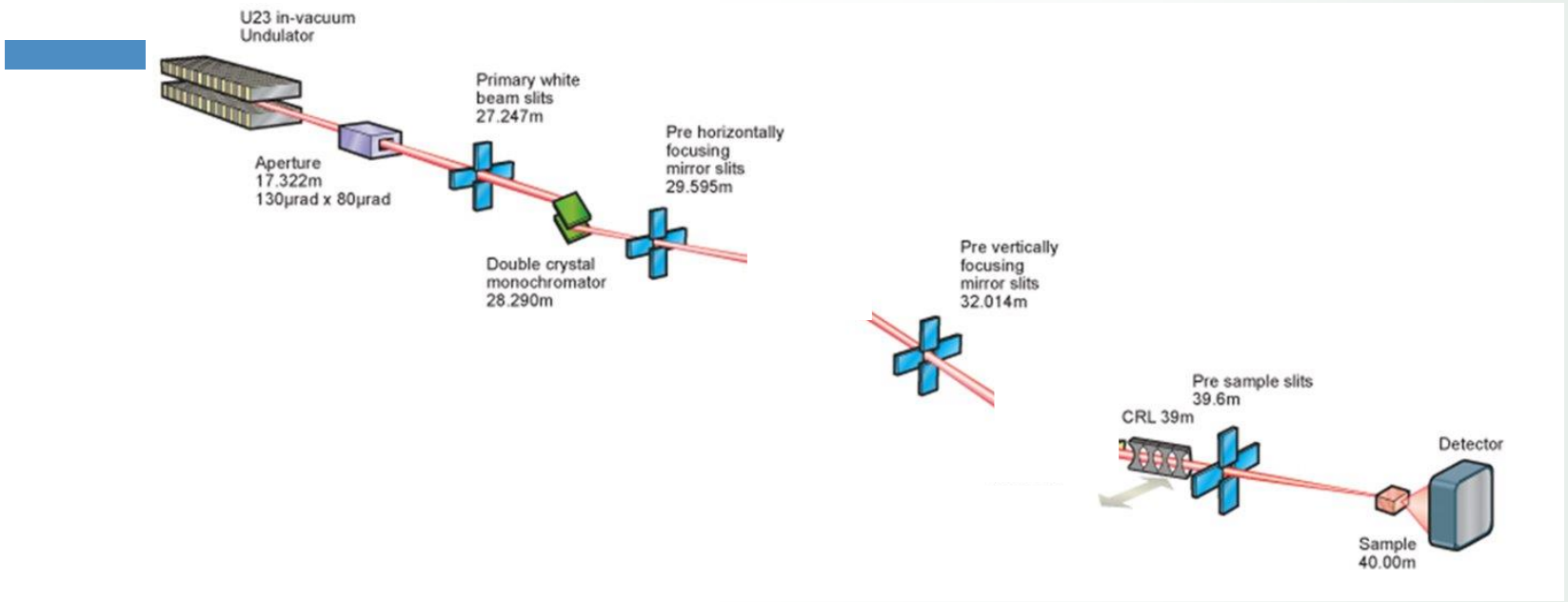
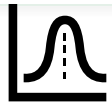


I04 beamline

A versatile & high-throughput instrument that delivers high quality diffraction data

Ralf Flaig
David Aragão
Pierpaolo Romano
Marco Mazzorana

Main reasons to use I04



- Variable focus & microfocus beam

- Variable energy

- High flux/flux density

- Extremely stable beam & hardware

- Multi-axis goniometry

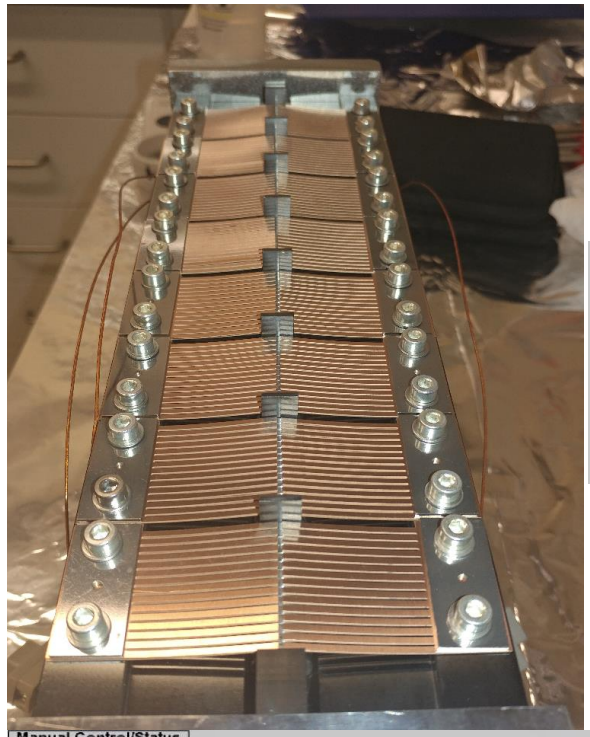
- Dose aware data collection

- Automation and high reliability

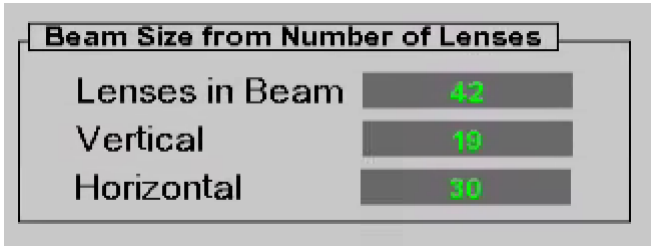
- Interactive locally and remote data collections as well as UDC

