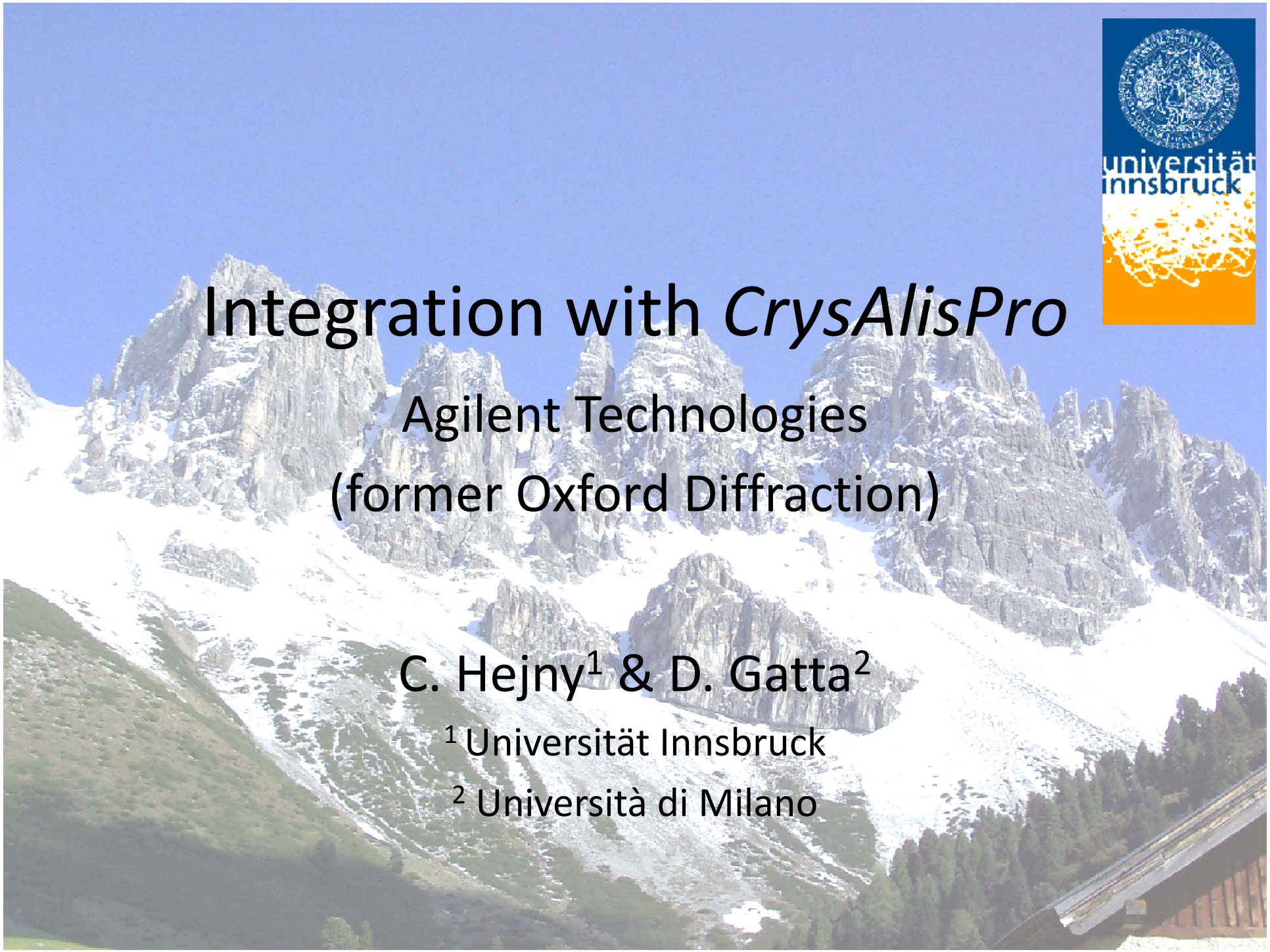


Integration with *CrysAlisPro*

Agilent Technologies
(former Oxford Diffraction)

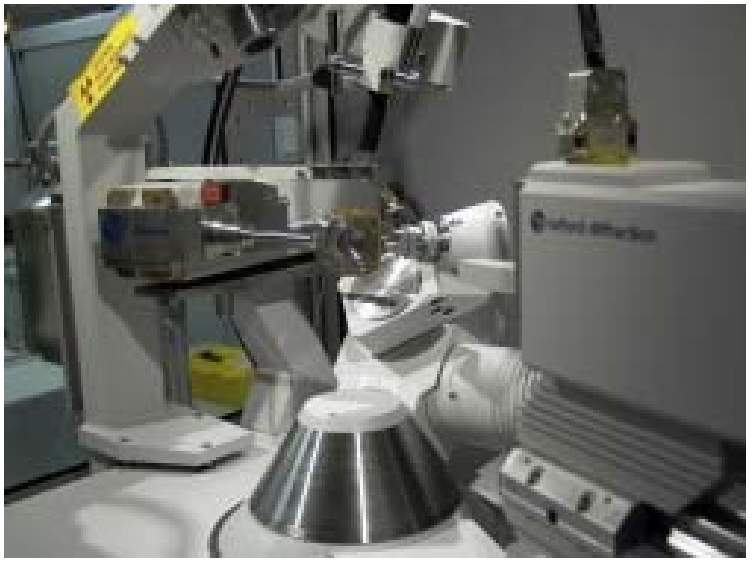
C. Hejny[1] & D. Gatta[2]

[1] Universität Innsbruck

[2] Università di Milano

http://www.oxford-diffraction.com/

Xcalibur ™ CCD



Xcalibur ™ PD

Gemini ™

SeperNova ™

Hi-flux dual wavelength micro-focus Mo & Cu X-ray sources

- *Support for up to 2Kg on the phi axis*
- *Better than 10 micron sphere of confusion*

Oxford Diffraction diffractometers driving program: CrysAlis^{Pro} ™

CrysAlisPro is accessible either via a graphical user interface or by a command line interface and can be operated under fully automatic, semi-automatic or **completely manual control**. A typical experiment is conducted in three stages:

1) **Automatic crystal screening** - A short pre-experiment of <5 minutes evaluates the crystal quality, providing unit cell and best exposure time information

2) **Strategy computation** - Sophisticated automatic strategy software calculates optimal conditions for fast, high quality, complete data collection

3) **Data collection and concurrent data reduction -** As data is collected, intelligent routines tune the parameters to give the best quality integrated data

http://www.oxford-diffraction.com/

CrysAlisPro also provides several specialist tools for dealing with non-standard and problematic crystals. These include:

* Movie based face indexation absorption correction
* Advanced unit cell finding
* Reciprocal space viewer
* Twinning
* Incommensurates
* High Pressure
* Powders

CrysAlisPro outputs data in HKLF format and interfaces directly with OLEX2, SHELX and third party data reduction packages including MOSFLM and XDS. CrysAlisPro is provided under a multi-site, multi-user licence.

