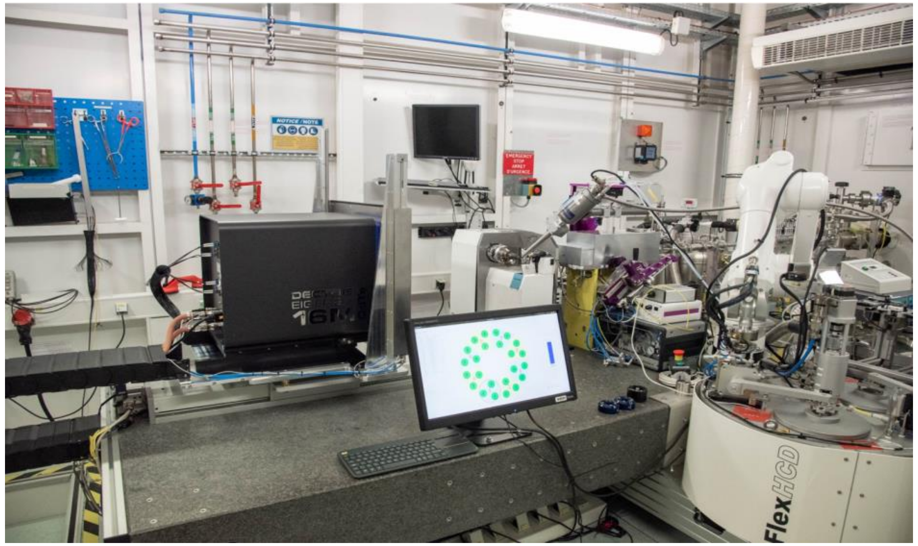
ID23-1 beamline report 2021

A. Popov & G. Santoni

ESRF | The European Synchrotron



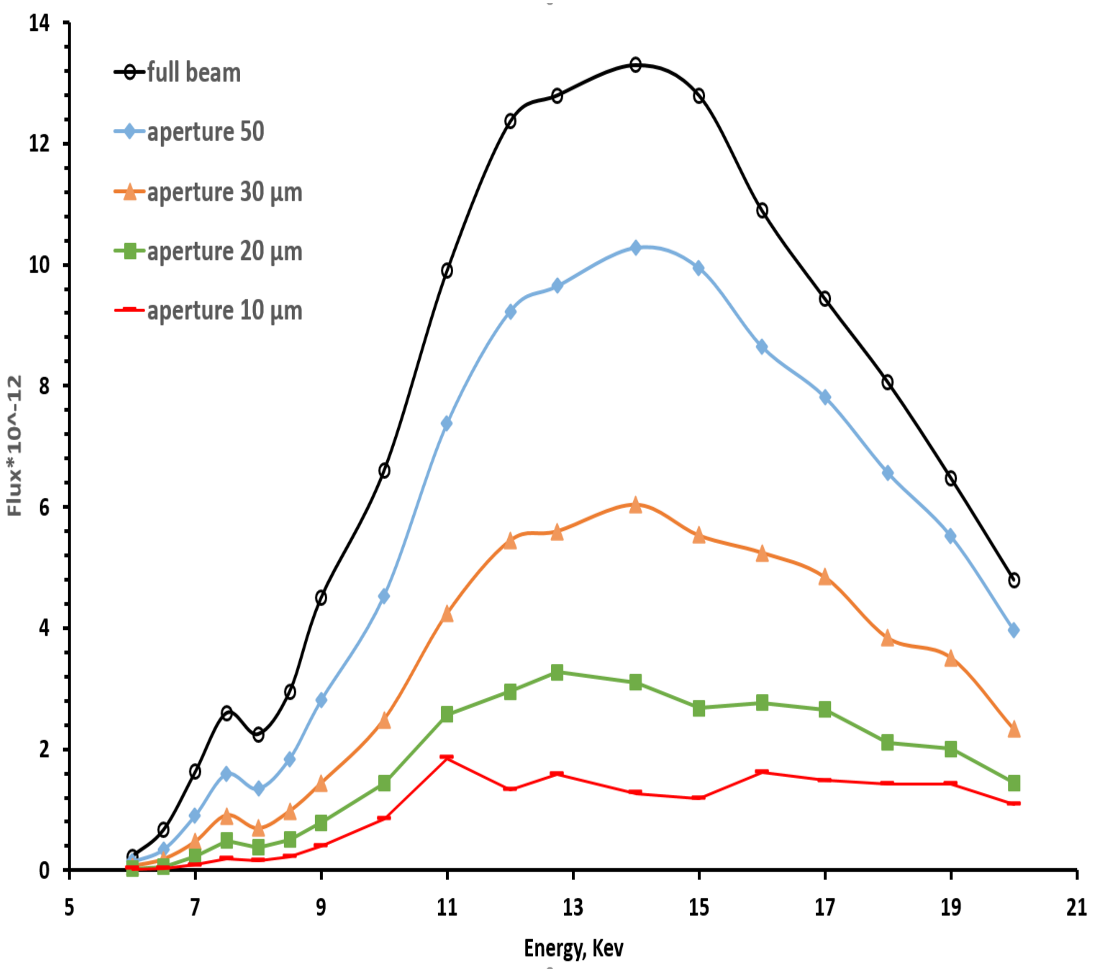
NEW EXPERIMENTAL SETUP

New ID23-1 sample environment is composed of a MD2-S micro-diffractometer and an EIGER2 X 16M detector with a CdTe sensor. The temperature at the sample position is controlled using new Cryostream 800 series cryocooler (temperature range 80–400K; Oxford Cryosystems, Oxford, UK).

