

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

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**Eric Pellegrini,**  
**Jonathan Fisher**  
**Laurent Chapon**

MLZ is a cooperation between:

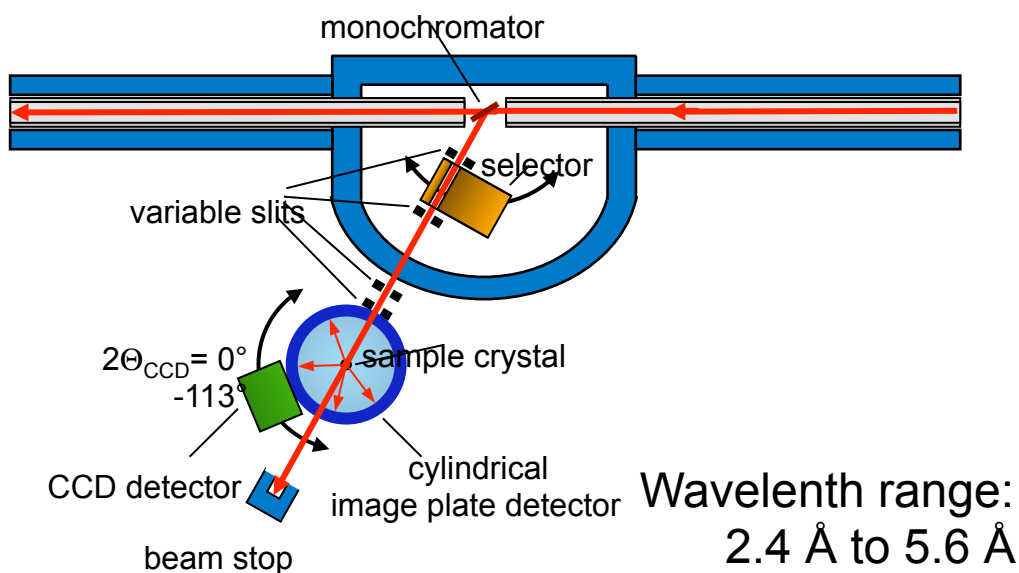
# Jürgen Miksch, Founder of BISS

- 1984 bis 1993 als stellvertretender Direktor der Evangelischen Akademie Tutzing

