NSXTool - new Software for Data Reduction at Monochromatic Single Crystal Diffractometers

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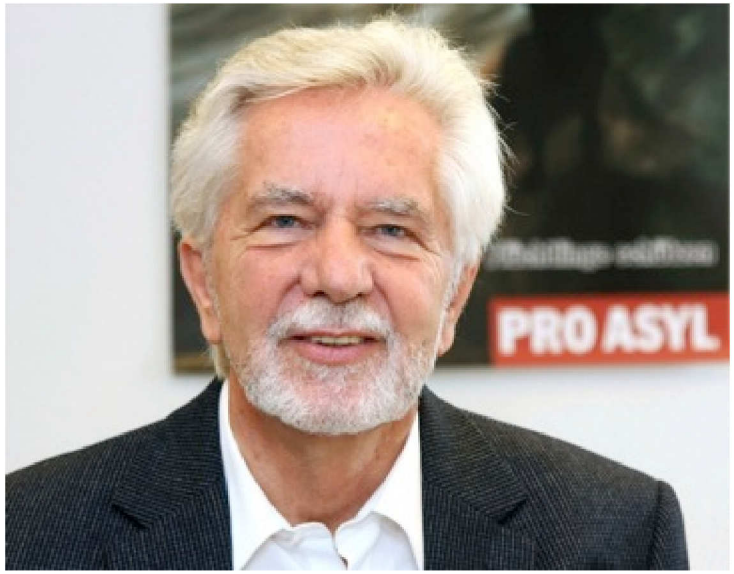
Laurent Chapon

MLZ is a cooperation between:
JÜLICH Forschungszentrum
Helmholtz-Zentrum Geesthacht Zentrum für Material- und Küstenforschung
Technical University of Munich

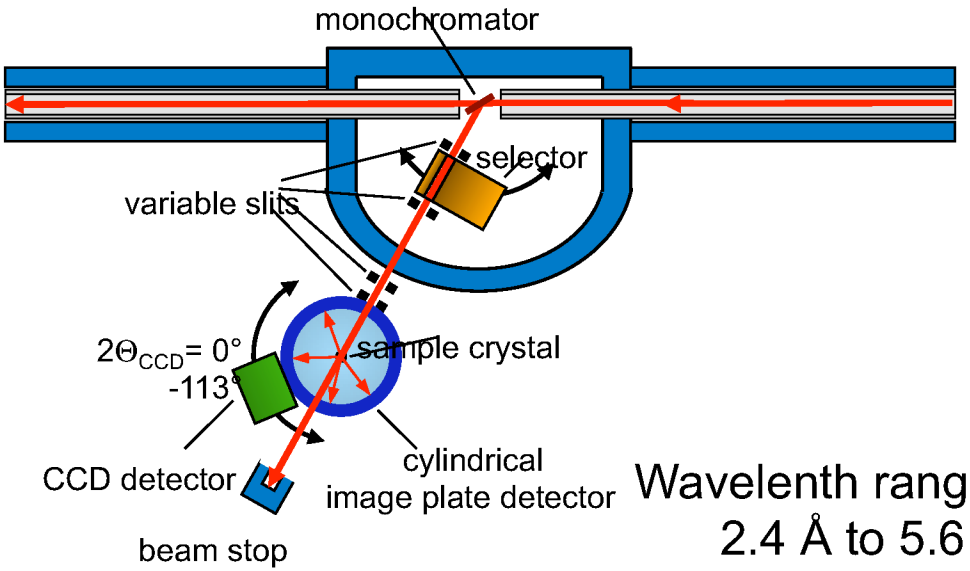
Jürgen Miksch, Founder of BISS

- 1984 bis 1993 als stellvertretender Direktor der Evangelischen Akademie Tutzing