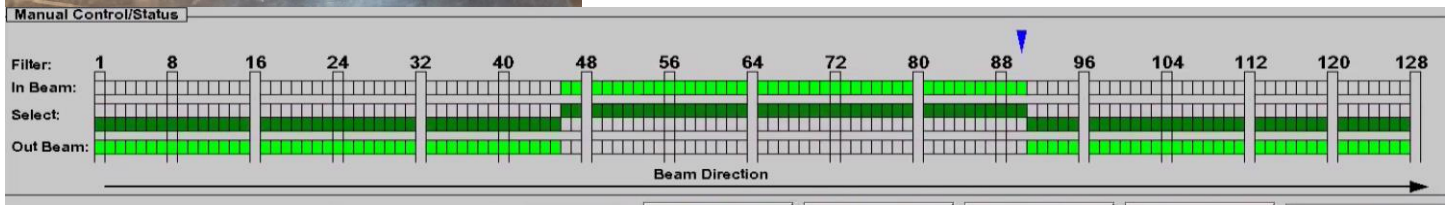
Variable focus & microfocus beam

CRL/lenses base mechanism to focus the beam



13 keV, #42 lenses, 19 um

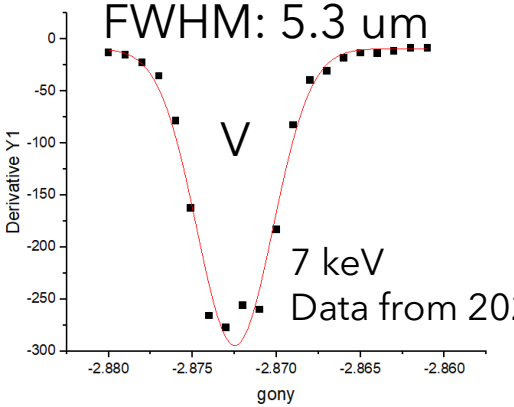


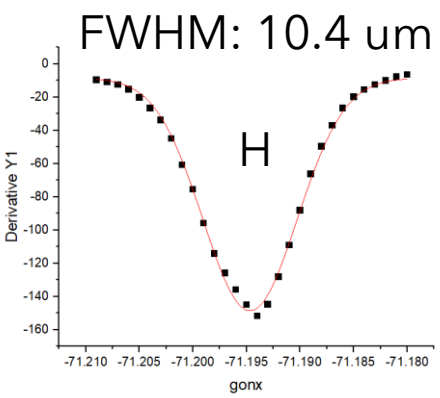


- Fast & easy change from a list of discrete beam sizes
  - Varies with energy but normally (5, 10, 15, 20, 30, 50, 75, 100 μm)

Operate from

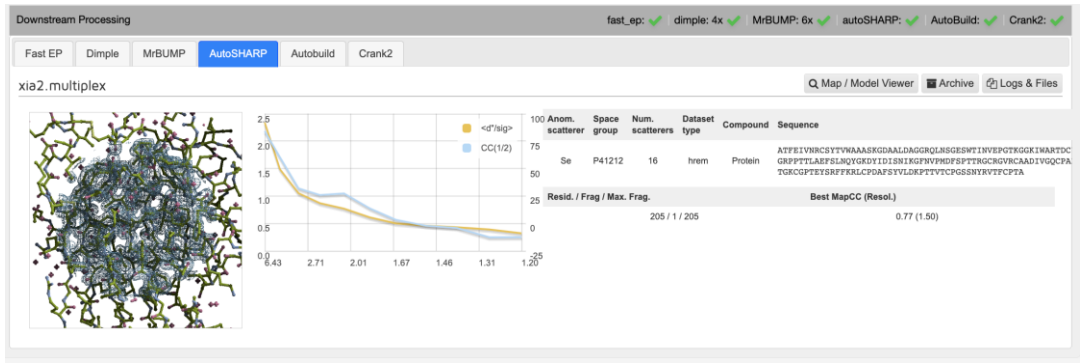
- microfocus level (e.g. 5 μm vertical)
- large beam level (E.g. 100 μm vertical)

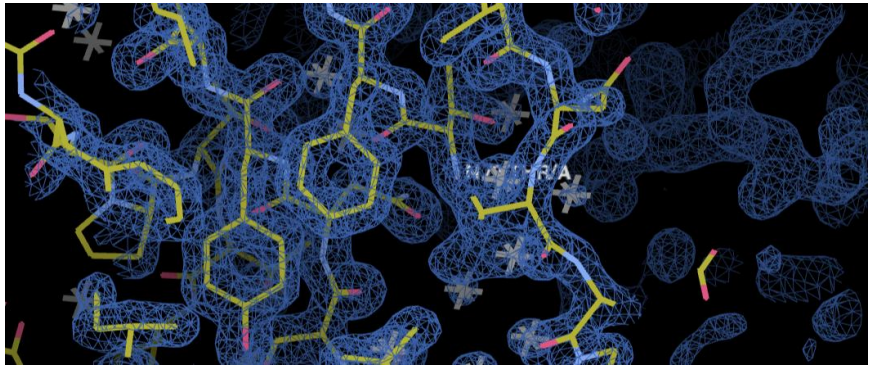




Variable energy (6000-18000 eV / 0.69 -1.55 Å)

- Phasing, edge scans, Fluorescence spectra

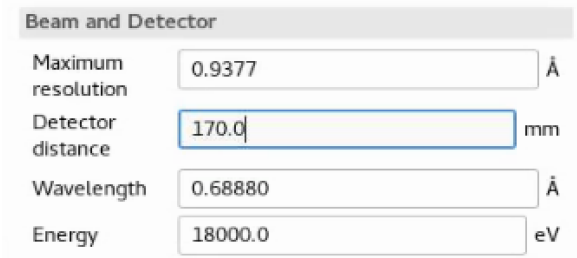


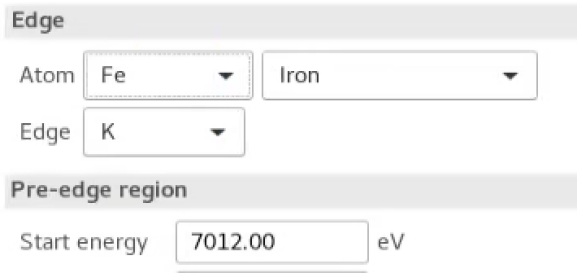


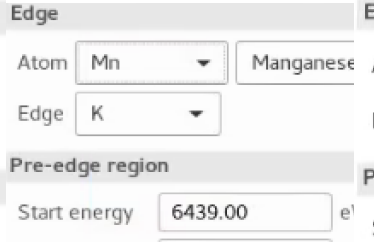
Good insulin crystals solve by S-SAD at 13 keV

