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

Tobias E. Schrader

Andreas Ostermann

Eric Pellegrini,
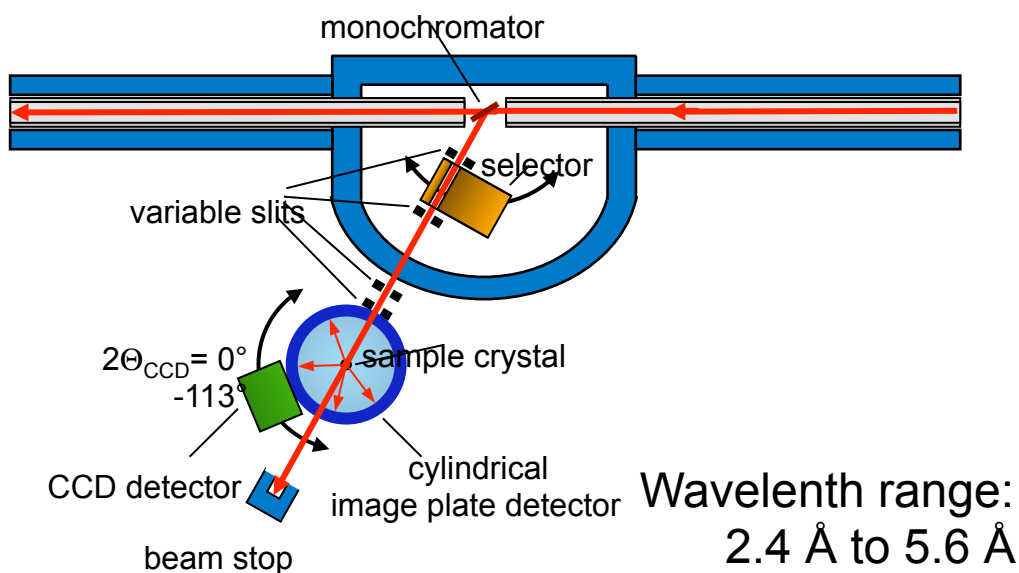
Jonathan Fisher

Laurent Chapon

Jürgen Miksch, Founder of BISS

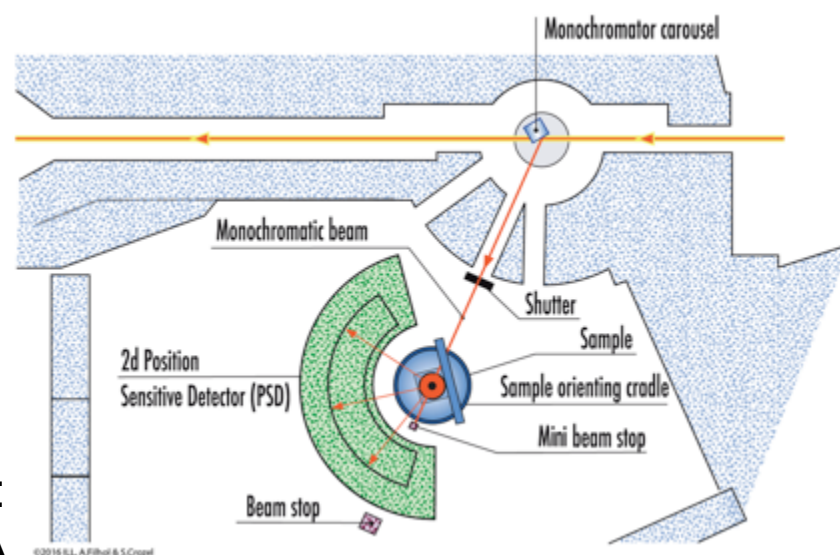- 1984 bis 1993 als stellvertretender Direktor der Evangelischen Akademie Tutzing

Instruments BIODIFF at MLZ and D19 at ILL

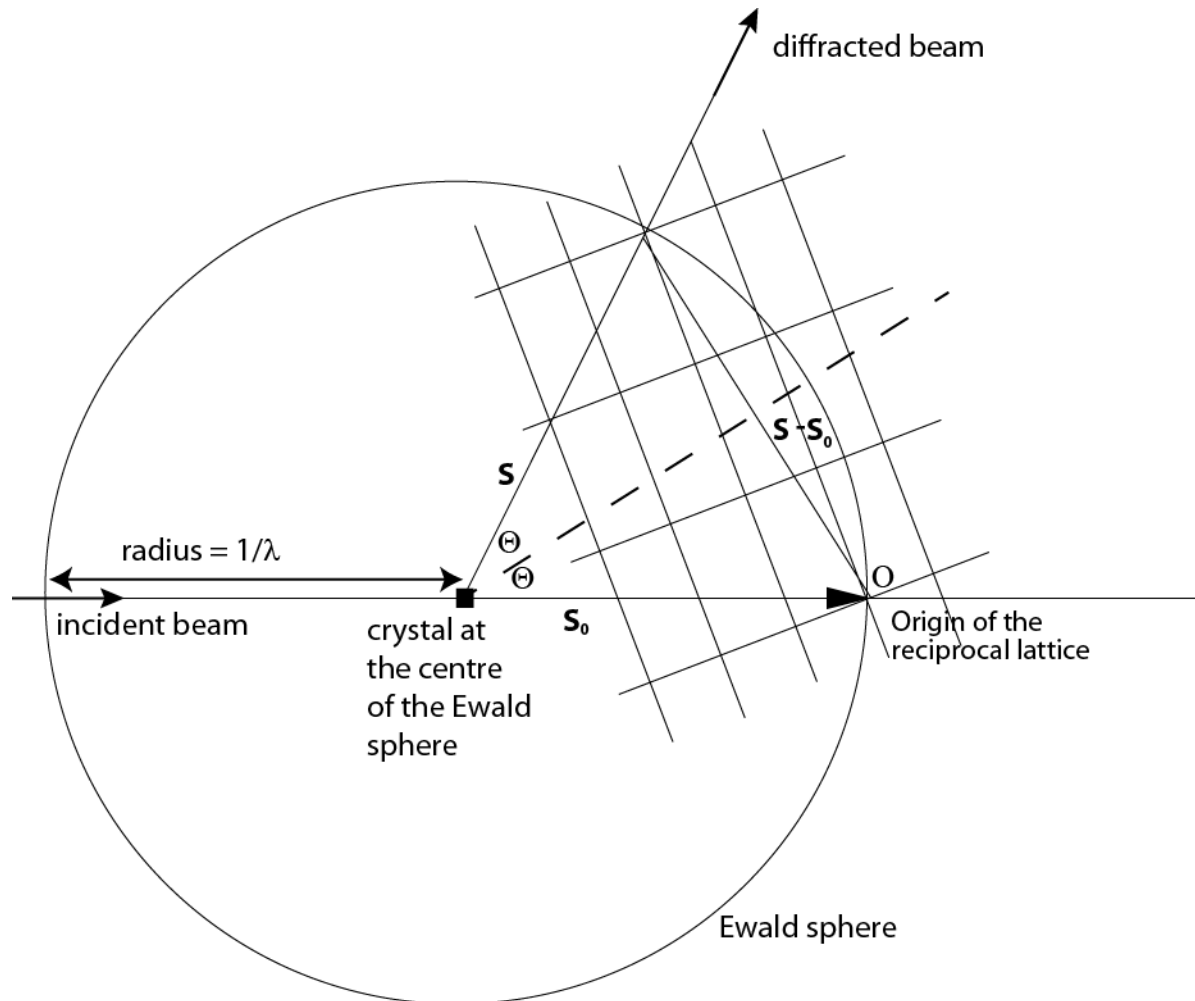- Both monochromatic neutron diffractometers with cylindrical detector