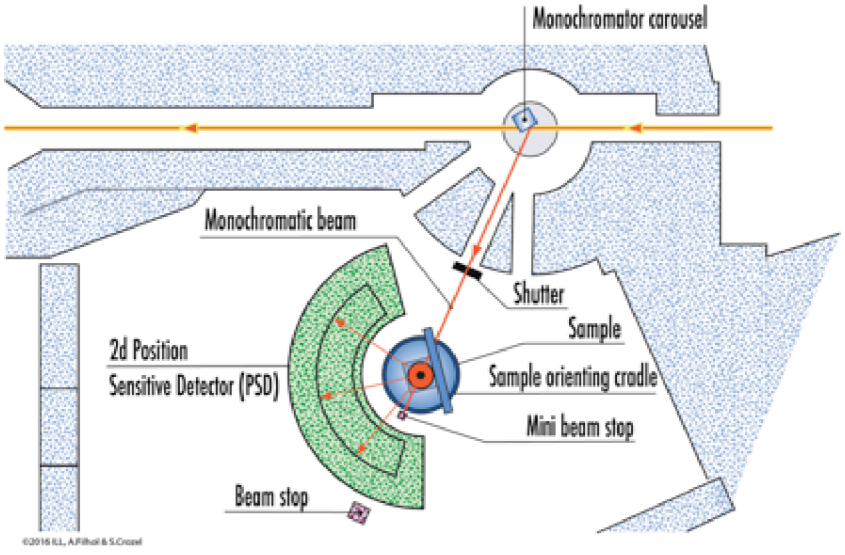


Instruments BIODIFF at MLZ and D19 at ILL

- Both monochromatic neutron diffractometers with cylindrical detector



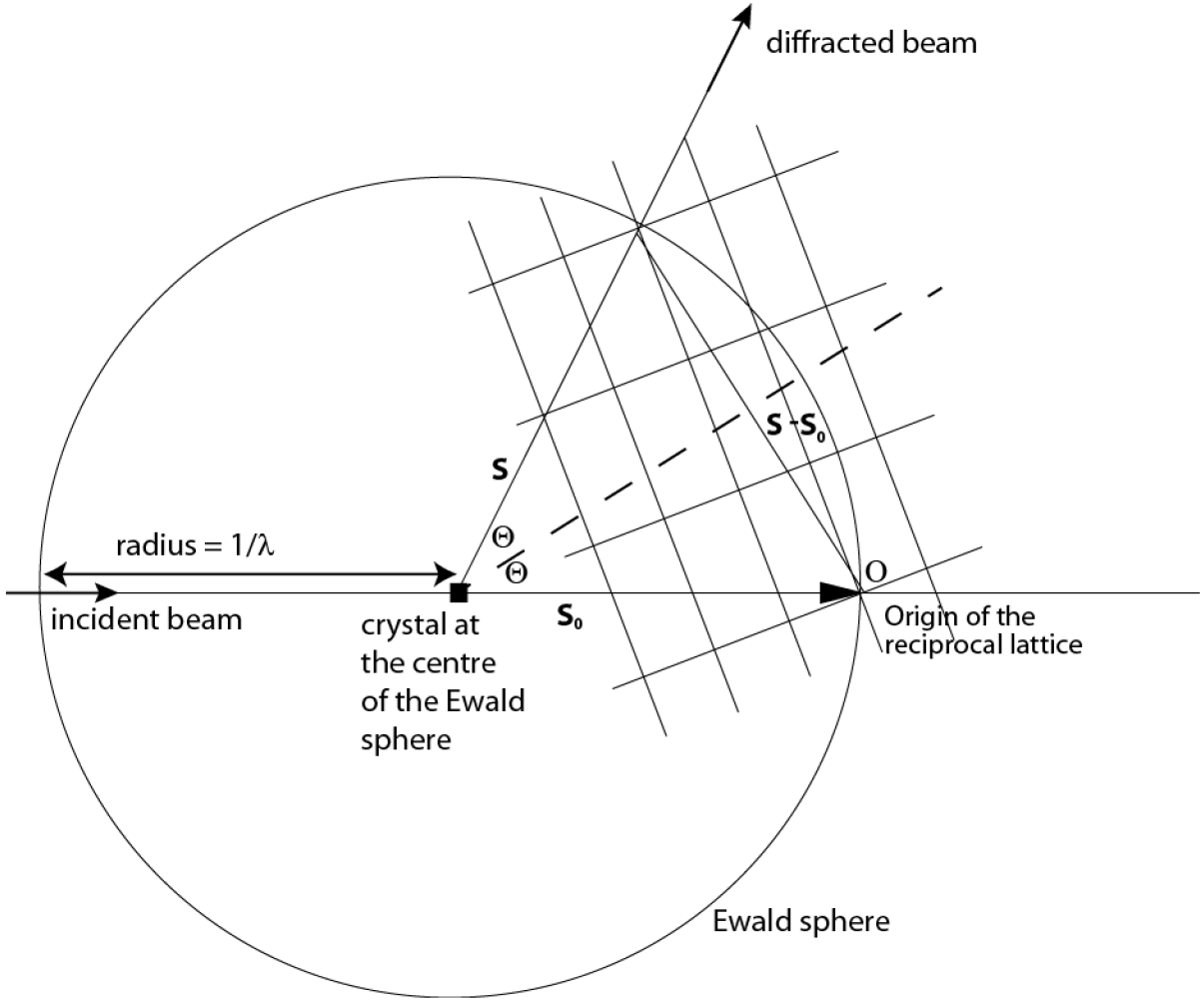
Wavelenth range:
2.4 Å to 5.6 Å



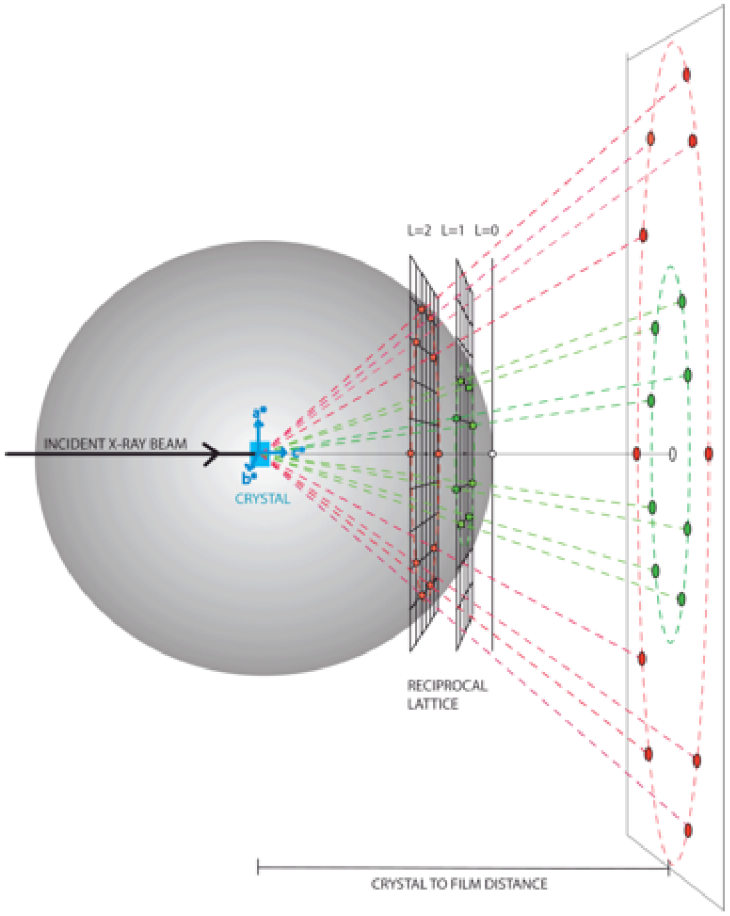
BIODIFF

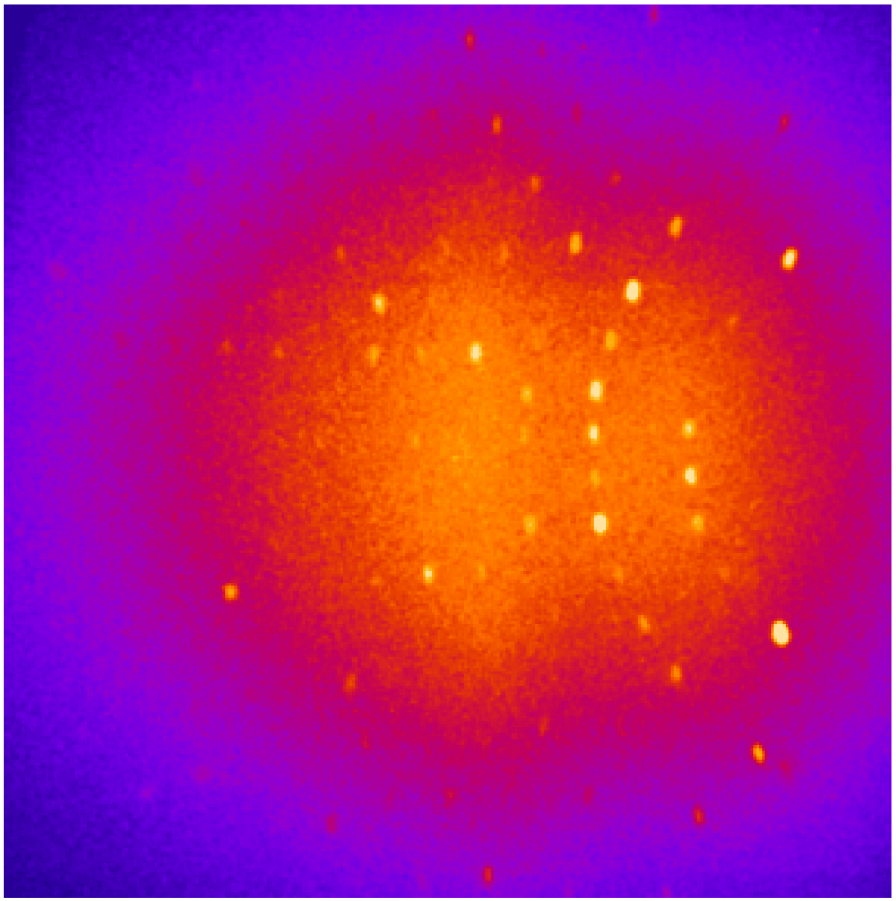
D19

Ewald construction and Bragg's Law



Myoglobin protein crystal (deuterated mother liquor) full data set recorded with CCD





prim. beam

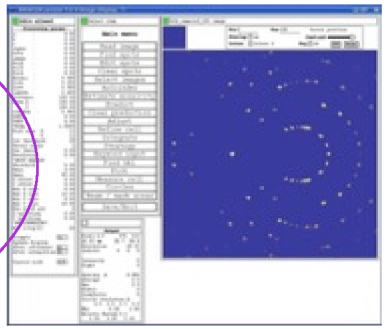
BioDiff: exposure time per frame: 20 minutes, sample: Myoglobin in deuterated mother liquor

Flow chart of data treatment and model building

Scans at varying crystal orientation
Scan := Series of detector images

Data reduction
- determination of crystal orientation, unit cell dimensions etc.
- Calculating integral of reflection intensities

hkl-list for each scan:
h k l Intensity Intensity error



-MOSFLM
-HKL-denzo
(comercial)

Scaling of each hkl list to match each other

-SCALA (CCP4-program package)

Unified hkl-list of measurement := complete data set

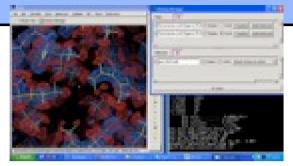
Calculation of a first map

Additional information from the solution of the phase problem

Structure refinement
-Refinement of atom coordinates displacements
- Calculation of scattering density maps (netrons) or electron density maps (x-rays)

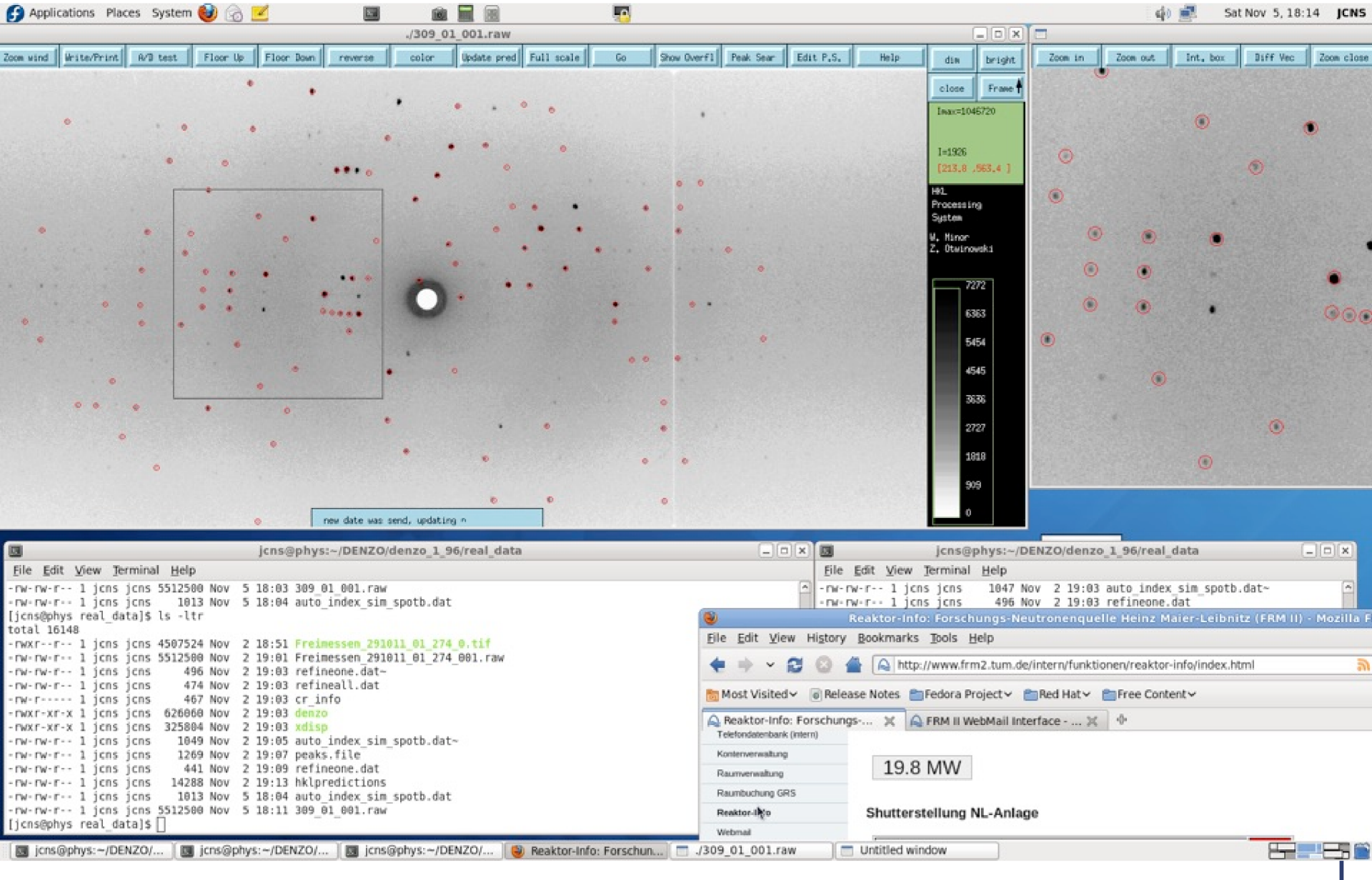
Map-plotting
- inspection of model to fit the map)
- real space changes and refinement to the model