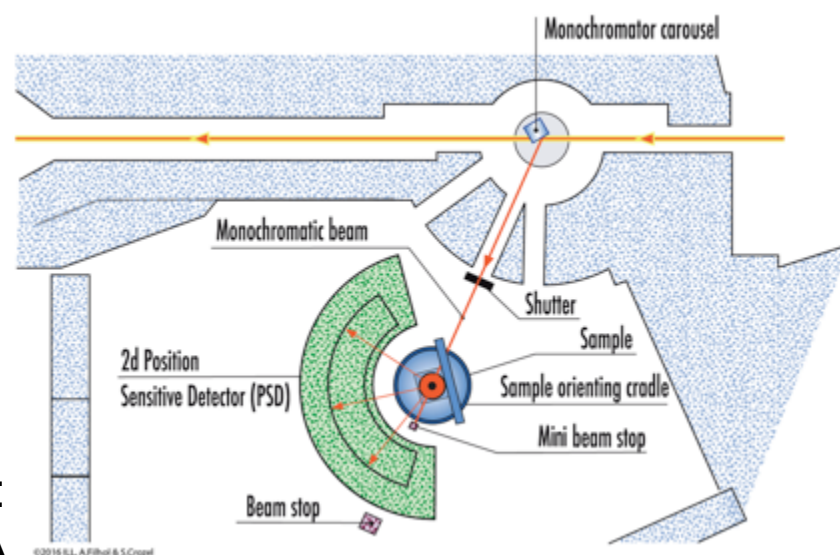


# Instruments BIODIFF at MLZ and D19 at ILL

- Both monochromatic neutron diffractometers with cylindrical detector

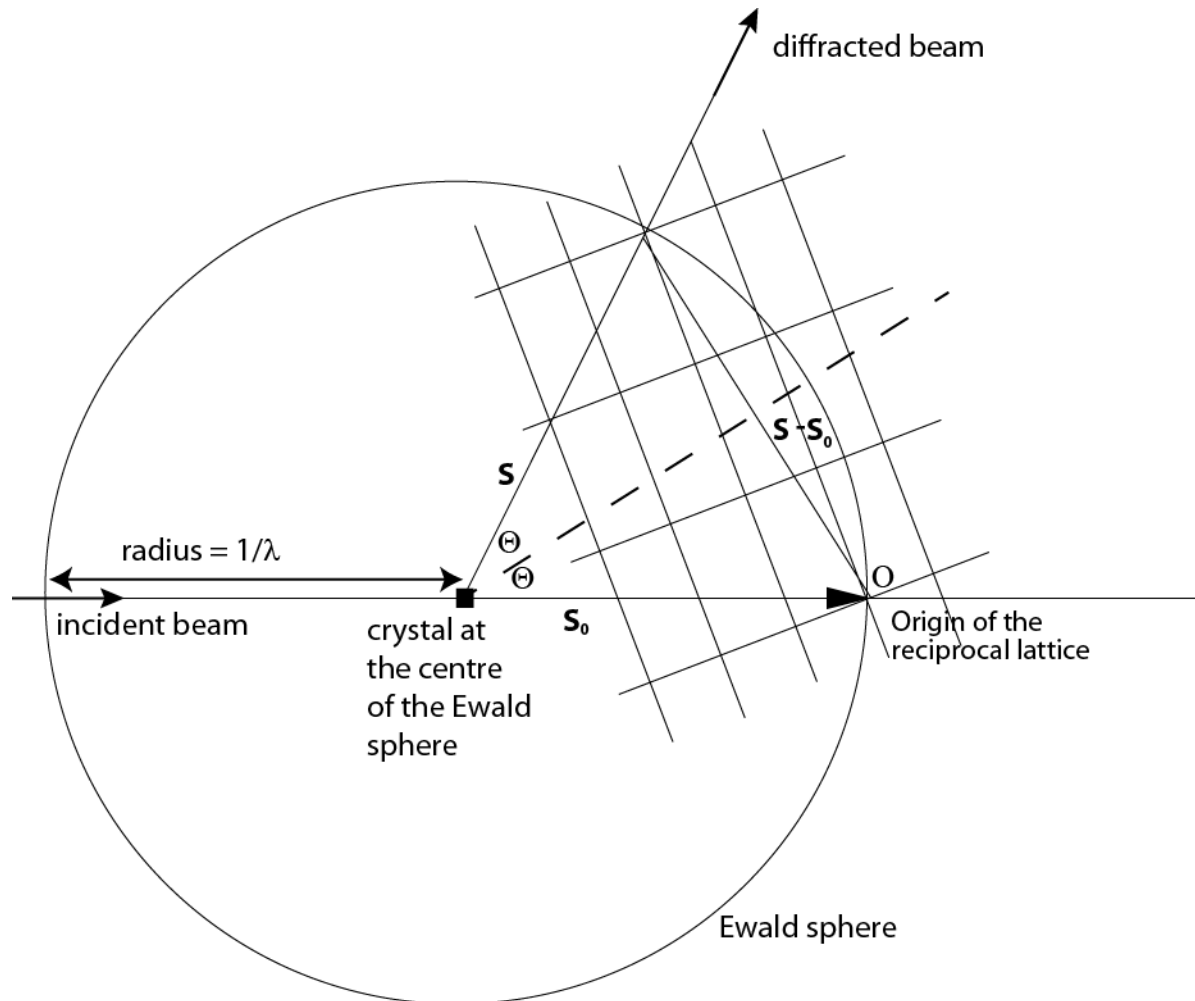


BIODIFF

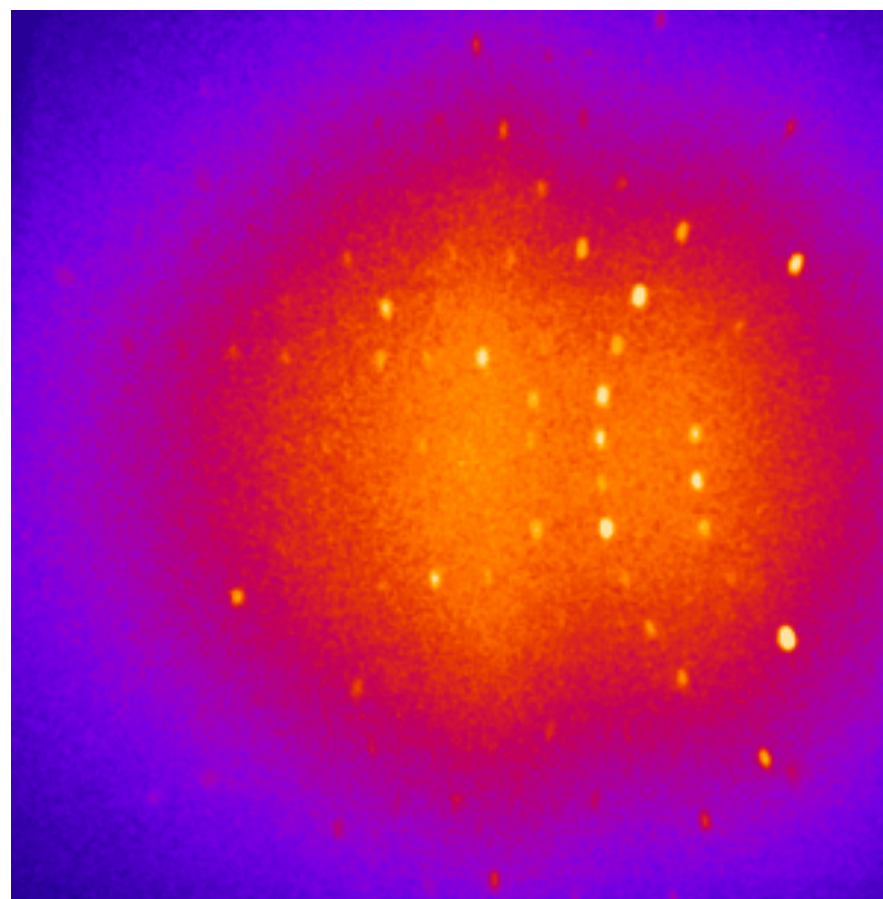
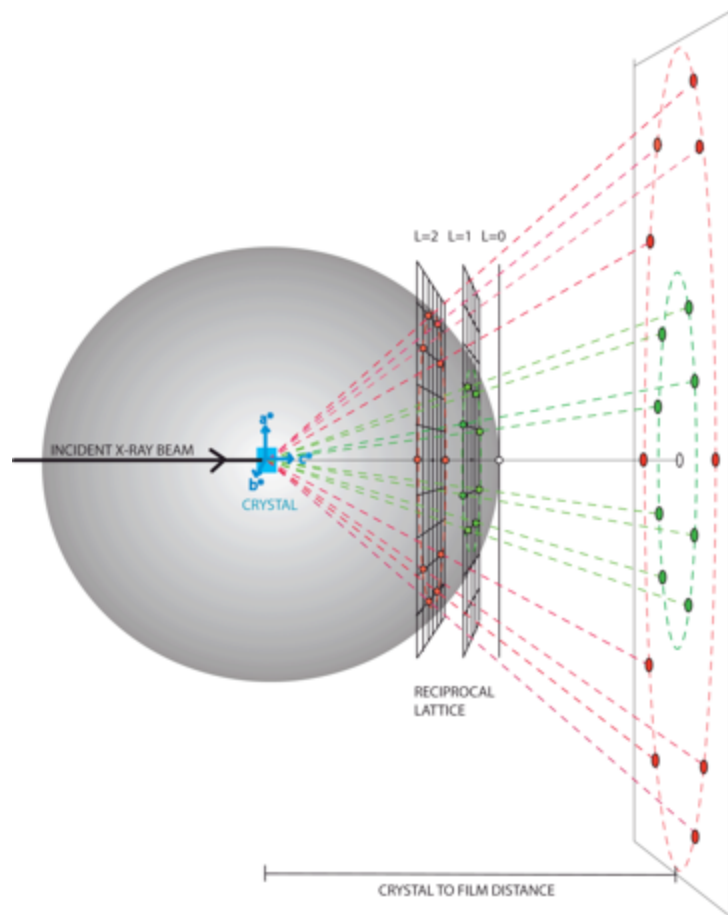


D19

# 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

Scaling of each hkl list to match each other

Unified hkl-list of measurement := complete data set

Calculation of a first map

-SCALA (CCP4-program package)

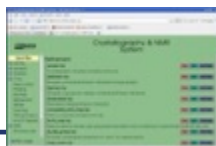
Additional information from the  
solution of the phase problem

**Structure refinement**

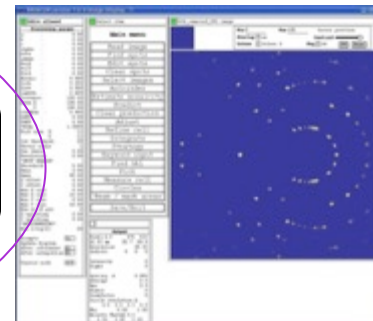
- Refinement of atom coordinates displacements
- Calculation of scattering density maps (neutrons) or electron density maps (x-rays)

**Map-plotting**

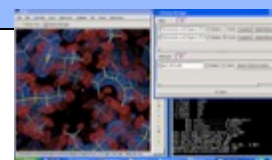
- inspection of model to fit the map)
- real space changes and refinement to the model



-nCNS  
-PHENIX



-MOSFLM  
-HKL-denzo  
(comercial)



-XtalView  
-Coot



# Peak search with hkl DENZO

The screenshot shows the hkl DENZO software interface. The main window displays a diffraction pattern with red spots. A zoomed-in view of a specific spot is shown on the right. The interface includes a menu bar, a toolbar, and a status bar. Below the main window, there are two terminal windows showing command-line output and a web browser window displaying the FRM II WebMail Interface.

**Terminal 1 (Left):**

```
jcns@phys:~/DENZO/denzo_1_96/real_data
File Edit View Terminal Help
-rw-rw-r-- 1 jcns jcns 5512500 Nov 5 18:03 309_01_001.raw
-rw-rw-r-- 1 jcns jcns 1013 Nov 5 18:04 auto_index_sim_spotb.dat
[jcns@phys real_data]$ ls -ltr
total 16148
-rwxr-xr-x 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-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 1269 Nov 2 19:07 peaks.file
-rw-rw-r-- 1 jcns jcns 441 Nov 2 19:09 refineone.dat
-rw-rw-r-- 1 jcns jcns 14288 Nov 2 19:13 hklpredictions
-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
[jcns@phys real_data]$
```

**Terminal 2 (Right):**

```
jcns@phys:~/DENZO/denzo_1_96/real_data
File Edit View Terminal Help
-rw-rw-r-- 1 jcns jcns 1047 Nov 2 19:03 auto_index_sim_spotb.dat
-rw-rw-r-- 1 jcns jcns 496 Nov 2 19:03 refineone.dat
```

**Web Browser (Bottom Right):**

Reaktor-Info: Forschungs-Neutronenquelle Heinz Maier-Leibnitz (FRM II) - Mozilla Firefox

File Edit View History Bookmarks Tools Help

http://www.frm2.tum.de/intern/funktionen/reaktor-info/index.html

Most Visited Release Notes Fedora Project Red Hat Free Content

Reaktor-Info: Forschungs-Neutronenquelle Heinz Maier-Leibnitz (FRM II) - Mozilla Firefox

Telefondatenbank (intern)

Kostenverwaltung

Raumerhaltung

Raumbuchung GRS

Reaktor-Info

Webmail

19.8 MW

Shutterstellung NL-Anlage

# auto-index

Applications Places System Sat Nov 5, 18:24 JCS

/home/jcns/DENZO/denzo\_1\_96/real\_data/309\_01\_001.raw

Zoom in Zoom out Int. box Diff Vec Zoom close

close Frame

imax=1046720  
I=1805  
[298.6, 566.6]