monochromator

selector

variable slits

$2\Theta_{CCD} = 0°$
-113°

sample crystal

CCD detector

cylindrical
image plate detector

beam stop

Wavelenth range:
2.4 Å to 5.6 Å

Monochromator carousel

Monochromatic beam

Shutter

Sample

2d Position
Sensitive Detector (PSD)

Sample orienting cradle
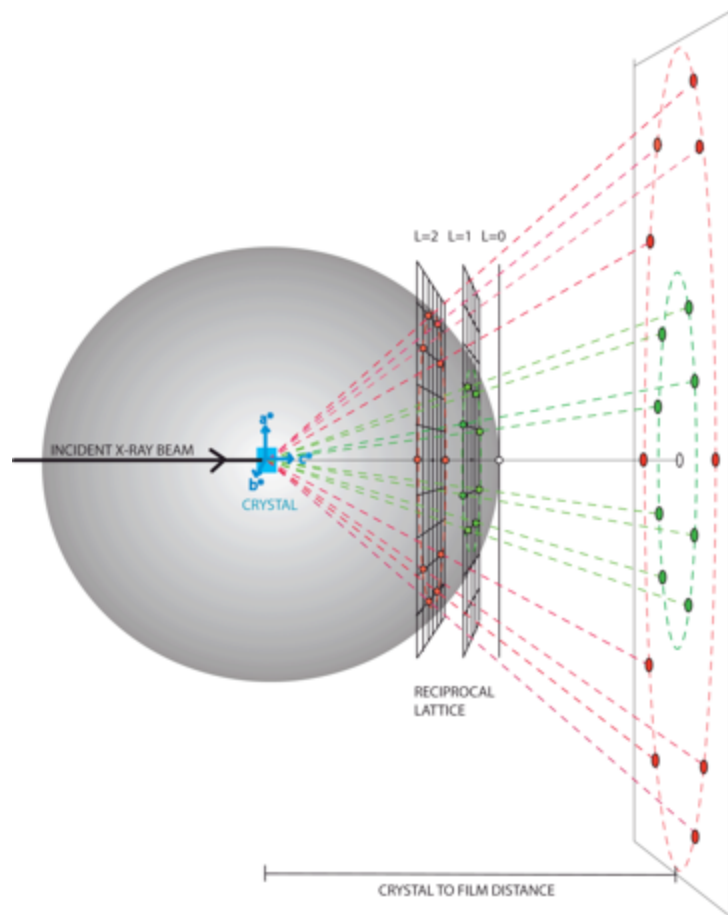
Mini beam stop

Beam stop

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BIODIFF

D19

Ewald construction and Bragg's Law

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