-nCNS
-PHENIX

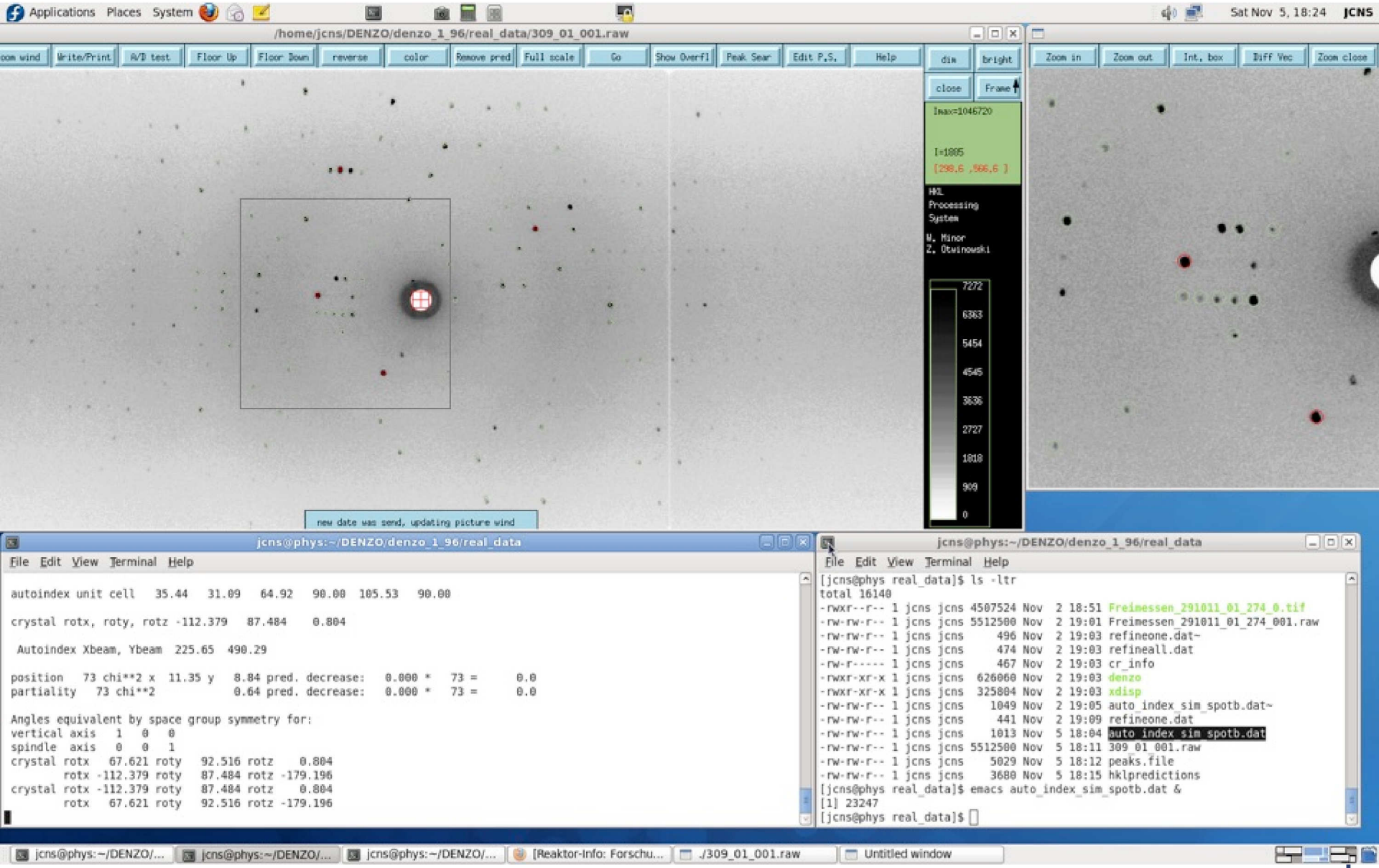


-XtalView
-Coot

Peak search with hkl DENZO



auto-index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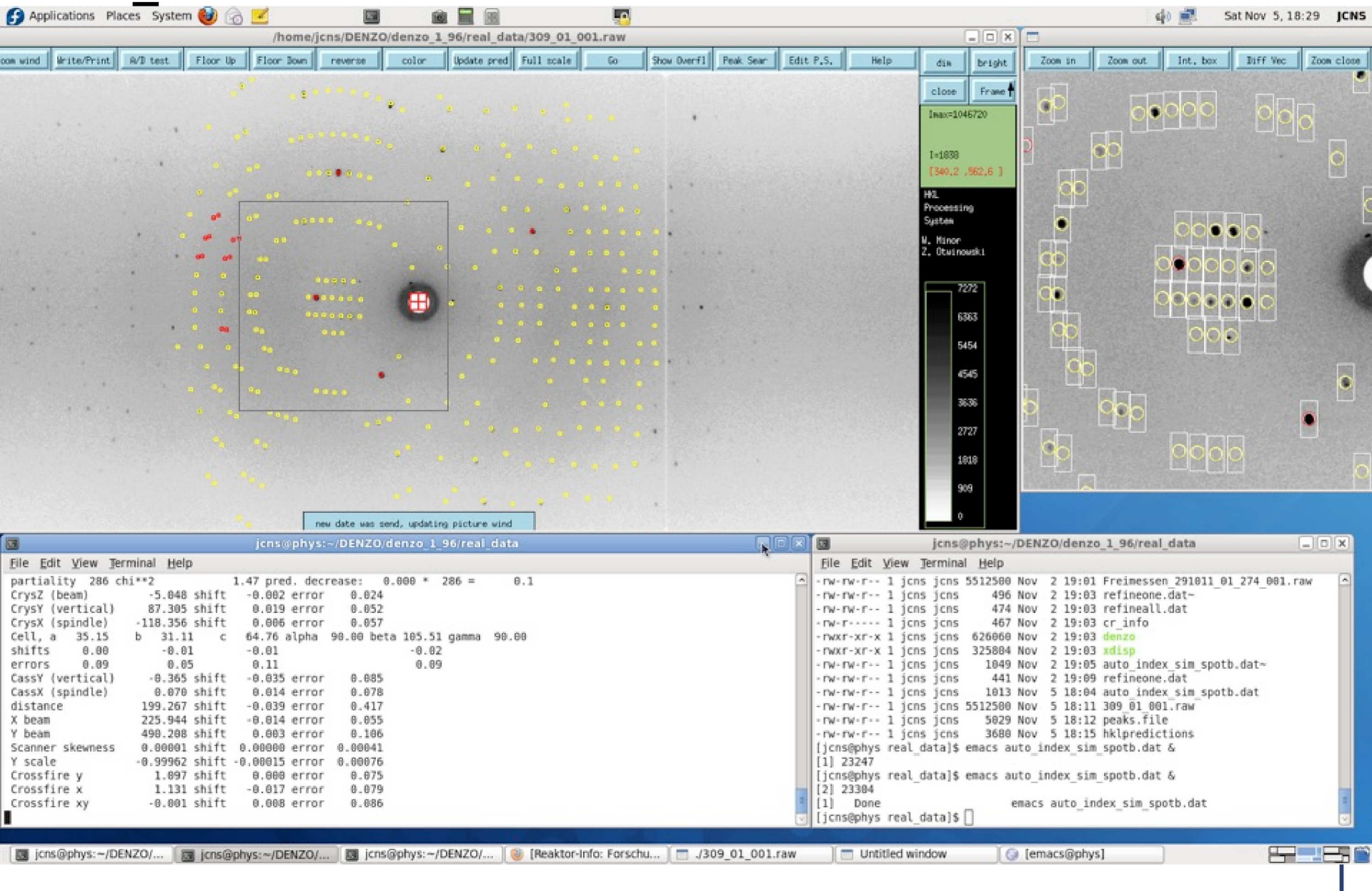
/home/jcns/DENZO/denzo_1_96/real_data/309_01_001.raw
Write/Print
A/D test
Floor Up
Floor Down
reverse
color
Remove pred
Full scale
Go
Show Overfl
Peak Sear
Edit P,S.
Help
dim
bright
close
Frame
Imax=1046720
I=1805
[298.6 ,966.6]
HKL
Processing
System
W. Minor
Z. Otwinowski
7272
6363
5454
4545
3636
2727
1818
909
0
Zoom in
Zoom out
Int. box
Diff Vec
Zoom close
new date was send, updating picture wind
jcns@phys:~/DENZO/denzo_1_96/real_data
File Edit View Terminal Help
autoindex unit cell 35.44 31.09 64.92 90.00 105.53 90.00
crystal rotx, roty, rotz -112.379 87.484 0.804
Autoindex Xbeam, Ybeam 225.65 490.29
position 73 chi**2 x 11.35 y 8.84 pred. decrease: 0.000 * 73 = 0.0
partiality 73 chi**2 0.64 pred. decrease: 0.000 * 73 = 0.0
Angles equivalent by space group symmetry for:
vertical axis 1 0 0
spindle axis 0 0 1
crystal rotx 67.621 roty 92.516 rotz 0.804
rotx -112.379 roty 87.484 rotz -179.196
crystal rotx -112.379 roty 87.484 rotz 0.804
rotx 67.621 roty 92.516 rotz -179.196
jcns@phys:~/DENZO/denzo_1_96/real_data
File Edit View Terminal Help
[jcns@phys real_data]$ ls -ltr
total 16140
-rwxr--r-- 1 jcns jcns 4507524 Nov 2 18:51 Freimessen_291011_01_274_0.tif
-rw-rw-r-- 1 jcns jcns 5512500 Nov 2 19:01 Freimessen_291011_01_274_001.raw
-rw-rw-r-- 1 jcns jcns 496 Nov 2 19:03 refineone.dat~
-rw-rw-r-- 1 jcns jcns 474 Nov 2 19:03 refineall.dat
-rw-r----- 1 jcns jcns 467 Nov 2 19:03 cr_info
-rwxr-xr-x 1 jcns jcns 626060 Nov 2 19:03 denzo
-rwxr-xr-x 1 jcns jcns 325884 Nov 2 19:03 xdisp
-rw-rw-r-- 1 jcns jcns 1049 Nov 2 19:05 auto_index_sim_spotb.dat~
-rw-rw-r-- 1 jcns jcns 441 Nov 2 19:09 refineone.dat
-rw-rw-r-- 1 jcns jcns 1013 Nov 5 18:04 auto_index_sim_spotb.dat
-rw-rw-r-- 1 jcns jcns 5512500 Nov 5 18:11 309_01_001.raw
-rw-rw-r-- 1 jcns jcns 5029 Nov 5 18:12 peaks.file
-rw-rw-r-- 1 jcns jcns 3680 Nov 5 18:15 hklpredictions
[jcns@phys real_data]$ emacs auto_index_sim_spotb.dat &
[1] 23247
[jcns@phys real_data]$
jcns@phys:~/DENZO/...
jcns@phys:~/DENZO/...
jcns@phys:~/DENZO/...
[Reaktor-Info: Forschu...
./309_01_001.raw
Untitled window

