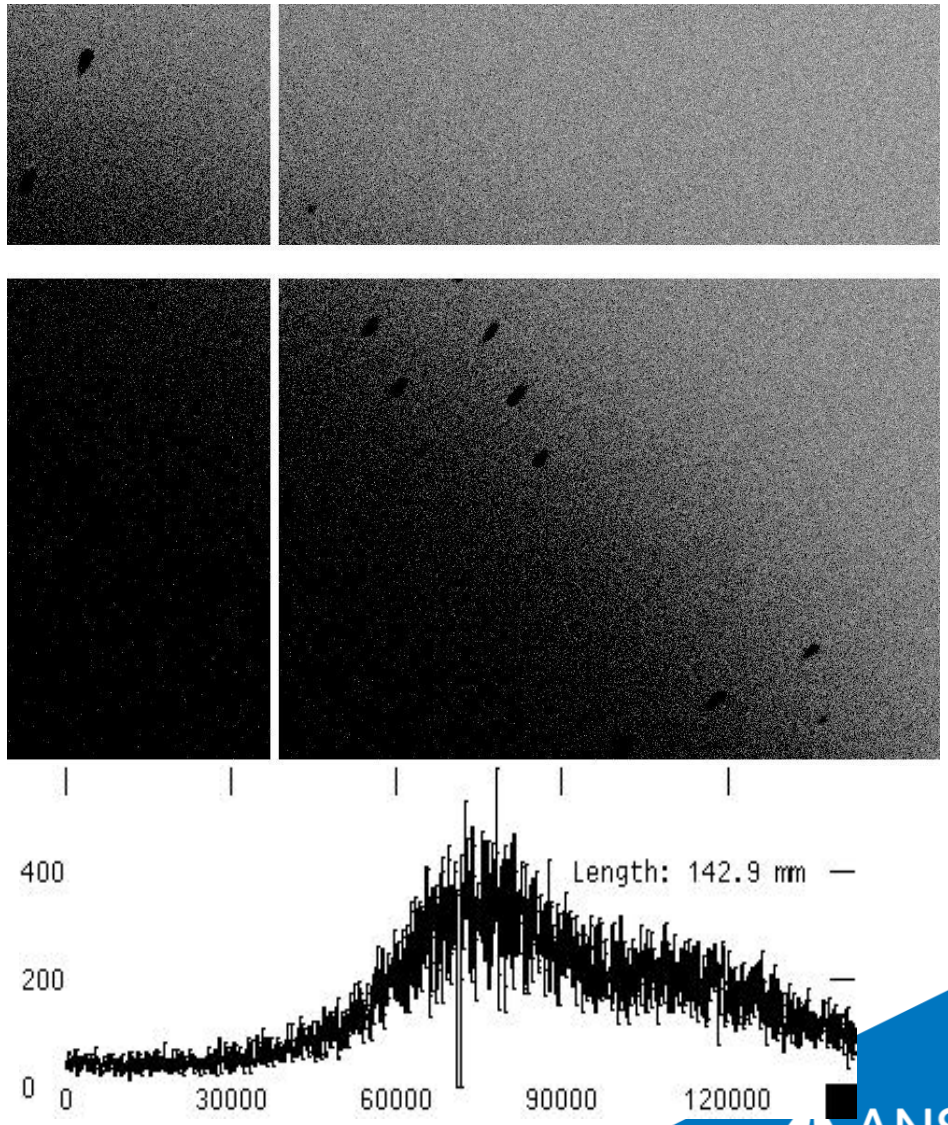
Impact of material in the beam path



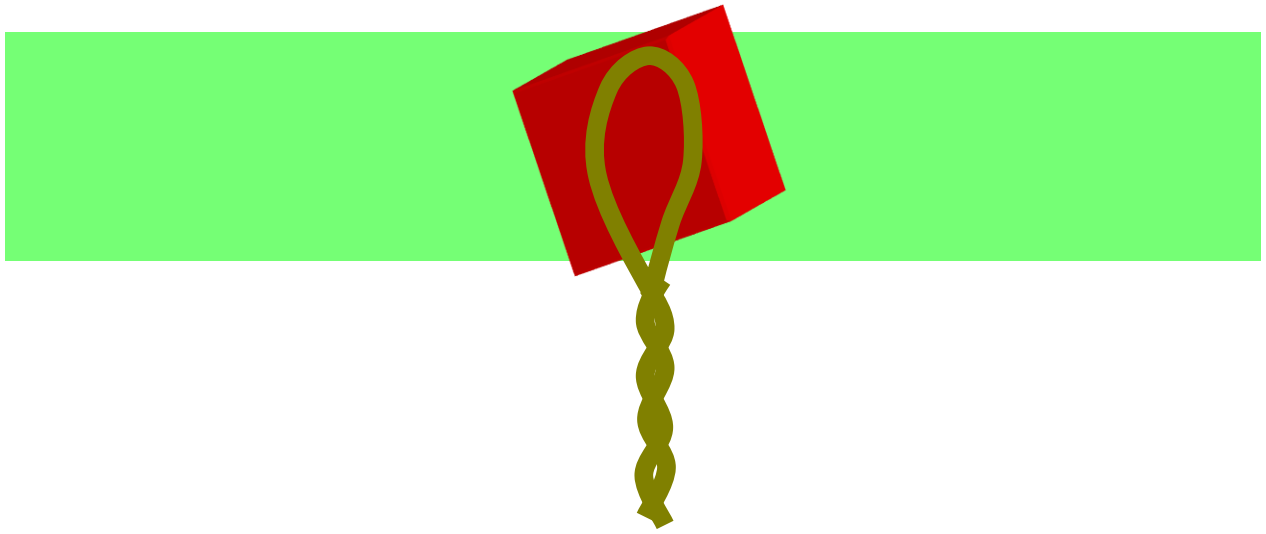
10 micron crystal

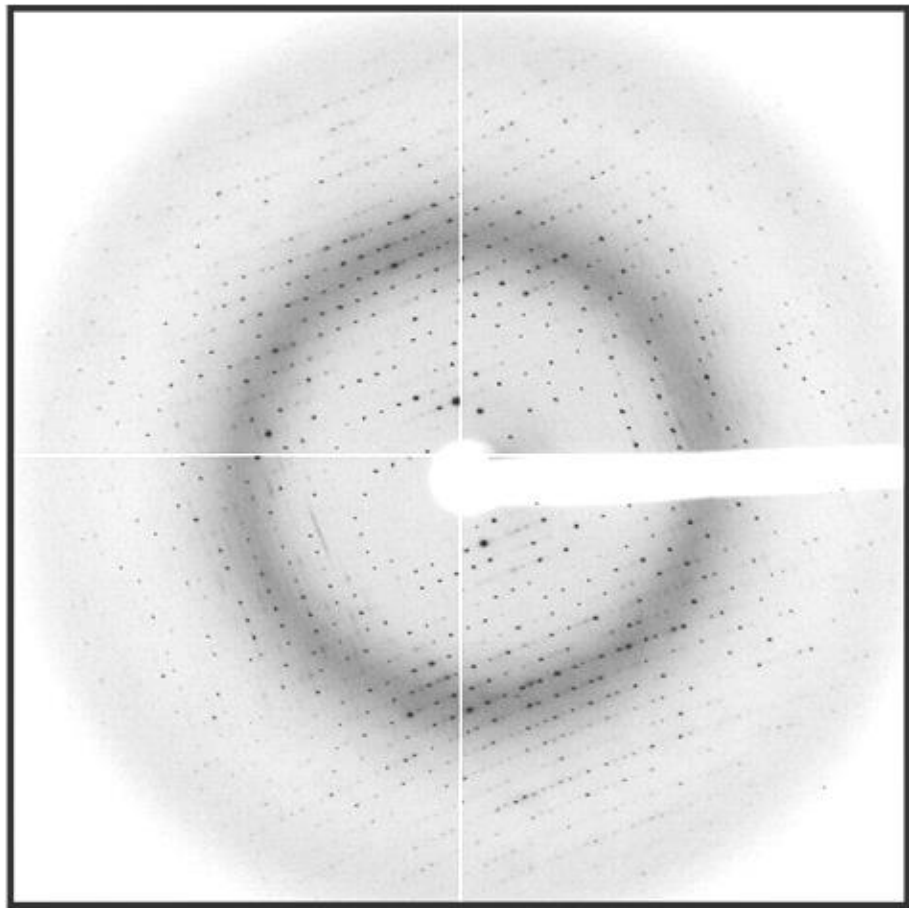
50 micron water





Big crystal and big beam – easy!