L=2 L=1 L=0

INCIDENT X-RAY BEAM

CRYSTAL

RECIPROCAL LATTICE

CRYSTAL TO FILM DISTANCE
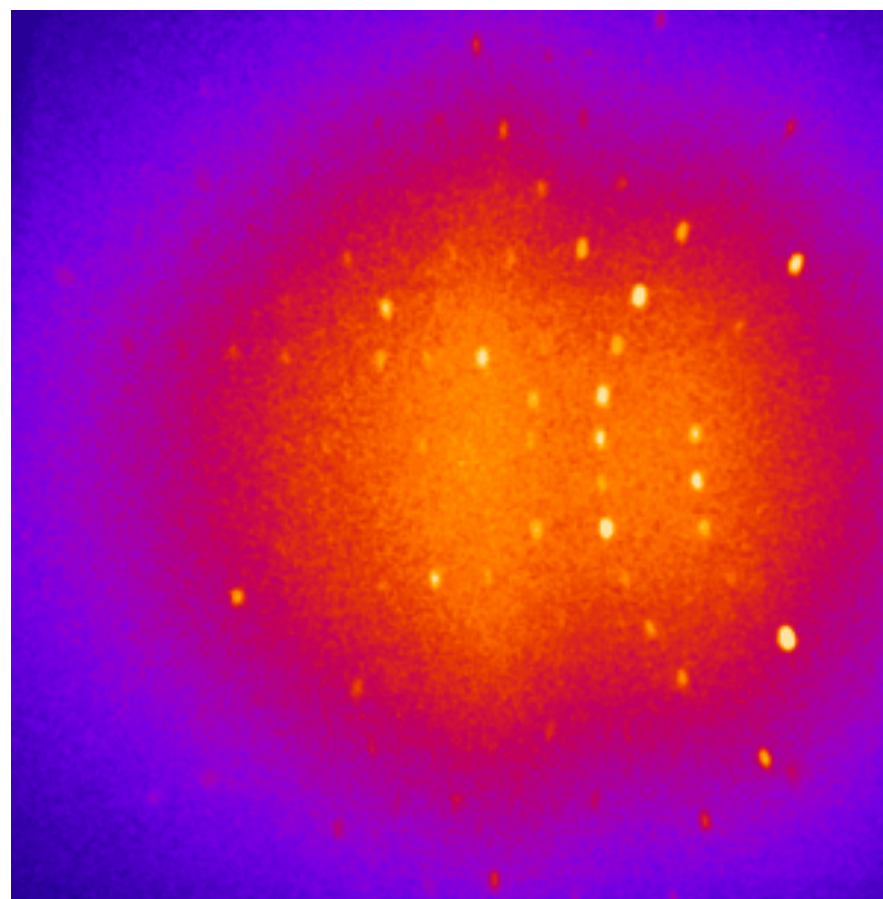
prim. beam

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

d_min=2.5 Å

d_min=1.5 Å

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
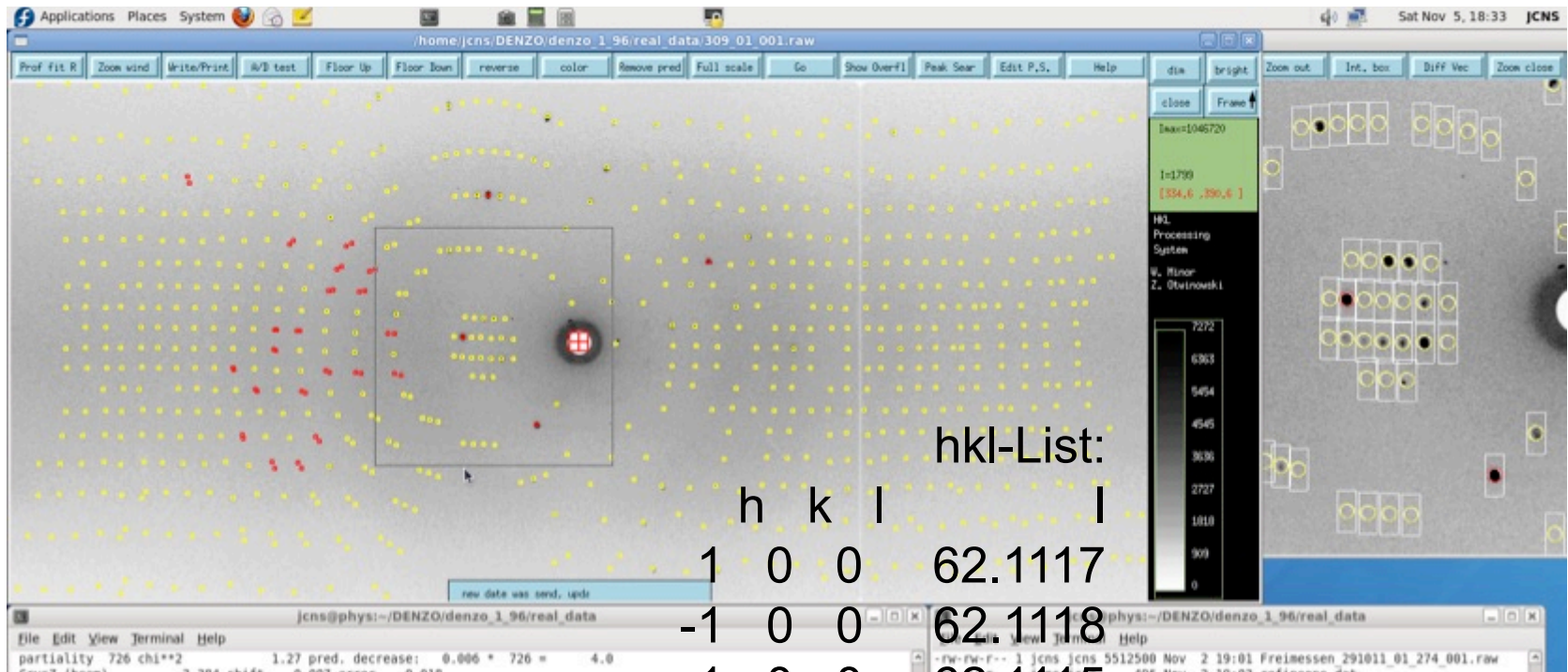
The „old" HKL2000 software

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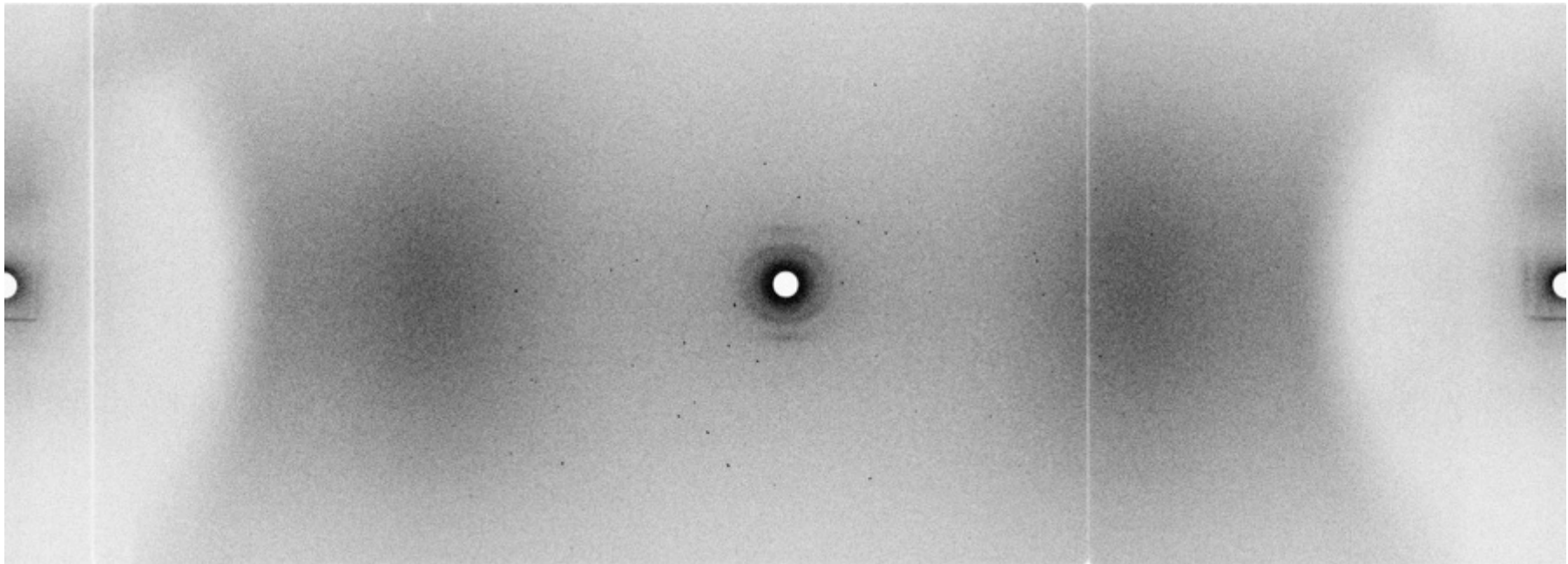
Special issues regarding neutron crystallography as compared to x-ray