d_min=2.5 Å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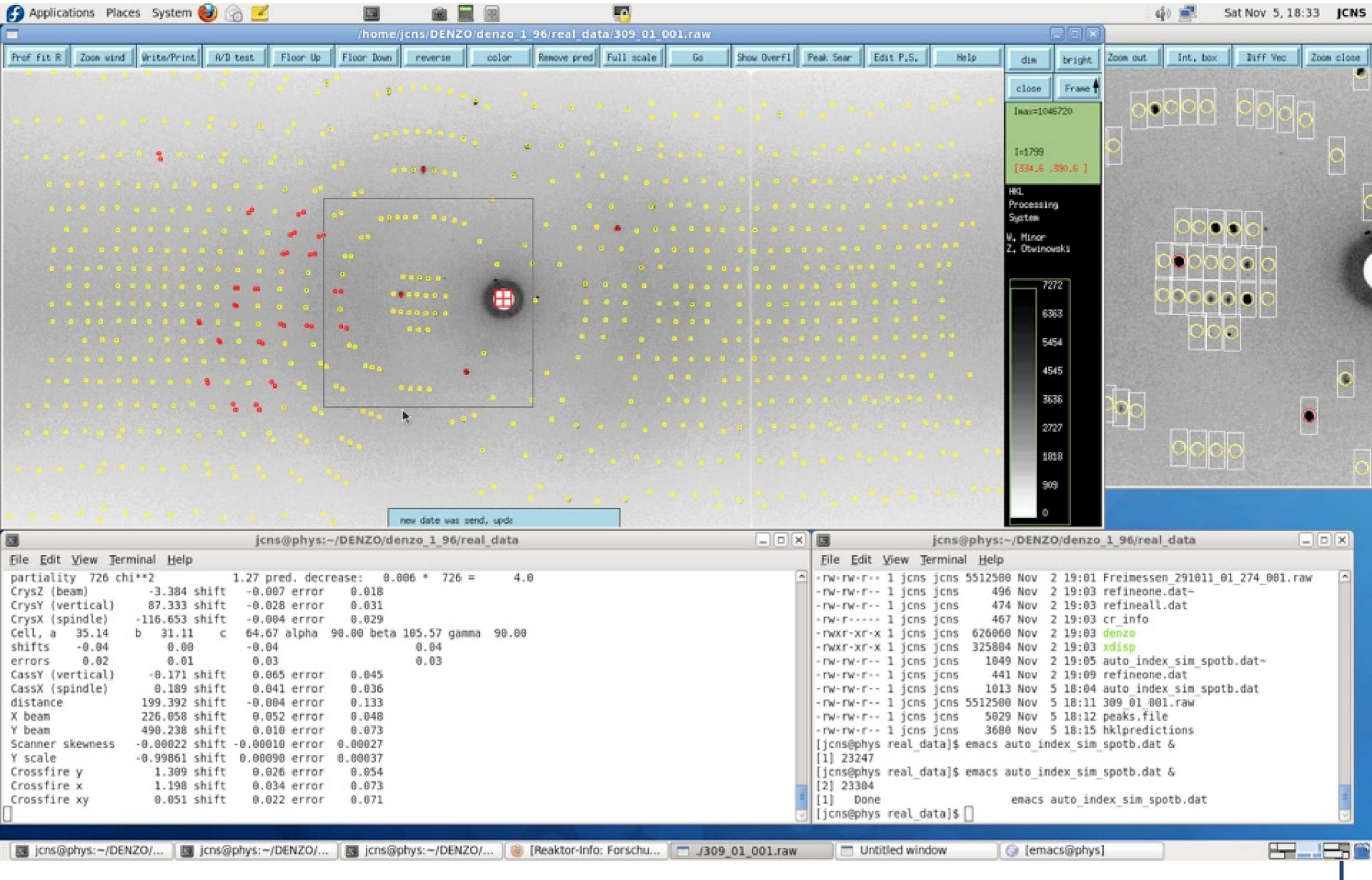
Sat Nov 5, 18:29 JCNS
/home/jcns/DENZO/denzo_1_96/real_data/309_01_001.raw
Write/Print
A/D test
Floor Up
Floor Down
reverse
color
Update pred
Full scale
Go
Show Overfl
Peak Sear
Edit P,S.
Help
dim
bright
close
Frame
Imax=1046720
I=1838
[340.2 ,962.6]
HKL
Processing
System
W. Minor
Z. Otwinowski
7272
6363
5454
4545
3636
2727
1818
909
0
Zoom in
Zoom out
Int. box
Diff Vec
Zoom close
new date was send, updating picture wind
jcns@phys:~/DENZO/denzo_1_96/real_data
File Edit View Terminal Help
partiality 286 chi**2 1.47 pred. decrease: 0.000 * 286 = 0.1
CrysZ (beam) -5.048 shift -0.002 error 0.024
CrysY (vertical) 87.305 shift 0.019 error 0.052
CrysX (spindle) -118.356 shift 0.006 error 0.057
Cell, a 35.15 b 31.11 c 64.76 alpha 90.00 beta 105.51 gamma 90.00
shifts 0.00 -0.01 -0.01 -0.02
errors 0.09 0.05 0.11 0.09
CassY (vertical) -0.365 shift -0.035 error 0.085
CassX (spindle) 0.070 shift 0.014 error 0.078
distance 199.267 shift -0.039 error 0.417
X beam 225.944 shift -0.014 error 0.055
Y beam 490.208 shift 0.003 error 0.106
Scanner skewness 0.00001 shift 0.00000 error 0.00041
Y scale -0.99962 shift -0.00015 error 0.00076
Crossfire y 1.097 shift 0.000 error 0.075
Crossfire x 1.131 shift -0.017 error 0.079
Crossfire xy -0.001 shift 0.008 error 0.086
jcns@phys:~/DENZO/denzo_1_96/real_data
File Edit View Terminal Help
-rw-rw-r-- 1 jcns jcns 5512580 Nov 2 19:01 Freimessen_291011_01_274_001.raw
-rw-rw-r-- 1 jcns jcns 496 Nov 2 19:03 refineone.dat~
-rw-rw-r-- 1 jcns jcns 474 Nov 2 19:03 refineall.dat
-rw-r----- 1 jcns jcns 467 Nov 2 19:03 cr_info
-rwxr-xr-x 1 jcns jcns 626060 Nov 2 19:03 denzo
-rwxr-xr-x 1 jcns jcns 325804 Nov 2 19:03 xdisp
-rw-rw-r-- 1 jcns jcns 1049 Nov 2 19:05 auto_index_sim_spotb.dat~
-rw-rw-r-- 1 jcns jcns 441 Nov 2 19:09 refineone.dat
-rw-rw-r-- 1 jcns jcns 1013 Nov 5 18:04 auto_index_sim_spotb.dat
-rw-rw-r-- 1 jcns jcns 5512580 Nov 5 18:11 309_01_001.raw
-rw-rw-r-- 1 jcns jcns 5029 Nov 5 18:12 peaks.file
-rw-rw-r-- 1 jcns jcns 3680 Nov 5 18:15 hklpredictions
[jcns@phys real_data]$ emacs auto_index_sim_spotb.dat &
[1] 23247
[jcns@phys real_data]$ emacs auto_index_sim_spotb.dat &
[2] 23304
[1] Done emacs auto_index_sim_spotb.dat
[jcns@phys real_data]$
jcns@phys:~/DENZO/...
jcns@phys:~/DENZO/...
jcns@phys:~/DENZO/...
[Reaktor-Info: Forschu...
./309_01_001.raw
Untitled window
[emacs@phys]

d_min=1.5 Å


Applications Places System
Sat Nov 5, 18:33 JCNS
/home/jcns/DENZO/denzo_1_96/real_data/309_01_001.raw
Prof fit R
Zoom wind
Write/Print
A/D test
Floor Up
Floor Down
reverse
color
Remove pred
Full scale
Go
Show Overfl
Peak Sear
Edit P.S.
Help
dim
bright
close
Frame
Imax=1046720
I=1799
[334.6 ,390.6]
HKL
Processing
System
W. Minor
Z. Otwinowski
7272
6363
5454
4545
3636
2727
1818
909
0
Zoom out
Int. box
Diff Vec
Zoom close
new date was send, upd
jcns@phys:~/DENZO/denzo_1_96/real_data
File Edit View Terminal Help
partiality 726 chi**2 1.27 pred. decrease: 0.006 * 726 = 4.0
CrysZ (beam) -3.384 shift -0.007 error 0.018
CrysY (vertical) 87.333 shift -0.028 error 0.031
CrysX (spindle) -116.653 shift -0.004 error 0.029
Cell, a 35.14 b 31.11 c 64.67 alpha 90.00 beta 105.57 gamma 90.00
shifts -0.04 0.00 -0.04 0.04
errors 0.02 0.01 0.03 0.03
CassY (vertical) -0.171 shift 0.065 error 0.045
CassX (spindle) 0.189 shift 0.041 error 0.036
distance 199.392 shift -0.004 error 0.133
X beam 226.058 shift 0.052 error 0.048
Y beam 490.238 shift 0.010 error 0.073
Scanner skewness -0.00022 shift -0.00010 error 0.00027
Y scale -0.99861 shift 0.00090 error 0.00037
Crossfire y 1.309 shift 0.026 error 0.054
Crossfire x 1.198 shift 0.034 error 0.073
Crossfire xy 0.051 shift 0.022 error 0.071
jcns@phys:~/DENZO/denzo_1_96/real_data
File Edit View Terminal Help
-rw-rw-r-- 1 jcns jcns 5512500 Nov 2 19:01 Freimessen_291011_01_274_001.raw
-rw-rw-r-- 1 jcns jcns 496 Nov 2 19:03 refineone.dat~
-rw-rw-r-- 1 jcns jcns 474 Nov 2 19:03 refineall.dat
-rw-r----- 1 jcns jcns 467 Nov 2 19:03 cr_info
-rwxr-xr-x 1 jcns jcns 626060 Nov 2 19:03 denzo
-rwxr-xr-x 1 jcns jcns 325804 Nov 2 19:03 xdisp
-rw-rw-r-- 1 jcns jcns 1049 Nov 2 19:05 auto_index_sim_spotb.dat~
-rw-rw-r-- 1 jcns jcns 441 Nov 2 19:09 refineone.dat
-rw-rw-r-- 1 jcns jcns 1013 Nov 5 18:04 auto_index_sim_spotb.dat
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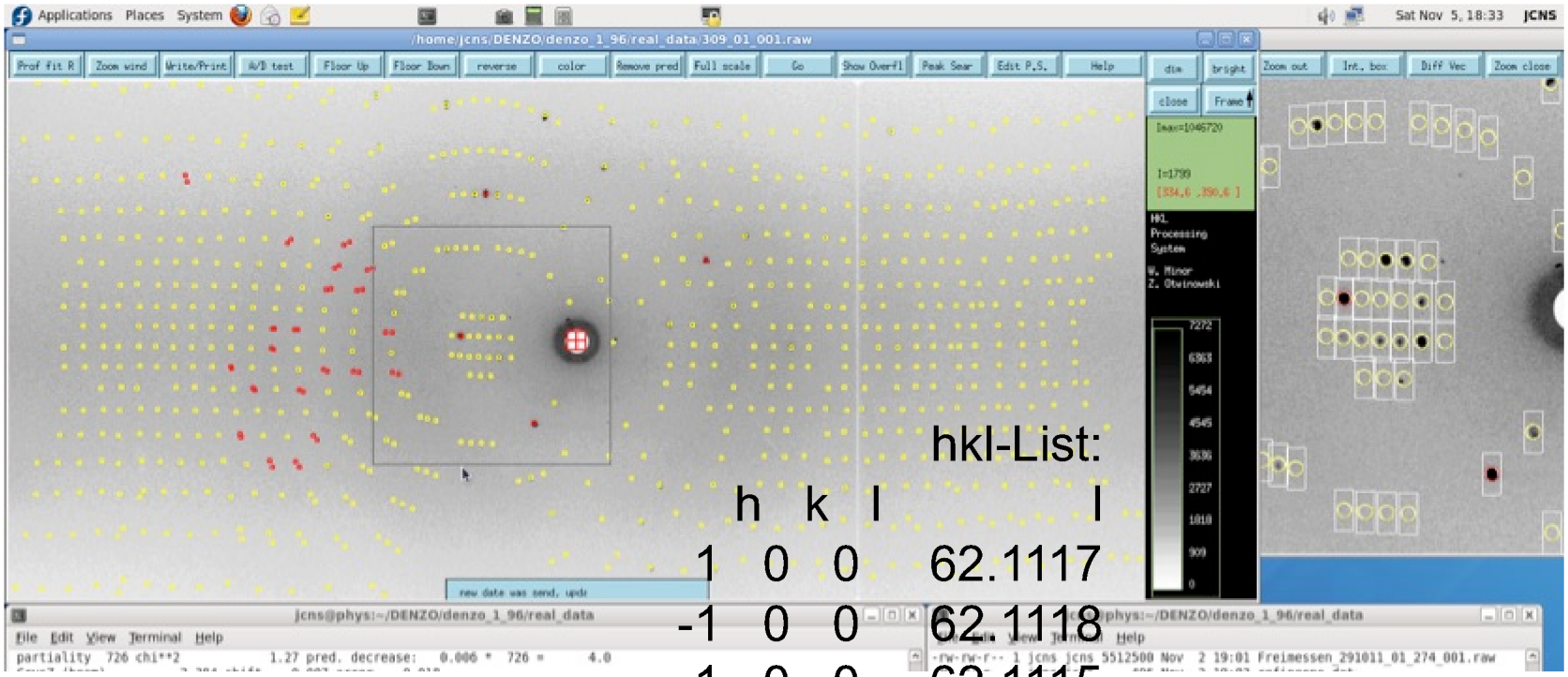
-rw-rw-r-- 1 jcns jcns 3680 Nov 5 18:15 hklpredictions
[jcns@phys real_data]$ emacs auto_index_sim_spotb.dat &
[1] 23247
[jcns@phys real_data]$ emacs auto_index_sim_spotb.dat &
[2] 23304
[1] Done emacs auto_index_sim_spotb.dat
[jcns@phys real_data]$
jcns@phys:~/DENZO/...
jcns@phys:~/DENZO/...
jcns@phys:~/DENZO/...
[Reaktor-Info: Forschu...
./309_01_001.raw
Untitled window
[emacs@phys]

Integration of partial Bragg peaks with the commercial software hkl-denzo up to d_{min}=1.5 Å



hkl-List:

h	k	l	I
1	0	0	62.1117
-1	0	0	62.1118
1	0	0	62.1115
-1	0	0	62.1120
0	0	-1	33.5555
1	0	-1	33.5589
0	0	1	33.5533
-1	0	1	33.5511

The „old“ HKL2000 software

- Not supported,
- Running under academic licence
- Need to re-new every 6 momths
- crashes frequently, if you press the buttons in the wrong order (also reboots the computer for you)
- No automatization, arbitrary choice of integration boxes…