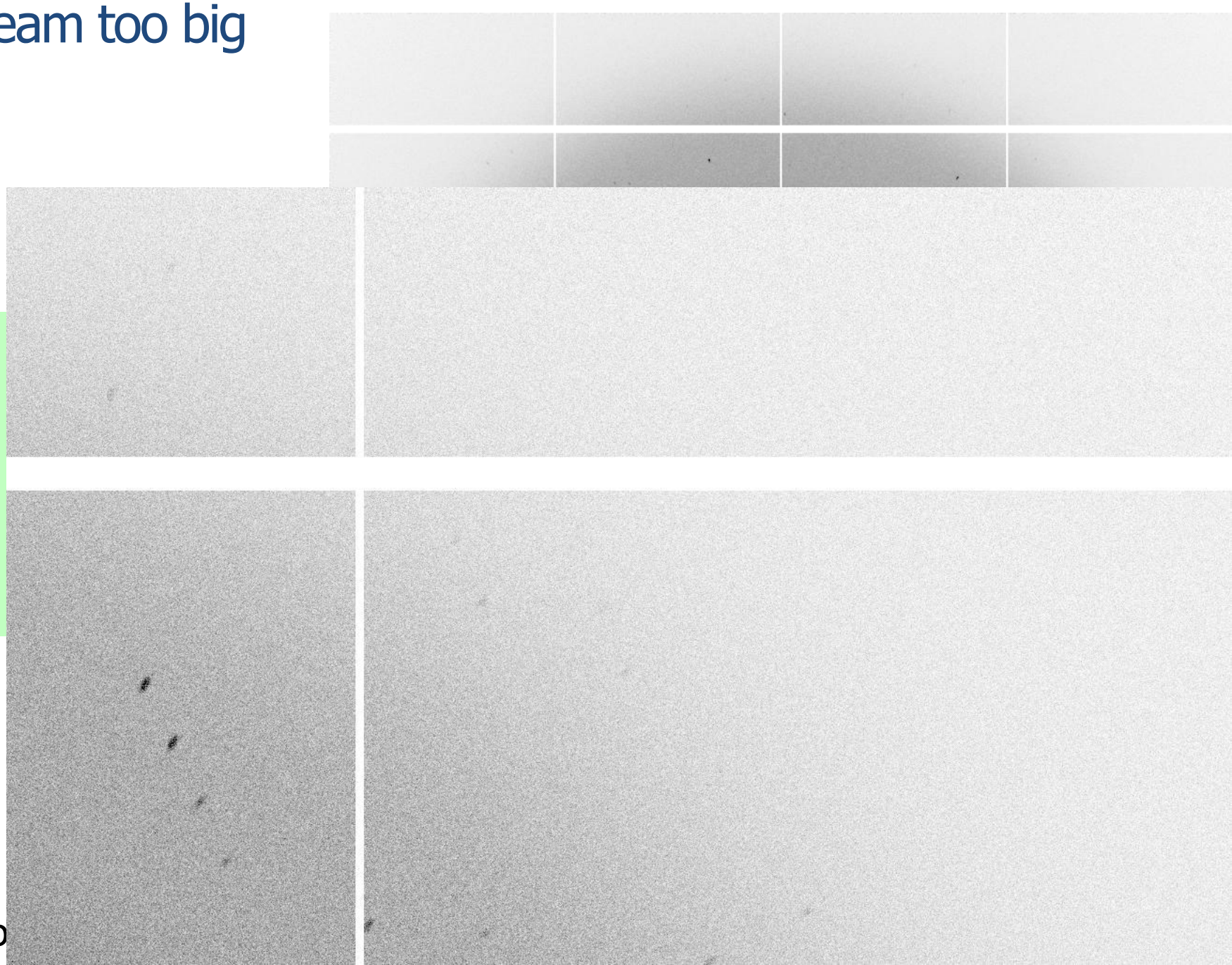
Images from James Holton and data from 8.3.1 at the ALS

Microfocus – beam too big

10 micron crystal

50 micron beam

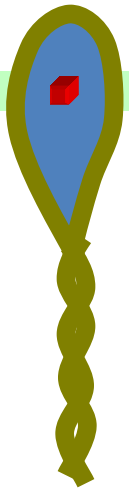
Loop graphic from James Holto

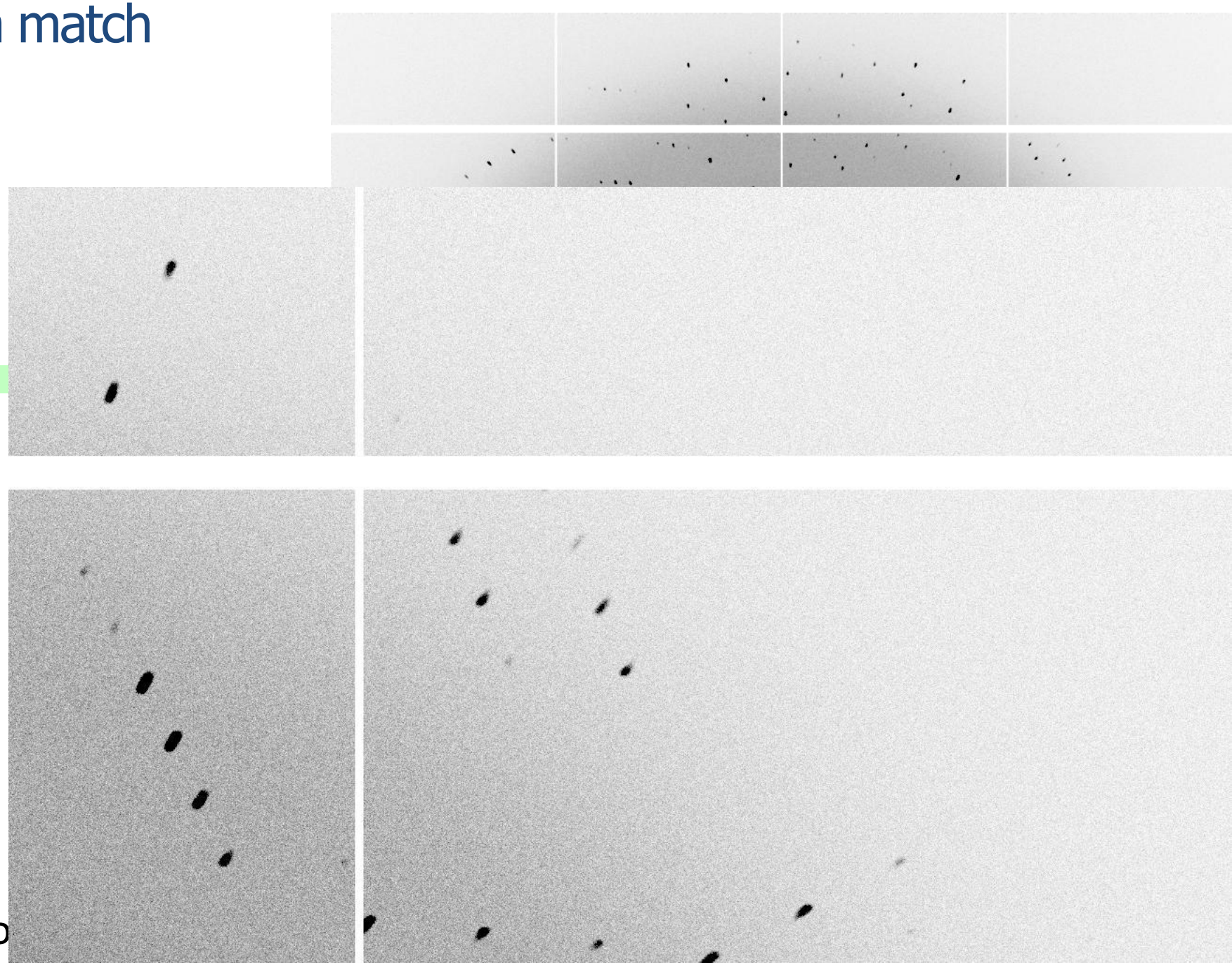


Microfocus – beam match

10 micron crystal

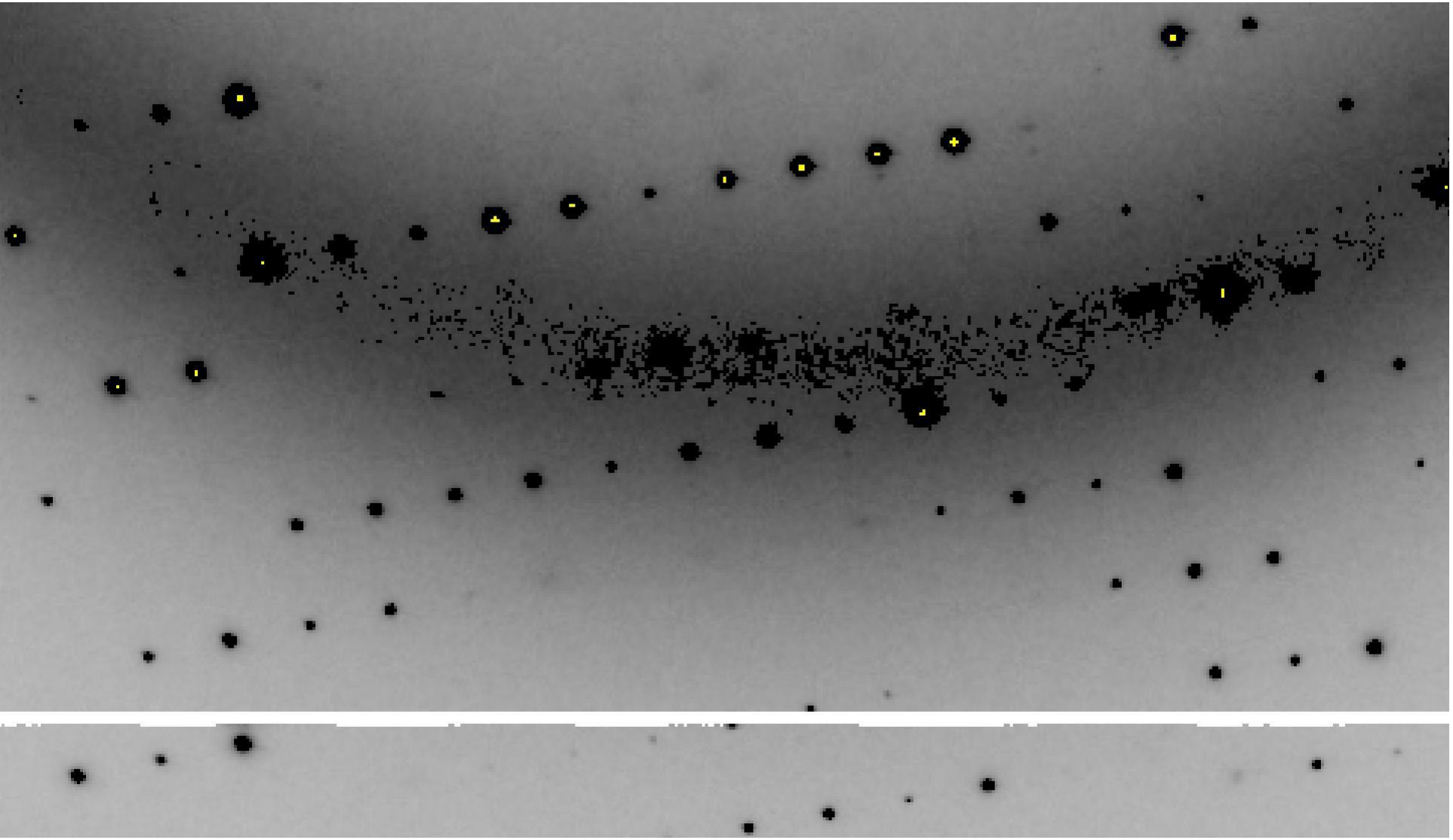
10 micron beam





Loop graphic from James Holto

Microfocus – finding the best bit of a crystal



Trypsin crystal

Tip of the same crystal
~ 5-10 microns thick

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Microfocus – finding the best bit of a crystal

So microfocus is important!