W2  
Processing  
System  
W. Minor  
Z. Otwinowski

7272  
6363  
5454  
4545  
3636  
2727  
1818  
909  
0

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

```

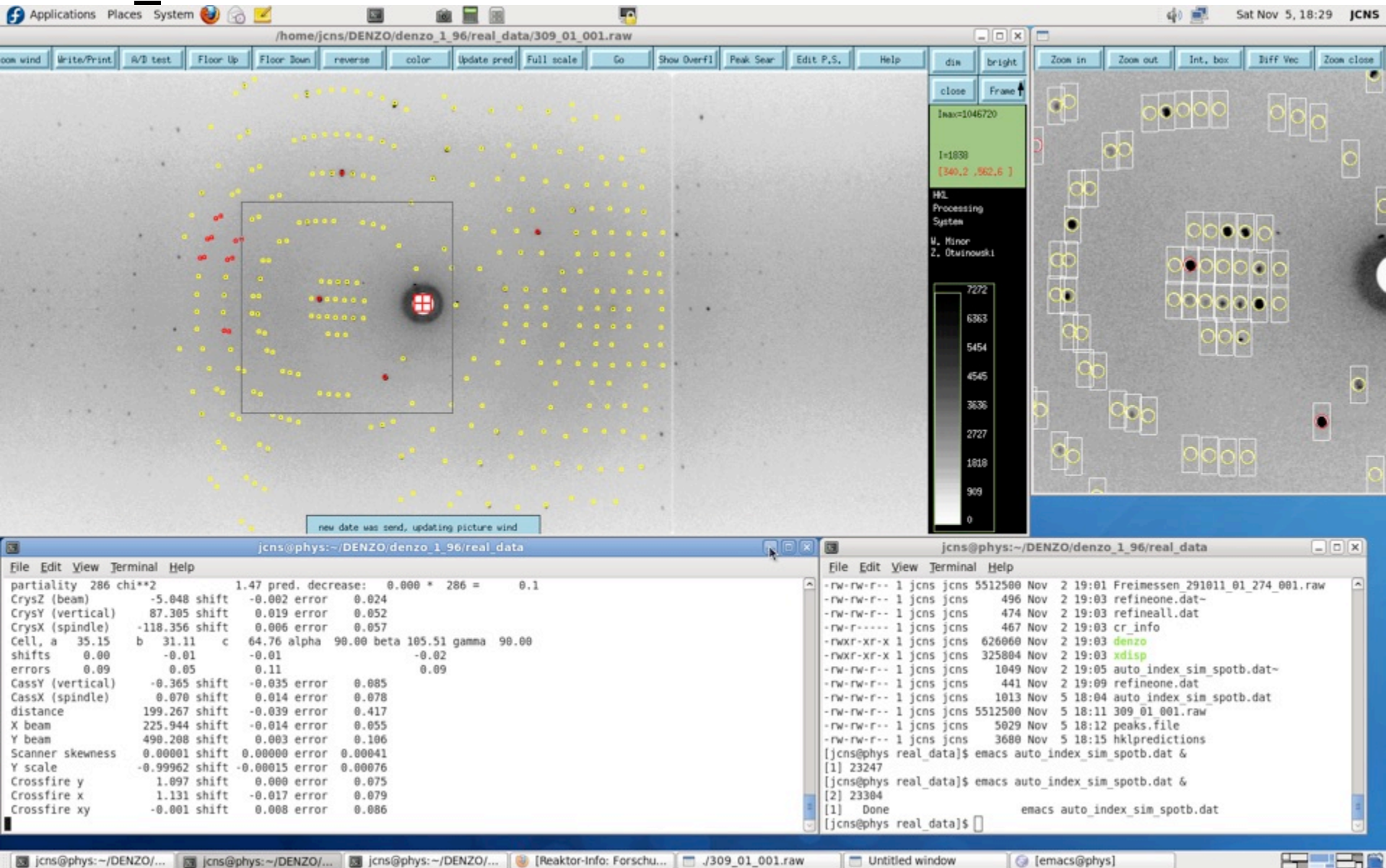
[jcns@phys real_data]$ ls -ltr
total 16140
-rwxr--r-- 1 jcns jcns 4507524 Nov 2 18:51 Freimessen_291011_01_274_0.tif
-rw-rw-r-- 1 jcns jcns 5512500 Nov 2 19:01 Freimessen_291011_01_274_001.raw
-rw-rw-r-- 1 jcns jcns 496 Nov 2 19:03 refineone.dat
-rw-rw-r-- 1 jcns jcns 474 Nov 2 19:03 refineall.dat
-rw-r--r-- 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 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]$

```

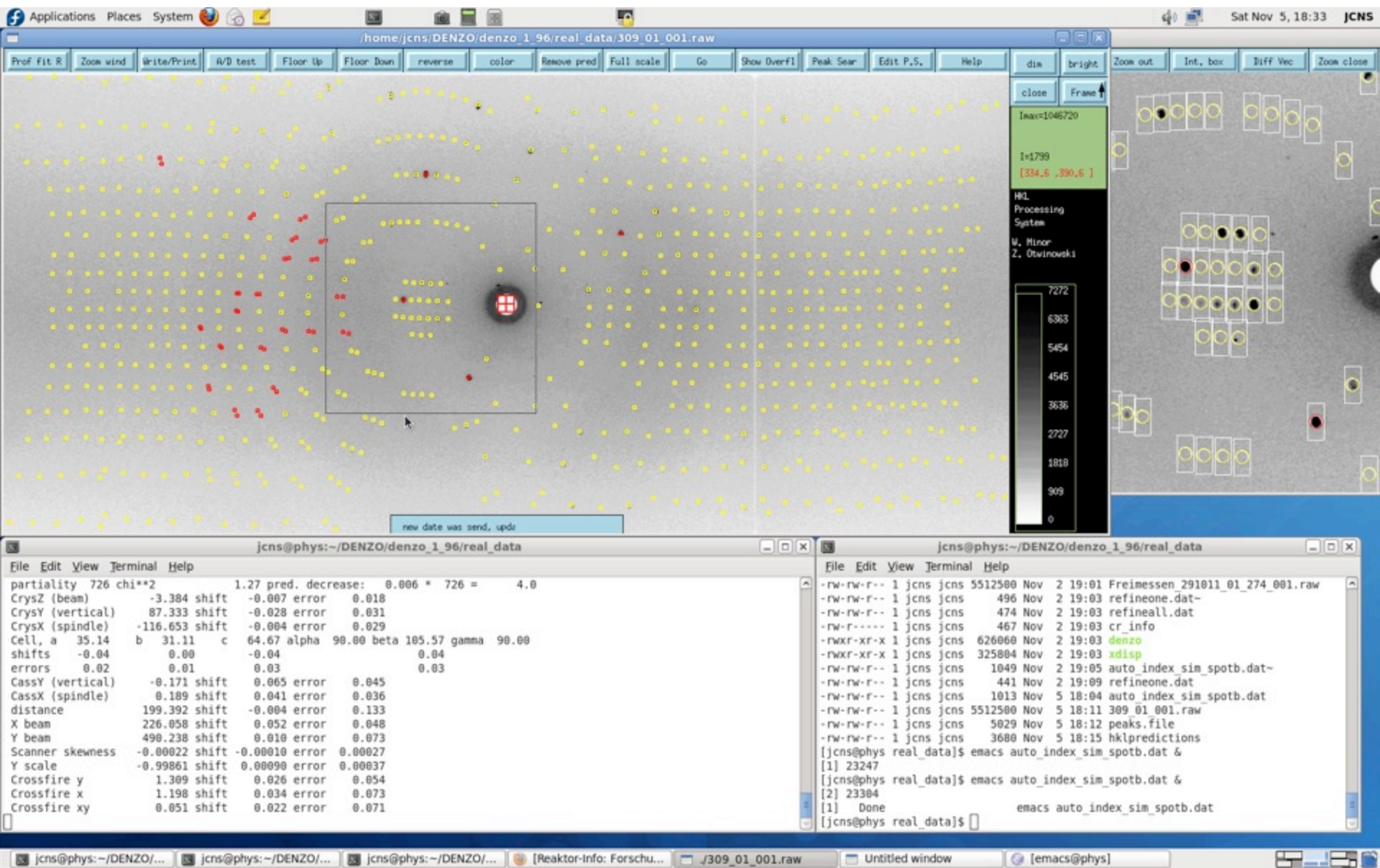
jcns@phys:~/DENZO/... jcns@phys:~/DENZO/... jcns@phys:~/DENZO/... [Reaktor-Info: Forschu... /309\_01\_001.raw Untitled window



# d<sub>min</sub>=2.5 Å

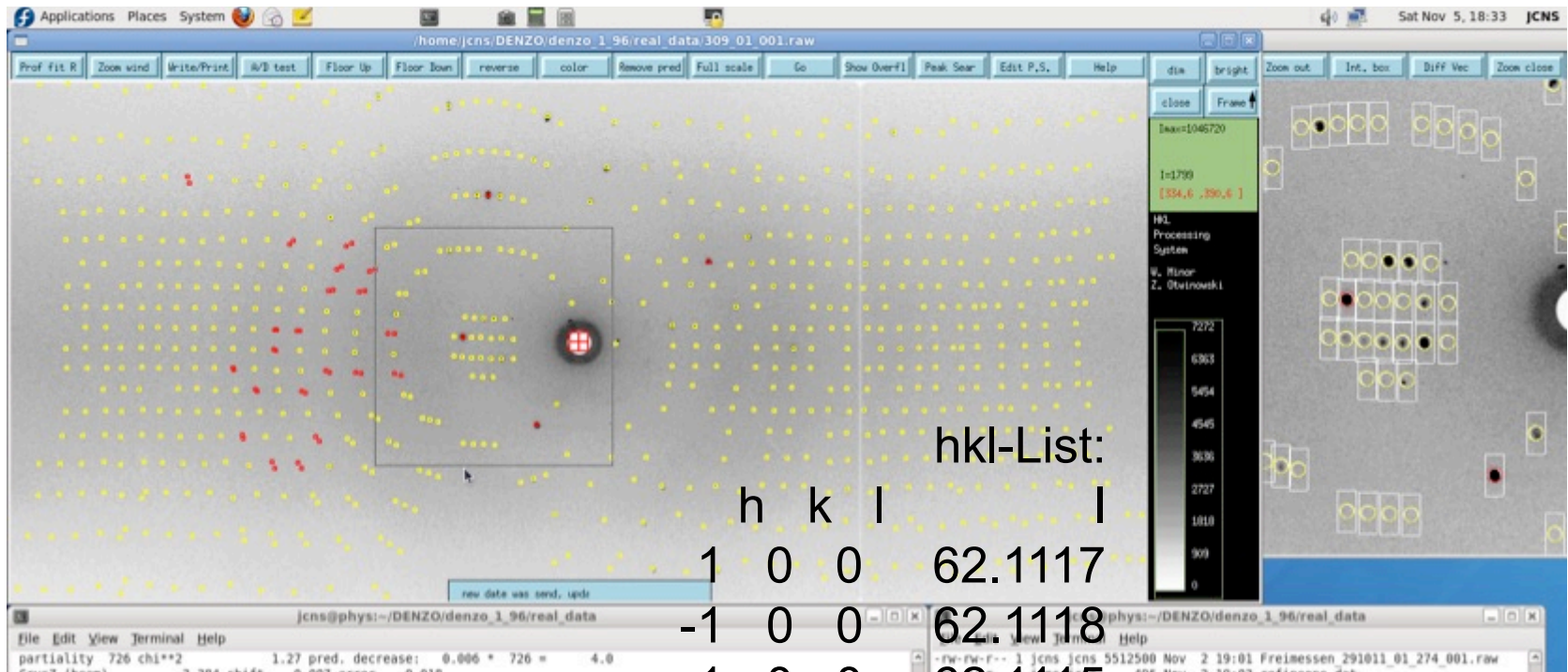


# $d_{\min}=1.5 \text{ \AA}$





# Integration of partial Bragg peaks with the commercial software hkl-denzo up to $d_{\min}=1.5 \text{ \AA}$



## The „old“ HKL2000 software

- Not supported,
- Running under academic licence
- Need to re-new every 6 months
- 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!**