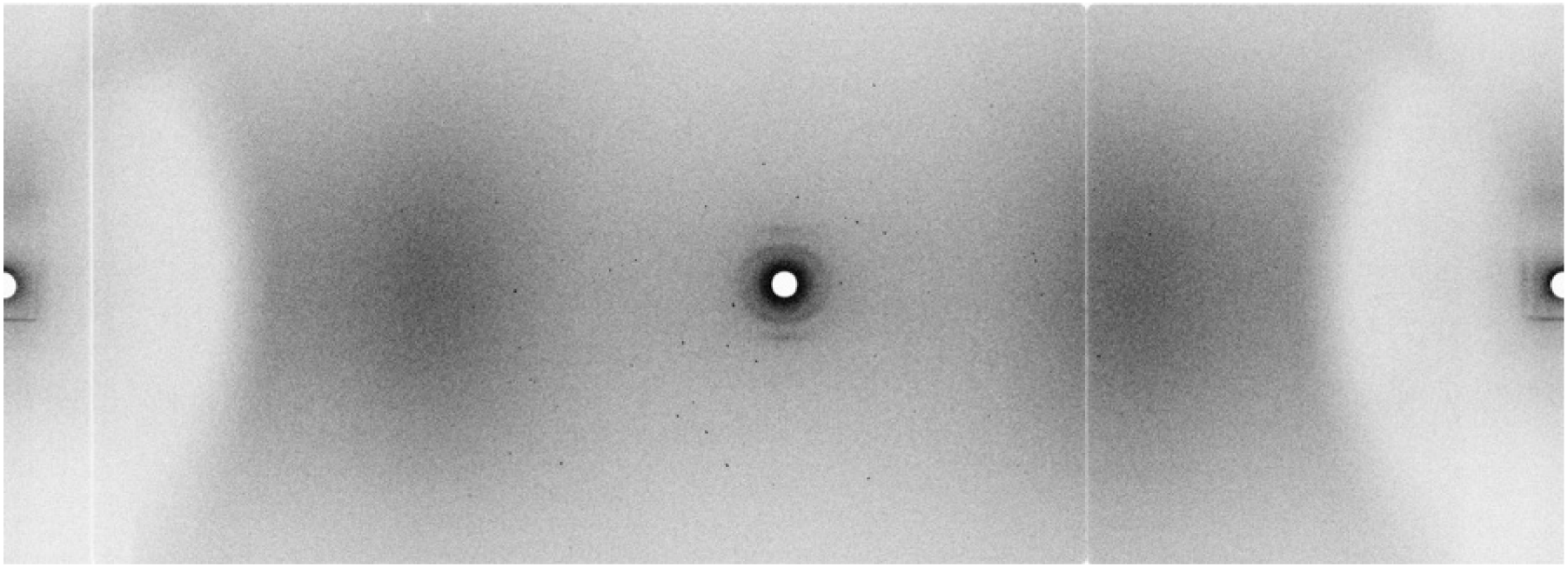
- Need something new!

Other alternatives

- XDS: no circular detector geometry
- IP mosflm
- DIALS: We are in contact with them…
- Mantid
- Leighton Coates: Recent Publication on 3-D
- ESS: Plans from Esko Oksanen for NMX
- …???

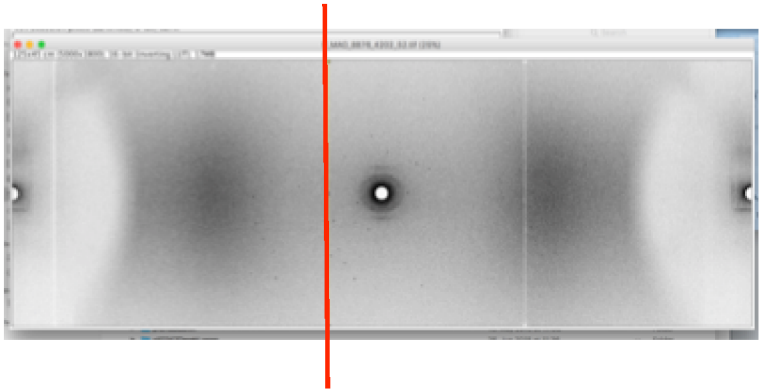
Outline of the problem

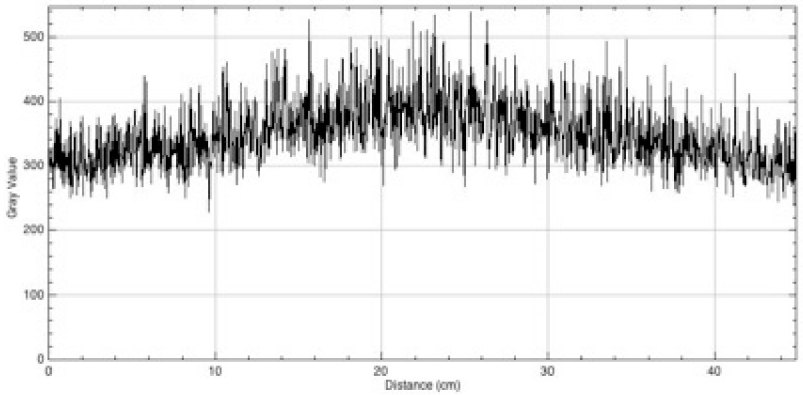
- We have to integrate the Bragg spots belonging to one Bragg reflection…

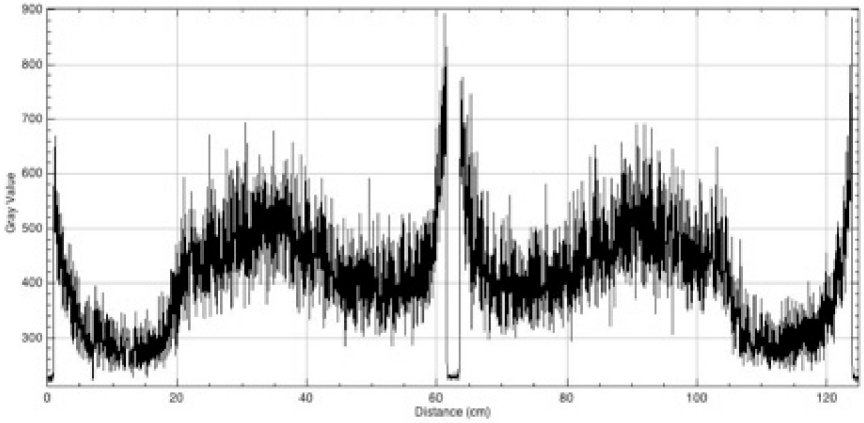


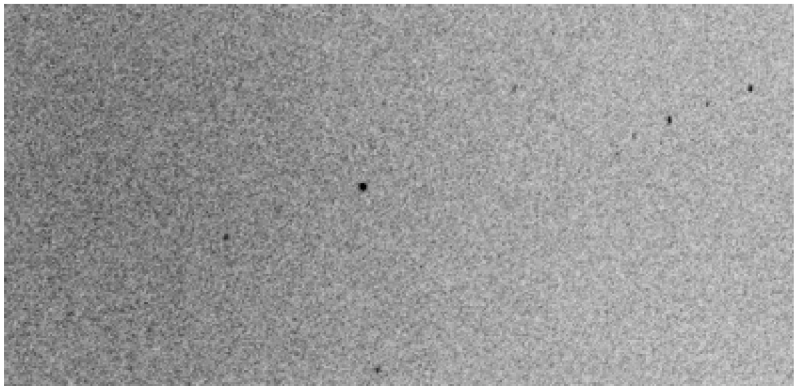
Special issues regarding neutron crystallography as compared to x-ray