- Background much more pronounced and not so homogenous
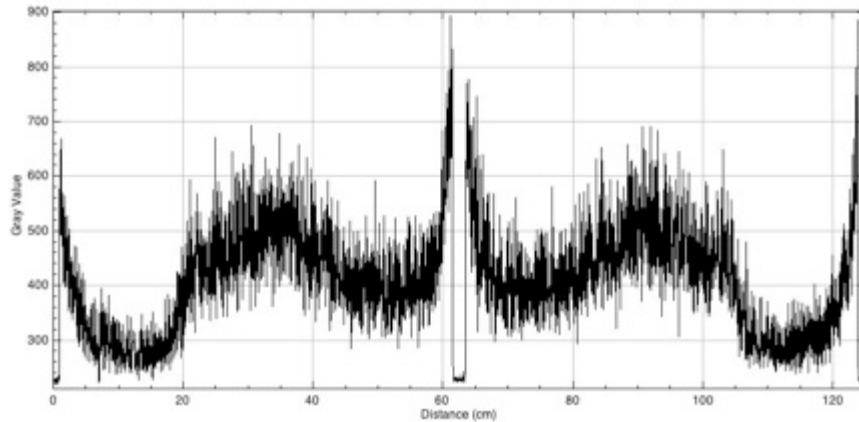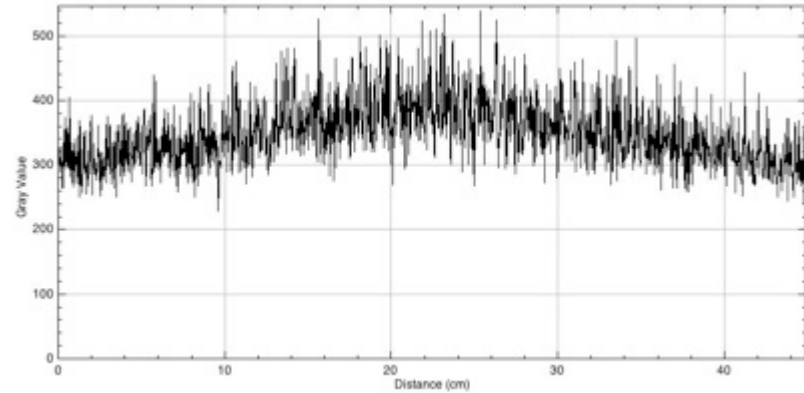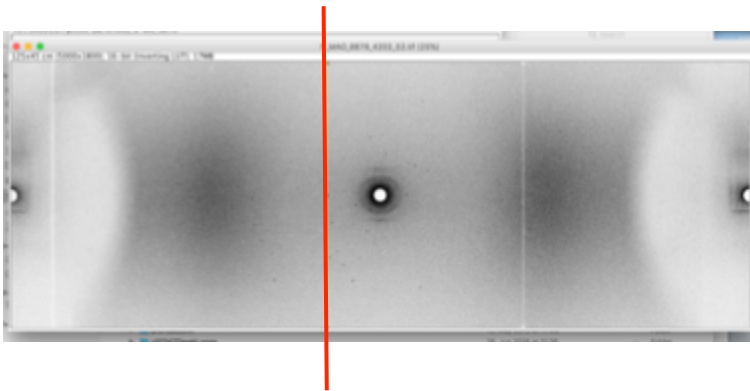
The x-ray case for comparison

- Horizontal cut showing the background
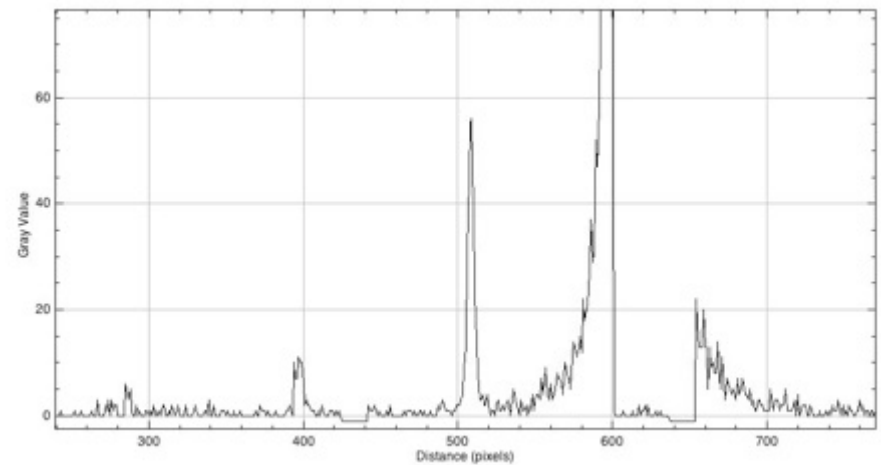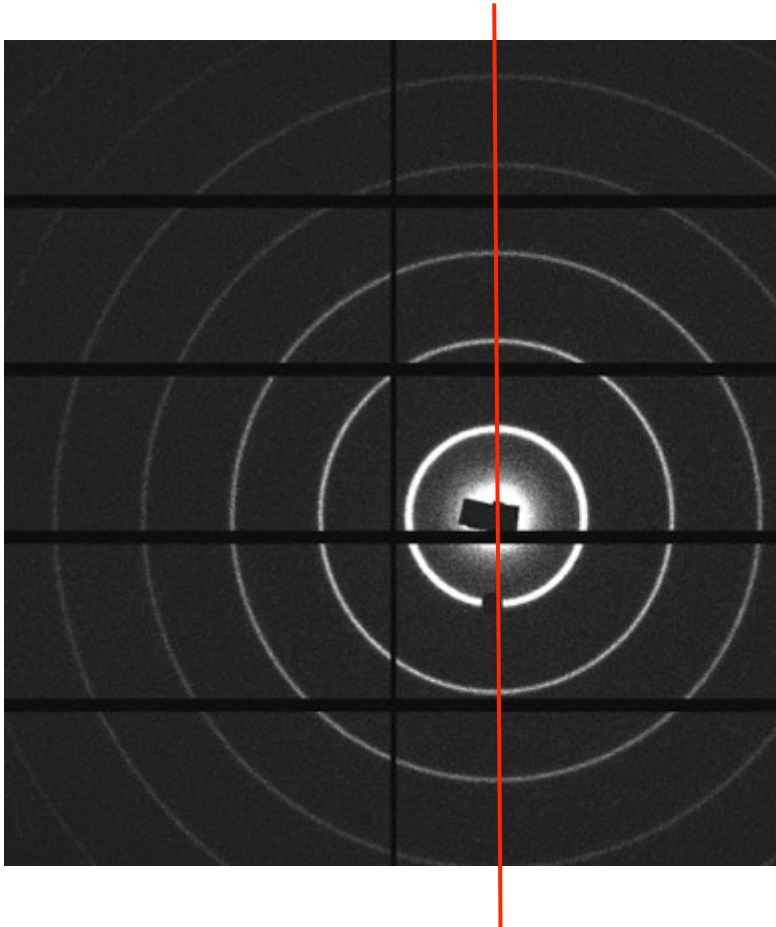
New Data Reduction Software: NXStool
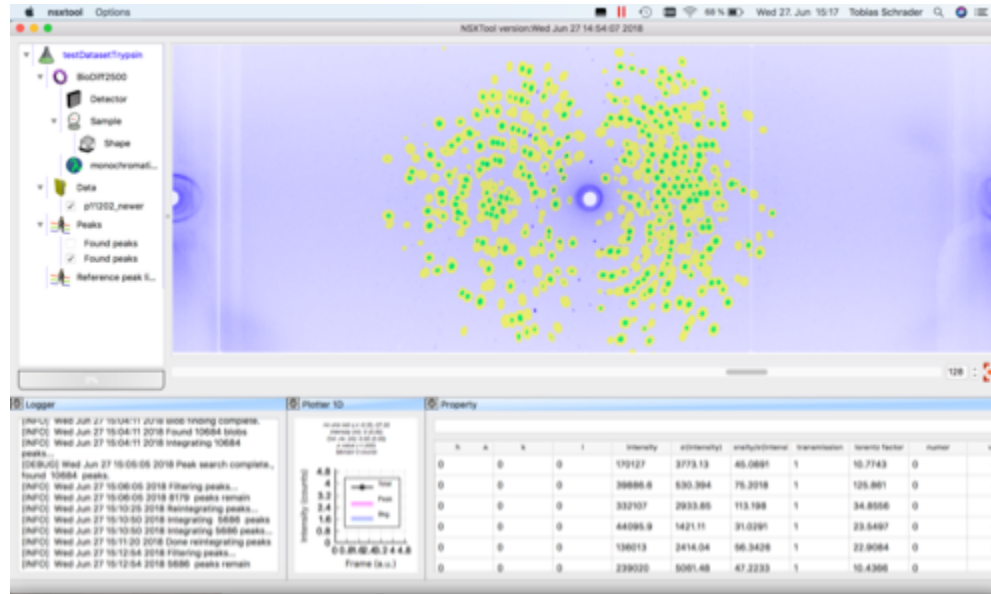
Allows to incorporate