(1) Introduction to *CrysAlisPro*

(2) General remarks

(3) Centre DAC

(4) Data collection

(5) Pre-analysis by user

(6) Best possible data reduction for high pressure data

Get good unit cell

Data reduction

Absorption correction

Good practice:

Measure crystal in air

Measure crystal in DAC

without pressure / pressure transmitting medium

Measure crystal with increasing pressure

Have a datasheet for each measurement

Have a datasheet for each data integration

Example of a data sheet for each measurement

Date:

Title (directory/filename)
Chem. composition
□ **DAC-front facing X-ray for $\omega = \varphi = \kappa = 0°$**

Used cell LH-0806-
Pressure [GPa]:
time between change of P and measurement
AP ruby
Indent of gasket:
Ø of gasket hole: 250 micron
Crystal size:
Image of crystal (directory/filename):
Detector distance: 90 mm
Exposure time: 60 sec
Generator setting (kV, mA): 50/40
Strategy of measurement:

Make an image of crystal on diamond before closing DAC

Take care about orientation of DAC on this image

Lattice parameter, S.G:

a	**b**	**c**
α	β	γ

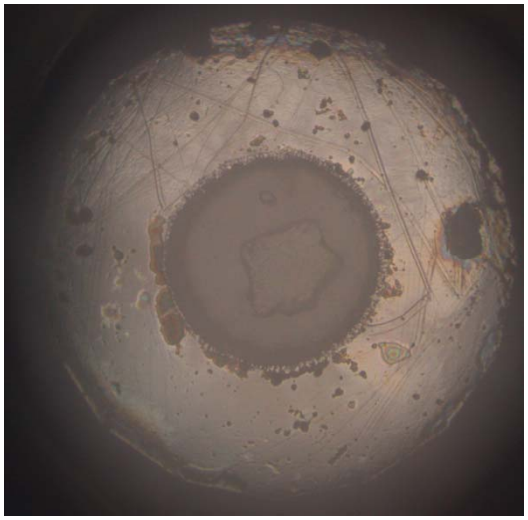
Notes:

Example of a data sheet for each measurement

Date:

<u>Title</u> (directory/filename)
Chem. composition
□ DAC-front facing X-ray for ω = φ = κ = 0°

Used cell LH-0806-
Pressure [GPa]:
time between change of P and measurement
AP ruby
Indent of gasket:
Ø of gasket hole: 250 micron
Crystal size:
Image of crystal (directory/filename):



Detector distance: 90 mm
Exposure time: 60 sec
Generator setting (kV, mA): 50/40
Strategy of measurement:

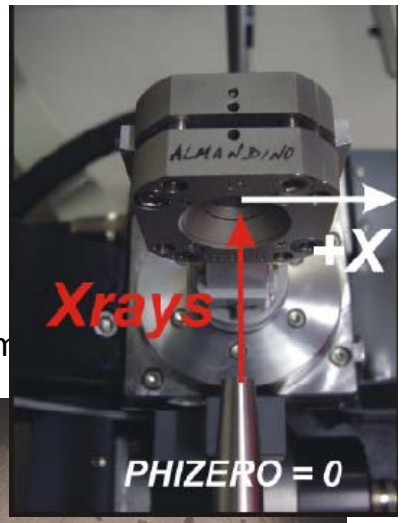
Lattice parameter, S.G:

a	**b**	**c**
α	β	γ

Notes:

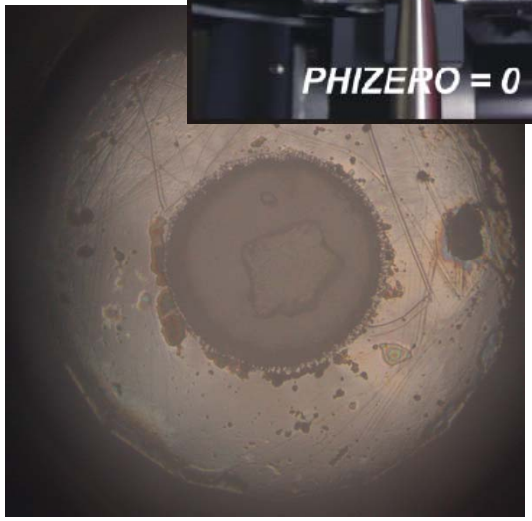
Example of a data sheet for each measurement

Date:



<u>Title</u> (directory/filename)
Chem. composition
√ DAC-front facing X-ray for ω = φ = κ = 0°

Used cell LH-0806-
Pressure [GPa]:
time between change of P and measurem
AP ruby
Indent of gasket:
Ø of gasket hole: 250 micron
Crystal size:
Image of crystal (directory/filename):



Detector distance: 90 mm
Exposure time: 60 sec
Generator setting (kV, mA): 50/40
Strategy of measurement:

Lattice parameter, S.G:

a	**b**	**c**
α	β	γ

Notes:

(1) Introduction to *CrysAlisPro*

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Get good unit cell

Data reduction

Absorption correction

Centre DAC

See notes on morning talk “Diffractometry Data Collection“

(1) Put on DAC in correct orientation

√ **DAC-front facing X-ray for $\omega = \varphi = \kappa = 0°$**

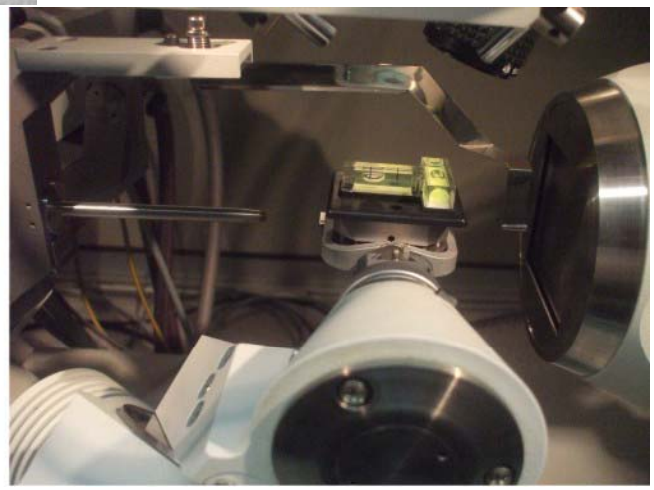
Centre DAC

See notes on morning talk “Diffractometry Data Collection“

(1) Put on DAC in correct orientation

(2) Look perpendicular to diamond surface:

Use command **gt e 0 0 90 90** in the command window



Rotate DAC until the face of the DAC is exactly horizontal, as measured by a spirit level. Gently tighten the height locking screw on the goniometer head.

Centre DAC

See notes on morning talk “Diffractometry Data Collection“

(1) Put on DAC in correct orientation

(2) Look perpendicular to diamond surface

(3) Visual pre-centering

Centre DAC

See notes on morning talk “Diffractometry Data Collection“

(1) Put on DAC in correct orientation

(2) Look perpendicular to diamond surface

(3) Visual pre-centering

(4) Precise Centre with X-ray beam

Type in the following commands

or write a macro *filename.mac* with these commands :

```
gt p -25
card raw on 0.1
ip copy dc2 dc1
gt p 25
card raw on 0.1
ip subtract dcc dc1 dc2
gt p 0
```

```
gt e 0 0 90 0
card raw on 0.1
ip copy dc2 dc1
gt e 0 0 -90 0
card raw on 0.1
ip subtract dcc dc1 dc2
gt e 0 0 0 0
```

Call the macro with the command **script *filename.mac***

Centre DAC

See notes on morning talk “Diffractometry Data Collection“

(1) Put on DAC in correct orientation

(2) Look perpendicular to diamond surface

(3) Visual pre-centering

(4) Precise Centre with X-ray beam

Type in the following commands

or write a macro *filename.mac* with these commands :

```
gt p -25
card raw on 0.1
ip copy dc2 dc1
gt p 25
card raw on 0.1
ip subtract dcc dc1 dc2
gt p 0
```

BEAMSTOP IN AFTER THAT!