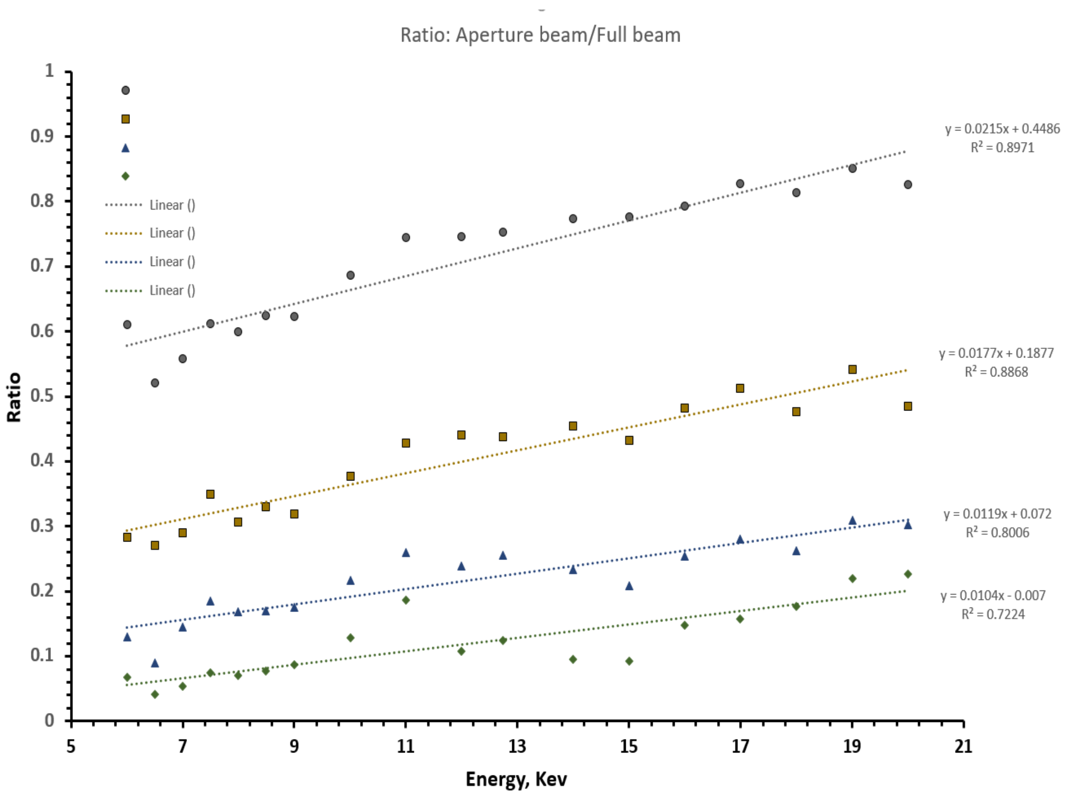
Diffractometer	MD2-S micro-diffractometer (Arinax).
Sample Changer	FlexHCD
Fast Shutter	Piezo-based millisecond shutter
Beam-defining apertures	Radius 50 µm, 30 µm, 20 µm, 10 µm
Fluorescence Detector	AXAS-A (KETEK GmbH).
Detector	EIGER2 X CdTe 16M, active area 311 x 327 mm^2 , 4148 x 4362 pixels (75 microns in size), 120 images per second
Experiment Control	MXCuBE3

EXPERIMENTAL SETUP

Photon flux (photons/s) at the ID23-1 sample position for different apertures as a function of energy. The storage ring current is ~200mA.

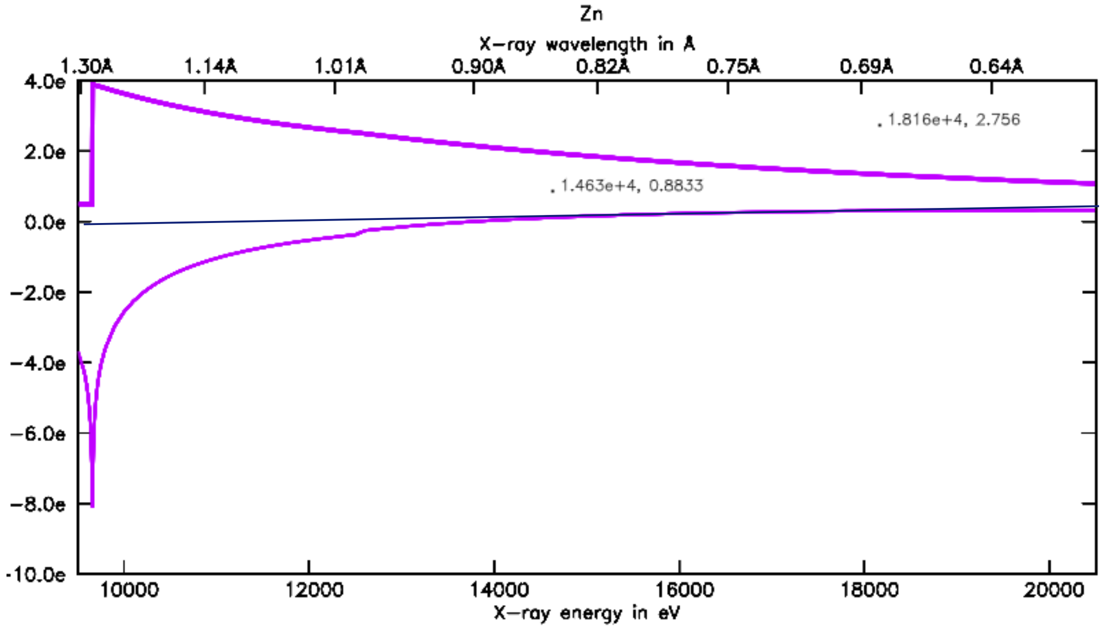


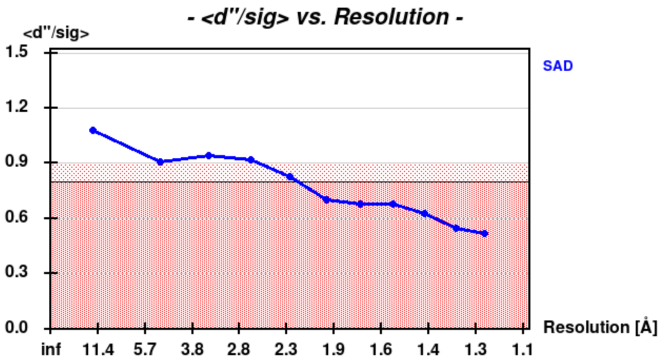
Photon flux reduction according to aperture size as a function of X-ray energy