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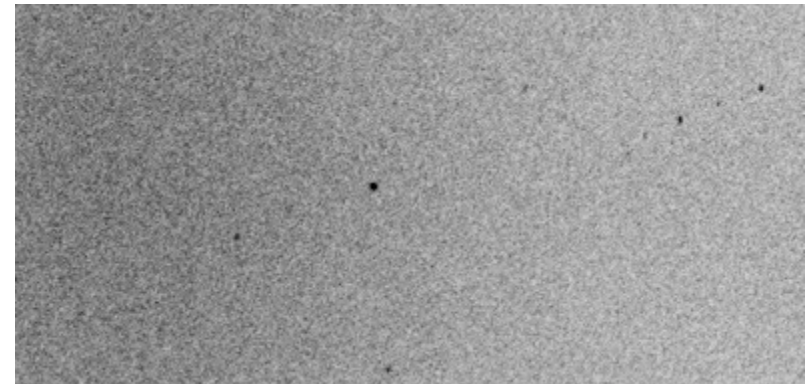- Pushing resolution limits
- Monitoring detector parameters and detecting detector problems

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
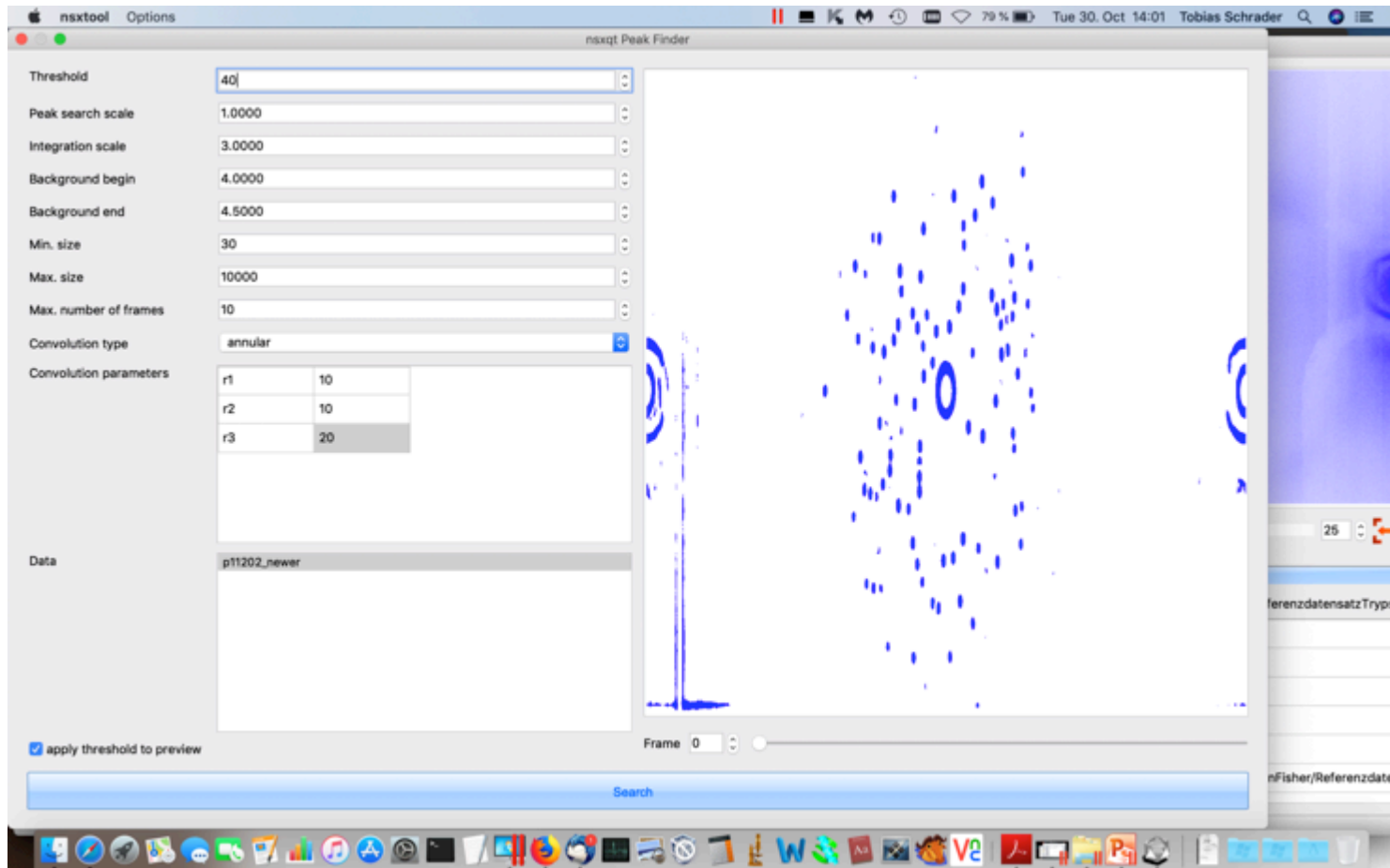
NXStool step by step
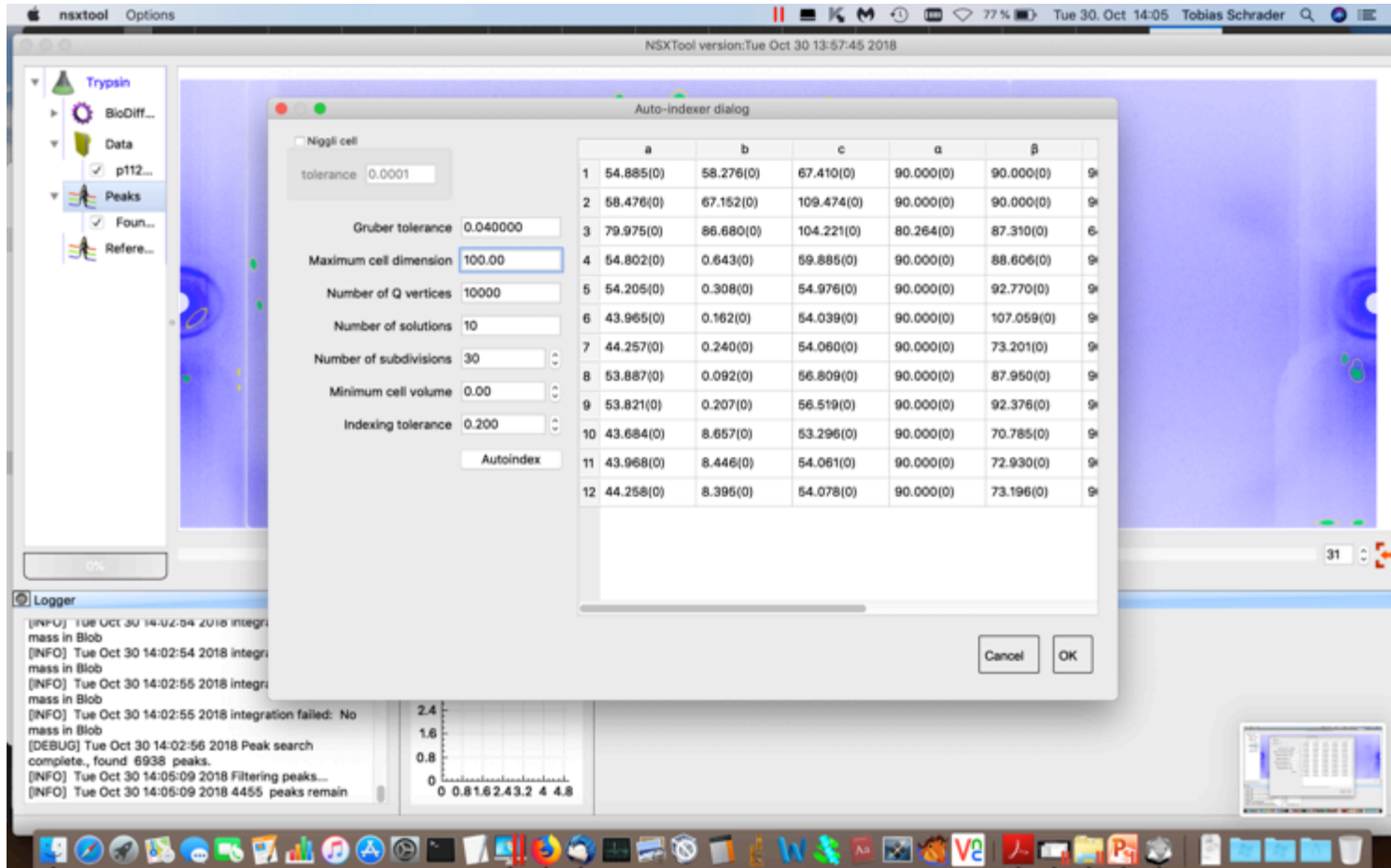
- Filtering of the peaks found by the image analysis
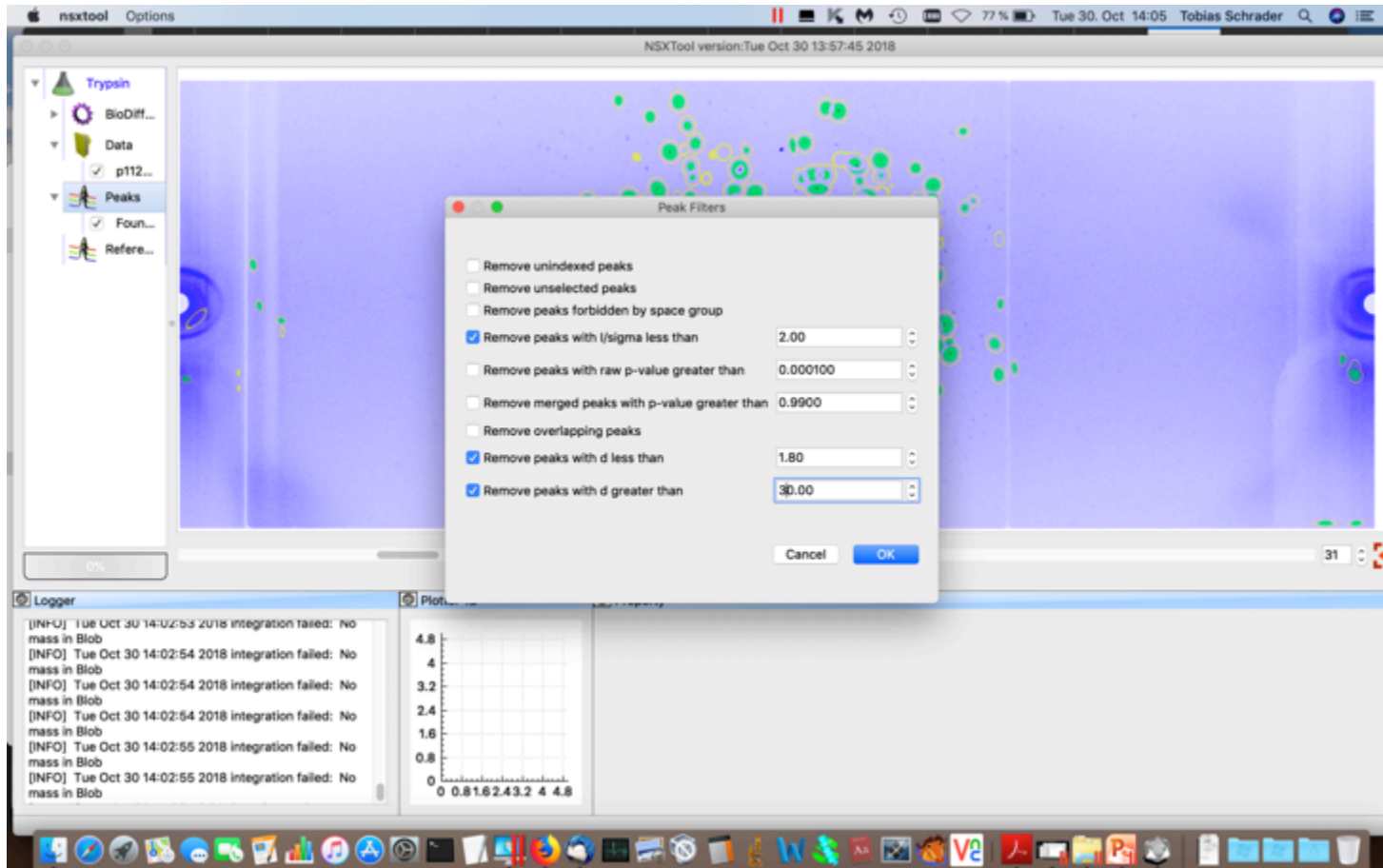
NXStool step by step