Call the macro with the command **script *filename.mac***

(1) Introduction to *CrysAlisPro*

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Absorption correction

Data collection:

2 options for full experiment:

(1) pre-designed runs

(2) Experiment strategy from *CrysAlisPro*

Data collection:

(1) Pre-set run file:

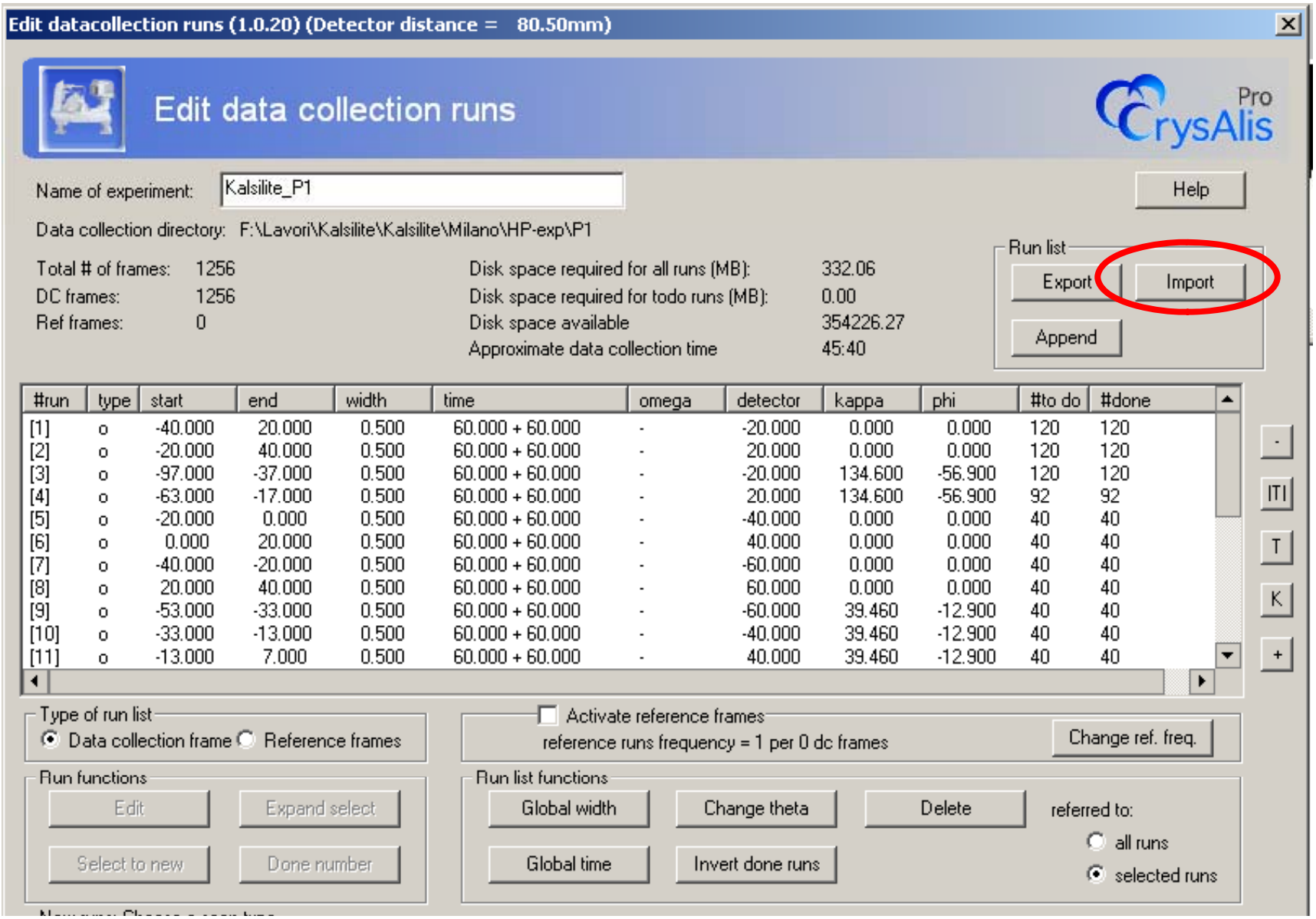
Diego Gatta has designed run files for data collection with the Sapphire-3 CCD set at *dd*=80mm and a DAC with a half-opening angle of 40 degrees:

DAC_psi40_dd80_tth60_full_sapphire3.run
DAC_psi40_dd80_tth80_full_sapphire3.run

Both run files attempt to cover all of accessible reciprocal space. If only one-half of that space is required, then the runs at negative values of 2-theta can be deleted.

I have a copy of this file for distribution.

Start/Stop ➔ New (no pre-experiment)



Data collection:

Optionally:

Prevent remeasuring on diamond reflection overflow:

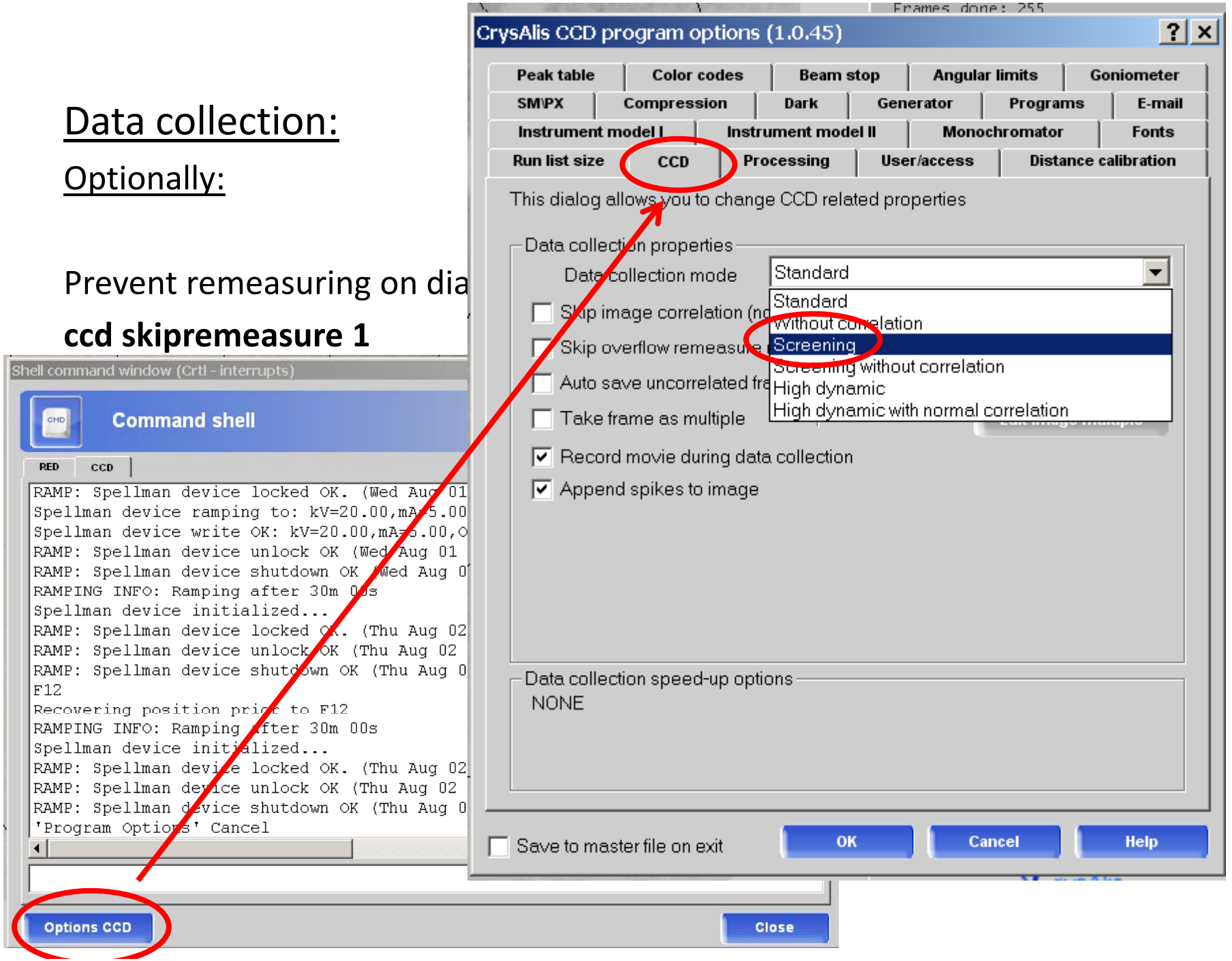
ccd skipremeasure 1

Data collection:

Optionally:

Prevent remeasuring on dia

ccd skipremeasure 1



Data collection:

Optionally:

ccd skipremeasure 1 to prevent remeasuring on diamond reflection overflow.