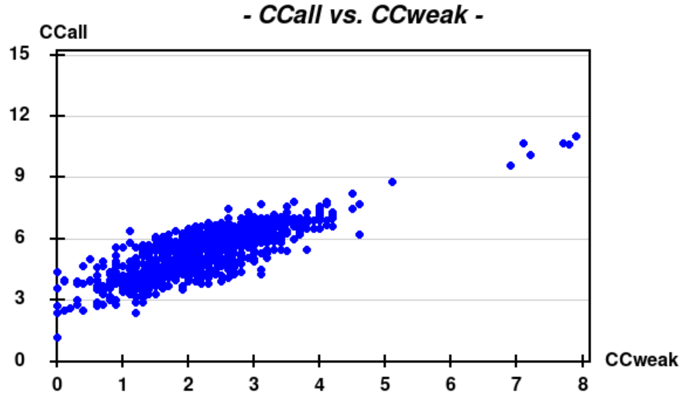


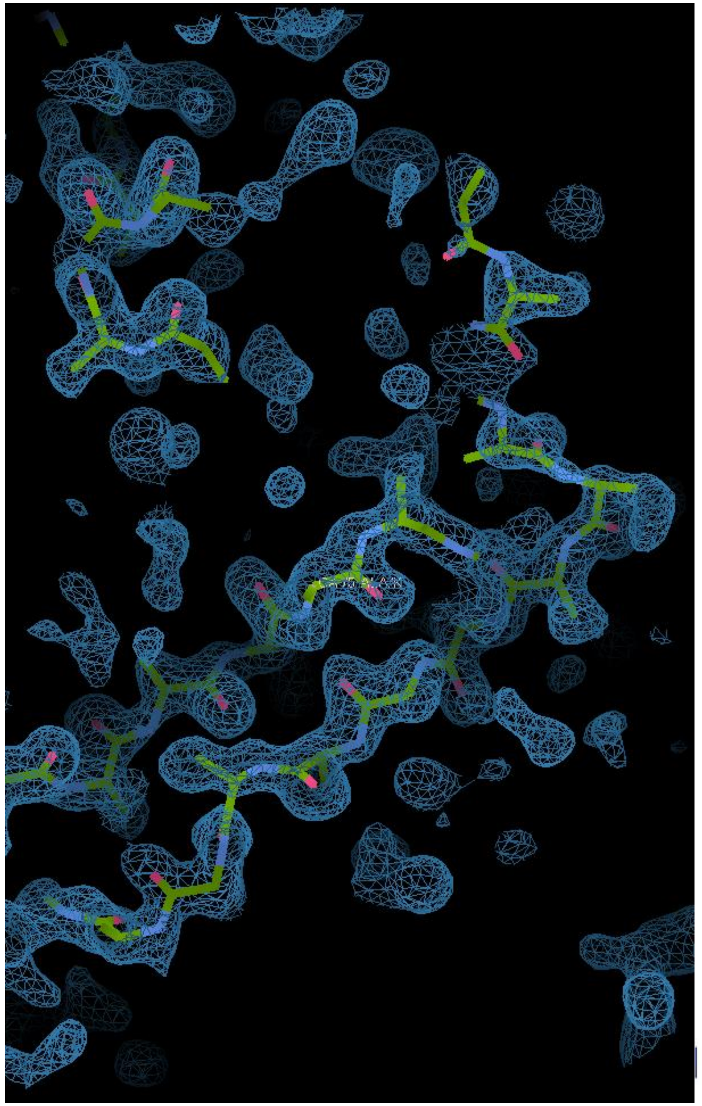
HIGHER X-RAY ENERGIES FOR MACROMOLECULAR CRYSTALLOGRAPHY

Thermolysin, 20 keV





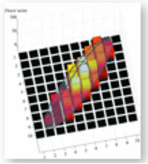




ESRF

METHOD DEVELOPMENTS: X-RAY CRYSTAL RECOGNITION

Acta Cryst. (2018). D**74**, 355-365
https://doi.org/10.1107/S2059798318002735

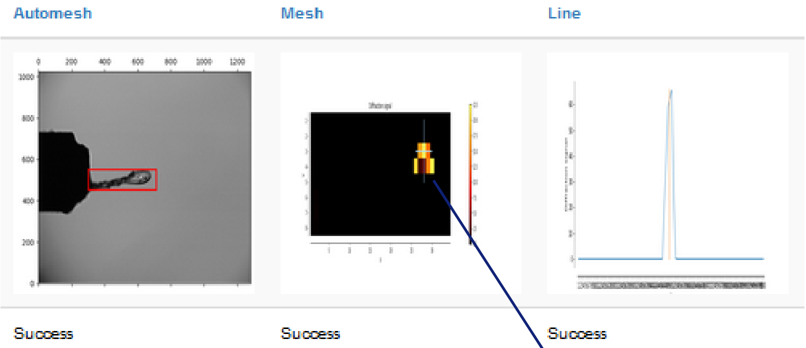


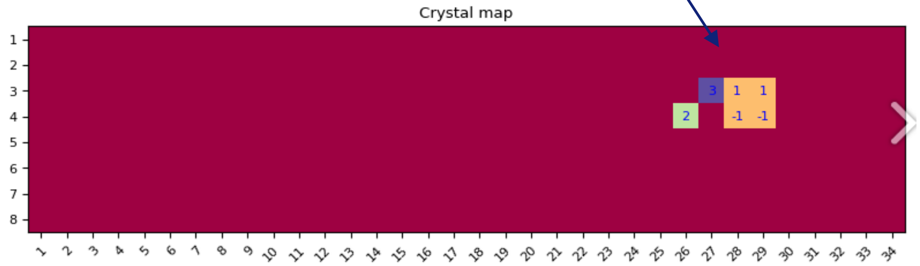
The complex analysis of X-ray mesh scans for macromolecular crystallography