- 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
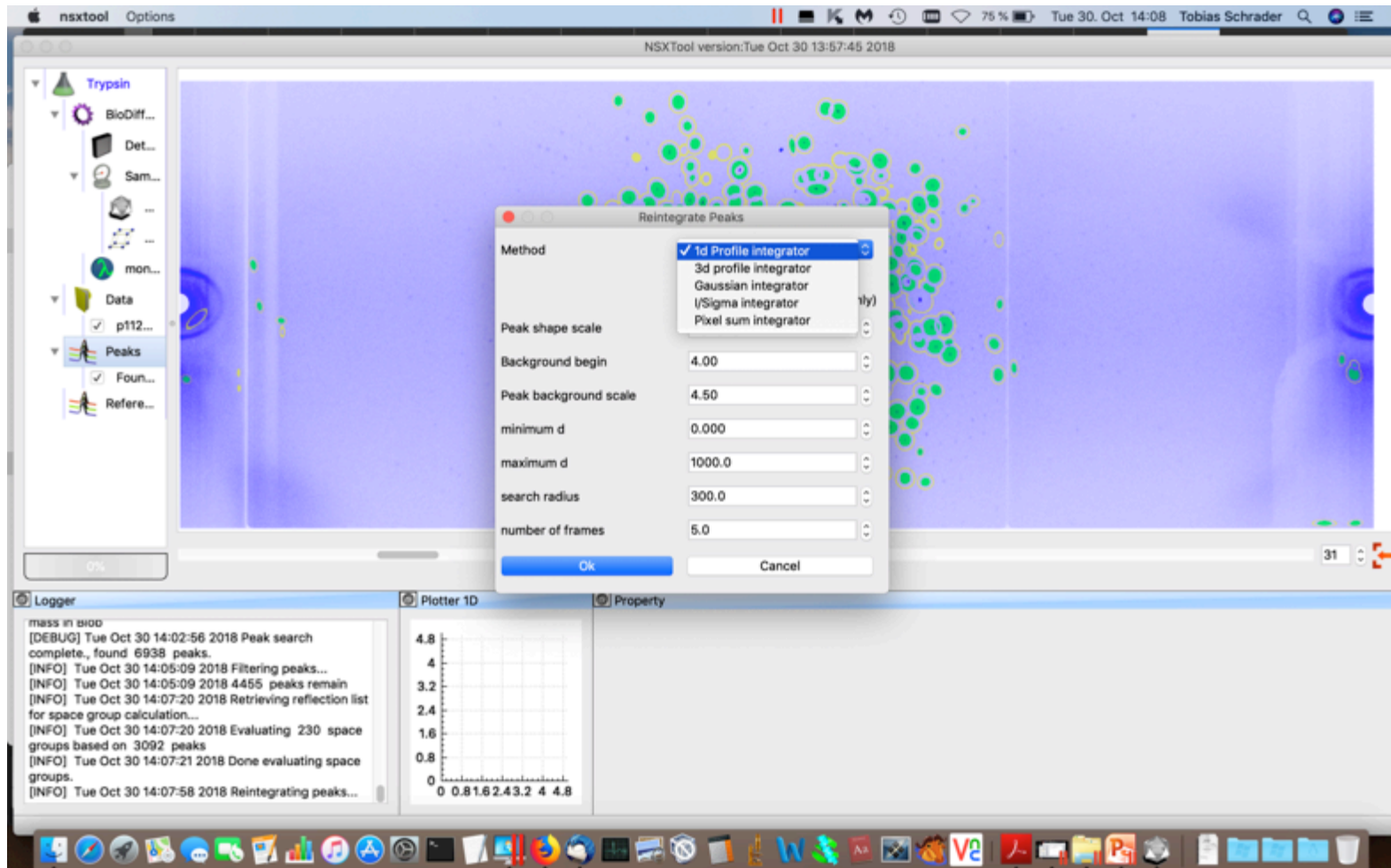
NXStool step by step