Collision test: **dc stest**

Data collection:

Pre-Experiment

Delete 3 Standard runs

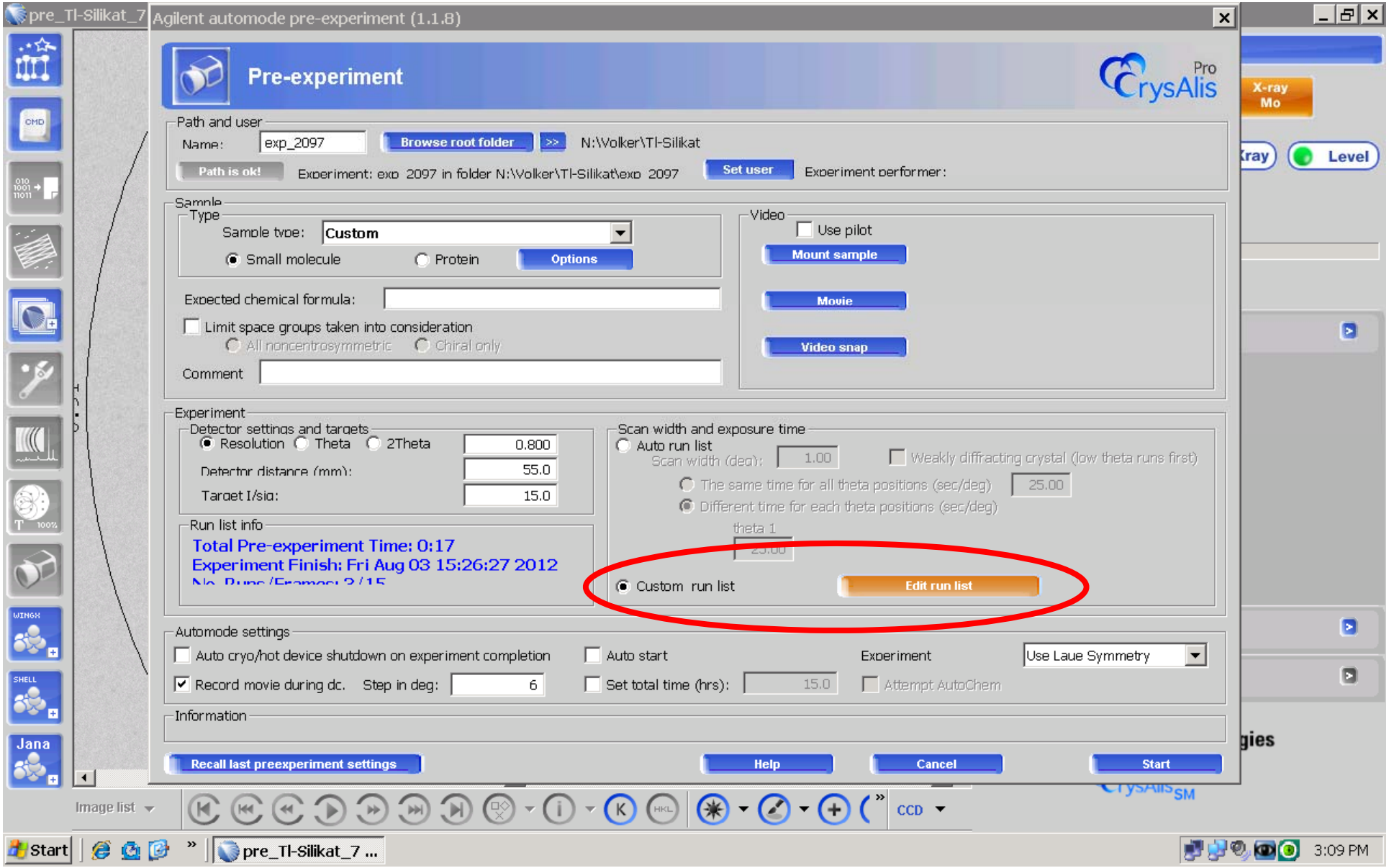
Make phi- or omega scan from -15 to +15

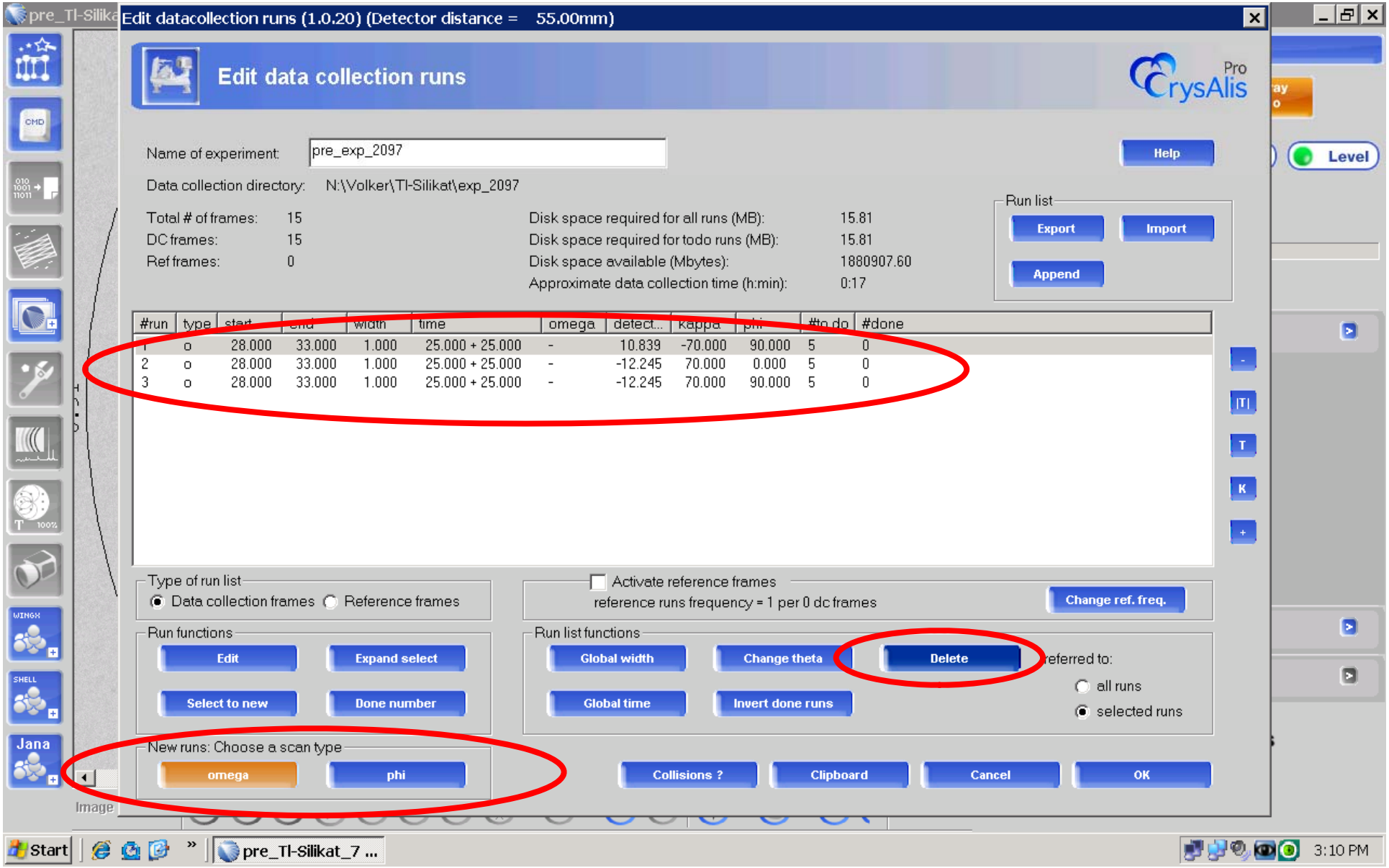
Good time to get an estimate of intensities: 10 - 30sec / frame

Width: 1°

Make sure you make no other changes after editing the runs!