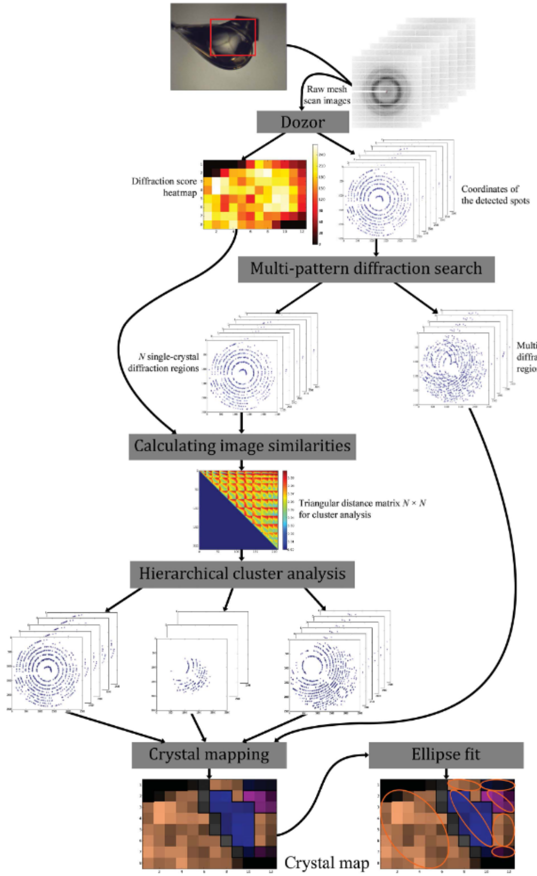
I. Melnikov, O. Svensson, G. Bourenkov, G. Leonard and A. Popov

In macromolecular crystallography, mesh (raster) scans are carried out either as part of X-ray-basec
which diffraction images can be collected. Here, the methods used in *MeshBest*, software which autc
produces a two-dimensional crystal map showing estimates of the dimensions, centre positions anc
presented. Sample regions producing diffraction images resulting from the superposition of more th

X-ray centering







igure 1
)verview of the workflow of the method. Each X-ray mesh scan produces $N_{rows} \times N_{columns}$ diffraction images. These are individually analysed by *Dozor*, vhich produces an estimate of the diffraction signal and determines a list of diffraction-spot coordinates and their partial intensities in each image.

ID23-1 MXPRESSE

osc 02-02-2022 14:12:20
/data/id23eh1/inhouse/opid231/20220202/RAW_DATA/Sample-2-1-04/

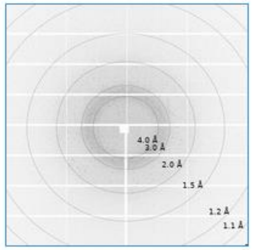
Summary | Beamline Parameters | Data Collections 5 | Sample | Last Collect Results 17 | Workflow 6

Workflow	MXPressE
Protein	
Sample	
Prefix	opid231
Run #	1
# Images (Total)	2440 (3105)
Transmission	100.0 %

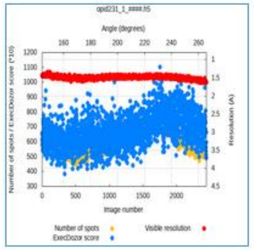
Res. (corner)	1.31 Å (1.03 Å)
En. (Wave.)	14.000 keV (0.8856 Å)
Omega range	0.05 °
Omega start (total)	144.00 ° (122°)
Exposure Time	0.016 s
Flux start	3.51e+11 ph/sec
Flux end	3.45e+11 ph/sec

P 21 21 21	Res.	Compl.	Rmerge
Overall	99.2%	74.4-1.5	4.1
Inner	96.7%	74.4-8.2	3.1
Outer	93.2%	1.52-1.50	109.6

a	b	c
70.13 Å	74.44 Å	77.54 Å
α	β	γ
90 °	90 °	90 °

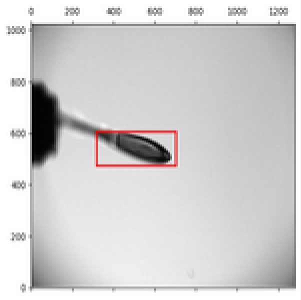
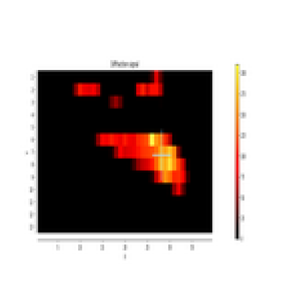
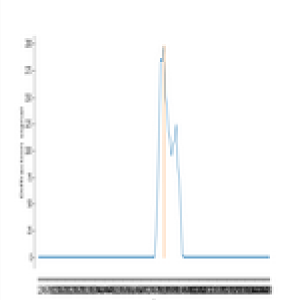
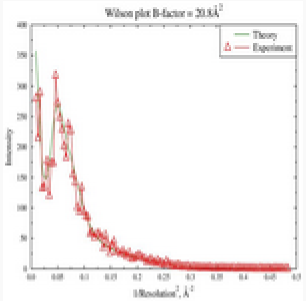
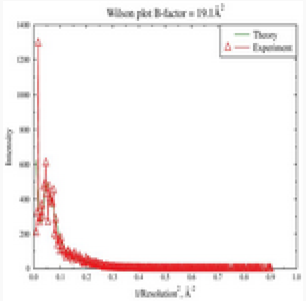