- Refinement of beam position and direction and other instrumental parameters
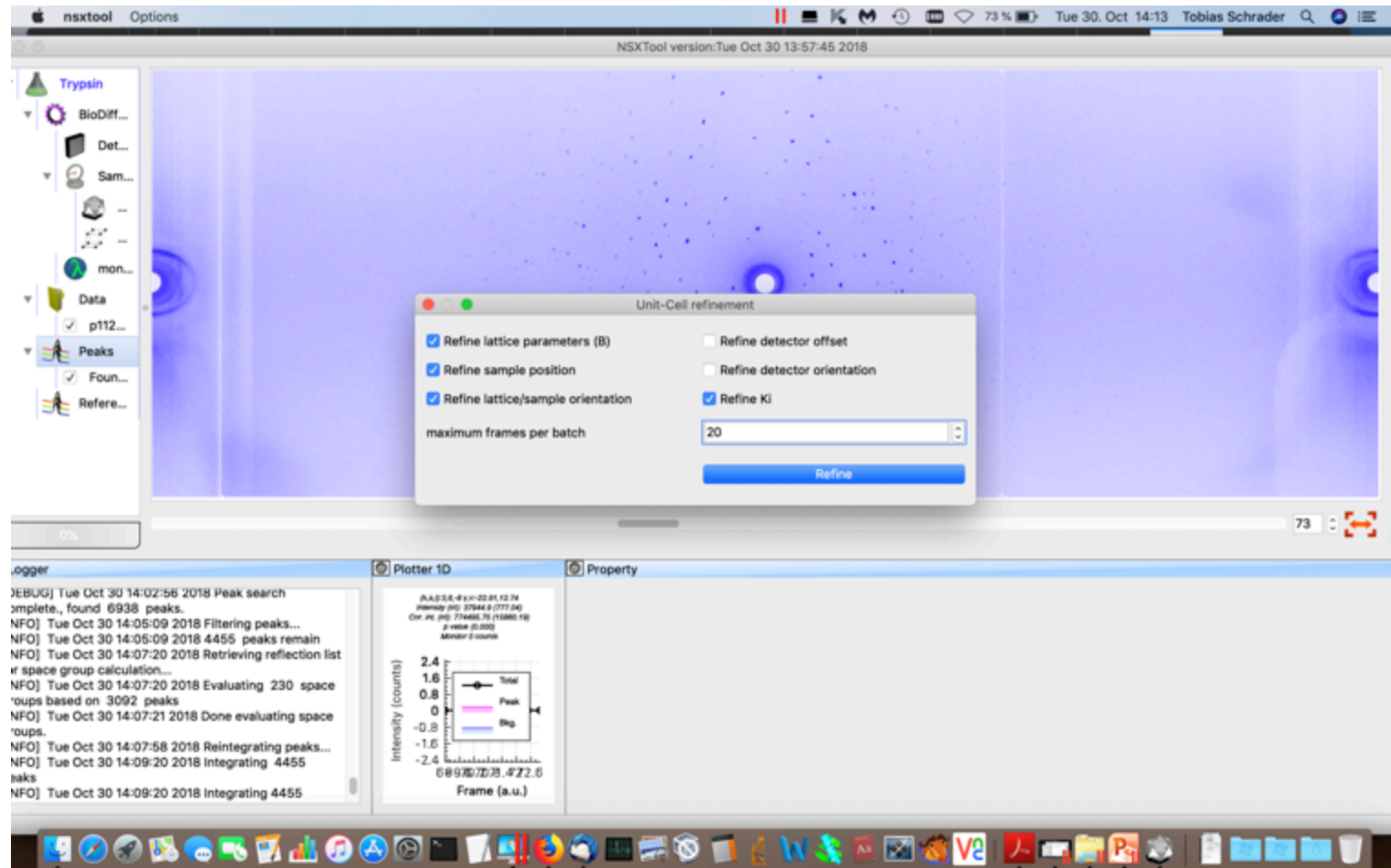
NXStool step by step

- Building a shape library for profile fitting
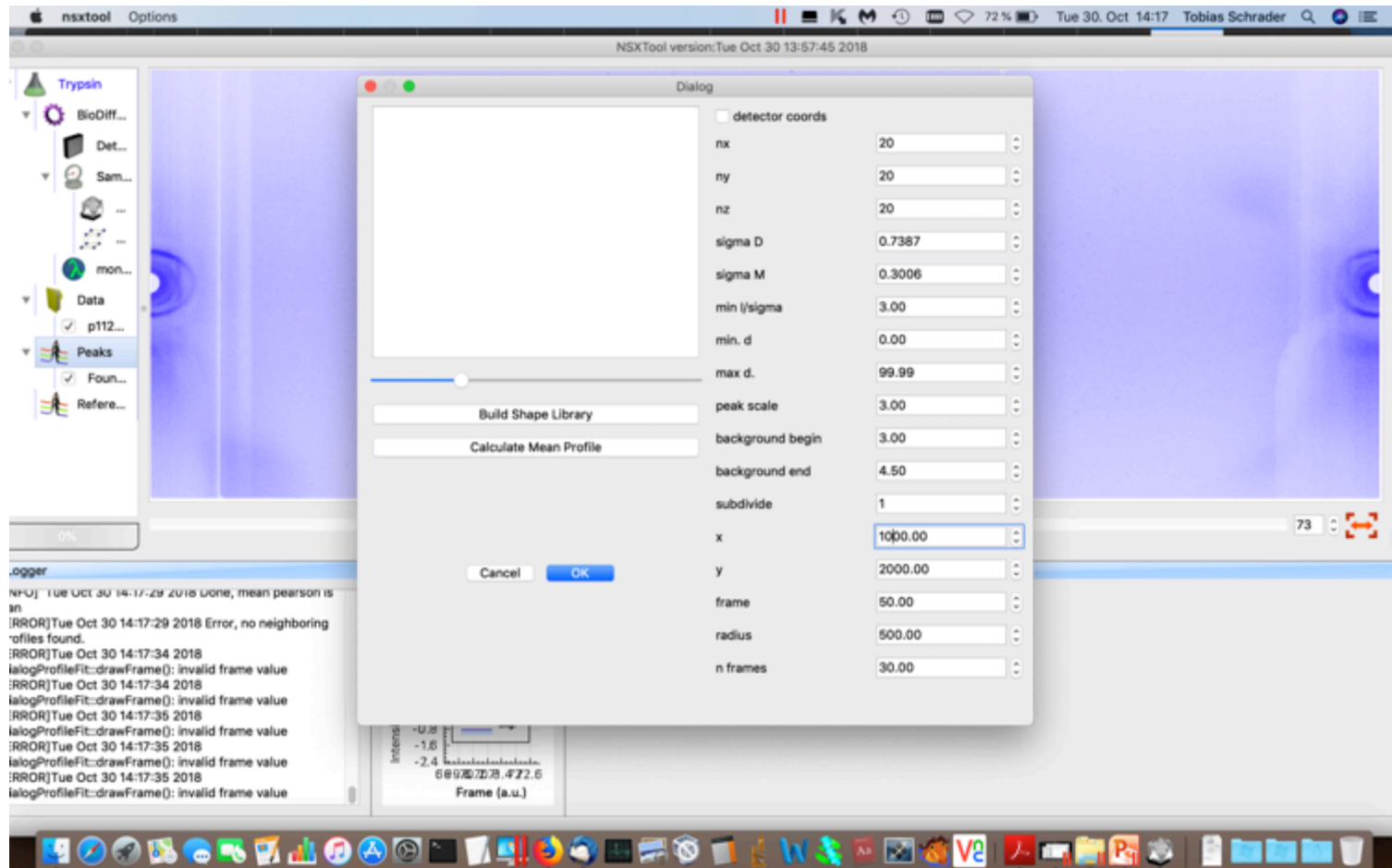
- Result of the integration

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 Lower limit	Upper Angstrom	Average I	Average error	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!!