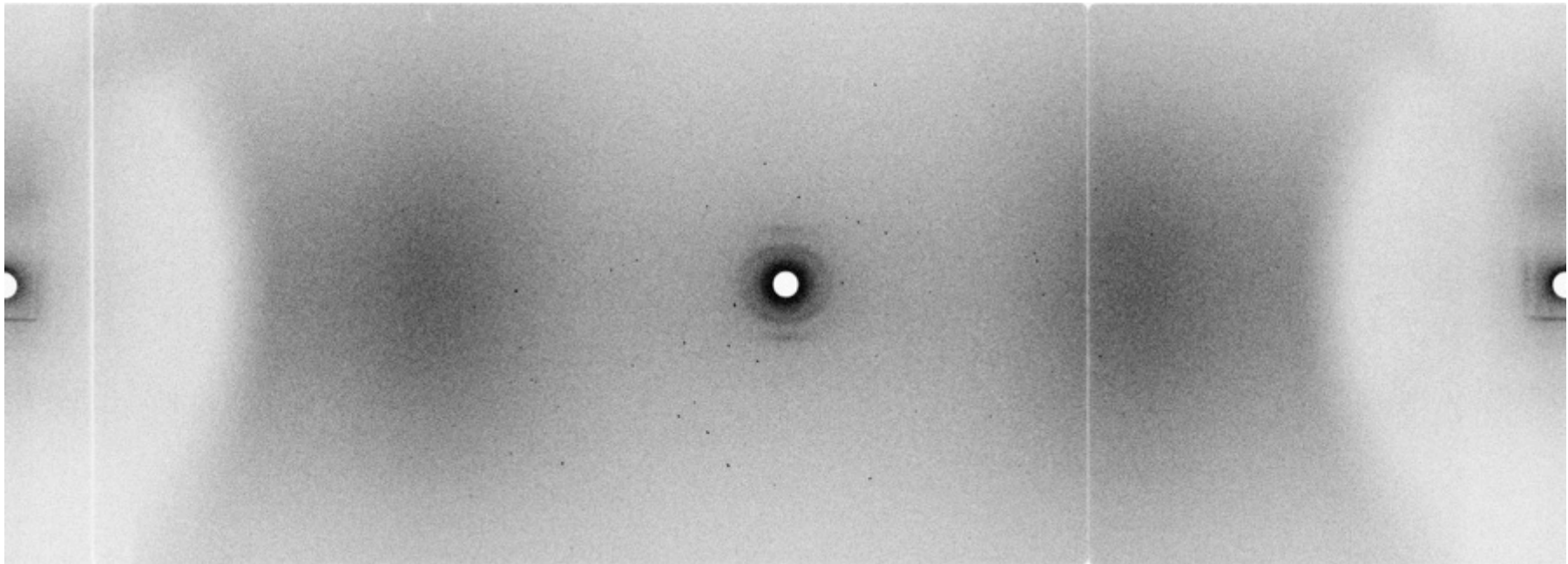


## Other alternatives

- 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
- ...???

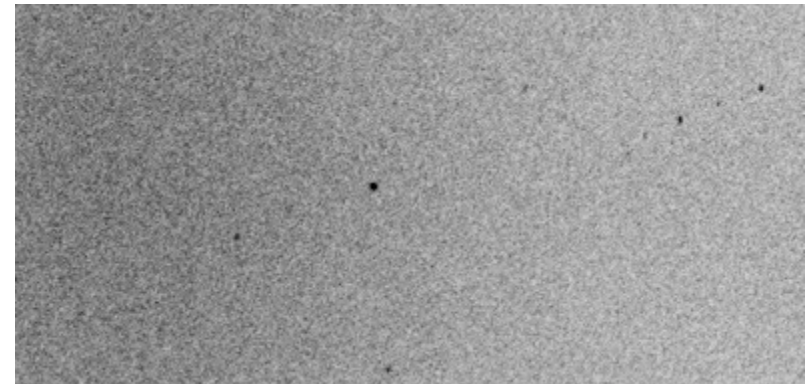
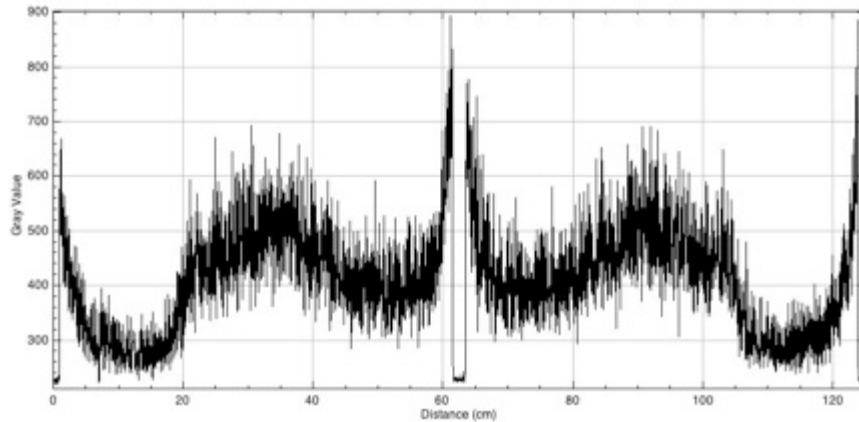
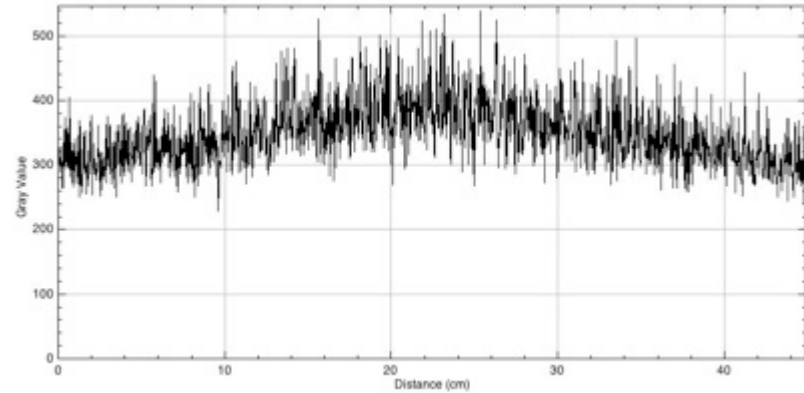
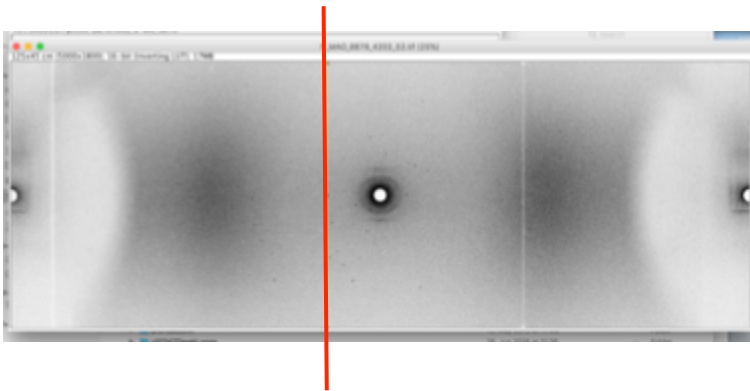
## Outline of the problem