Hover the mouse over for alternate view

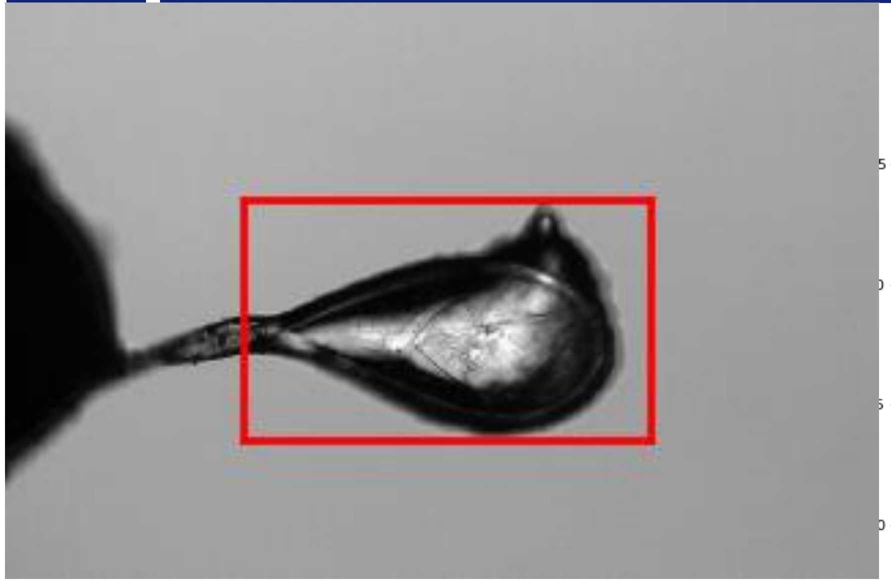


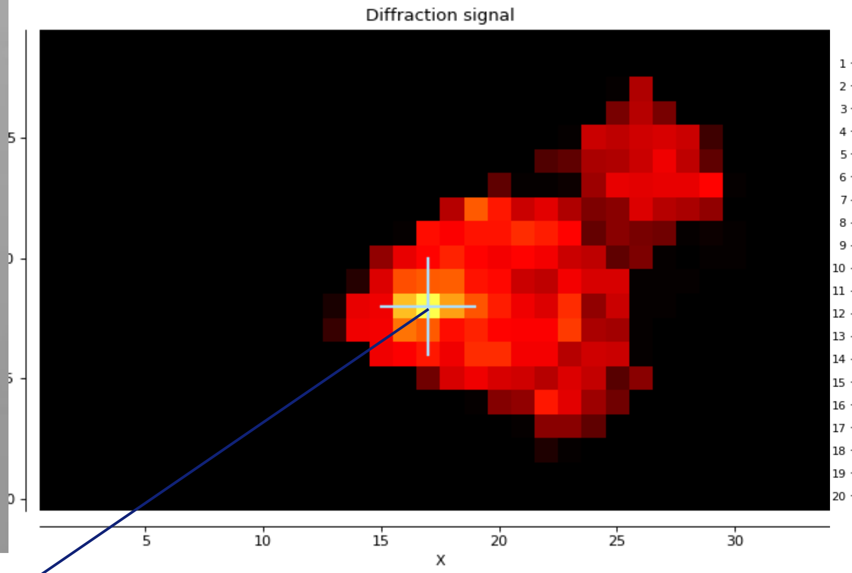
Comments:

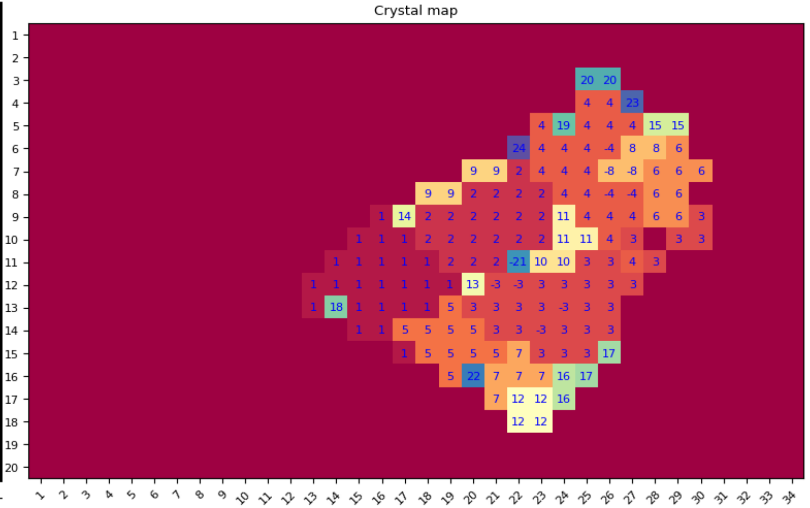
osc 02-02-2022 14:12:20
/data/id23eh1/inhouse/opid231/20220202/RAW_DATA/Sample-2-1-04/

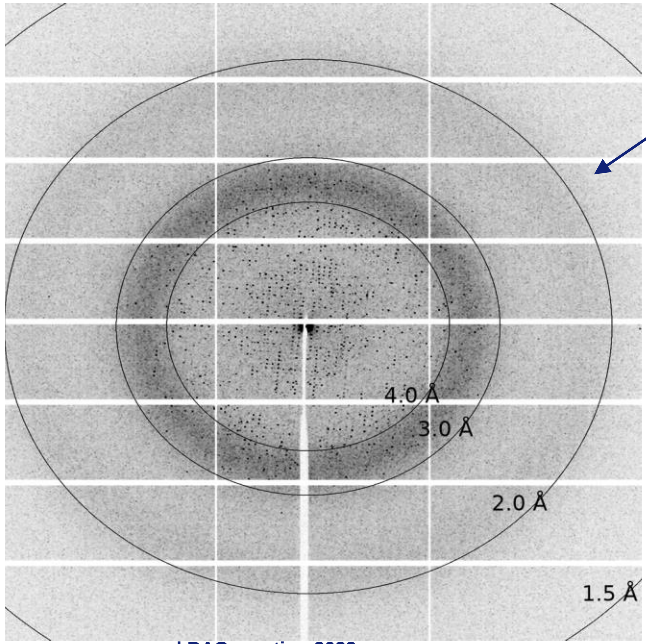
Snapshots	Automesh	Mesh	Line	Characterisation	Characterisation
					