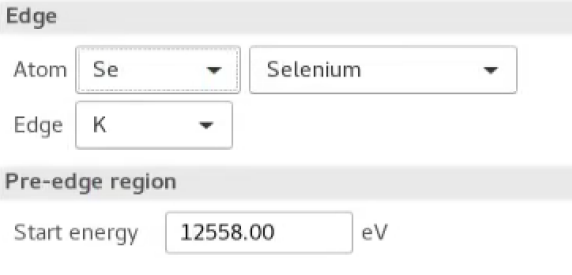
Reaches most common edges: Cu, Fe, Mn, Se, Ni, Br, Os, etc

- Can get diffraction up to 0.94 Å resolution (edge of detector) by going to maximum energy

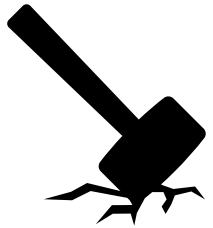




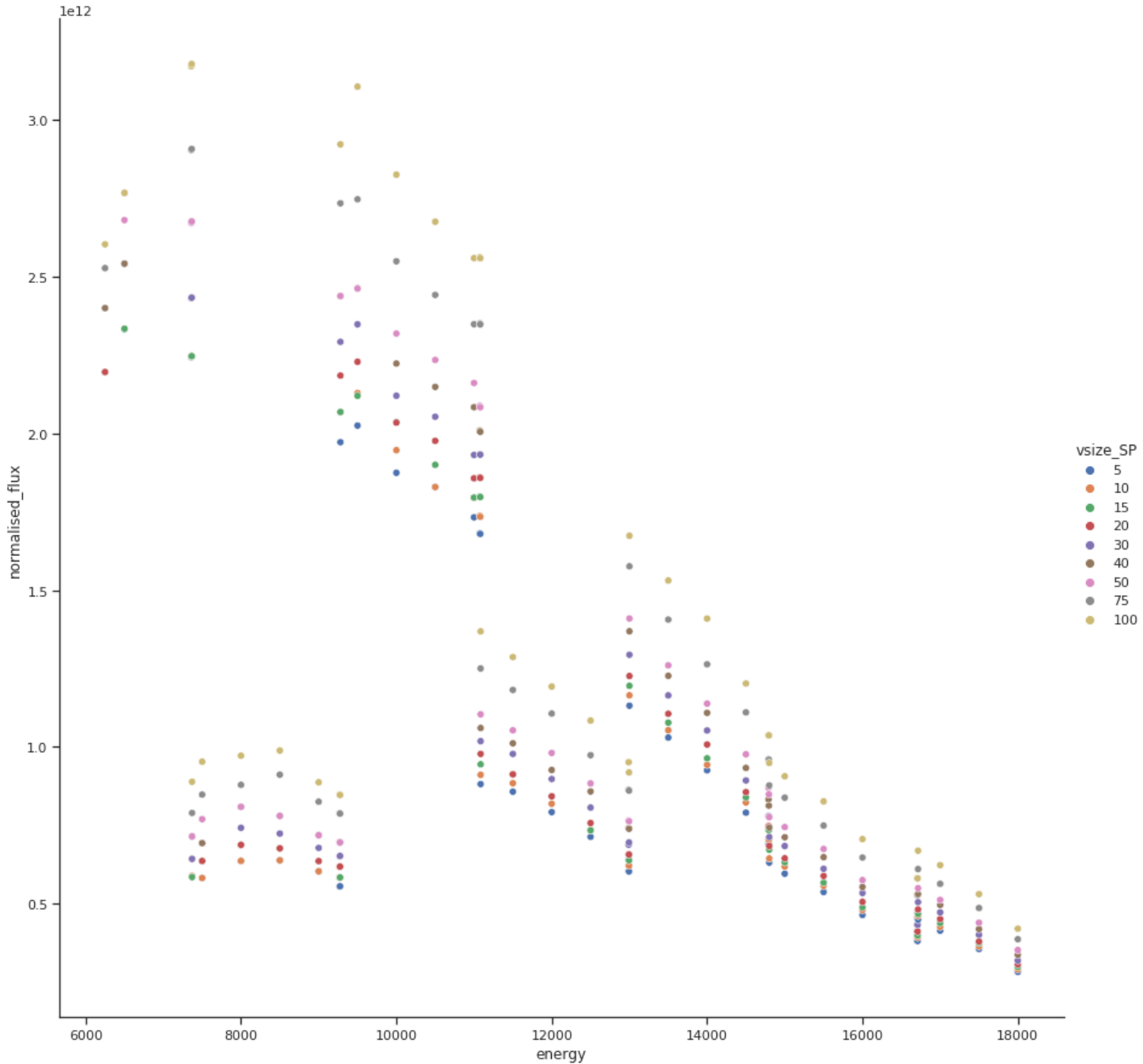


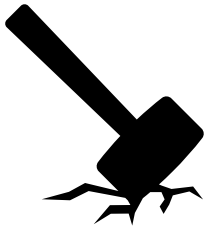


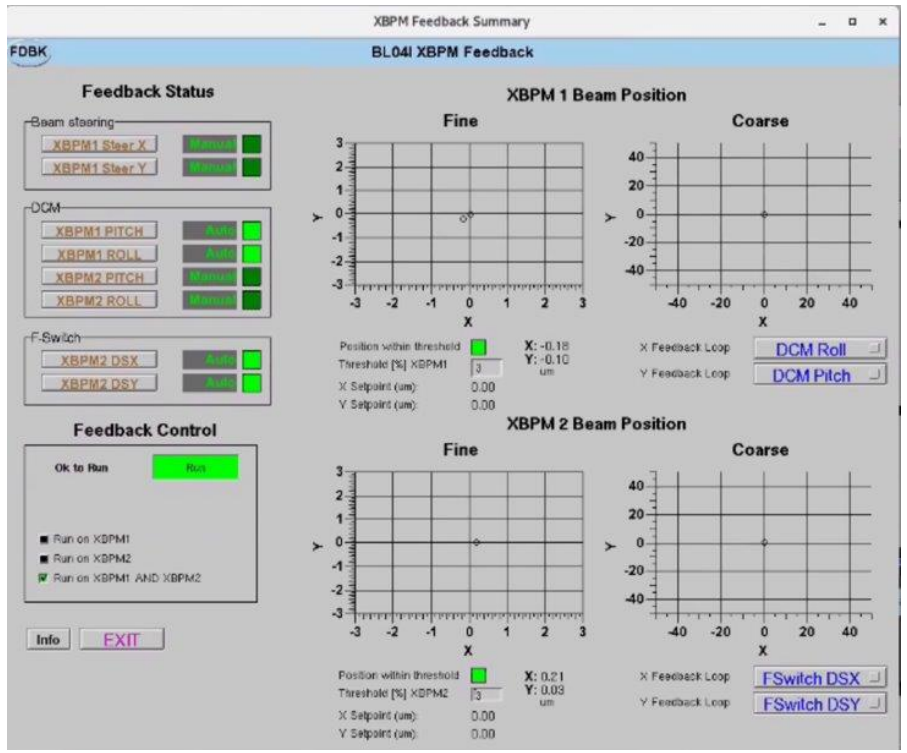
High flux/flux density



- Flux profile with energy & beam sizes
- Eiger has a flux density limit
  - For well diffracting samples the Eiger count rate limit is reached

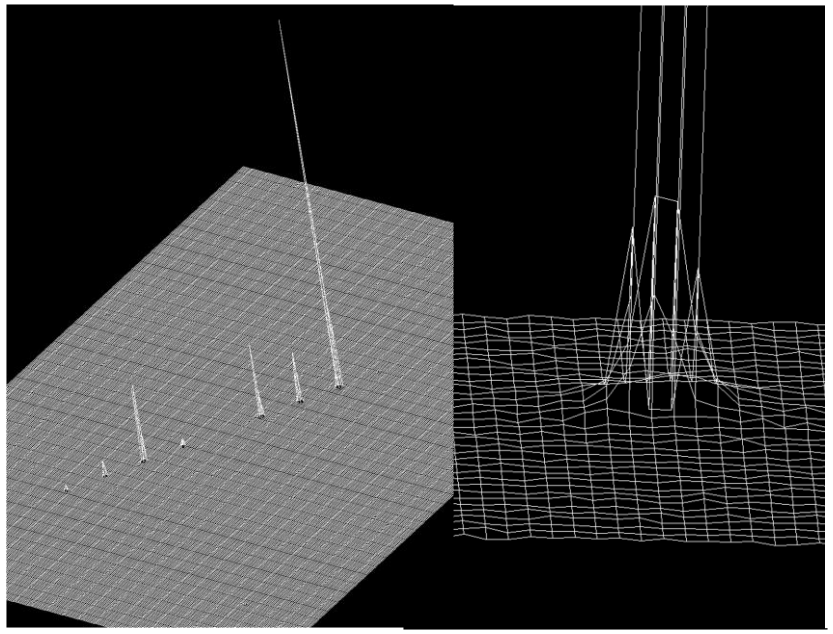

1e12
3.0
2.5
2.0
1.5
1.0
0.5
normalised_flux
6000
8000
10000
12000
14000