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



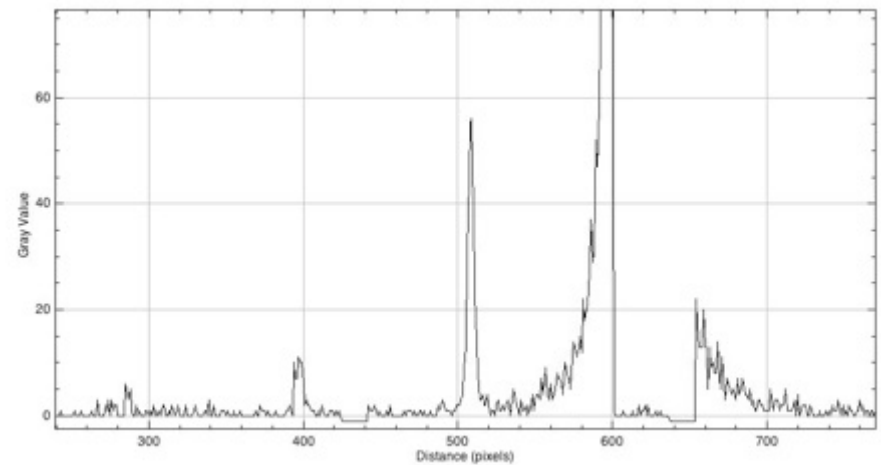
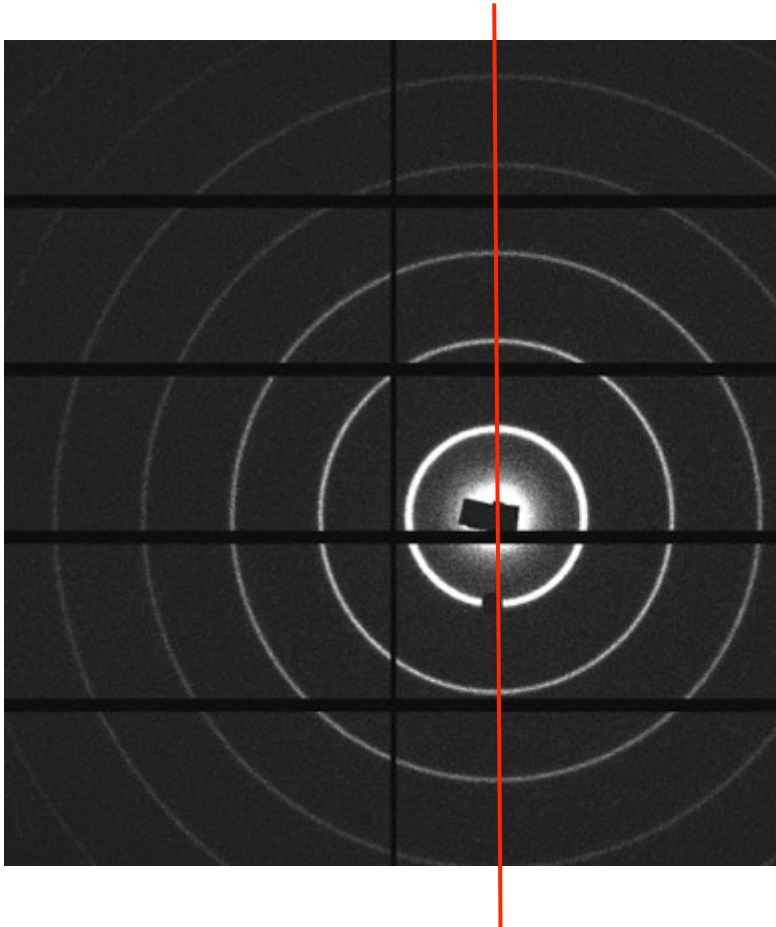
# Special issues regarding neutron crystallography as compared to x-ray