Success	Success	Success	Success	Success	Success

X-RAY CRYSTAL RECOGNITION DOZORM2 –two or more scans analysis



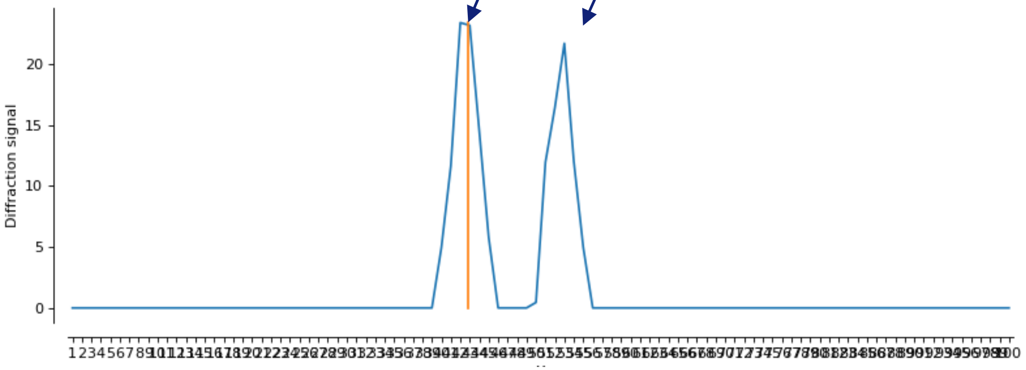


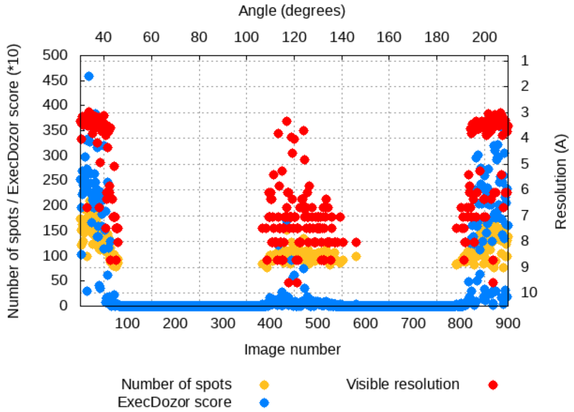




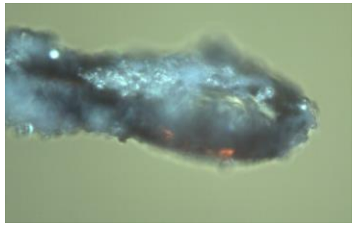
90 º ?

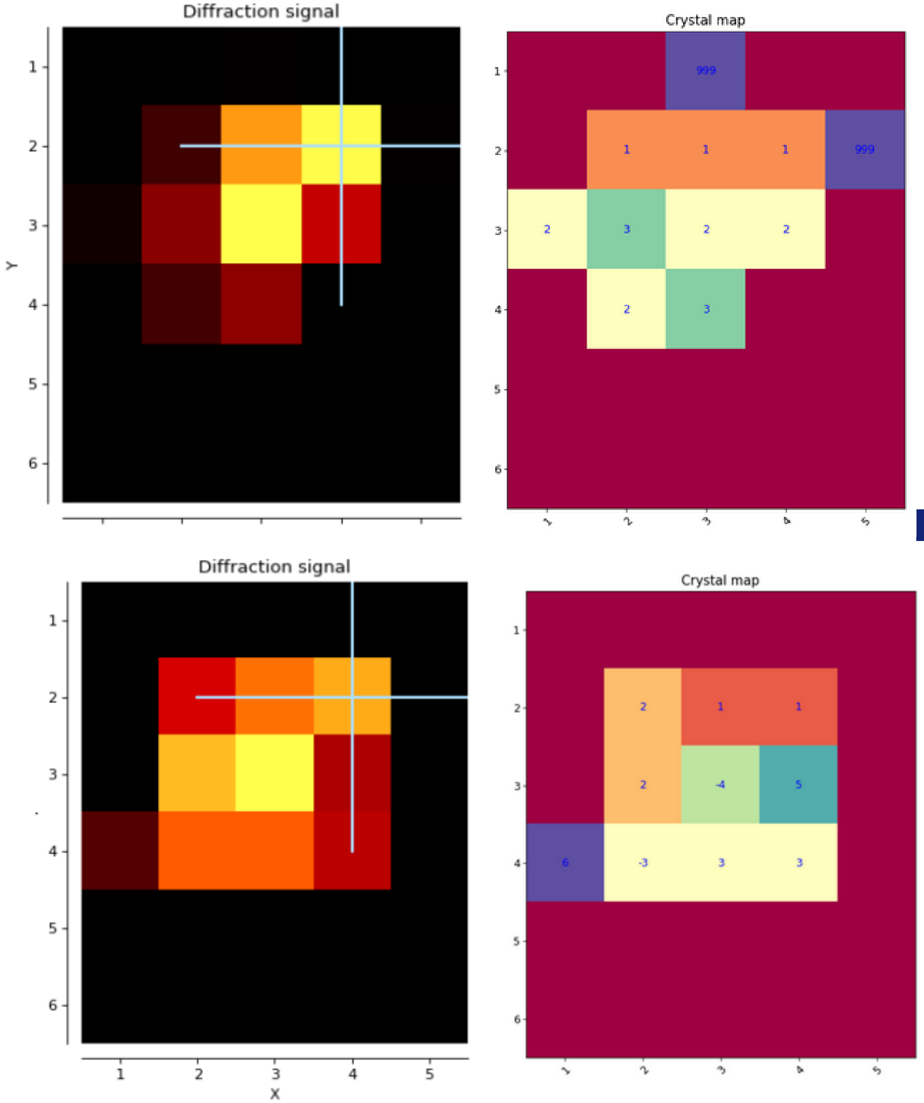
?