- Focal size ~=xtal size
- Smaller beams = harder engineering
- Rule of 10ths

Deciding on minimum beam size has a huge impact on beamline design

Key parameters for good MX experiment

Beamline:
Energy
Divergence (HxV)
Dispersion
Spot size
Flux
Flux stability
Positional stability

Crystal:
Size
Mosaicity
Cell dimensions
Space group
Resolution
Surrounding material

Endstation:
Goniometer SOC
Alignment of crystal/beam/camera/goniometer
Shutter jitter
Variation in Omega speed
Cryostream alignment
Cryostream vibration
Detector noise/count-rate/charge-sharing
Drift (thermal/mechanical)

Other Optical parameters not important such as:
Parasitic scattering
Energy resolution
Energy scanning speed

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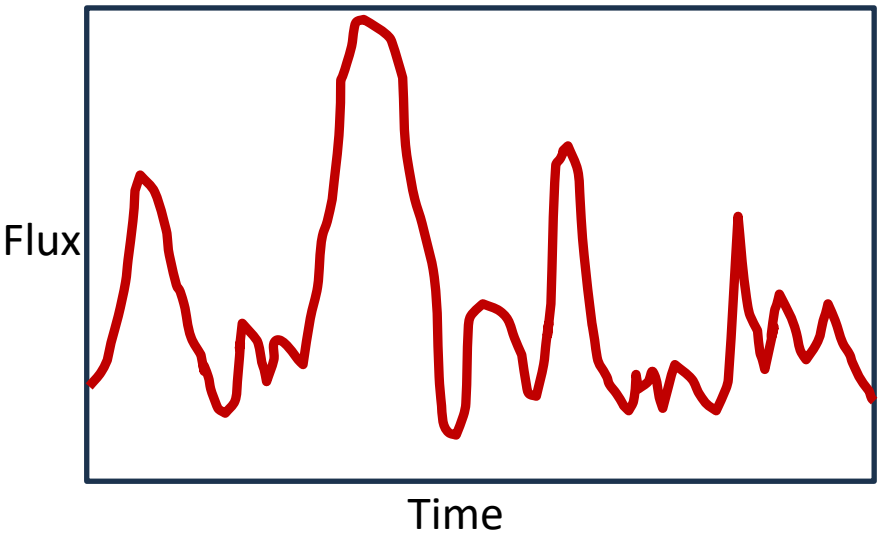
Vibration and Stability



Vibration:

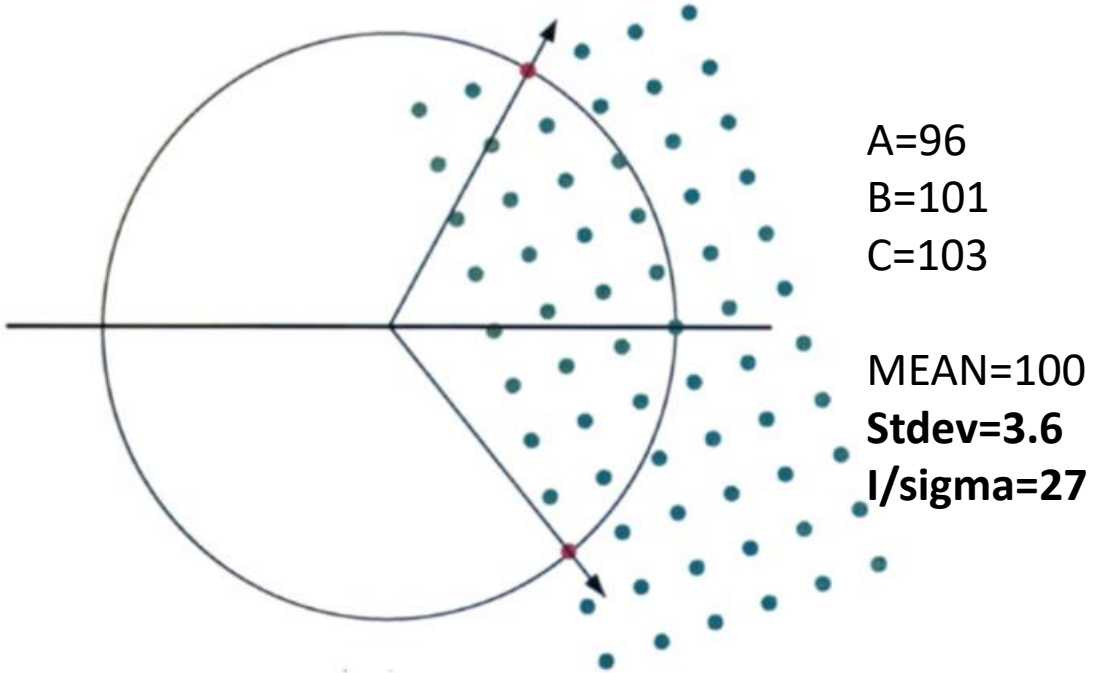
Stable optics and equipment

As few vertical deflection optics as possible

High natural frequency

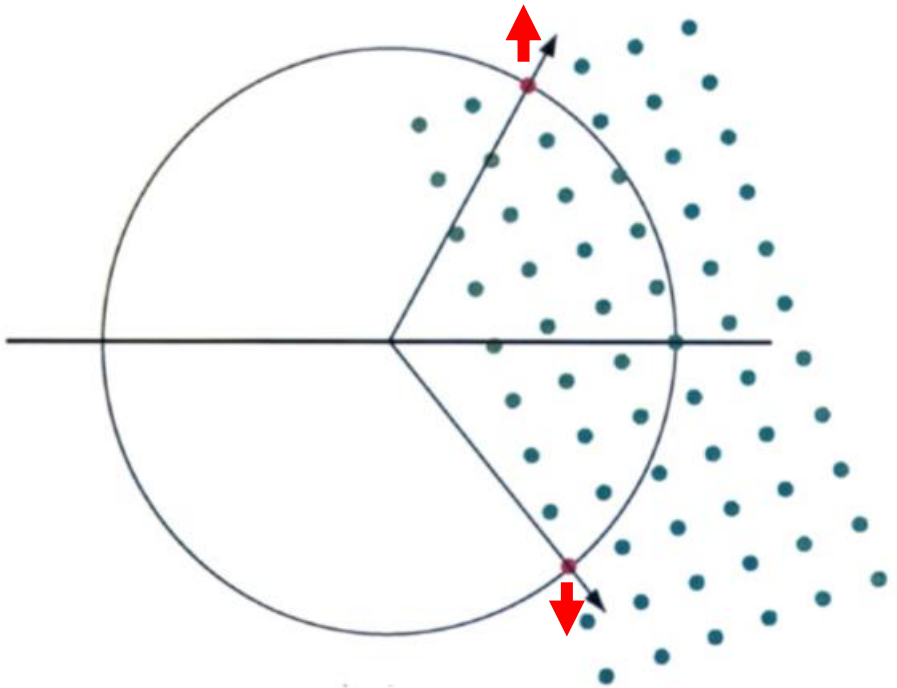
Design to reduce susceptibility to vibration (HDMM, SSA etc)