- Background much more pronounced and not so homogenous



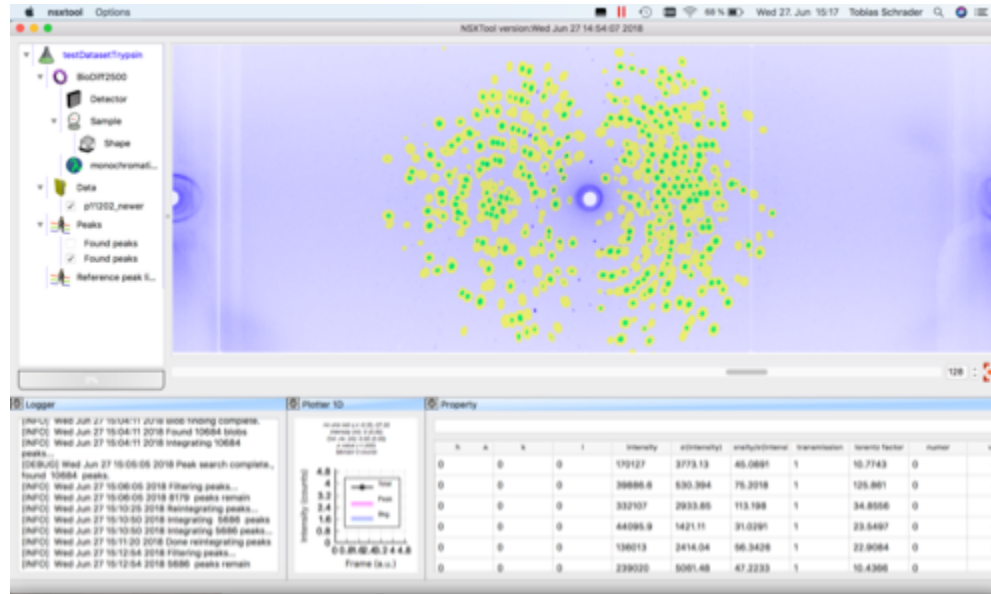
# The x-ray case for comparison

- 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



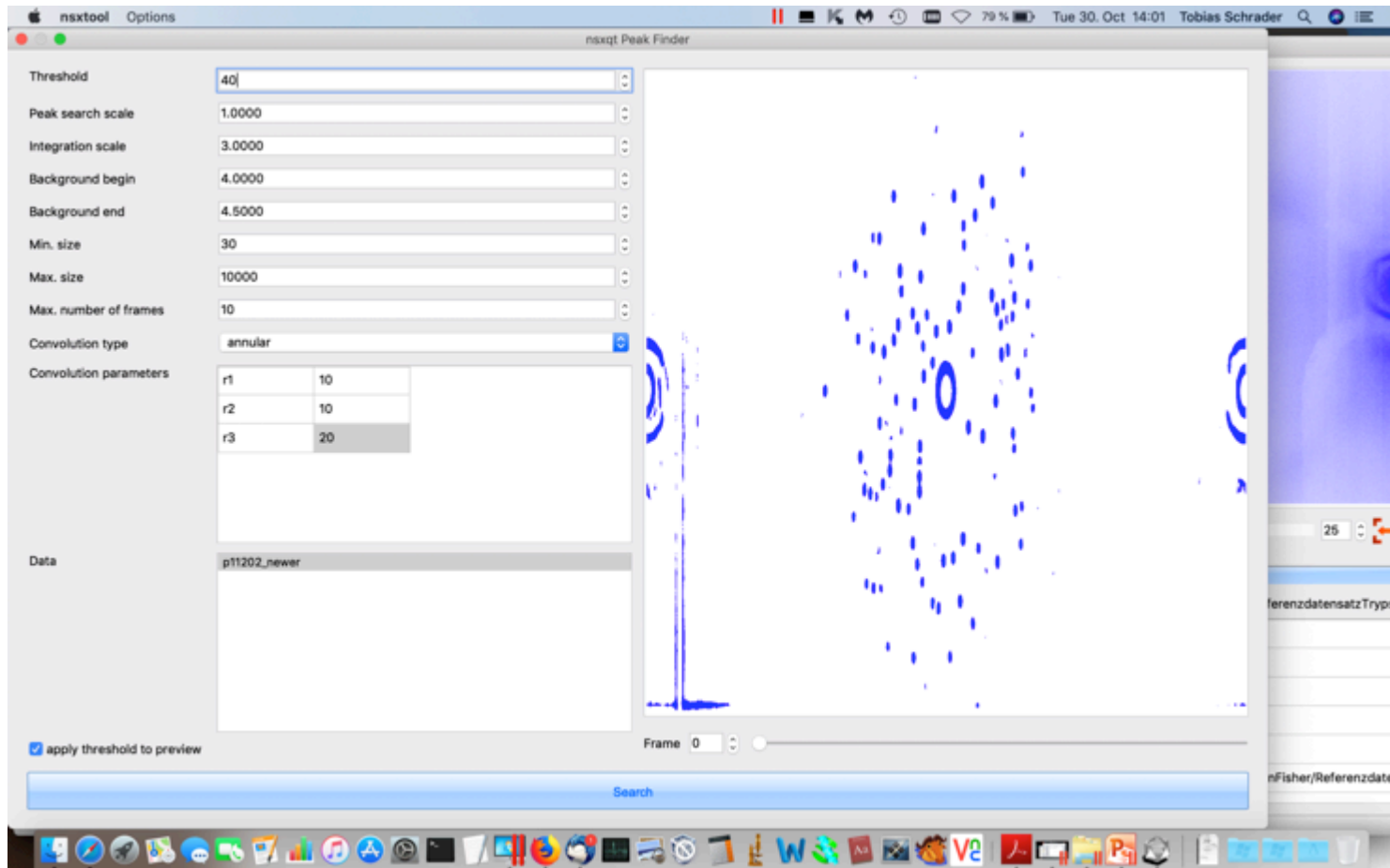
Eric Pellegrini

Jonathan Fisher



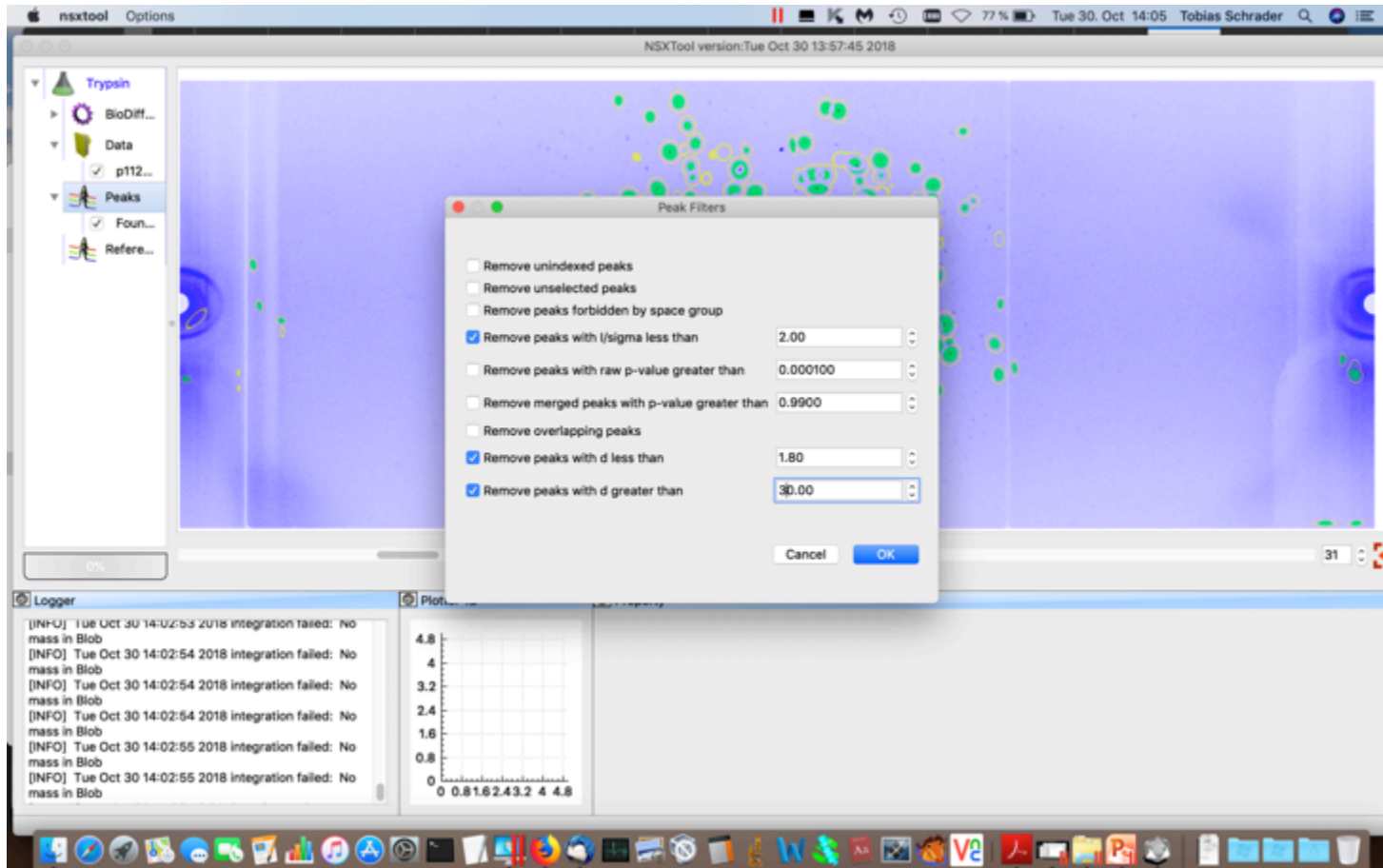
# NXStool step by step

- Peak finding based on image analysis



# NXStool step by step

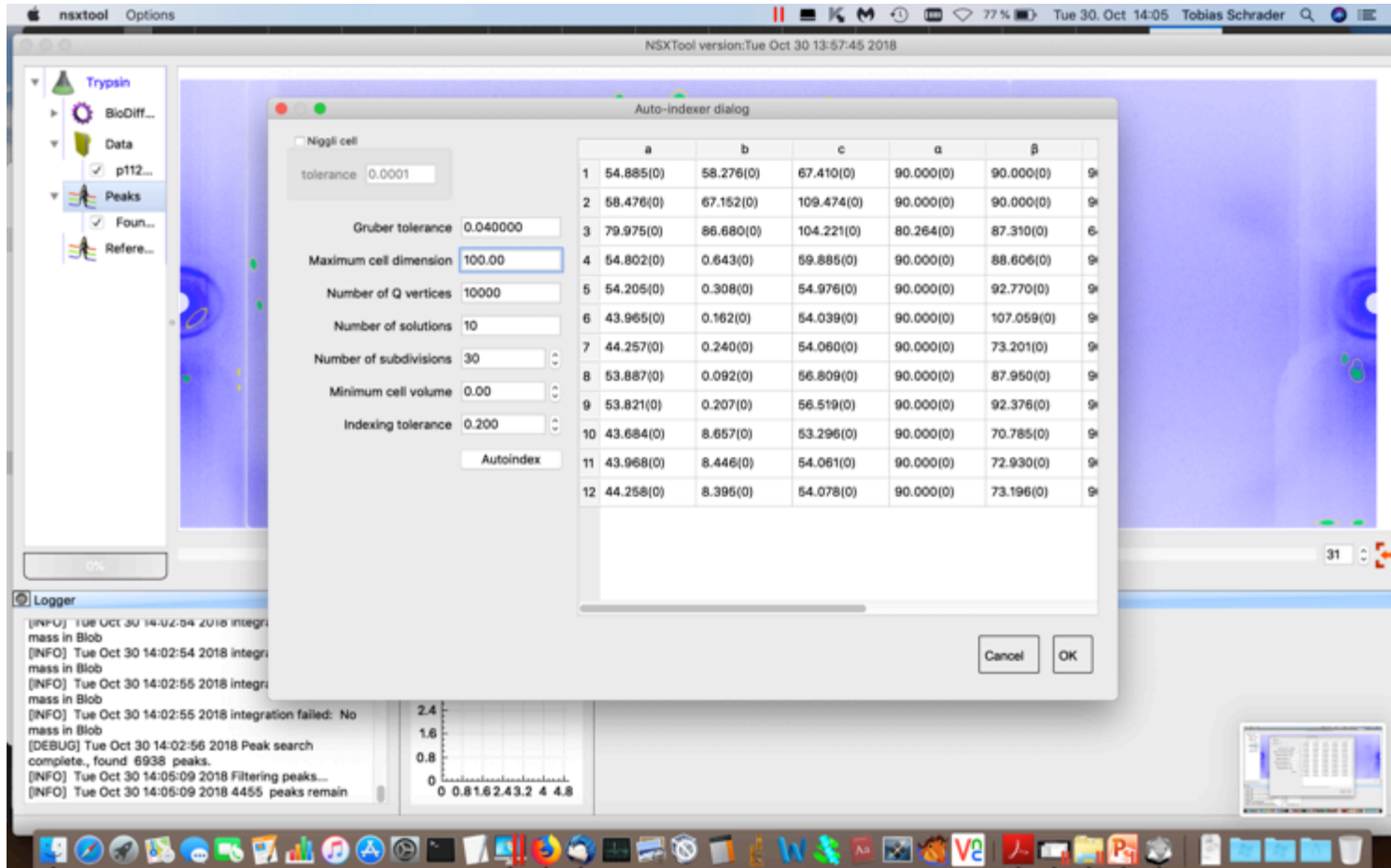
- Filtering of the peaks found by the image analysis





# NXStool step by step

- Indexing of the Peaks, finding the right unit cell



Auto-indexer dialog

☐ Niggli cell

tolerance 0.0001

Gruber tolerance 0.040000

Maximum cell dimension 100.00

Number of Q vertices 10000

Number of solutions 10

Number of subdivisions 30

Minimum cell volume 0.00

Indexing tolerance 0.200

Autoindex

	a	b	c	$\alpha$	$\beta$	
1	54.885(0)	58.276(0)	67.410(0)	90.000(0)	90.000(0)	9
2	58.476(0)	67.152(0)	109.474(0)	90.000(0)	90.000(0)	9
3	79.975(0)	86.680(0)	104.221(0)	80.264(0)	87.310(0)	6
4	54.802(0)	0.643(0)	59.885(0)	90.000(0)	88.606(0)	9
5	54.205(0)	0.308(0)	54.976(0)	90.000(0)	92.770(0)	9
6	43.965(0)	0.162(0)	54.039(0)	90.000(0)	107.059(0)	9
7	44.257(0)	0.240(0)	54.060(0)	90.000(0)	73.201(0)	9
8	53.887(0)	0.092(0)	56.809(0)	90.000(0)	87.950(0)	9
9	53.821(0)	0.207(0)	56.519(0)	90.000(0)	92.376(0)	9
10	43.684(0)	8.657(0)	53.296(0)	90.000(0)	70.785(0)	9
11	43.968(0)	8.446(0)	54.061(0)	90.000(0)	72.930(0)	9
12	44.258(0)	8.395(0)	54.078(0)	90.000(0)	73.196(0)	9