(in this case CrysAlisPro goes back to the 3 standard runs for a pre-experiment)


pre_Tl-Silikat_7
Agilent automode pre-experiment (1.1.8)
Pre-experiment
Pro CrysAlis
Path and user
Name: exp_2097
Browse root folder
N:\Volker\Tl-Silikat
Path is ok!
Experiment: exp_2097 in folder N:\Volker\Tl-Silikat\exp_2097
Set user
Experiment performer:
Sample
Type
Sample type: Custom
Small molecule
Protein
Options
Expected chemical formula:
Limit space groups taken into consideration
All noncentrosymmetric
Chiral only
Comment
Video
Use pilot
Mount sample
Movie
Video snap
Experiment
Detector settings and targets
Resolution
Theta
2Theta
0.800
Detector distance (mm):
55.0
Target I/sig:
15.0
Run list info
Total Pre-experiment Time: 0:17
Experiment Finish: Fri Aug 03 15:26:27 2012
Scan width and exposure time
Auto run list
Scan width (deg):
1.00
Weakly diffracting crystal (low theta runs first)
The same time for all theta positions (sec/deg)
25.00
Different time for each theta positions (sec/deg)
theta 1
Custom run list
Edit run list
Automode settings
Auto cryo/hot device shutdown on experiment completion
Auto start
Experiment
Use Laue Symmetry
Record movie during dc.
Step in deg:
6
Set total time (hrs):
15.0
Attempt AutoChem
Information
Recall last preexperiment settings
Help
Cancel
Start
Image list
CCD
Start
pre_Tl-Silikat_7 ...
3:09 PM


Edit datacollection runs (1.0.20) (Detector distance = 55.00mm)
Edit data collection runs
CrysAlis Pro
Name of experiment: pre_exp_2097
Help
Data collection directory: N:\Volker\TI-Silikat\exp_2097
Total # of frames: 15
DC frames: 15
Ref frames: 0
Disk space required for all runs (MB): 15.81
Disk space required for todo runs (MB): 15.81
Disk space available (Mbytes): 1880907.60
Approximate data collection time (h:min): 0:17
Run list
Export
Import
Append
#run type start end width time omega detect... kappa phi #to do #done
1 o 28.000 33.000 1.000 25.000 + 25.000 - 10.839 -70.000 90.000 5 0
2 o 28.000 33.000 1.000 25.000 + 25.000 - -12.245 70.000 0.000 5 0
3 o 28.000 33.000 1.000 25.000 + 25.000 - -12.245 70.000 90.000 5 0
Type of run list
Data collection frames
Reference frames
Activate reference frames
reference runs frequency = 1 per 0 dc frames
Change ref. freq.
Run functions
Edit
Expand select
Select to new
Done number
Run list functions
Global width
Change theta
Delete
Global time
Invert done runs
referred to:
all runs
selected runs
New runs: Choose a scan type
omega
phi
Collisions ?
Clipboard
Cancel
OK
Start
pre_TI-Silikat_7 ...
3:10 PM

Data collection:

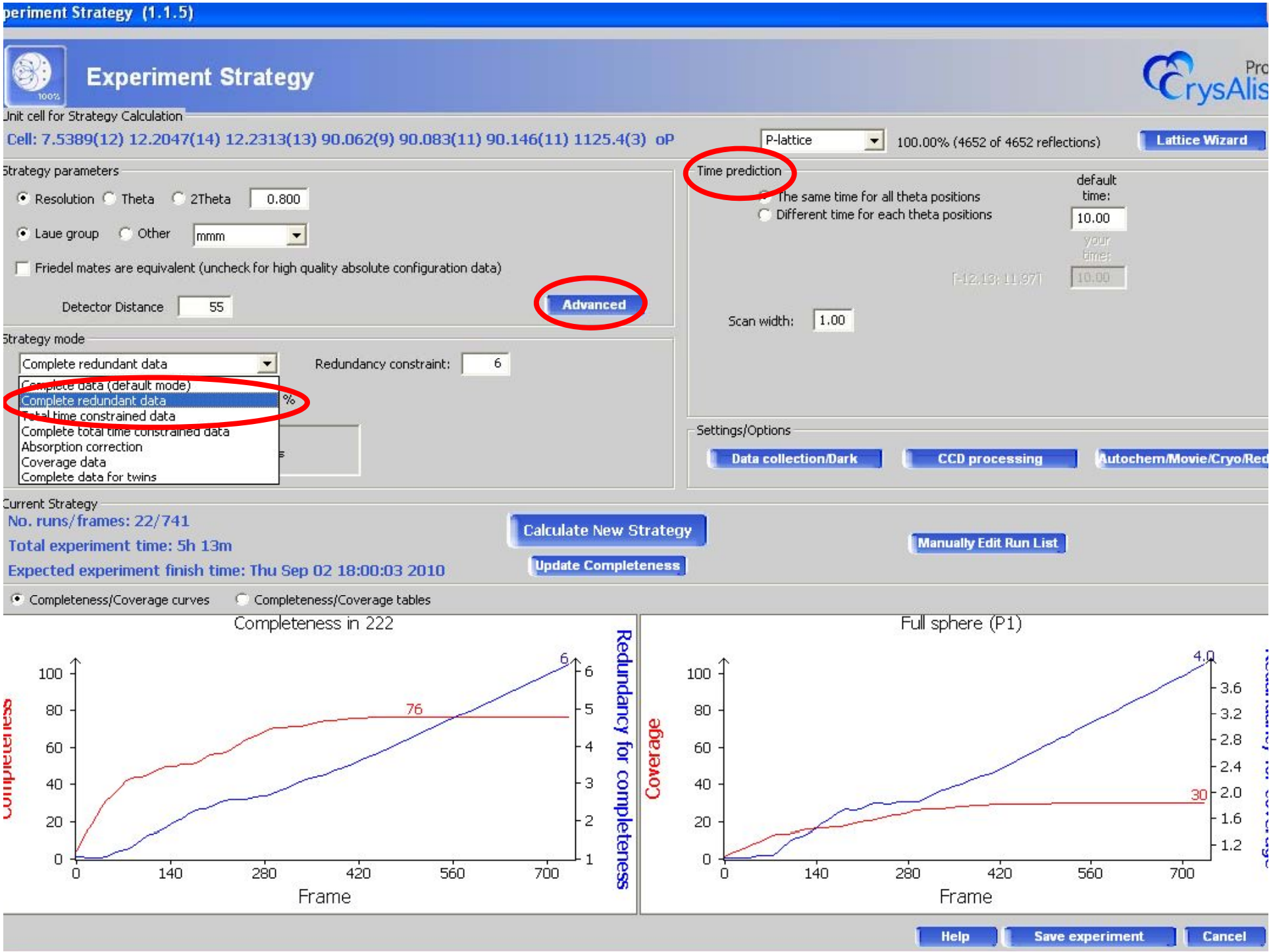
(2) Use CrysAlisPro for Experiment strategy