- Background much more pronounced and not so homogenous



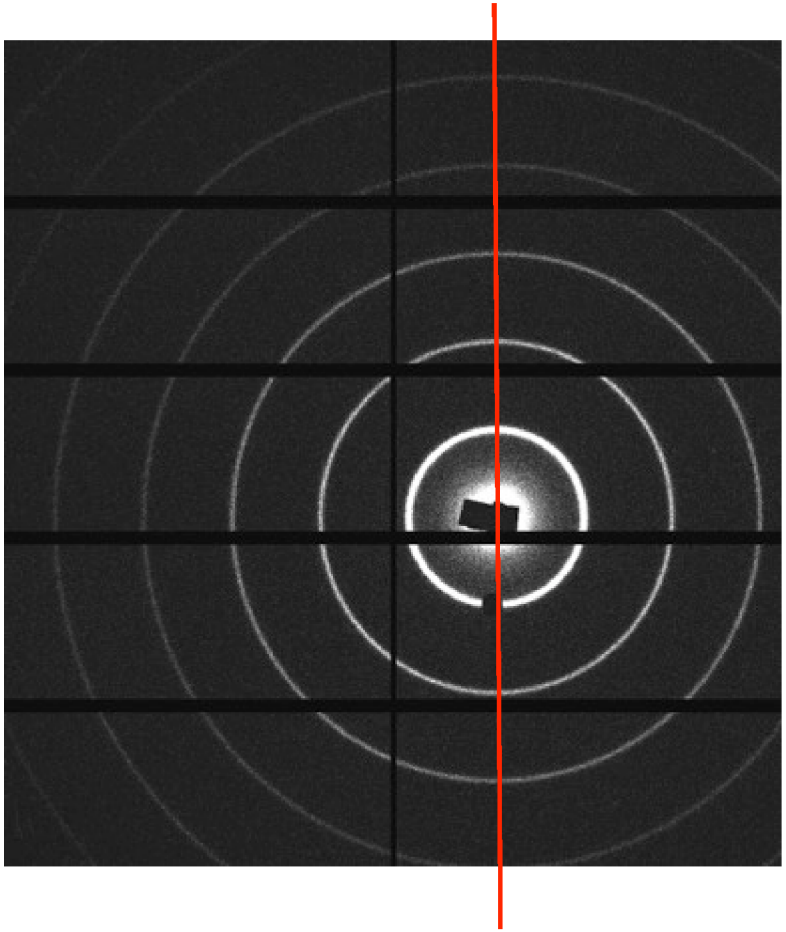


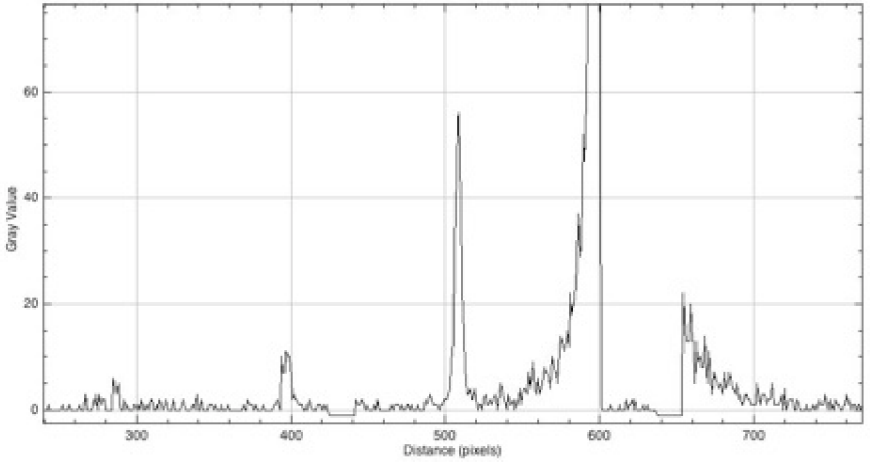




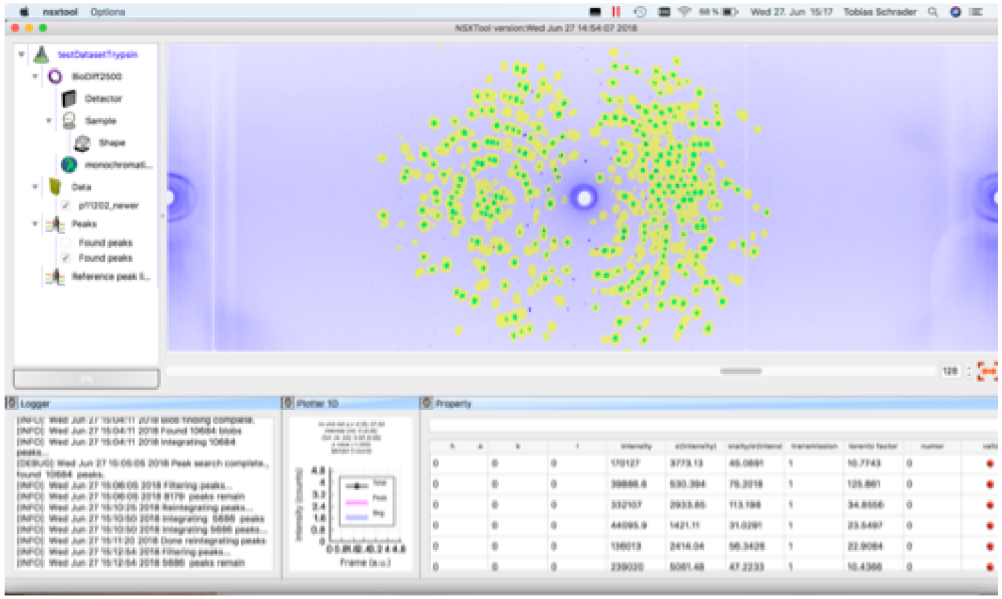
The x-ray case for comparison

- Horizontal cut showing the background





New Data Reduction Software: NXStool



Allows to incorporate

- Absorption correction of crystal with convex hull
- Systematic optimization of integration box size by python scripting
- Pushing resolution limits
- Monitoring detector parameters and detecting detector problems

To be completed…

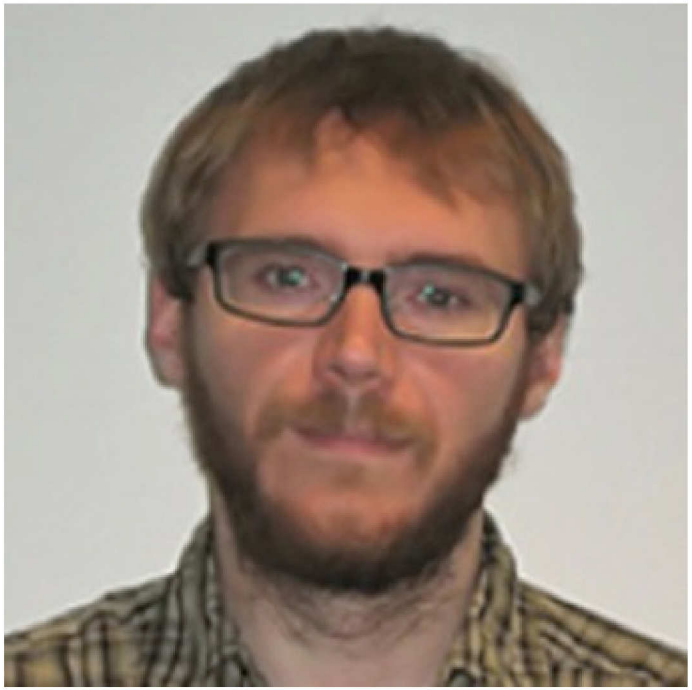
- Incorporation of a strategy software
- open source, well documented