Cancel OK

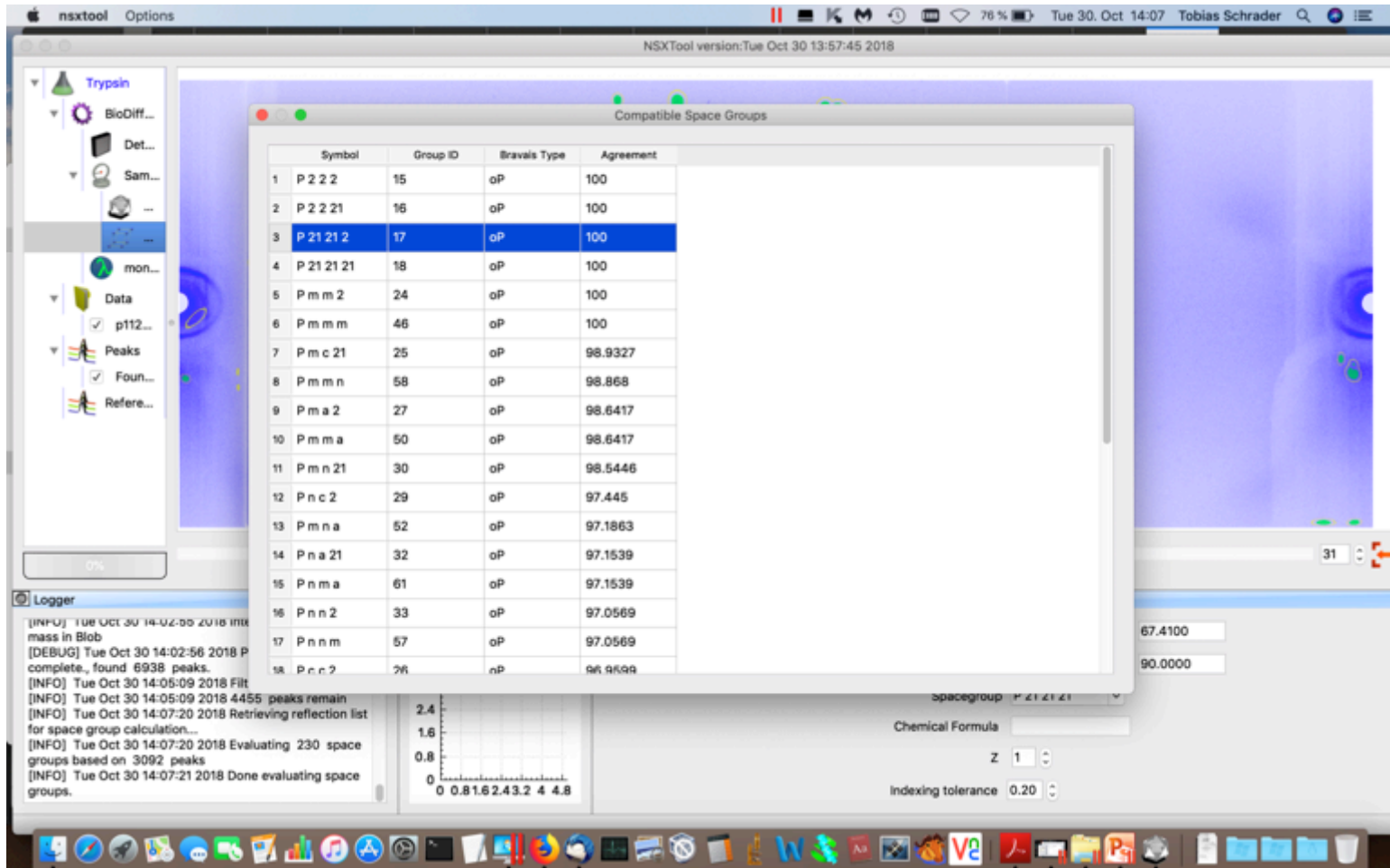
Logger

[INFO] Tue Oct 30 14:02:54 2018 integr...  
 mass in Blob  
 [INFO] Tue Oct 30 14:02:54 2018 integr...  
 mass in Blob  
 [INFO] Tue Oct 30 14:02:55 2018 integr...  
 mass in Blob  
 [INFO] Tue Oct 30 14:02:55 2018 integration failed: No  
 mass in Blob  
 [DEBUG] Tue Oct 30 14:02:56 2018 Peak search  
 complete., found 6938 peaks.  
 [INFO] Tue Oct 30 14:05:09 2018 Filtering peaks...  
 [INFO] Tue Oct 30 14:05:09 2018 4455 peaks remain

2.4  
1.6  
0.8  
0  
0 0.8 1.6 2.4 3.2 4 4.8

# NXStool step by step

- Selecting the space group



NSXTool version: Tue Oct 30 13:57:45 2018

Compatible Space Groups

	Symbol	Group ID	Bravais Type	Agreement
1	P 2 2 2	15	oP	100
2	P 2 2 21	16	oP	100
3	P 21 21 2	17	oP	100
4	P 21 21 21	18	oP	100
5	P m m 2	24	oP	100
6	P m m m	46	oP	100
7	P m c 21	25	oP	98.9327
8	P m m n	58	oP	98.868
9	P m a 2	27	oP	98.6417
10	P m m a	50	oP	98.6417
11	P m n 21	30	oP	98.5446
12	P n c 2	29	oP	97.445
13	P m n a	52	oP	97.1863
14	P n a 21	32	oP	97.1539
15	P n m a	61	oP	97.1539
16	P n n 2	33	oP	97.0569
17	P n n m	57	oP	97.0569
18	P c c 2	26	nP	96.9699

Logger

[INFO] Tue Oct 30 14:02:35 2018 Int mass in Blob  
 [DEBUG] Tue Oct 30 14:02:56 2018 P complete., found 6938 peaks.  
 [INFO] Tue Oct 30 14:05:09 2018 Fit  
 [INFO] Tue Oct 30 14:05:09 2018 4455 peaks remain  
 [INFO] Tue Oct 30 14:07:20 2018 Retrieving reflection list for space group calculation...  
 [INFO] Tue Oct 30 14:07:20 2018 Evaluating 230 space groups based on 3092 peaks  
 [INFO] Tue Oct 30 14:07:21 2018 Done evaluating space groups.