Avoid plant vibration



A=96
B=101
C=103

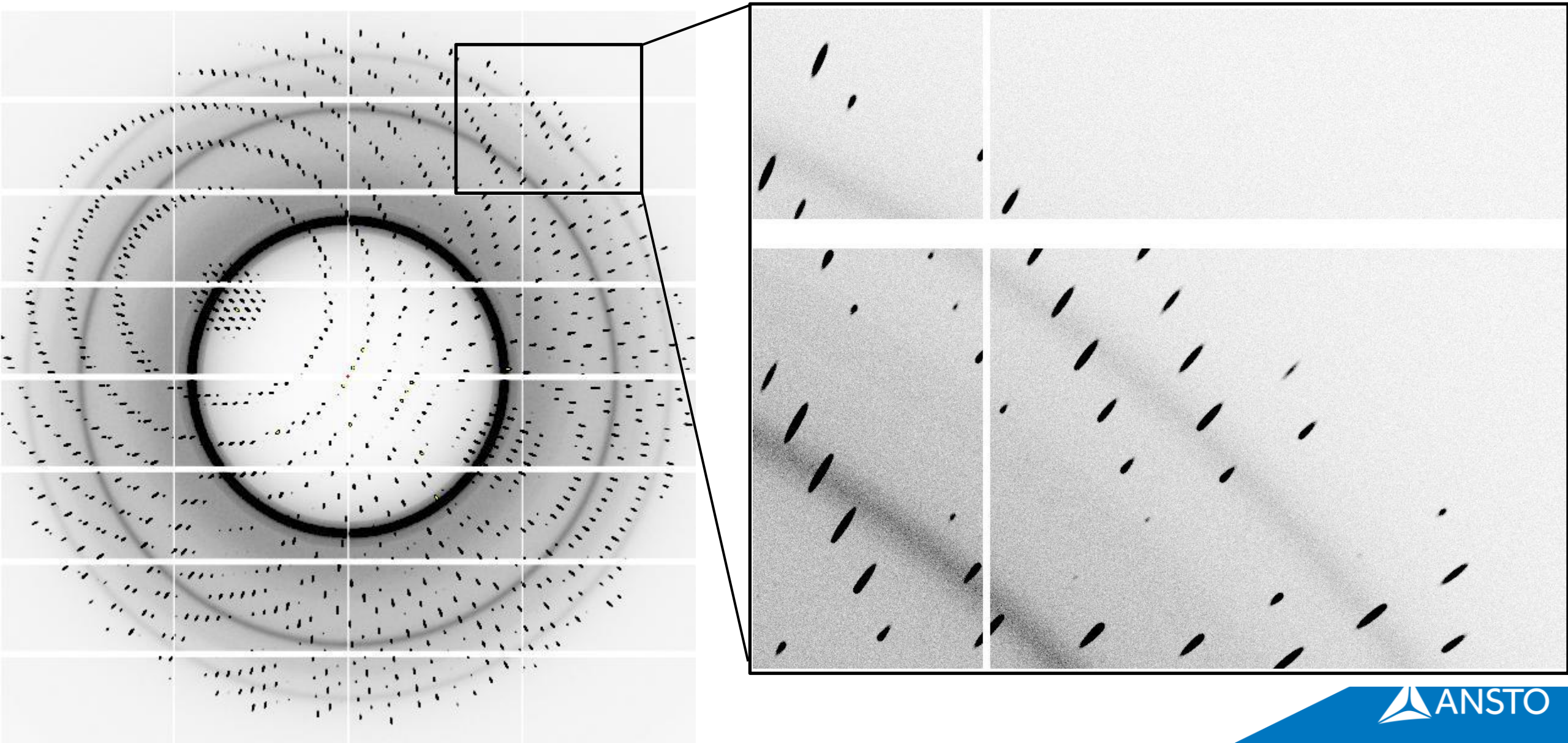
MEAN=100
Stdev=3.6
I/sigma=27



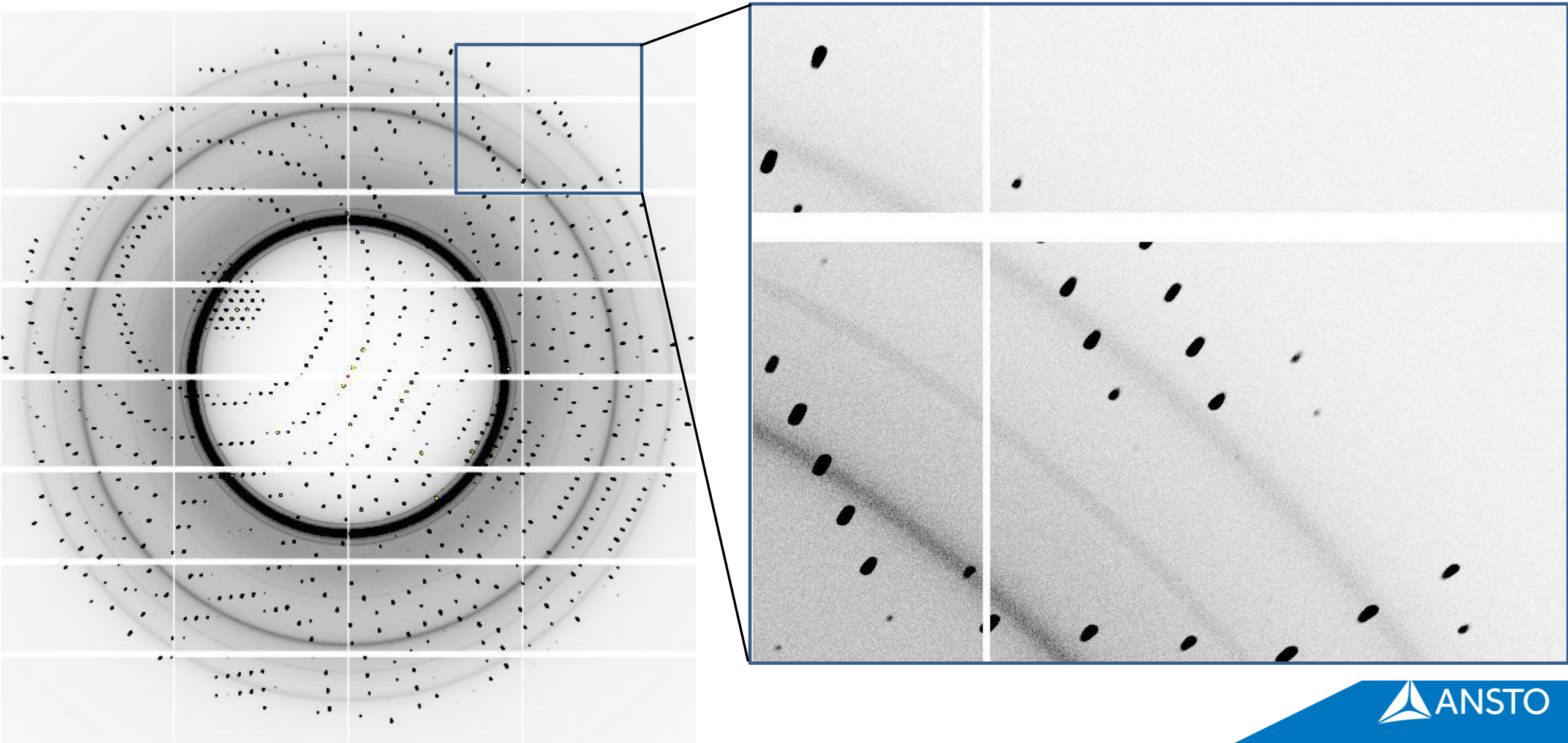
A=78
B=95
C=127

MEAN=100
Stdev=24.9
I/sigma=4

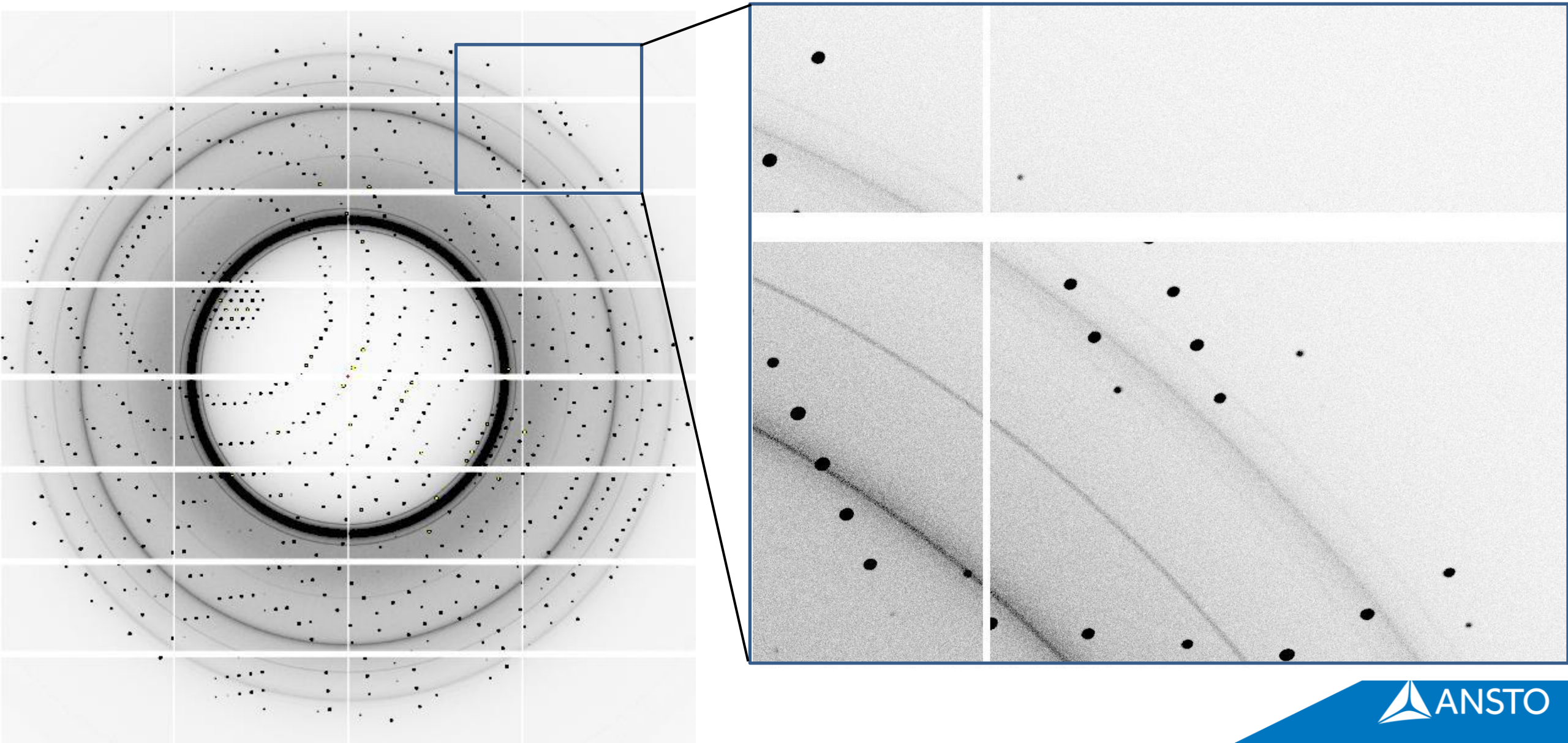
What does a DMM mean for MX: DMM at 3%bandpass