People involved

- Laurent Chapon

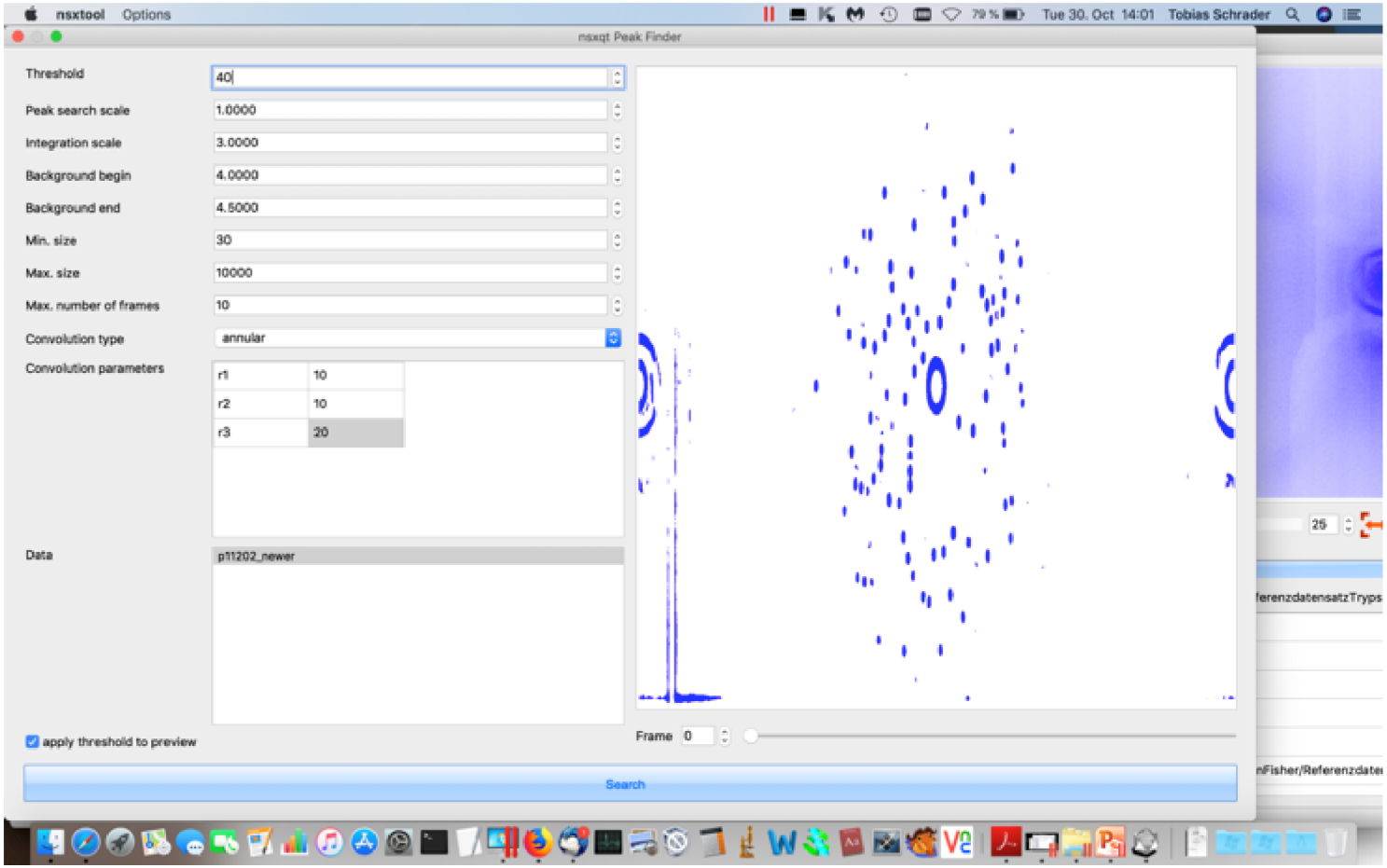
Eric Pellegrini

Jonathan Fisher



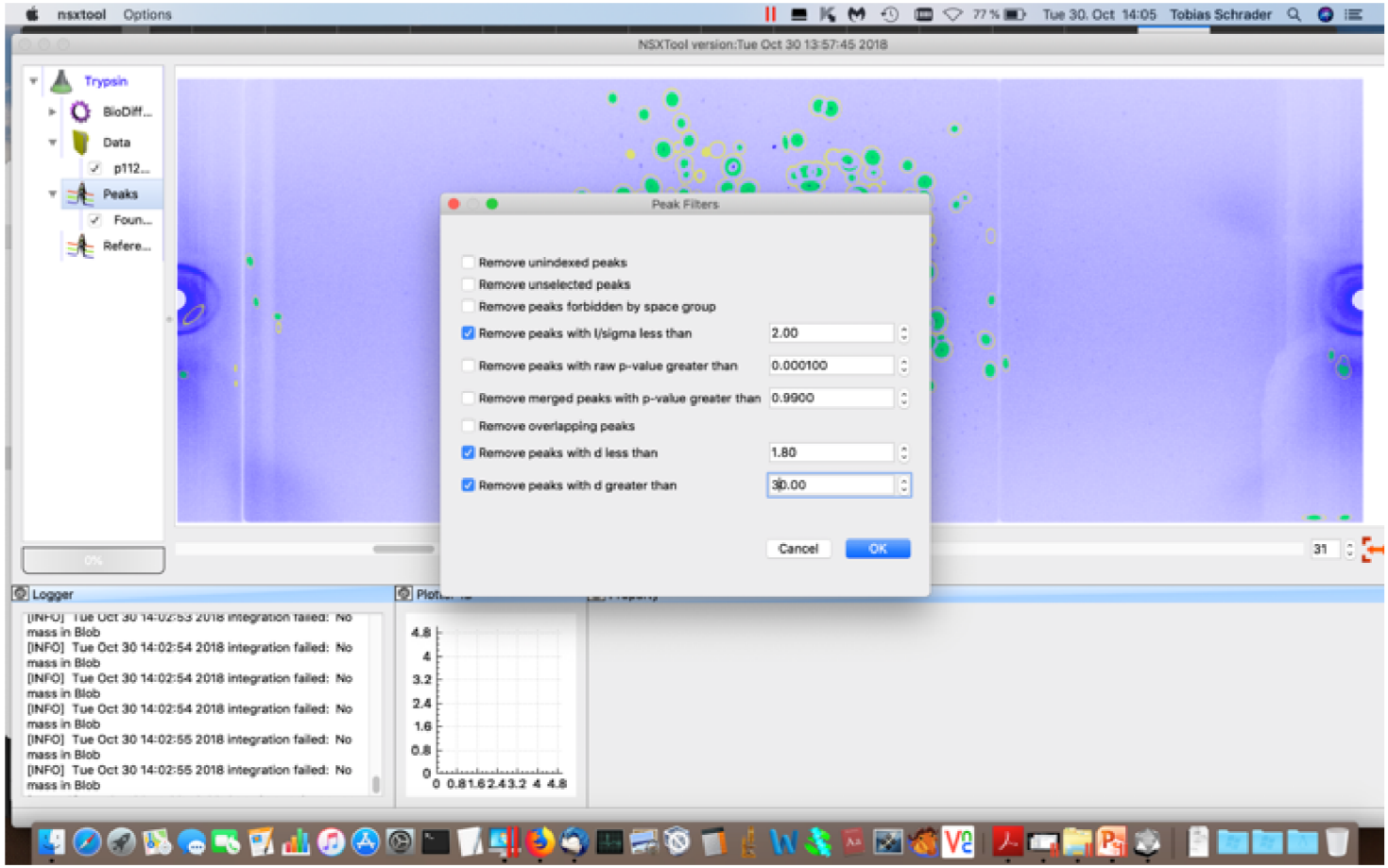
NXStool step by step

- Peak finding based on image analysis



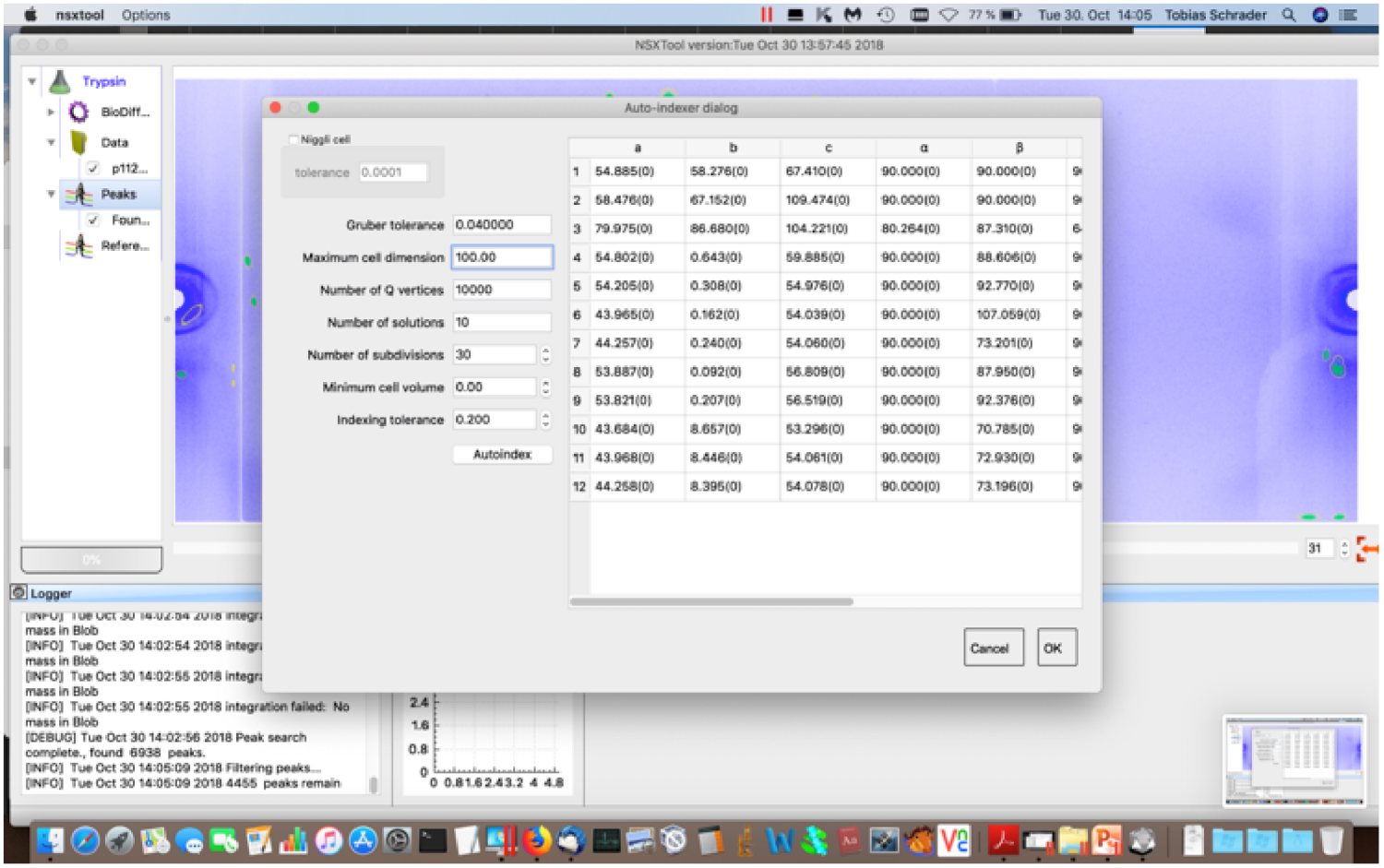
NXStool step by step

- Filtering of the peaks found by the image analysis



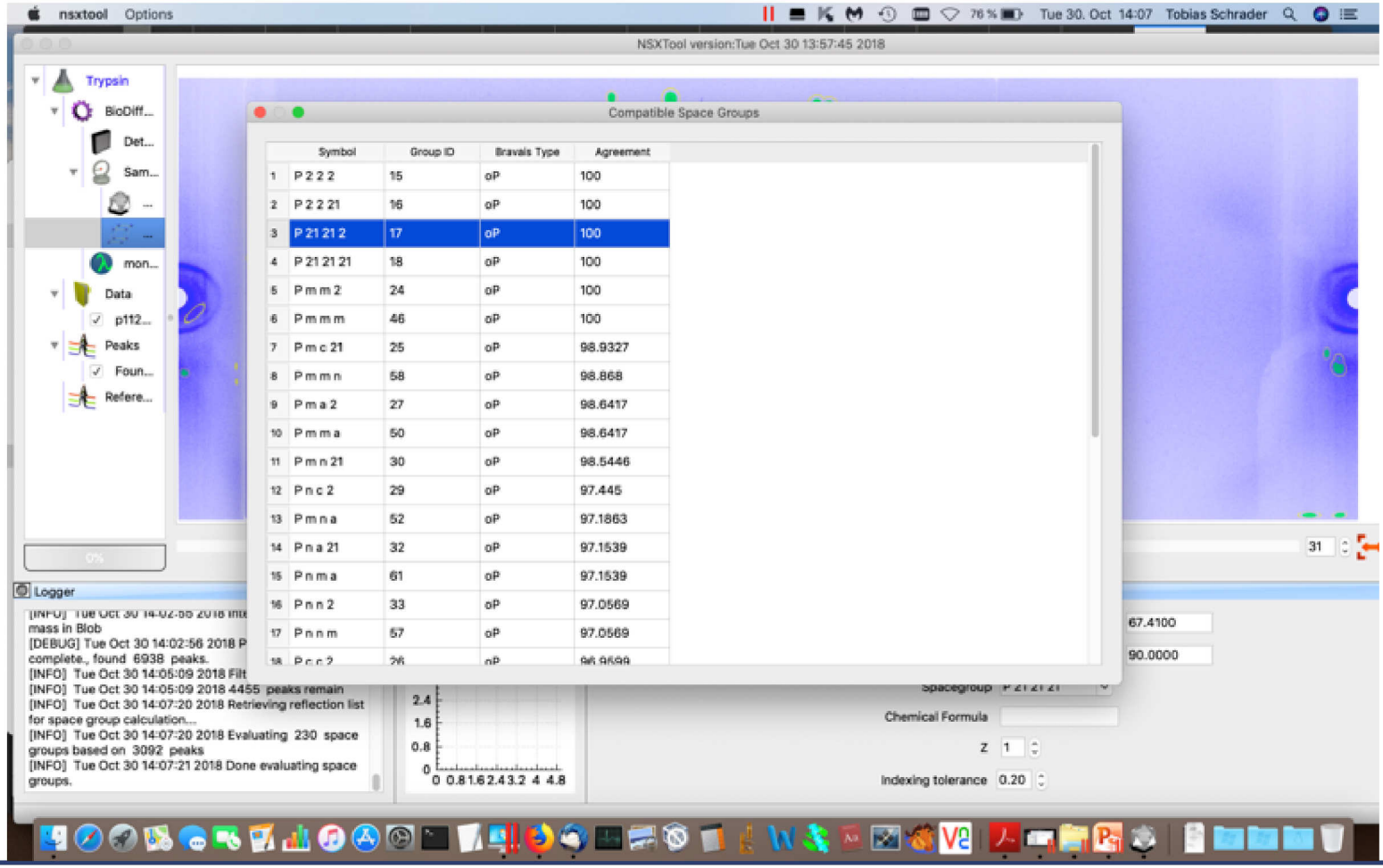
NXStool step by step

- Indexing of the Peaks, finding the right unit cell



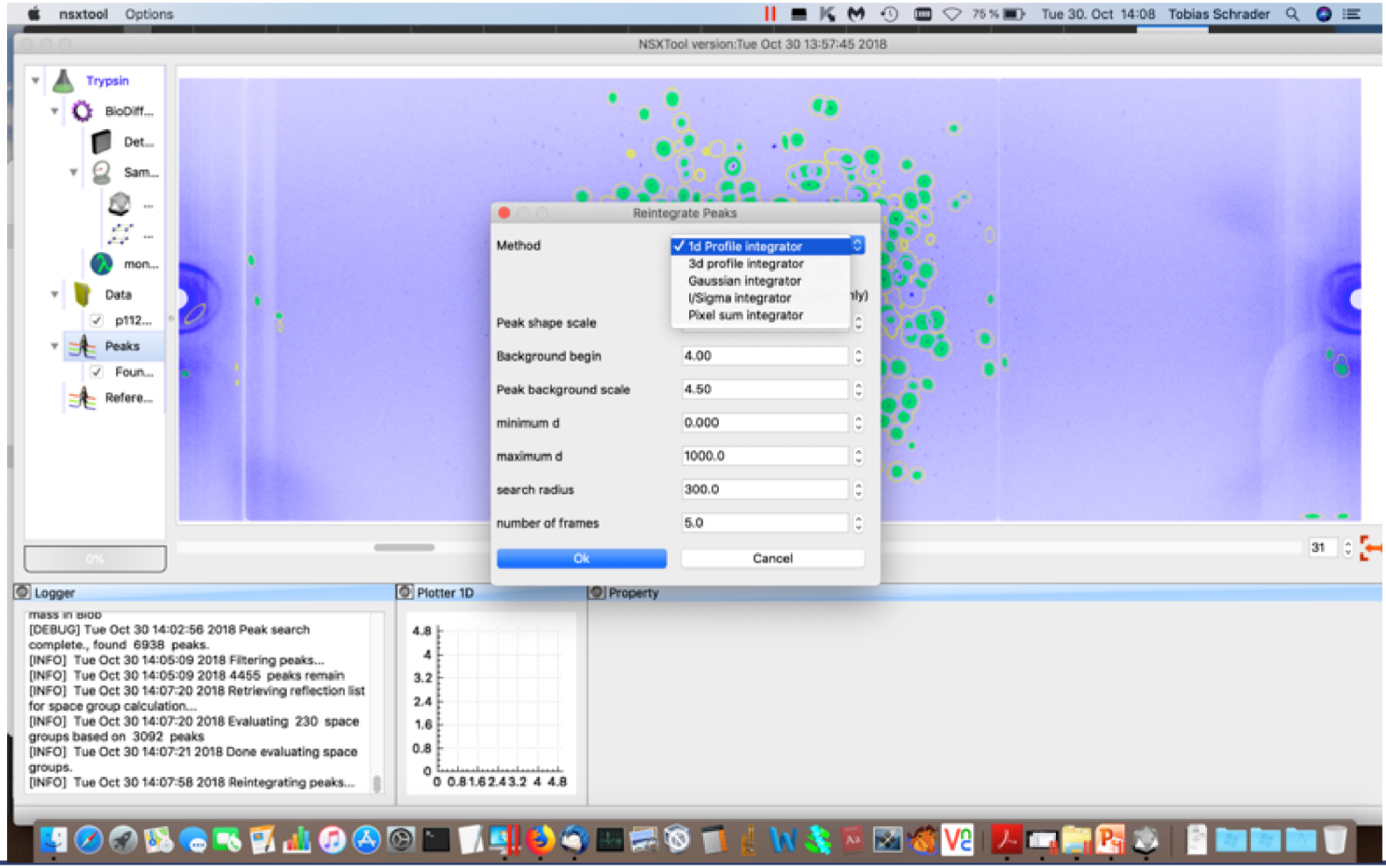
NXStool step by step

- Selecting the space group



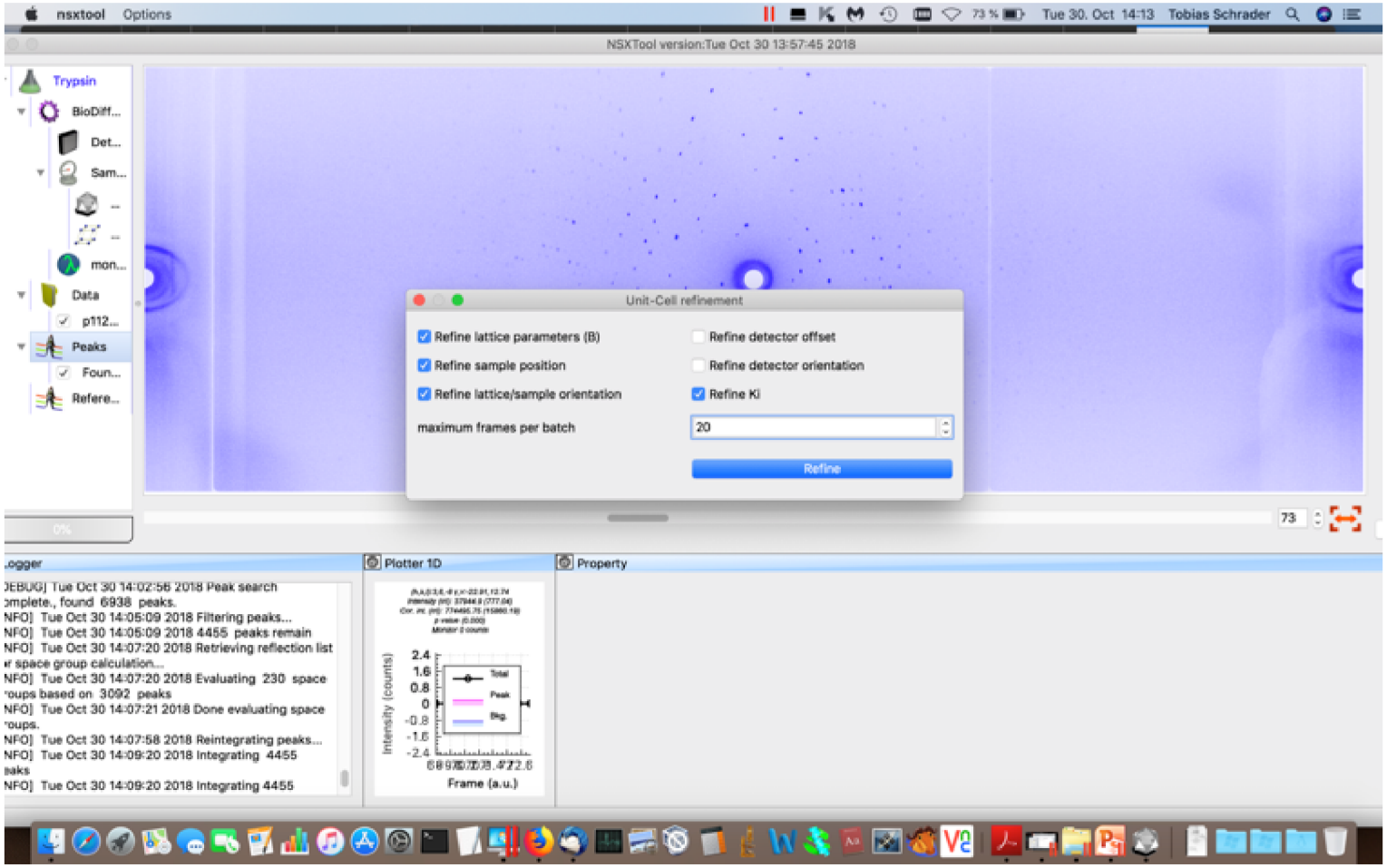
NXStool step by step

- Selecting the integration method



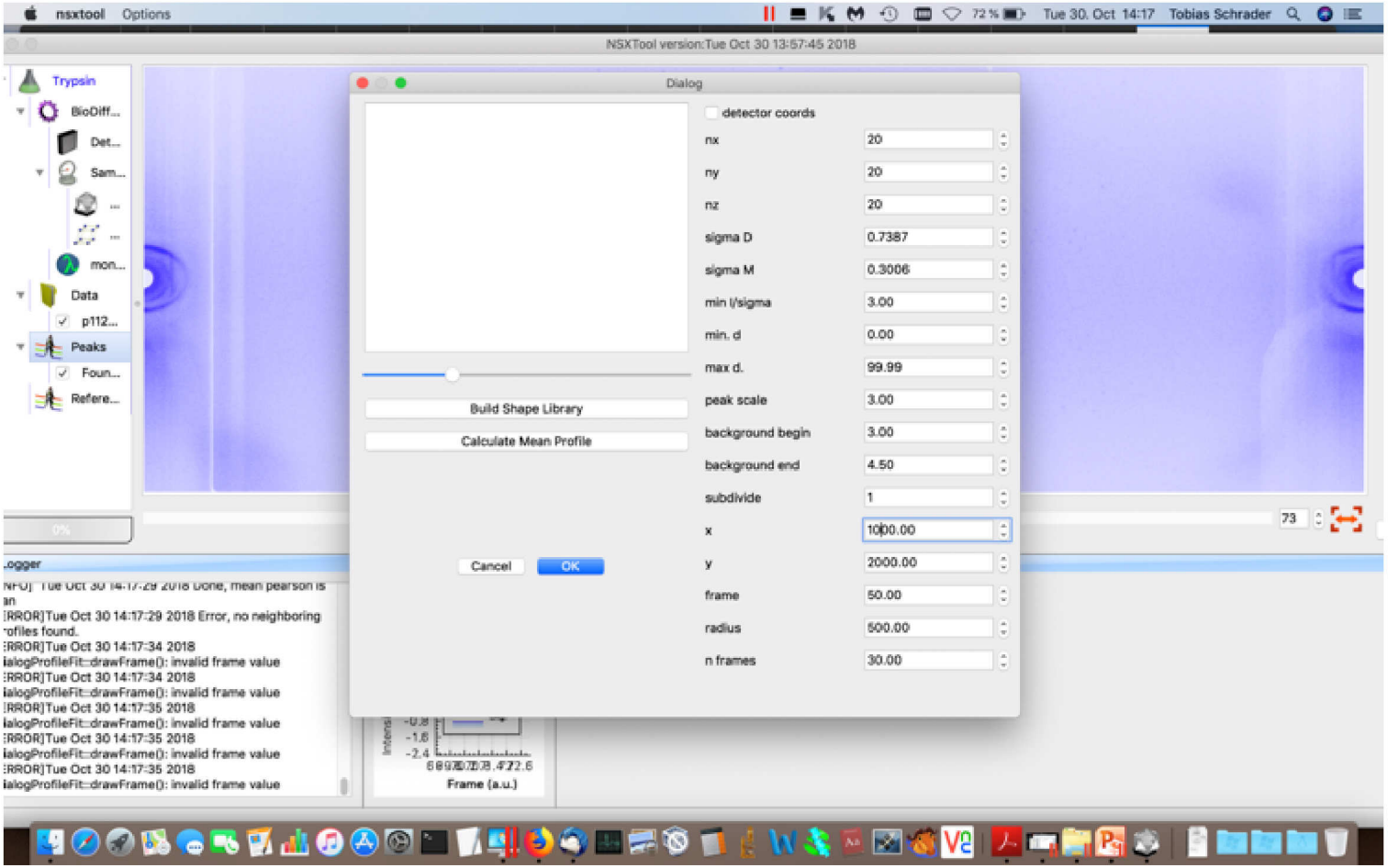
NXStool step by step

- Refinement of beam position and direction and other instrumental parameters



NXStool step by step

- Building a shape library for profile fitting



NXStool step by step

- Result of the integration

dmax	dmin	nobs	nmerged	redundancy	Rmeas	Rmeas(est.)	CChalf
19.78	4.19	1612	1153	1.398	0.166	0.016	0.853
4.19	3.33	1187	859	1.382	0.167	0.019	0.837
3.33	2.91	630	484	1.302	0.146	0.022	0.855
2.91	2.64	203	158	1.285	0.177	0.028	0.787
2.64	2.45	100	81	1.235	0.169	0.029	0.818
2.45	2.31	68	58	1.172	0.382	0.056	0.509
2.31	2.19	41	33	1.242	0.104	0.035	0.889
2.19	2.10	18	13	1.385	0.135	0.033	0.866
2.10	2.02	6	5	1.200	0.899	0.054	nan
2.02	1.95	5	4	1.250	0.230	0.040	nan
19.78	1.95	3871	2845	1.361	0.165	0.019	0.842

- Result of HKL2000, the old Software in use:

Shell limit	Lower Angstrom	Upper	Average I	Average error	Average stat.	Norm. Chi**2	Linear R-fac	Square R-fac	Rmeas	Rpim	CC1/2	CC*