16000
18000
energy
vsize_SP
5
10
15
20
30
40
50
75
100



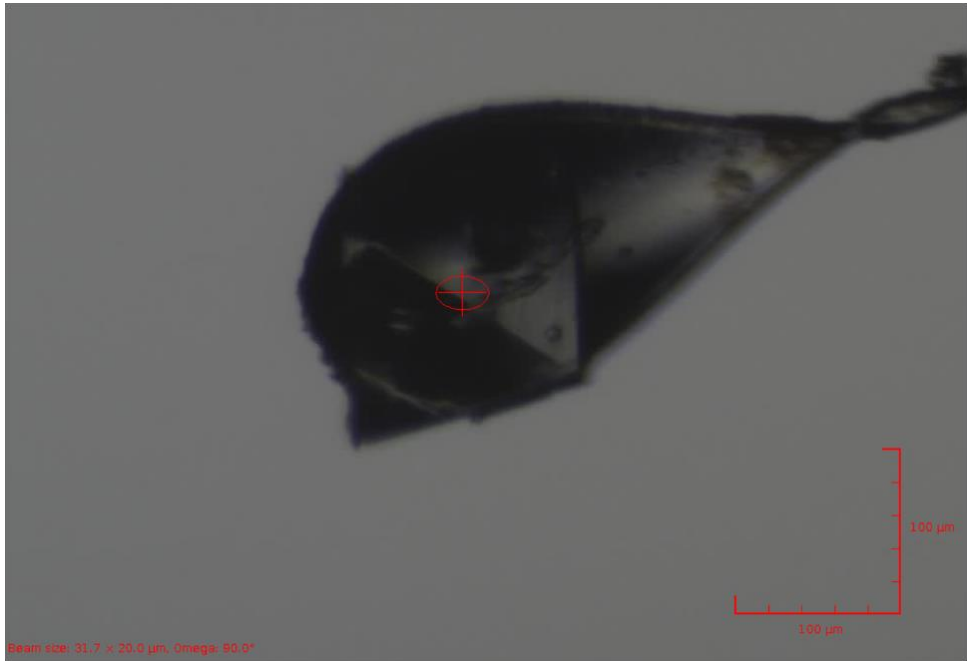

XBPM Feedback Summary
FDBK
BL04I XBPM Feedback
Feedback Status
Beam steering
XBPM1 Steer X
XBPM1 Steer Y
Manual
DCM
XBPM1 PITCH
XBPM1 ROLL
XBPM2 PITCH
XBPM2 ROLL
Auto
F-Switch
XBPM2 DSX
XBPM2 DSY
Feedback Control
Ok to Run
Run
Run on XBPM1
Run on XBPM2
Run on XBPM1 AND XBPM2
Info
EXIT
XBPM 1 Beam Position
Fine
Coarse
Position within threshold
Threshold [%] XBPM1
X Setpoint (um):
Y Setpoint (um):
X: -0.18
Y: -0.10
X Feedback Loop
Y Feedback Loop
DCM Roll
DCM Pitch
XBPM 2 Beam Position
Threshold [%] XBPM2
X: 0.21
Y: 0.03
FSwitch DSX
FSwitch DSY