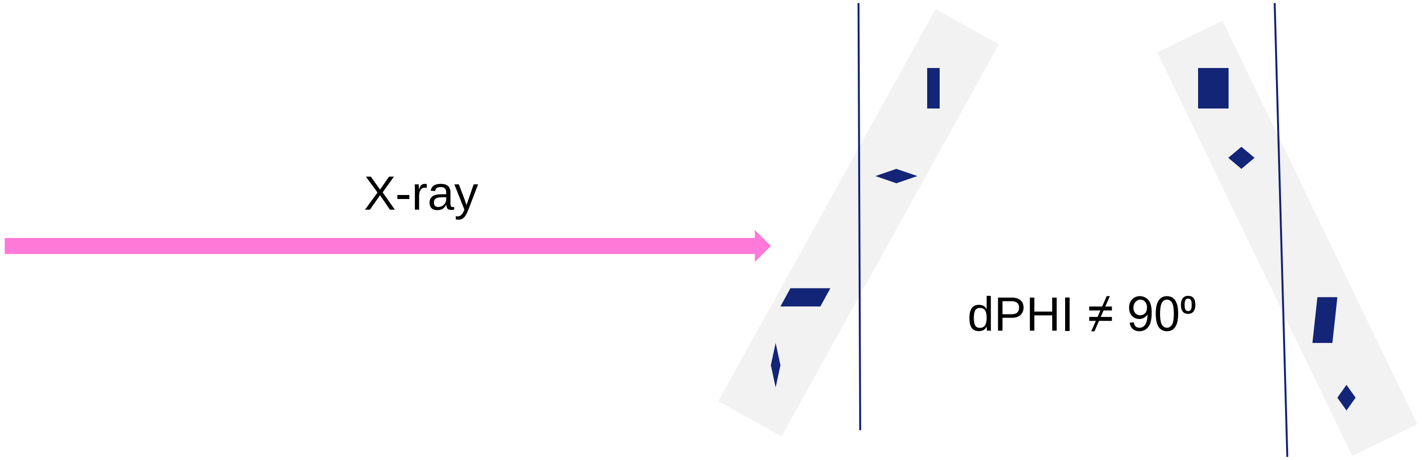
DOZORM2 –TWO OR MORE SCANS ANALYSIS



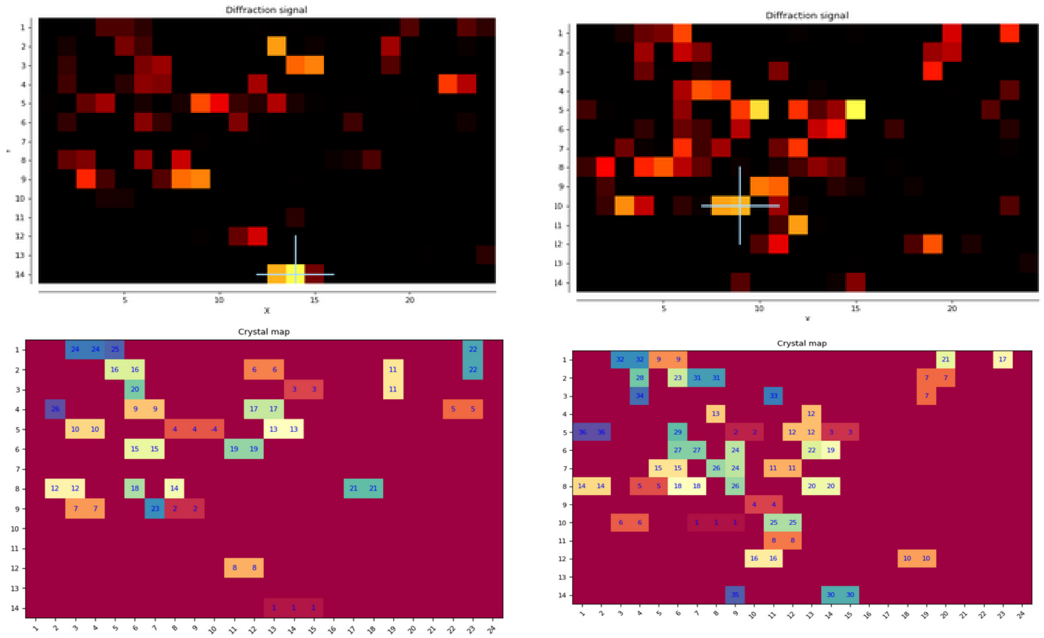



```
 Program dozorM /A.Popov,G.Bourenkov & I.Melnikov /
 Version 2.1.5 //  03.02.2022
 Copyright 2020 by Alexander Popov and Gleb Bourenkov

                  SCAN 1
                  ======

   Total N.of crystals in Loop =  3
Cryst Aperture Central  Coordinate  Int/Sig  N.of Images CRsize Score  Helic   Start     Finish     Int/Sig
number size     image      X    Y           All  dX  dY   X   Y   sum          x     y     x     y   helical
------------------------------------------------------------------------------------------------------------> X
    1    20.0       7    4.0    2.0   53.8   3   3   1   3   1    42.9  YES     2     2     4     2    78.6
    2    20.0      13    3.0    3.0   53.2   4   4   2   4   1    34.0  YES     1     3     4     3   132.6
    3    20.0      12    2.0    3.0   15.8   2   2   2   1   1    10.3   NO

                  SCAN 2
                  ======

   Total N.of crystals in Loop =  6
Cryst Aperture Central  Coordinate  Int/Sig  N.of Images CRsize Score  Helic   Start     Finish     Int/Sig
number size     image      X    Y           All  dX  dY   X   Y   sum          x     y     x     y   helical
------------------------------------------------------------------------------------------------------------> X
    1    20.0       7    4.0    2.0   27.1   2   2   1   2   1    16.6   NO
    2    20.0      12    2.0    3.0   27.7   2   1   2   1   2    13.7   NO
    3    20.0      17    4.0    4.0   11.7   1   1   1   1   1     3.5   NO
    4    20.0      14    4.0    3.0   10.6   1   1   1   1   1     3.2   NO
    5    20.0      20    1.0    4.0    5.4   1   1   1   1   1     1.6   NO
    6    20.0      13    3.0    3.0    2.3   1   1   1   1   1     1.4   NO
-------------------------------------------------------------------------------------

                3D COORDINATES
                ===============

Numb Score   Crystal Aperture    ScaneCoordinate     CrSize,micron      RealCoordin.micron    Alfa    sampX  sampY   phiy
     aver.  Sc1  Sc2   size    X      Y1      Y2   dX    dY1   dY2      X       Y      Z
----------------------------------------------------------------------------------------------------------------------------
   1   29.8   1    1    20.0   4.0    2.0     2.0   60.0  20.0  20.0   17.6  -17.6  -15.6  135.000  0.1326 0.1714 0.4804
   2   25.3   2    1    20.0   3.5    3.0     2.0   80.0  20.0  20.0   17.6    2.4   -5.6   82.152  0.1326 0.1914 0.4904
   3   23.2   1    3    20.0   4.0    2.0     4.0   60.0  20.0  20.0  -22.4  -17.6  -15.6  231.907  0.0926 0.1714 0.4804
   4   23.1   1    4    20.0   4.0    2.0     3.0   60.0  20.0  20.0   -2.4  -17.6  -15.6  187.848  0.1126 0.1714 0.4804
   5   17.7   2    6    20.0   3.0    3.0     3.0   80.0  20.0  20.0    3.1    2.4    4.4  591.907  0.1181 0.1914 0.5004
   6   12.0   3    2    20.0   2.0    3.0     3.0   20.0  20.0  40.0    0.3    2.4   24.4  547.848  0.1153 0.1914 0.5204
```