50.00	3.23		7052.9	216.6	166.1	1.593	0.034	0.117	0.043	0.026	0.942	0.985
3.23	2.56		3943.3	243.3	226.3	1.403	0.061	0.058	0.077	0.046	0.991	0.998
2.56	2.24		2919.6	321.9	315.4	1.505	0.099	0.101	0.128	0.080	0.968	0.992
2.24	2.04		2634.7	384.7	380.1	1.418	0.123	0.123	0.161	0.103	0.950	0.987
2.04	1.89		2324.2	416.1	412.6	1.160	0.141	0.132	0.188	0.123	0.946	0.986
1.89	1.78		1894.2	422.0	419.7	1.211	0.173	0.167	0.235	0.157	0.905	0.975
1.78	1.69		1596.5	407.5	405.8	1.154	0.194	0.185	0.261	0.173	0.894	0.972
1.69	1.62		1284.7	376.6	375.4	1.211	0.222	0.211	0.294	0.191	0.871	0.965
1.62	1.55		1003.5	335.2	334.4	1.187	0.263	0.253	0.348	0.225	0.814	0.947
1.55	1.50		732.3	289.9	289.4	1.203	0.314	0.316	0.417	0.272	0.714	0.913
All reflections			2847.7	332.0	320.6	1.368	0.085	0.114	0.111	0.070		

Current status

- Programme runs on our Mac-Computers
- Support available from Eric Pellegrini at ILL
- Python scripting available but not rolled out to Andreas and me
- R-factors are still a bit worse than in the old denzo/HKL2000 case
- New programmer is needed to remove all remaining bugs and to do some more testing…
- **Close contact and many discussions with this programmer is needed**

- Ideas: To use McStas Calculations for improving the prediction of the centre of the predicted reflections
- Better absorption correction by using the convex hull of the crystal in correcting the Bragg intensities

Thanks

- Jonathan Fisher
- Andreas Ostermann
- Eric Pellegrini
- Laurent Chapon
- Joachim Wuttke

- and you for the attention to my talk!!