High flux/flux density

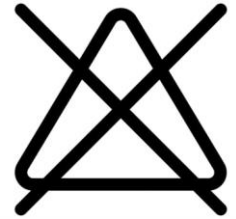
- Eiger has a flux density limit (see https://www.dectris.com/features/features-eiger-r/outstanding-count-rate-performance/)
  - For well diffracting samples (e.g thicker) the Eiger count rate limit is exceeded



22	20	16	26	32	25	36	39	34	18	25	15
16	18	23	26	35	48	52	40	41	45	14	19
17	17	13	27	31	55	83	86	66	56	29	21
13	8	13	23	38	176	549	362	164	46	25	21
17	17	16	22	52	627	-1	57155	386	32	24	21
19	12	34	28	65	611	-1	61246	248	38	30	26
15	16	19	27	64	214	429	260	70	39	21	19
16	23	25	33	43	73	97	95	50	39	28	24
9	22	25	23	35	50	38	37	42	26	25	15
20	19	13	13	29	31	31	34	25	37	27	20
17	11	13	21	25	18	25	21	29	11	27	16
20	9	17	14	18	20	22	36	20	28	27	14
14	18	15	23	16	20	17	18	18	11	19	17
9	13	23	12	18	14	17	27	13	14	22	16
18	15	20	17	18	17	21	19	18	12	15	16



Extremely stable beam & hardware



360° / 7.2 s / 500 Hz

Ω Osc: 0.10°

No. Images: 3600

Wavelength: 0.9537Å

Dose: 1.50MGy

Beamsize: 32x20µm

Feb 2023 • Data quality in general very high at fastest speed

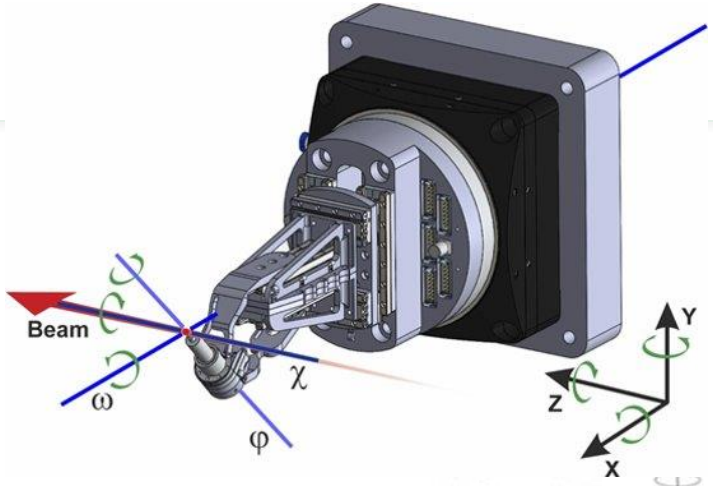
Shell	Observations	Unique	Resolution	Rmeas	I/sig(I)	CC Half	Completeness	Multiplicity
outerShell	44923	1841	1.35 - 1.39	0.755	4.2	0.9	97.0	24.4
innerShell	8126	364	6.05 - 28.03	0.023	134.0	1.0	99.1	22.3
overall	682225	26292	1.35 - 28.03	0.051	37.6	1.0	99.8	25.9

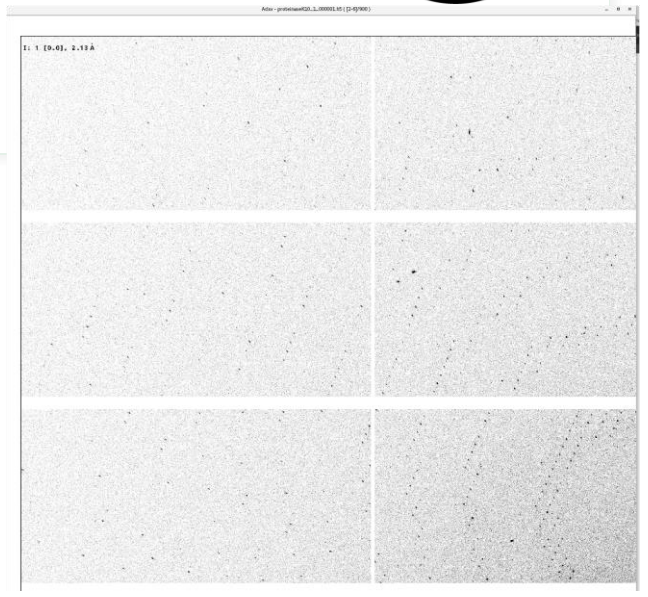
ISa
41.92

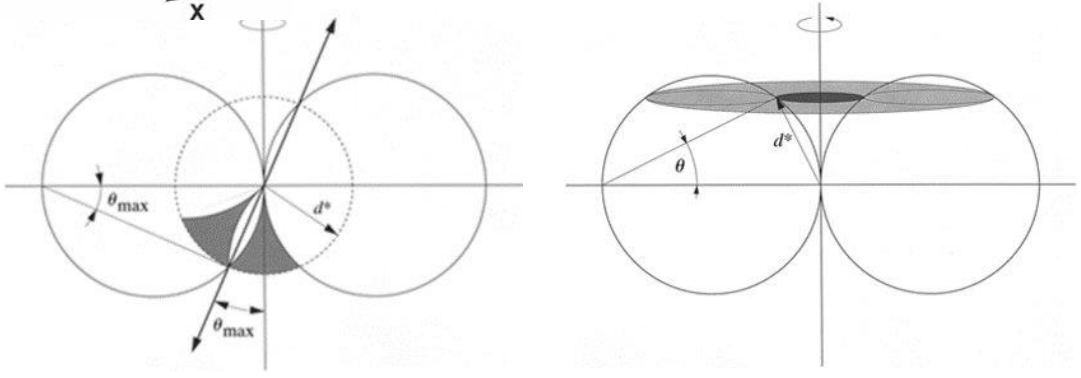
• Data quality even higher when lower speed and slightly larger beam size