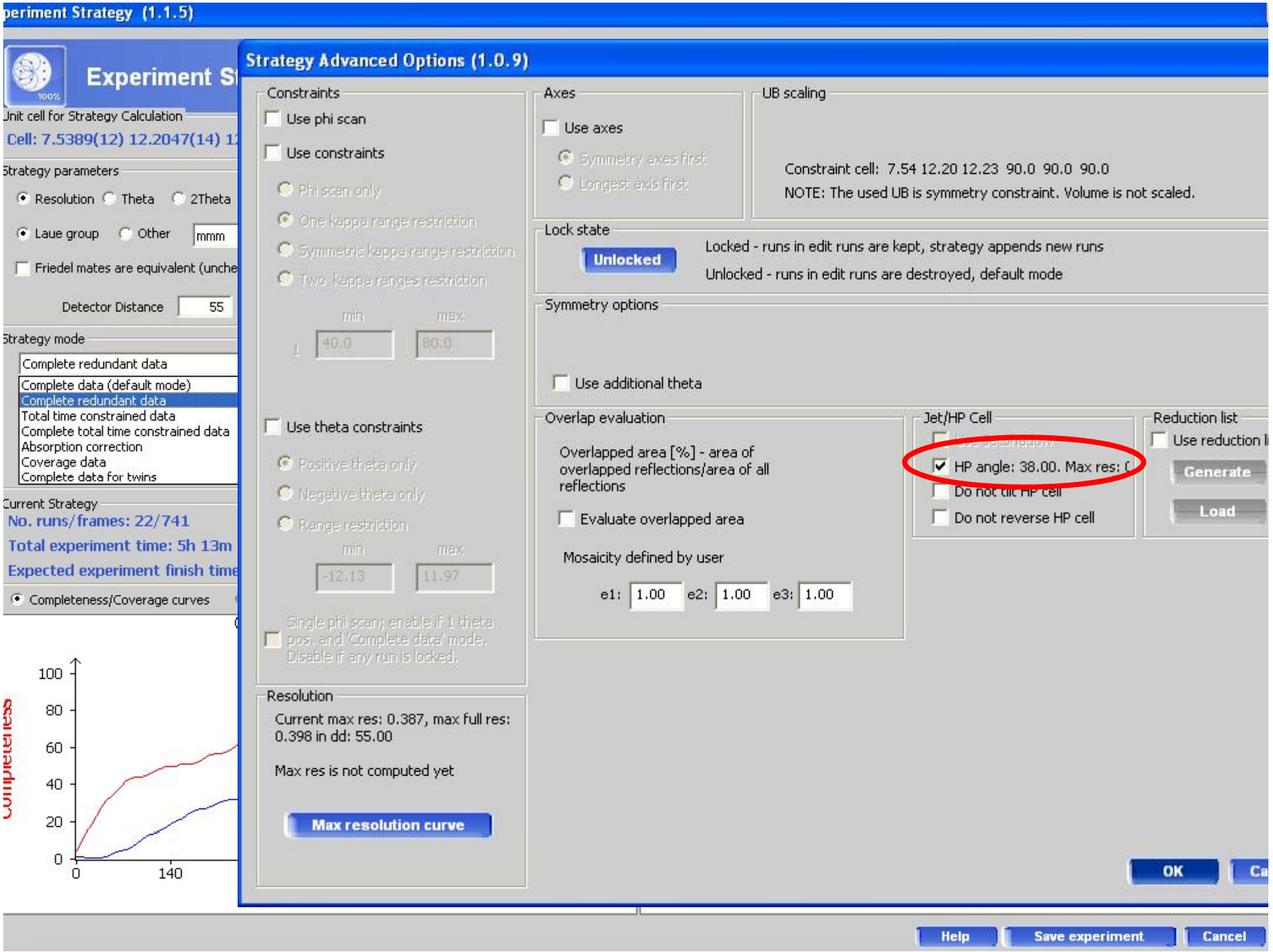
IMPORTANT:

Looooong enough exposure time!!!!

REDUNDANT data

Advanced: HP opening angle: 40° -dependant on used cell!


periment Strategy (1.1.5)
Experiment Strategy
Unit cell for Strategy Calculation
Cell: 7.5389(12) 12.2047(14) 12.2313(13) 90.062(9) 90.083(11) 90.146(11) 1125.4(3) oP
P-lattice
100.00% (4652 of 4652 reflections)
Lattice Wizard
Strategy parameters
Resolution
Theta
2Theta
0.800
Laue group
Other
mmm
Friedel mates are equivalent (uncheck for high quality absolute configuration data)
Detector Distance
55
Advanced
Time prediction
The same time for all theta positions
Different time for each theta positions
default time:
10.00
your time:
10.00
Scan width:
1.00
Strategy mode
Complete redundant data
Redundancy constraint:
6
Complete data (default mode)
Complete redundant data
Total time constrained data
Complete total time constrained data
Absorption correction
Coverage data
Complete data for twins
Settings/Options
Data collection/Dark
CCD processing
Current Strategy
No. runs/frames: 22/741
Total experiment time: 5h 13m
Expected experiment finish time: Thu Sep 02 18:00:03 2010
Calculate New Strategy
Update Completeness
Manually Edit Run List
Completeness/Coverage curves
Completeness/Coverage tables
Completeness in 222
Completeness
Redundancy for completeness
Frame
Full sphere (P1)
Coverage
Frame
Help
Save experiment
Cancel


periment Strategy (1.1.5)
Experiment St
Unit cell for Strategy Calculation
Cell: 7.5389(12) 12.2047(14) 1
Strategy parameters
Resolution
Theta
2Theta
Laue group
Other
mmm
Friedel mates are equivalent (unche
Detector Distance
55
Strategy mode
Complete redundant data
Complete data (default mode)
Complete redundant data
Total time constrained data
Complete total time constrained data
Absorption correction
Coverage data
Complete data for twins
Current Strategy
No. runs/frames: 22/741
Total experiment time: 5h 13m
Expected experiment finish time
Completeness/Coverage curves
Completeness
100
80
60
40
20
0
0
140
Strategy Advanced Options (1.0.9)
Constraints
Use phi scan
Use constraints
Phi scan only
One kappa range restriction
Symmetric kappa range restriction
Two kappa ranges restriction
min
max
1
40.0
80.0
Use theta constraints
Positive theta only
Negative theta only
Range restriction
min
max
-12.13
11.97
Single phi scan; enable if 1 theta pos. and 'Complete data' mode. Disable if any run is locked.
Resolution
Current max res: 0.387, max full res: 0.398 in dd: 55.00
Max res is not computed yet
Max resolution curve
Axes
Use axes
Symmetry axes first
Longest axis first
UB scaling
Constraint cell: 7.54 12.20 12.23 90.0 90.0 90.0
NOTE: The used UB is symmetry constraint. Volume is not scaled.
Lock state
Unlocked
Locked - runs in edit runs are kept, strategy appends new runs
Unlocked - runs in edit runs are destroyed, default mode
Symmetry options
Use additional theta
Overlap evaluation
Overlapped area [%] - area of overlapped reflections/area of all reflections
Evaluate overlapped area
Mosaicity defined by user
e1: 1.00
e2: 1.00
e3: 1.00
Jet/HP Cell
HP angle: 38.00. Max res:
Do not tilt HP cell
Do not reverse HP cell
Reduction list
Use reduction l
Generate
Load
OK
Ca
Help
Save experiment
Cancel

(1) Introduction to *CrysAlisPro*

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Get good unit cell

Data reduction

Absorption correction

Pre-Analysis by the user:

(1) Look through all frames:

Is there exposure on all frames

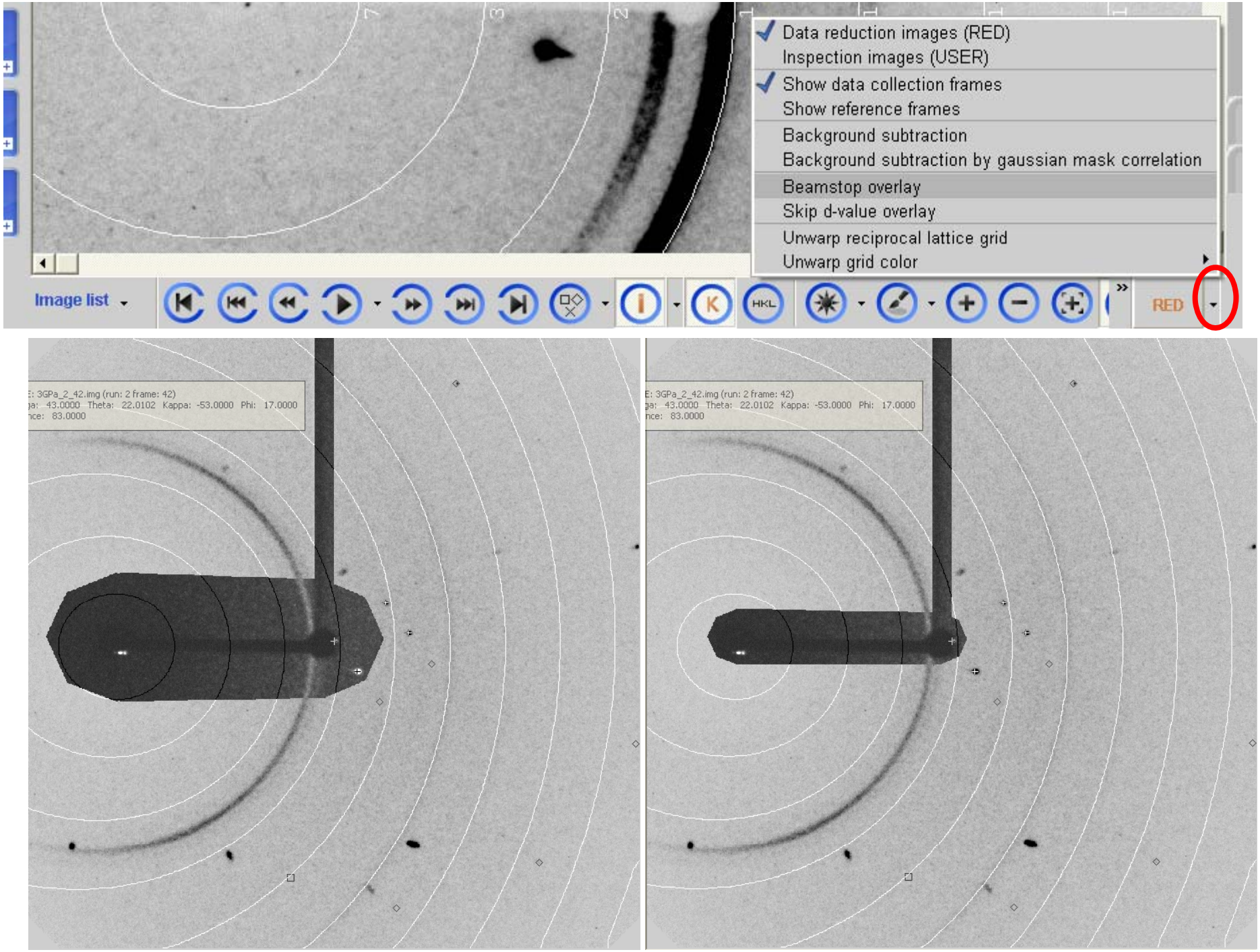
or are some frames shadowed by the cell?

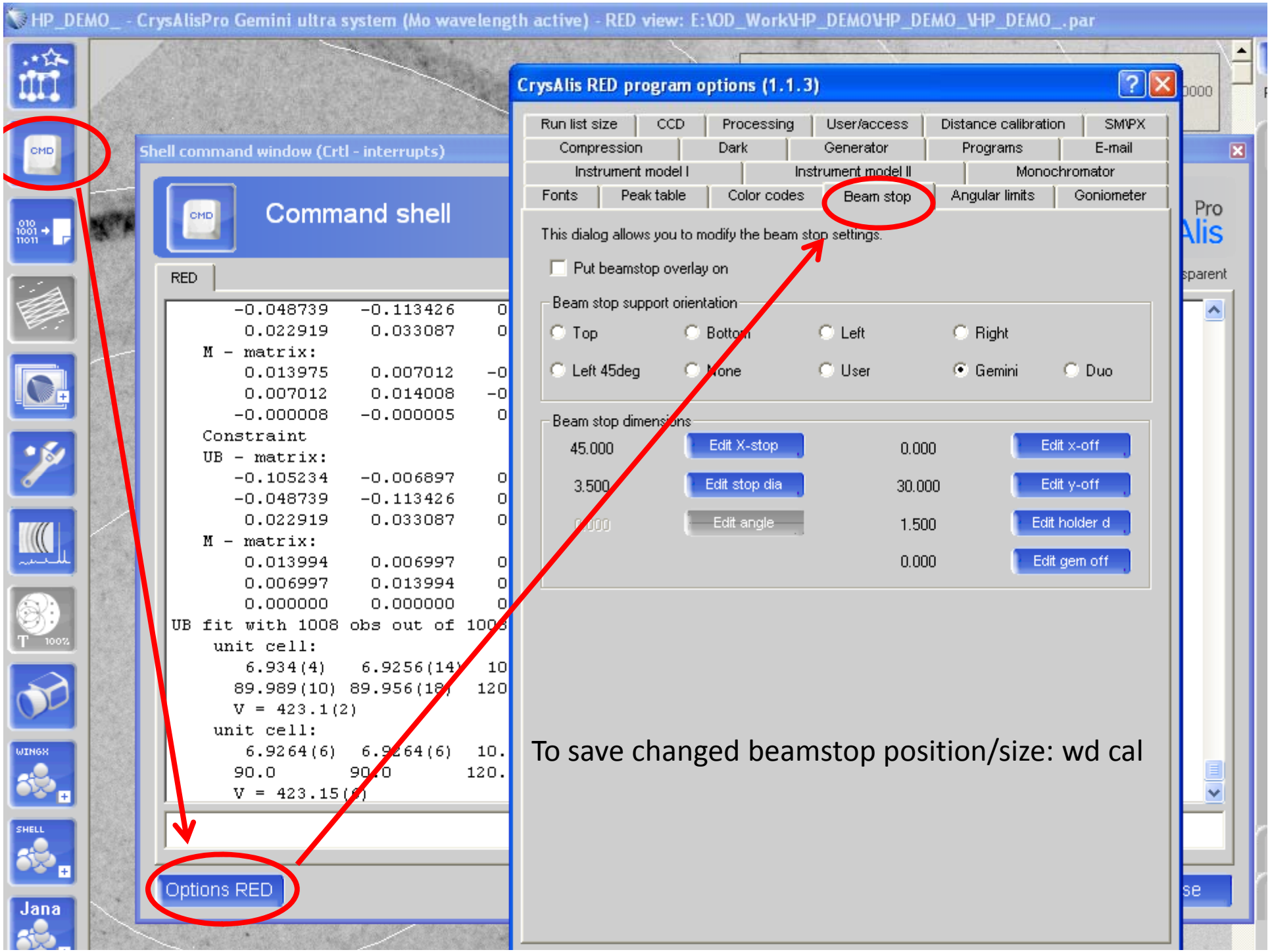
USE ONLY FRAMES WITH SAMPLE REFLECTIONS!

(2) Is the beamstop shadow in the correct place and of good size?