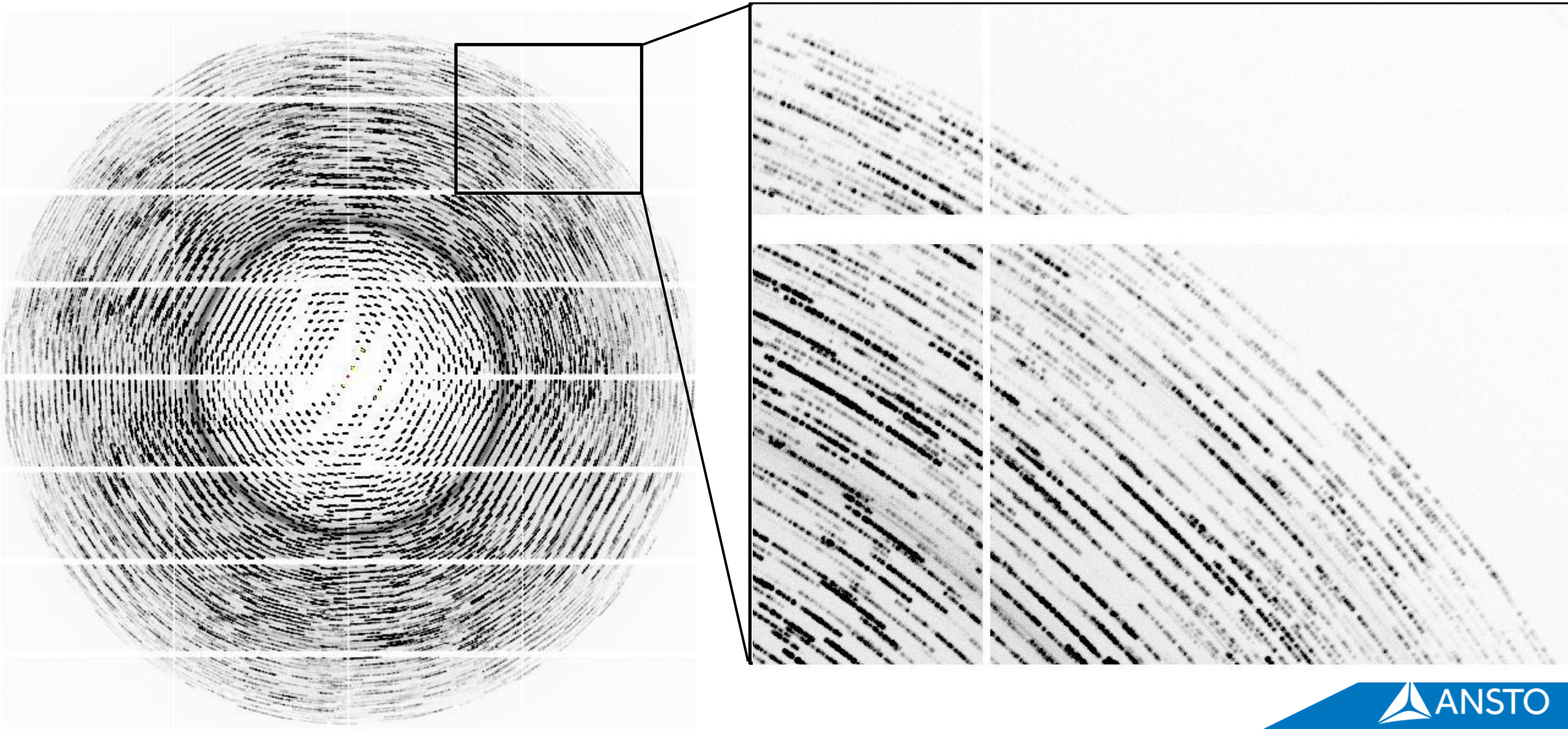
What does a DMM mean for MX: DMM with 1% bandpass



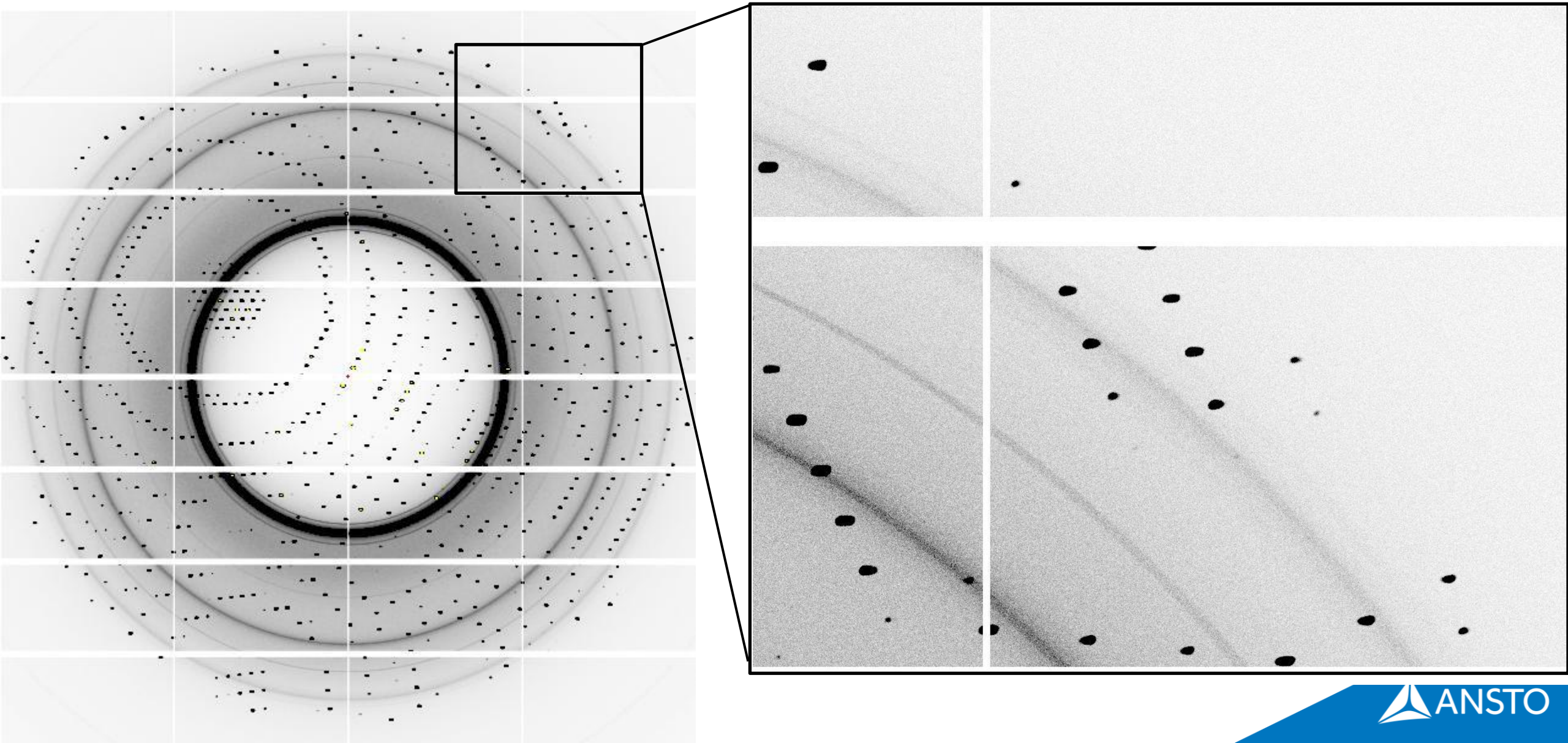
What does a DMM mean for MX: Si111 DCM (2e-4 bandpass)