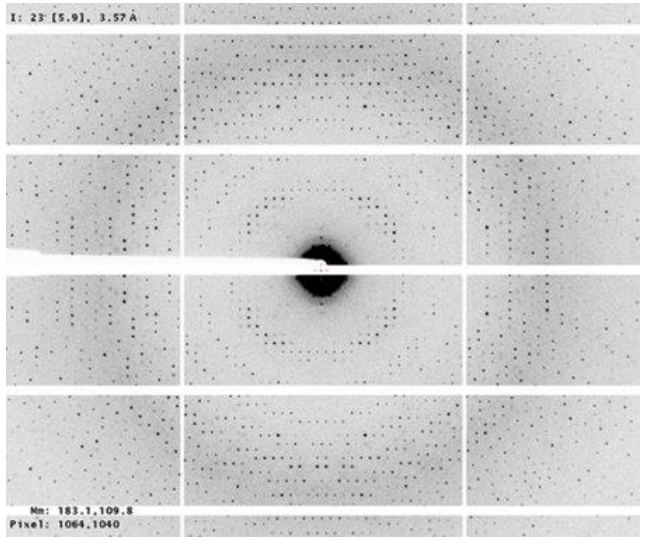
Dose (Mgy)	1.5	1.5	1.5	1.5	1.33	0.72	0.46	0.37
Beam size (um HxV)	32x20	43x30	54x40	63x50	32x20	43x30	54x40	63x50
Speed (Hz)	500	500	500	370	500	500	500	500
ISa	33.8	38.8	43.6	44.4	41.6	45.0	46.7	45.9

Multi-axis goniometry



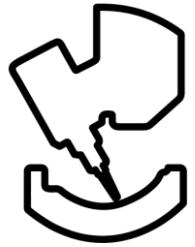






- Blind region
- Use of corners for completeness (e.g. higher res)
- High symmetry cell axis aligned

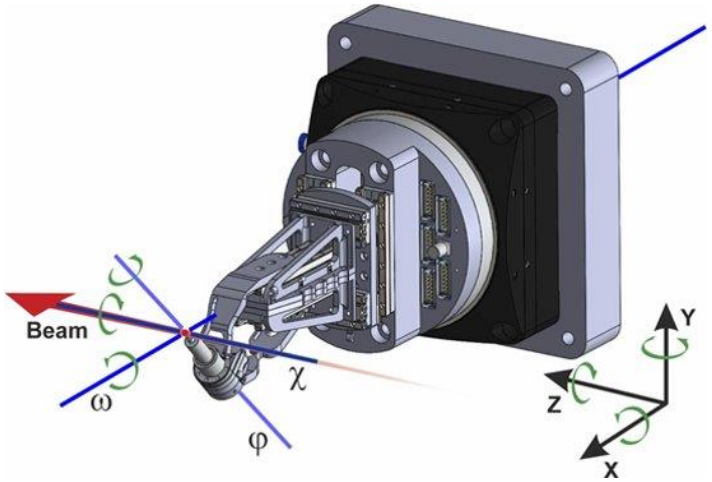
Multi-axis goniometry



- Lower systematic errors & redundancy
  - Spread of the dose is something that makes sense with modern detectors. Using multi-axis makes sense too because only adds and does not remove
- Complicated experiments (e.g. custom phasing recipe)

Omega start (°)	Omega Oscillation (°)	Omega oscillation per frame (°)	Chi (°)	Phi (°)
0	2x180 (360)	0.1	0	0
0	2x180 (360)	0.1	30	0

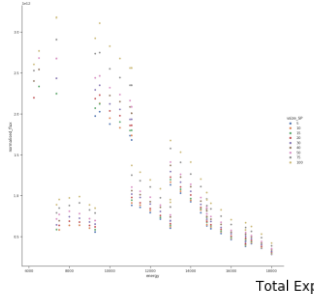
Omega Start (°)	Omega Oscillatio (°)	Omega Delta (°)	Chi (°)	Phi (°)
0.00	0.100	0.00	44.467	148.321
0.00	0.100	0.00	0.000	0.000
0.00	0.100	0.00	45.000	0.000
0.00	0.100	0.00	45.000	120.000
0.00	0.100	0.00	45.000	240.000



Dose aware data collection

- Dose as an extra information
- Dose has a tool to drive the experiment
- Dose is critical to setup an experiment with different beam size, energy or when flux is different from usual

Dose aware data collection



Start	0.00 °
Oscillation	0.100 °
Total oscillation	360.0 °
Delta	0.00 °
Chi	0.000 °
Phi	0.000 °