DOZORM2 –two or more scans analysis



Mesh&Collect



3D COORDINATES

Numb	Score	Crystal		Aperture	ScaneCoordinate			CrSize,micron			RealCoordin.micron			Alfa			
	aver.	Sc1	Sc2	size	X	Y1	Y2	dX	dY1	dY2	X	Y	Z				
1	136.0	1	5	50.0	14.4	14.0	5.0	57.0	19.0	19.0	148.6	-227.6	-124.6	46.872	0.4022	-0.4162	-0.5045
2	112.1	2	1	50.0	8.6	9.1	10.0	57.0	19.0	19.0	101.1	44.5	-15.6	326.221	0.3547	-0.1441	-0.3955
3	100.8	4	1	50.0	9.0	5.0	10.0	57.0	19.0	19.0	41.2	148.3	-22.1	275.531	0.2948	-0.0403	-0.4020
4	104.2	3	5	30.0	14.9	3.0	5.0	38.0	19.0	19.0	-14.0	53.9	-134.6	245.437	0.2396	-0.1347	-0.5145
5	47.6	8	2	20.0	7.0	3.0	4.0	57.0	19.0	19.0	-19.1	24.8	14.9	222.364	0.2345	-0.1638	-0.3650
6	56.7	7	7	20.0	3.0	9.0	10.0	38.0	19.0	19.0	100.3	45.9	90.9	325.418	0.3539	-0.1427	-0.2890
7	60.0	6	13	20.0	12.5	2.0	5.0	38.0	19.0	19.0	-28.8	79.5	-89.6	240.100	0.2248	-0.1091	-0.4695
8	75.3	11	7	20.0	3.5	5.0	10.0	38.0	19.0	19.0	41.2	148.3	81.4	275.531	0.2948	-0.0403	-0.2985
9	50.9	9	9	20.0	12.0	12.0	11.0	38.0	19.0	19.0	149.8	-1.8	-80.1	350.685	0.4034	-0.1904	-0.4600
10	45.1	5	24	20.0	22.0	4.0	5.0	38.0	19.0	19.0	0.8	28.3	-270.1	261.550	0.2544	-0.1603	-0.6500
11	51.4	10	10	20.0	6.0	4.0	1.0	38.0	19.0	19.0	-19.8	-88.1	33.9	92.645	0.2338	-0.2767	-0.3460
12	40.0	12	11	20.0	19.0	2.0	12.0	38.0	38.0	19.0	7.1	283.3	-213.1	261.443	0.2607	0.0947	-0.5930
13	36.1	13	13	20.0	12.5	5.0	5.0	38.0	19.0	19.0	15.5	2.7	-89.6	340.000	0.2691	-0.1859	-0.4695
14	24.0	15	15	20.0	6.0	6.0	7.0	38.0	19.0	19.0	40.6	35.4	33.9	308.938	0.2942	-0.1532	-0.3460
15	29.8	16	15	20.0	5.5	2.0	7.0	38.0	19.0	19.0	-18.5	137.8	43.4	252.340	0.2351	-0.0508	-0.3365
16	29.7	21	11	20.0	18.5	8.0	12.0	38.0	19.0	19.0	95.8	129.7	-203.6	296.452	0.3494	-0.0589	-0.5835
17	21.6	14	26	20.0	8.0	8.0	7.0	19.0	19.0	19.0	70.1	-15.8	-4.1	2.721	0.3237	-0.2044	-0.3840
18	20.8	17	25	20.0	11.5	4.0	10.0	38.0	19.0	19.0	26.4	173.9	-70.6	268.643	0.2800	-0.0147	-0.4505
19	18.6	25	15	20.0	5.5	1.0	7.0	38.0	19.0	19.0	-33.3	163.4	43.4	248.476	0.2203	-0.0252	-0.3365
20	17.5	18	22	20.0	6.0	8.0	2.0	19.0	19.0	19.0	44.5	-161.4	33.9	64.589	0.2981	-0.3500	-0.3460
21	29.0	19	25	20.0	11.0	6.0	10.0	38.0	19.0	19.0	56.0	122.7	-61.1	284.529	0.3096	-0.0659	-0.4410
22	16.4	20	29	20.0	3.0	8.0	7.0	38.0	19.0	19.0	70.1	-15.8	90.9	2.721	0.3237	-0.2044	-0.2890
23	13.1	22	24	20.0	22.5	1.0	5.0	19.0	38.0	19.0	-43.6	105.1	-279.6	237.489	0.2100	-0.0835	-0.6595
24	12.7	26	20	20.0	20.0	1.0	1.0	19.0	19.0	19.0	-64.1	-11.3	-232.1	160.000	0.1895	-0.1999	-0.6120
25	21.2	23	32	20.0	7.0	9.0	2.0	38.0	19.0	19.0	59.3	-187.0	14.9	62.414	0.3129	-0.3756	-0.3650
26	15.7	24	33	20.0	4.0	1.0	1.0	38.0	19.0	19.0	-64.1	-11.3	71.9	160.000	0.1895	-0.1999	-0.3080
27	9.7	27	38	20.0	2.0	4.0	9.0	19.0	19.0	19.0	21.3	144.8	109.9	268.369	0.2749	-0.0438	-0.2700