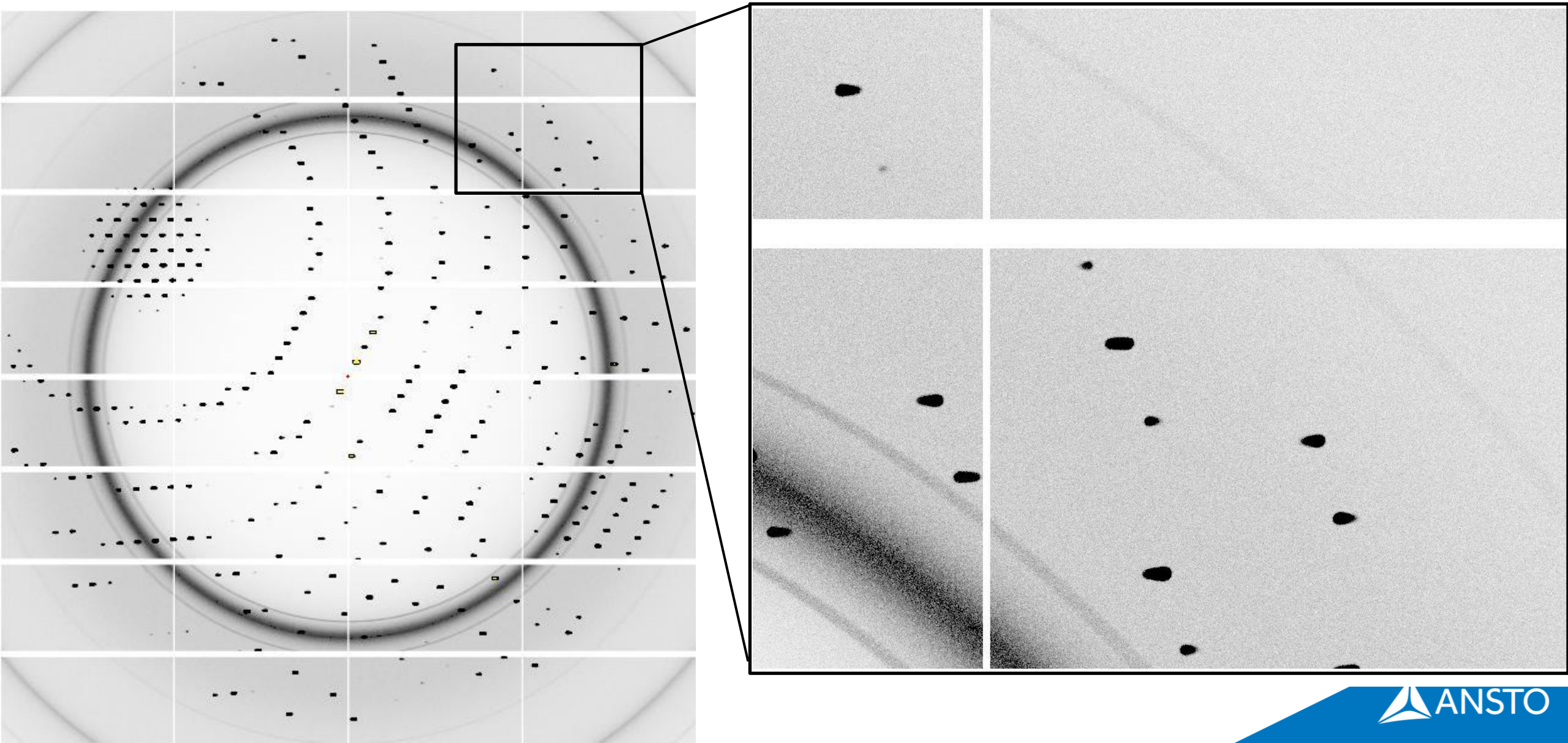
Mosaicity (set to 2.5%)



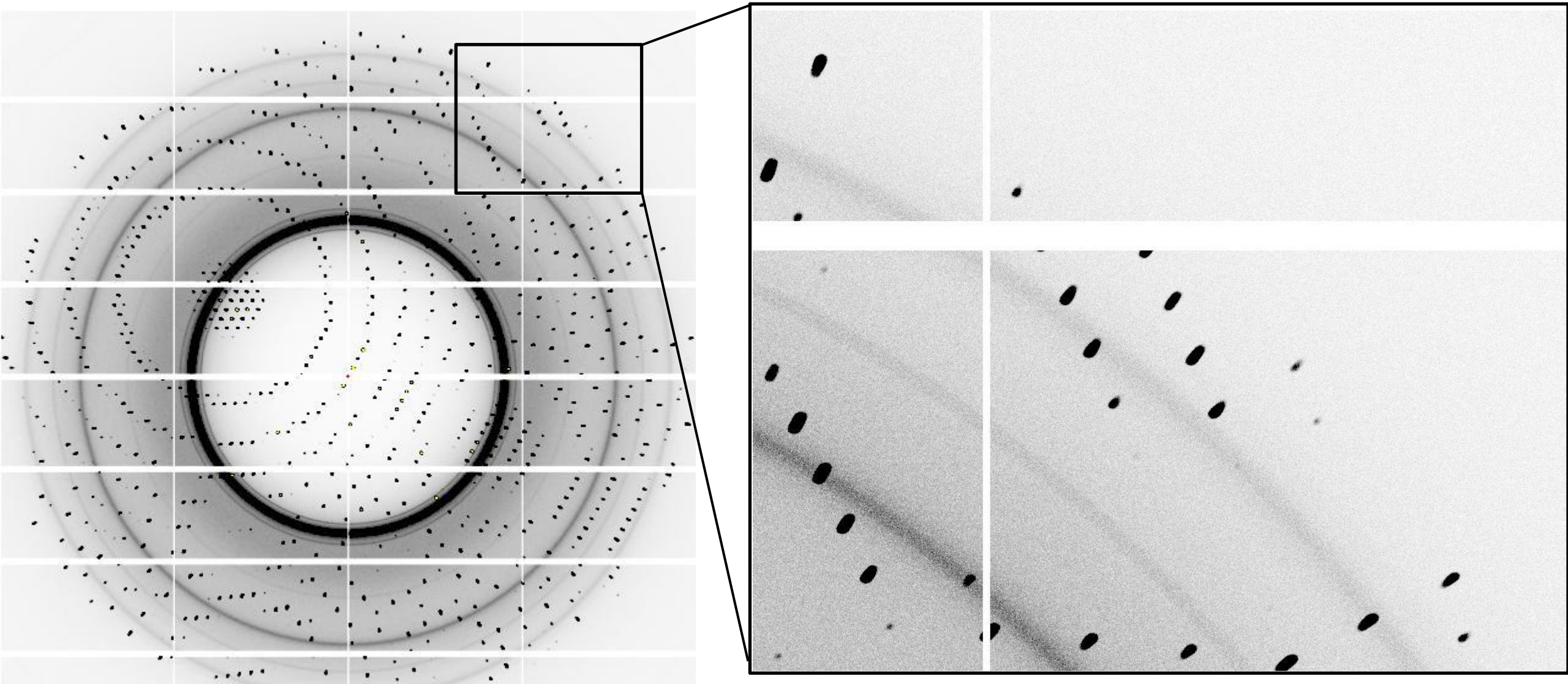
Divergence (horizontal set to 4 mrad, vertical 1mrad)



Divergence (horizontal set to 4 mrad, detector moved back)