Dose and Exposure

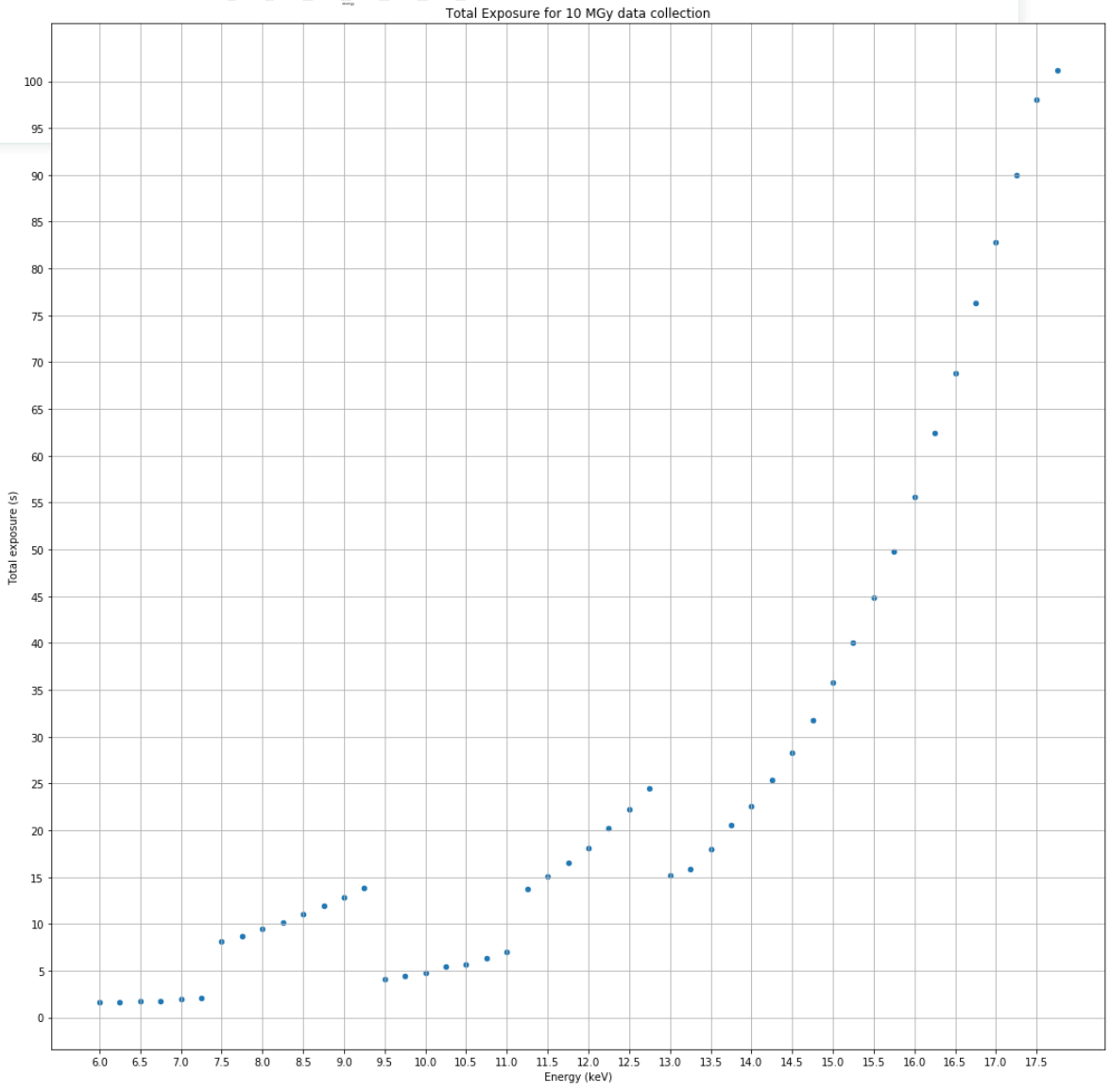
Number of Images	3600
	◉ Set Target Dose
	○ Set Target Exposure
Exposure Time	0.0030 s
Total Exposure Time	10.8 s
Dose / Dataset	7 MGy
First Image Number	1

Beam and Detector

Maximum resolution	1.9023 Å
Detector distance	289.3 mm
Wavelength	0.95373 Å
Energy	12999.9 eV
	☐ Use current energy
Transmission	100.000000 %

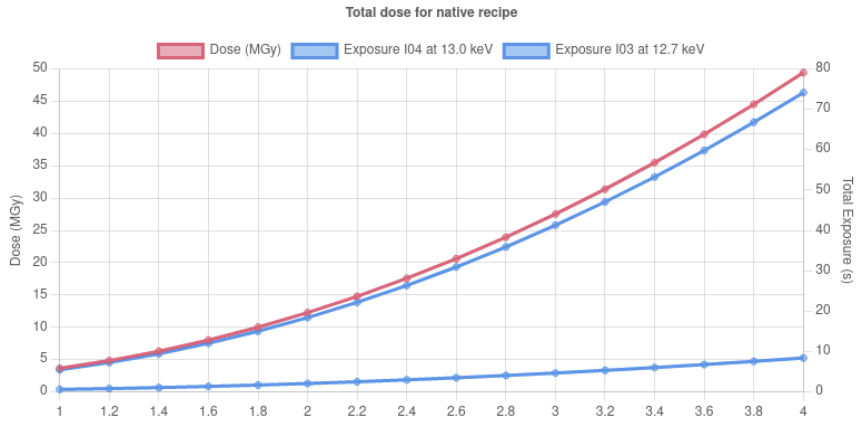
Aperture and Beamstop

Beamstop	Standard
Horizontal beam size	31.73 µm
Vertical beam size	20 µm
	☑ Use current beam size



Dose

- Changing beam size



Data Collection Settings

Position 2

Files

Visit directory
/dls/i04/data/2021/cm28182-1
Folder Configure Defaults
${date}/${proteinacronym}/${samplename}/
20210308/TestThermolysin/Themolysin2
Prefix Configure Defaults
${samplename}
Themolysin2
Automatic run number
Run number 0
Comment

Flux and Dose (beta version)

Predicted Flux 3.237e+11 ph s⁻¹
Average Absorption Coefficient 0.185775
Calculated Dose / Dataset 22.8239 MGy

Sample Position

Image

Number of images 3600
Exposure time 0.0100 s
Total exposure time 36.0 s
First image number 1

Beam and Detector

Maximum resolution 1.3369 Å
Detector distance 170.0 mm
Wavelength 0.97949 Å
Energy 12658.0 eV
Use current energy
Transmission 100.000000 %

Aperture and Beamstop

Beamstop Standard
Horizontal beam size 18.8 µm
Vertical beam size 10 µm
Use current beam size

Data Collection Settings

Position 2

Files

Visit directory
/dls/i04/data/2021/cm28182-1
Folder Configure Defaults
${date}/${proteinacronym}/${samplename}/
20210308/TestThermolysin/Themolysin2
Prefix Configure Defaults
${samplename}
Themolysin2
Automatic run number
Run number 0
Comment

Flux and Dose (beta version)

Predicted Flux 3.237e+11 ph s⁻¹
Average Absorption Coefficient 0.185775
Calculated Dose / Dataset 6.7680 MGy

Sample Position

Image

Number of images 3600
Exposure time 0.0100 s
Total exposure time 36.0 s
First image number 1