Spacegroup: P 21 21 2

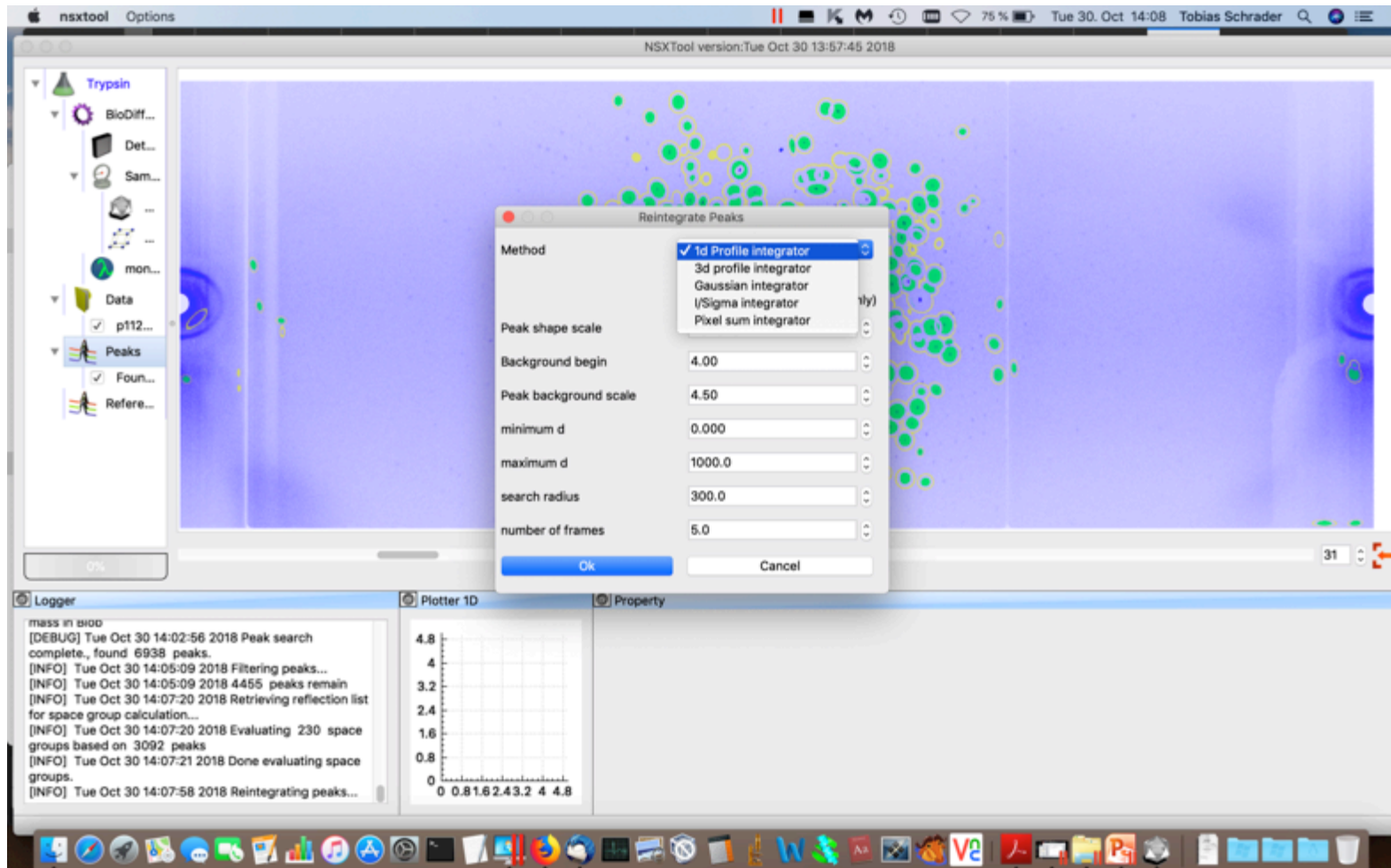
Chemical Formula

Z 1

Indexing tolerance 0.20

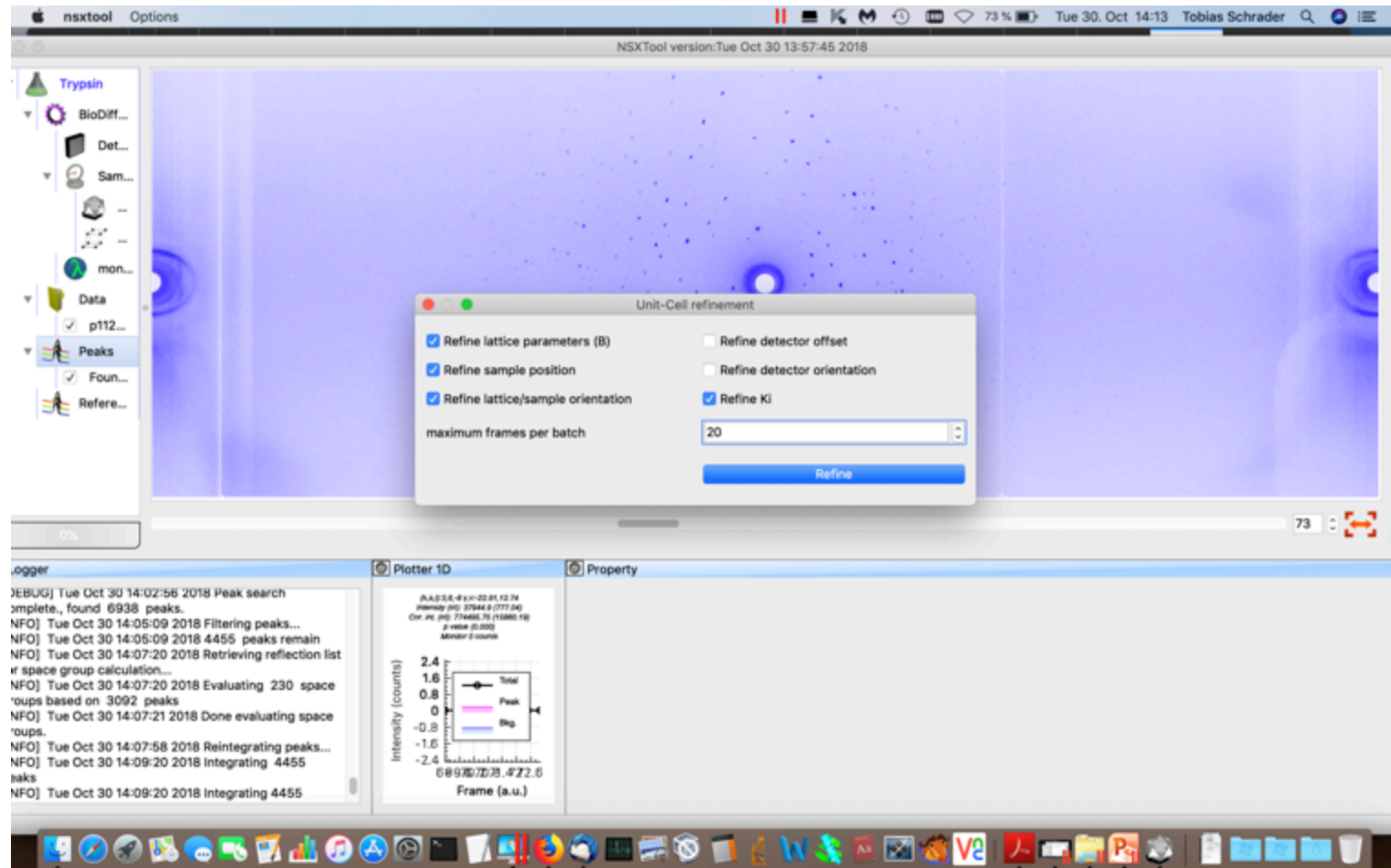
# NXStool step by step

- Selecting the integration method



# NXStool step by step

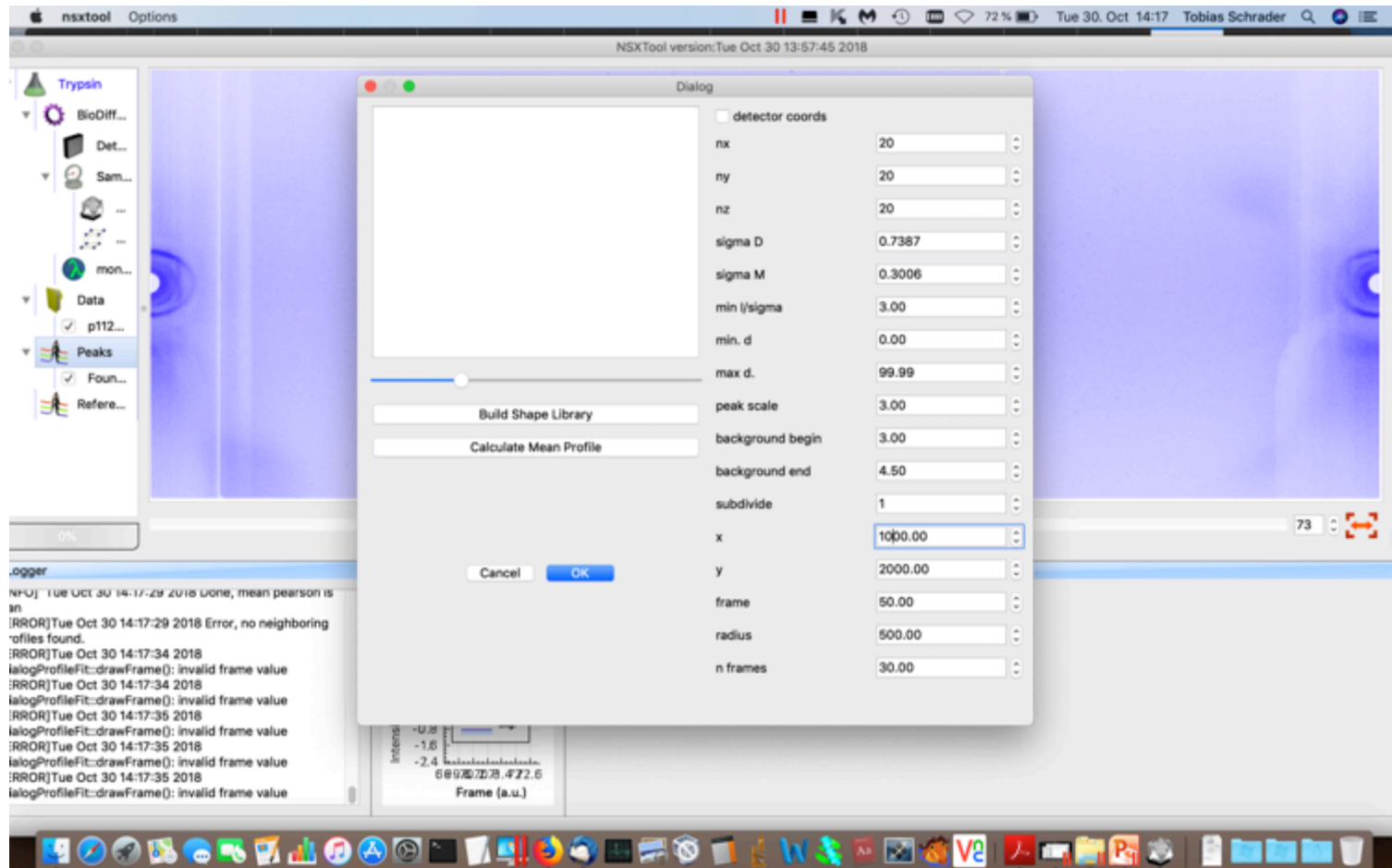
- Refinement of beam position and direction and other instrumental parameters





# NXStool step by step

- Building a shape library for profile fitting



- 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 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!!