MX3 as modelled, 1% bandpass, 2mrad horiz divergence, 0.1% mosaicity



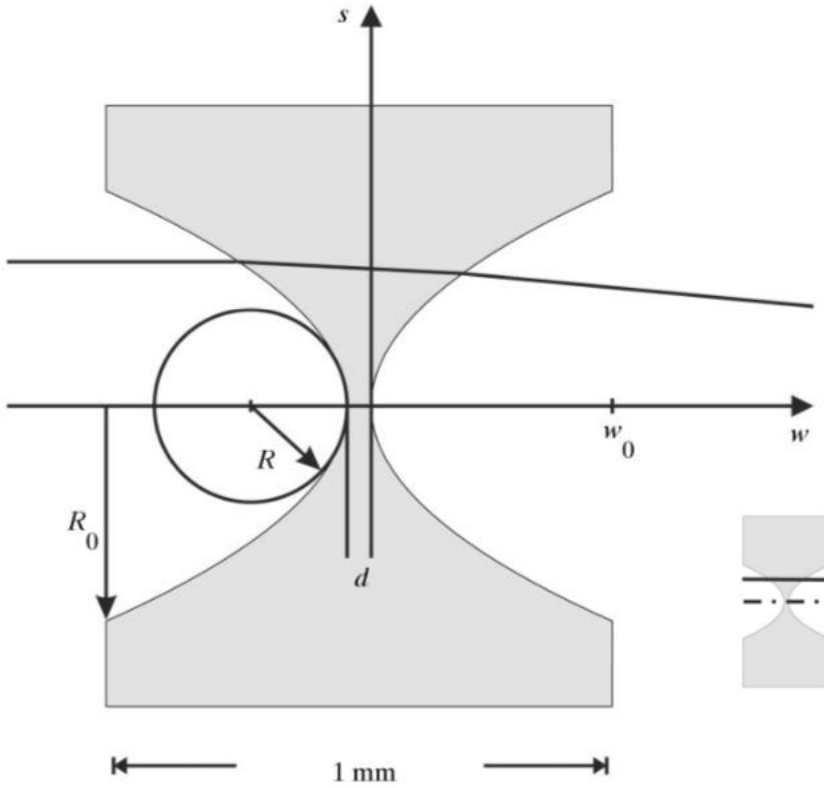
Defocusing

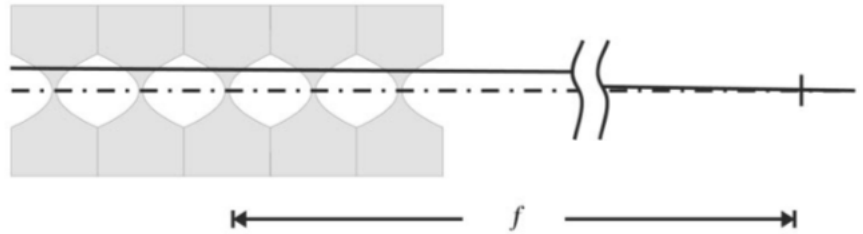
Ideally match beam size to crystal

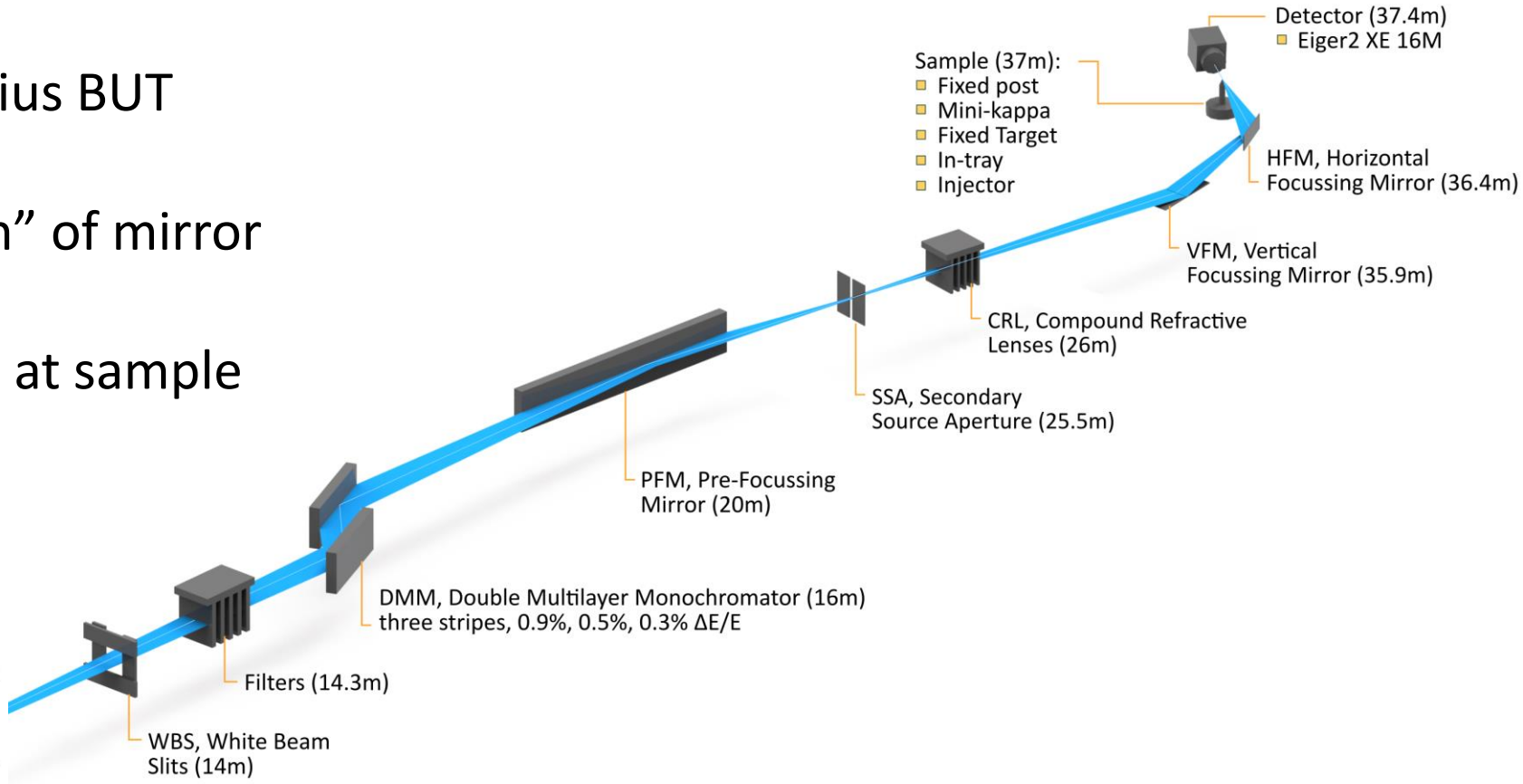
Mirrors: Can change bend radius BUT

- Can change pitch of mirror
- Can change “lateral position” of mirror

Lead to shift of beam position at sample

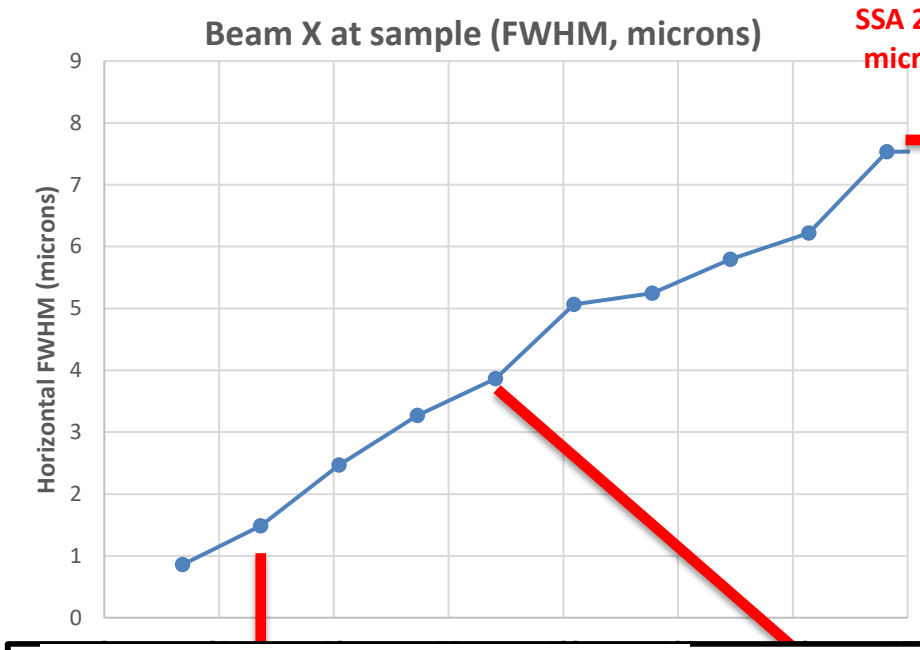




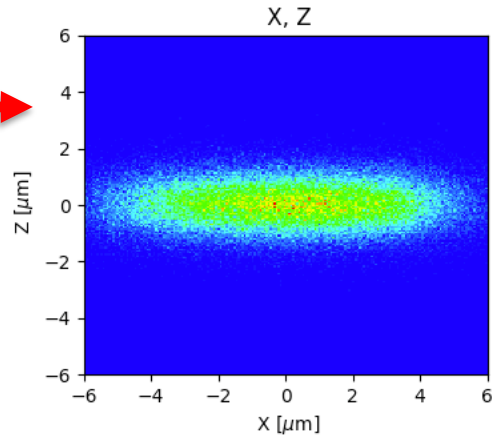


From Lengeler et al. “Imaging by parabolic refractive lenses in the hard X-ray region” J. Synchrotron Rad. (1999). 6, 1153-1167.