Beam and Detector

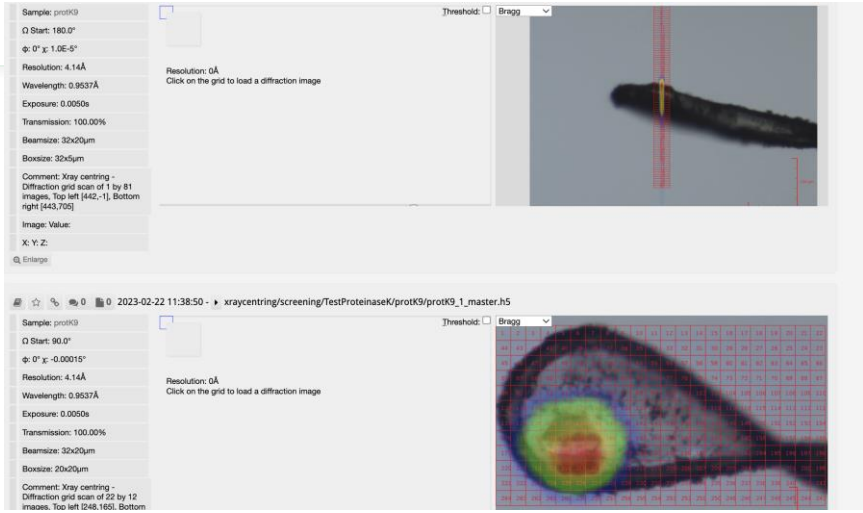
Maximum resolution 1.3369 Å
Detector distance 170.0 mm
Wavelength 0.97949 Å
Energy 12658.0 eV
Use current energy
Transmission 100.000000 %

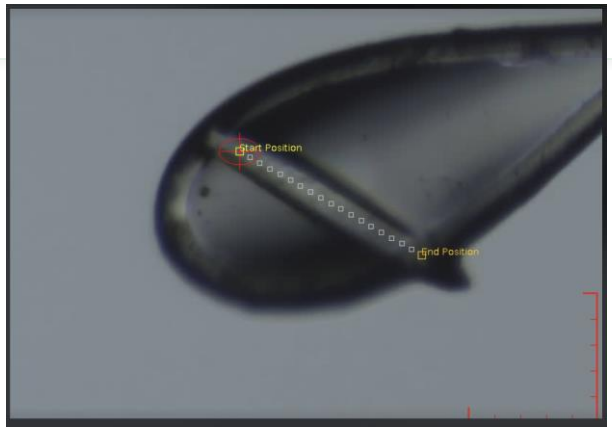
Aperture and Beamstop

Beamstop Standard
Horizontal beam size 31.7 µm
Vertical beam size 20 µm
Use current beam size

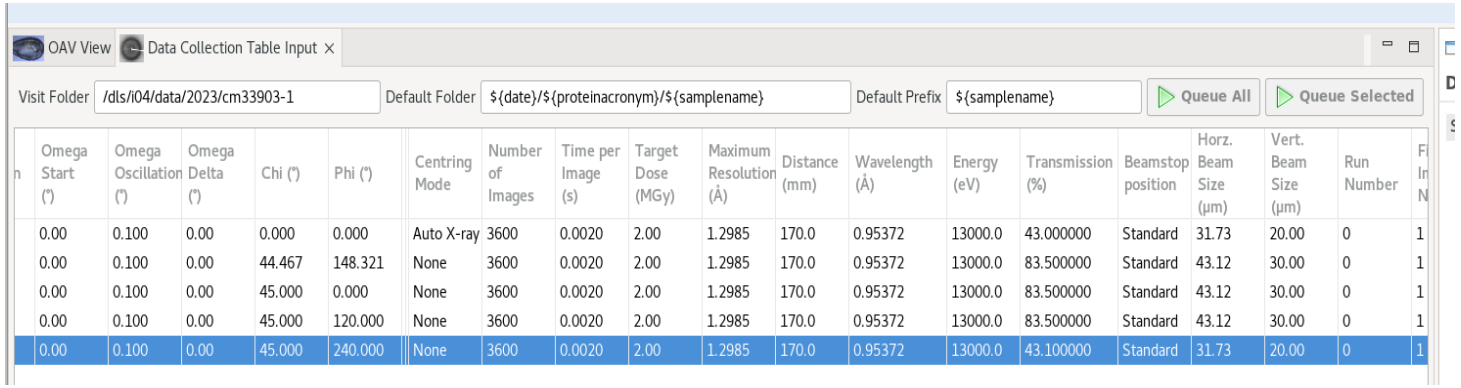
Automation and high reliability

- Xray centering
- Line scans
- Data collection queue
- UDC





Omega Start (°)	Omega Oscillatio (°)	Omega Delta (°)	Chi (°)	Phi (°)
0.00	0.100	0.00	44.467	148.321
0.00	0.100	0.00	0.000	0.000
0.00	0.100	0.00	45.000	0.000
0.00	0.100	0.00	45.000	120.000
0.00	0.100	0.00	45.000	240.000



Omega Start (°)	Omega Oscillation (°)	Omega Delta (°)	Chi (°)	Phi (°)	Centring Mode	Number of Images	Time per Image (s)	Target Dose (MGy)	Maximum Resolution (Å)	Distance (mm)	Wavelength (Å)	Energy (eV)	Transmission (%)	Beamstop position	Horz. Beam Size (µm)	Vert. Beam Size (µm)	Run Number
0.00	0.100	0.00	0.000	0.000	Auto X-ray	3600	0.0020	2.00	1.2985	170.0	0.95372	13000.0	43.000000	Standard	31.73	20.00	0
0.00	0.100	0.00	44.467	148.321	None	3600	0.0020	2.00	1.2985	170.0	0.95372	13000.0	83.500000	Standard	43.12	30.00	0
0.00	0.100	0.00	45.000	0.000	None	3600	0.0020	2.00	1.2985	170.0	0.95372	13000.0	83.500000	Standard	43.12	30.00	0
0.00	0.100	0.00	45.000	120.000	None	3600	0.0020	2.00	1.2985	170.0	0.95372	13000.0	83.500000	Standard	43.12	30.00	0
0.00	0.100	0.00	45.000	240.000	None	3600	0.0020	2.00	1.2985	170.0	0.95372	13000.0	43.100000	Standard	31.73	20.00	0

At our fastest speed (7.2 seconds exposure per dataset) we do 2 pucks (32 pins) per hour (single 360° wedge). This equates to 750 samples loaded and a data set collected in a day.

Interactive locally and remote data collections as well as UDC




- We train local users (*)


- We help remote users if requested (e.g via zoom) (*)


- We operate all currently available UDC recipes

(*) Please remember to note that, at scheduling time, so that a suitable time and local contact is allocated