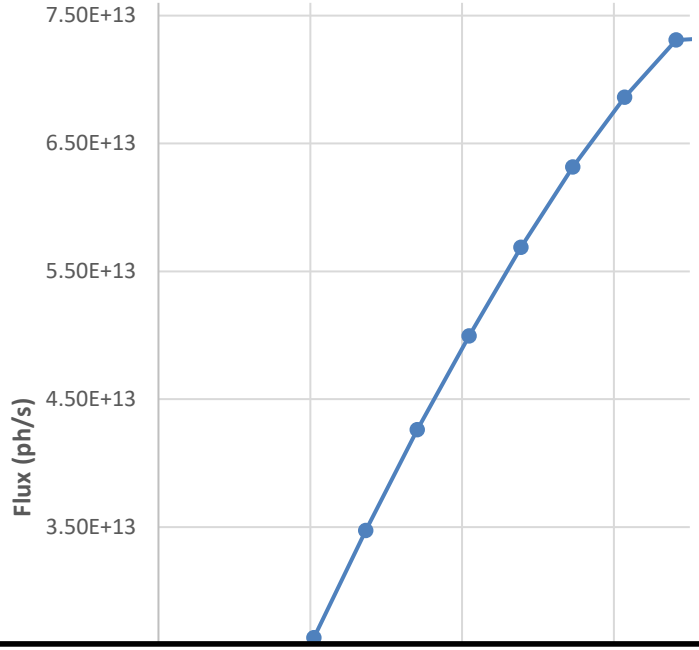
MX3 Beam performance



SSA 2000 microns



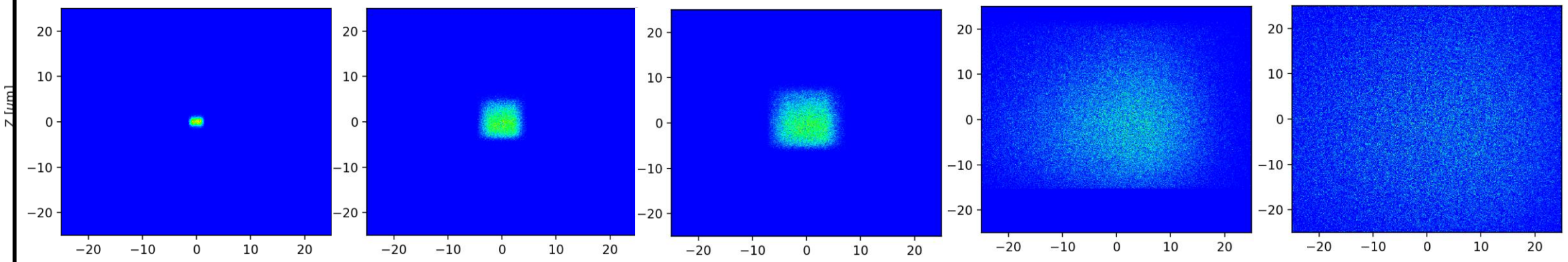
14 x 2 microns
1e^{14} ph/s



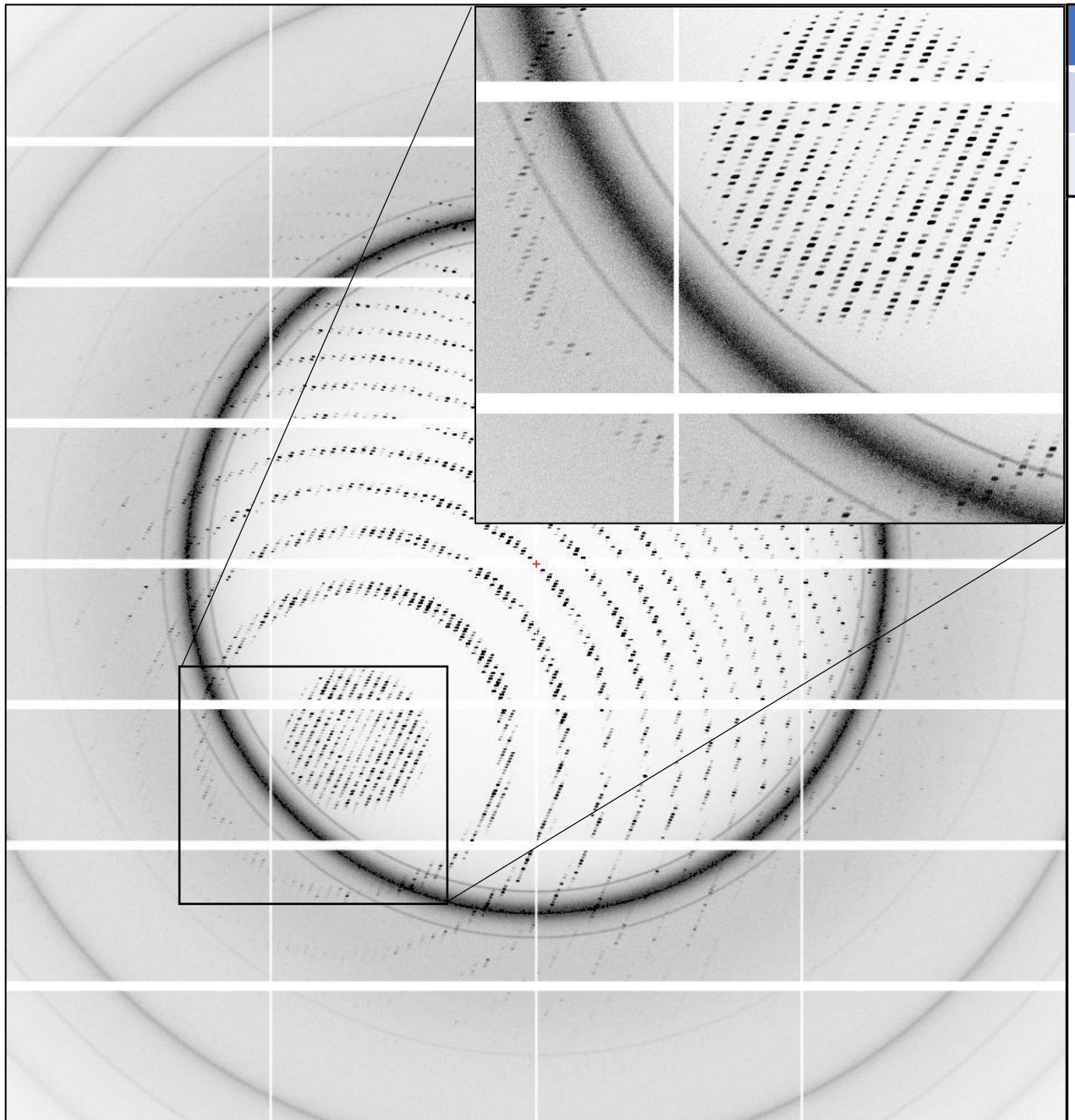
SSA 85

Horizontal and Vertical CRLs

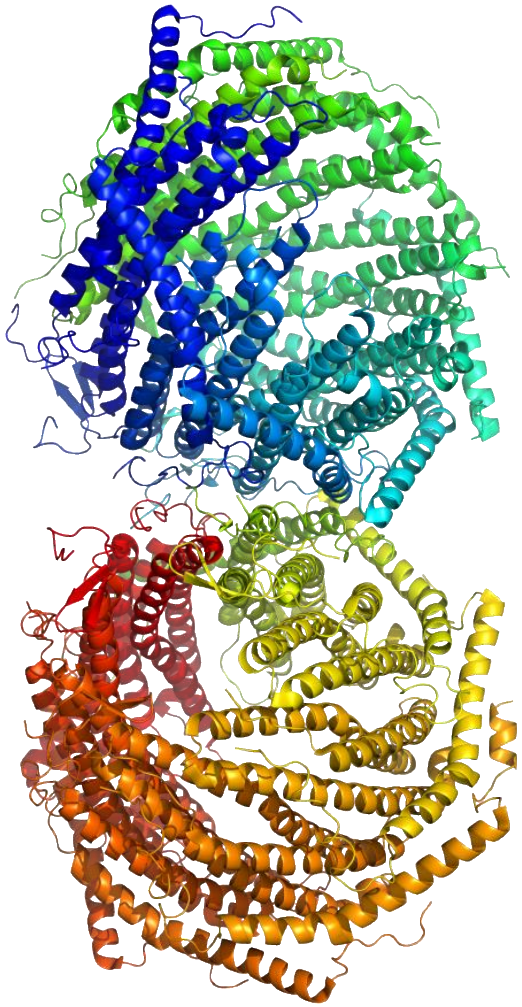
2 x 2 | 6 x 6 | 9 x 9 | 22 x 22 | 42 x 42



What we hope to be able to do with real samples



Length (Å)	Angle (°)
a/b= 166.485	α/β = 90.00
c = 399.162	γ = 120.00



Australian Government

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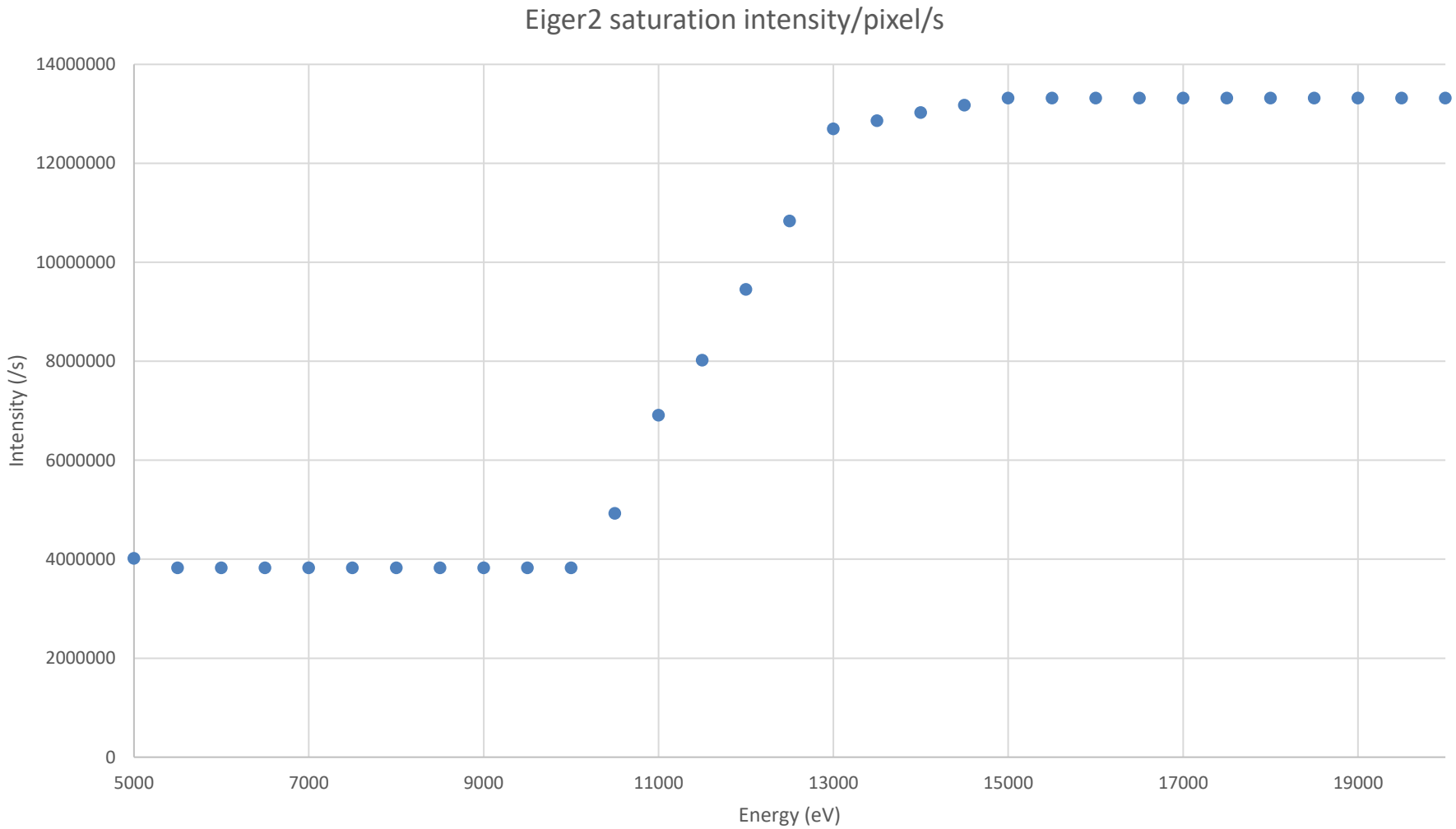
Thank you for your attention

Eiger2 pixel saturation



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

Maximum cell edges.

To date 2229 PDB entries from the two MX beamlines at the AS.
4 contain a unit cell length larger than 266 A (3 viral capsids and vascular adhesion protein 1).
Average of 96.8 A

A design requirement to be able to separate 400 A cell edges is reasonable.