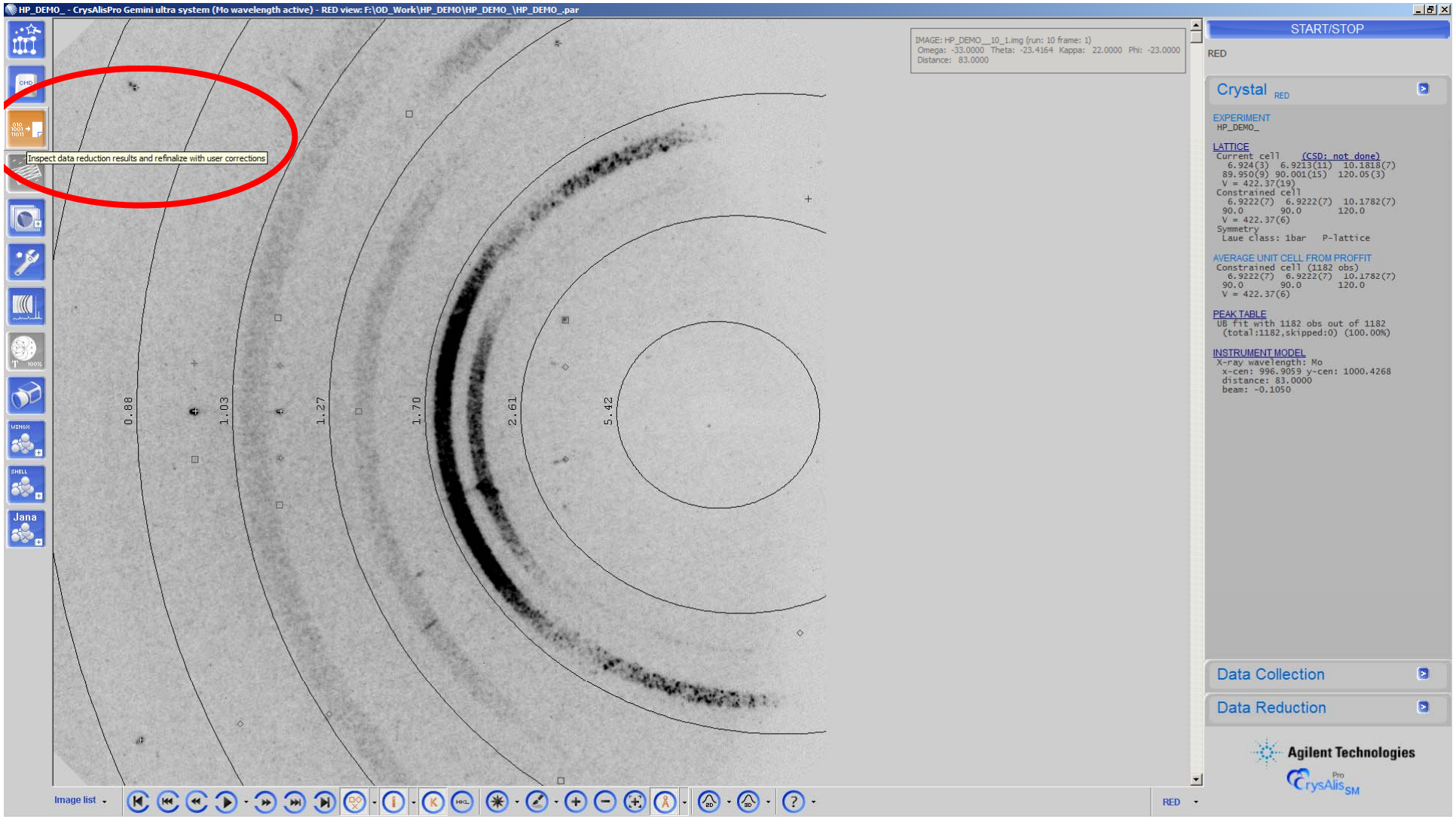
(if change necessary: save with **wd cal**)

(3) Look at results from automatic data reduction


Data reduction images (RED)
Inspection images (USER)
Show data collection frames
Show reference frames
Background subtraction
Background subtraction by gaussian mask correlation
Beamstop overlay
Skip d-value overlay
Unwarp reciprocal lattice grid
Unwarp grid color
Image list
HKL
RED
E: 3GPa_2_42.img (run: 2 frame: 42)
ga: 43.0000 Theta: 22.0102 Kappa: -53.0000 Phi: 17.0000
nce: 83.0000


HP_DEMO_ - CrysAlisPro Gemini ultra system (Mo wavelength active) - RED view: E:\OD_Work\HP_DEMO\HP_DEMO_\HP_DEMO_.par
Shell command window (Crtl - interrupts)
Command shell
RED
-0.048739 -0.113426 0
0.022919 0.033087 0
M - matrix:
0.013975 0.007012 -0
0.007012 0.014008 -0
-0.000008 -0.000005 0
Constraint
UB - matrix:
-0.105234 -0.006897 0
-0.048739 -0.113426 0
0.022919 0.033087 0
M - matrix:
0.013994 0.006997 0
0.006997 0.013994 0
0.000000 0.000000 0
UB fit with 1008 obs out of 100
unit cell:
6.934(4) 6.9256(14) 10
89.989(10) 89.956(18) 120
V = 423.1(2)
unit cell:
6.9264(6) 6.9264(6) 10.
90.0 90.0 120.
V = 423.15(6)
Options RED
CrysAlis RED program options (1.1.3)
Run list size
CCD
Processing
User/access
Distance calibration
SM\PX
Compression
Dark
Generator
Programs
E-mail
Instrument model I
Instrument model II
Monochromator
Fonts
Peak table
Color codes
Beam stop
Angular limits
Goniometer
This dialog allows you to modify the beam stop settings.
Put beamstop overlay on
Beam stop support orientation
Top
Bottom
Left
Right
Left 45deg
None
User
Gemini
Duo
Beam stop dimensions
45.000
Edit X-stop
0.000
Edit x-off
3.500
Edit stop dia
30.000
Edit y-off
Edit angle
1.500
Edit holder d
0.000
Edit gem off
To save changed beamstop position/size: wd cal


HP_DEMO_ - CrysAlisPro Gemini ultra system (Mo wavelength active) - RED view: F:\OD_Work\HP_DEMO\HP_DEMO_\HP_DEMO_.par
Inspect data reduction results and refinalize with user corrections
IMAGE: HP_DEMO__10_1.img (run: 10 frame: 1)
Omega: -33.0000 Theta: -23.4164 Kappa: 22.0000 Phi: -23.0000
Distance: 83.0000
0.88
1.03
1.27
1.70
2.61
5.42
START/STOP
RED
Crystal RED
EXPERIMENT
HP_DEMO_
LATTICE
Current cell (CSD: not done)
6.924(3) 6.9213(11) 10.1818(7)
89.950(9) 90.001(15) 120.05(3)
V = 422.37(19)
Constrained cell
6.9222(7) 6.9222(7) 10.1782(7)
90.0 90.0 120.0
V = 422.37(6)
Symmetry
Laue class: 1bar P-lattice
AVERAGE UNIT CELL FROM PROFFIT
Constrained cell (1182 obs)
6.9222(7) 6.9222(7) 10.1782(7)
90.0 90.0 120.0
V = 422.37(6)
PEAK TABLE
UB fit with 1182 obs out of 1182
(total:1182,skipped:0) (100.00%)
INSTRUMENT MODEL
X-ray wavelength: Mo
x-cen: 996.9059 y-cen: 1000.4268
distance: 83.0000
beam: -0.1050
Data Collection
Data Reduction
Agilent Technologies
CrysAlisPro
Image list
RED
Look at all frames
with information on run# and frame#
with prediction of reflection positions


HP_DEMO_ - CrysAlisPro Gemini ultra system (Mo wavelength active) - RED view: F:\OD_Work\HP_DEMO\HP_DEMO_\HP_DEMO_.par
Inspect data reduction results and refinalize with user corrections
IMAGE: HP_DEMO__10_1.img (run: 10 frame: 1)
Omega: -33.0000 Theta: -23.4164 Kappa: 22.0000 Phi: -23.0000
Distance: 83.0000
0.88
1.03
1.27
1.70
2.61
5.42
START/STOP
RED
Crystal RED
EXPERIMENT
HP_DEMO_
LATTICE
Current cell (CSD: not done)
6.924(3) 6.9213(11) 10.1818(7)
89.950(9) 90.001(15) 120.05(3)
V = 422.37(19)
Constrained cell
6.9222(7) 6.9222(7) 10.1782(7)
90.0 90.0 120.0
V = 422.37(6)
Symmetry
Laue class: 1bar P-lattice
AVERAGE UNIT CELL FROM PROFFIT
Constrained cell (1182 obs)
6.9222(7) 6.9222(7) 10.1782(7)
90.0 90.0 120.0
V = 422.37(6)
PEAK TABLE
UB fit with 1182 obs out of 1182
(total:1182,skipped:0) (100.00%)
INSTRUMENT MODEL
X-ray wavelength: Mo
x-cen: 996.9059 y-cen: 1000.4268
distance: 83.0000
beam: -0.1050
Data Collection
Data Reduction
Agilent Technologies
CrysAlisPro
Image list
RED

Dc rrp

Inspect data collection and reduction results

CrysAlis Pro

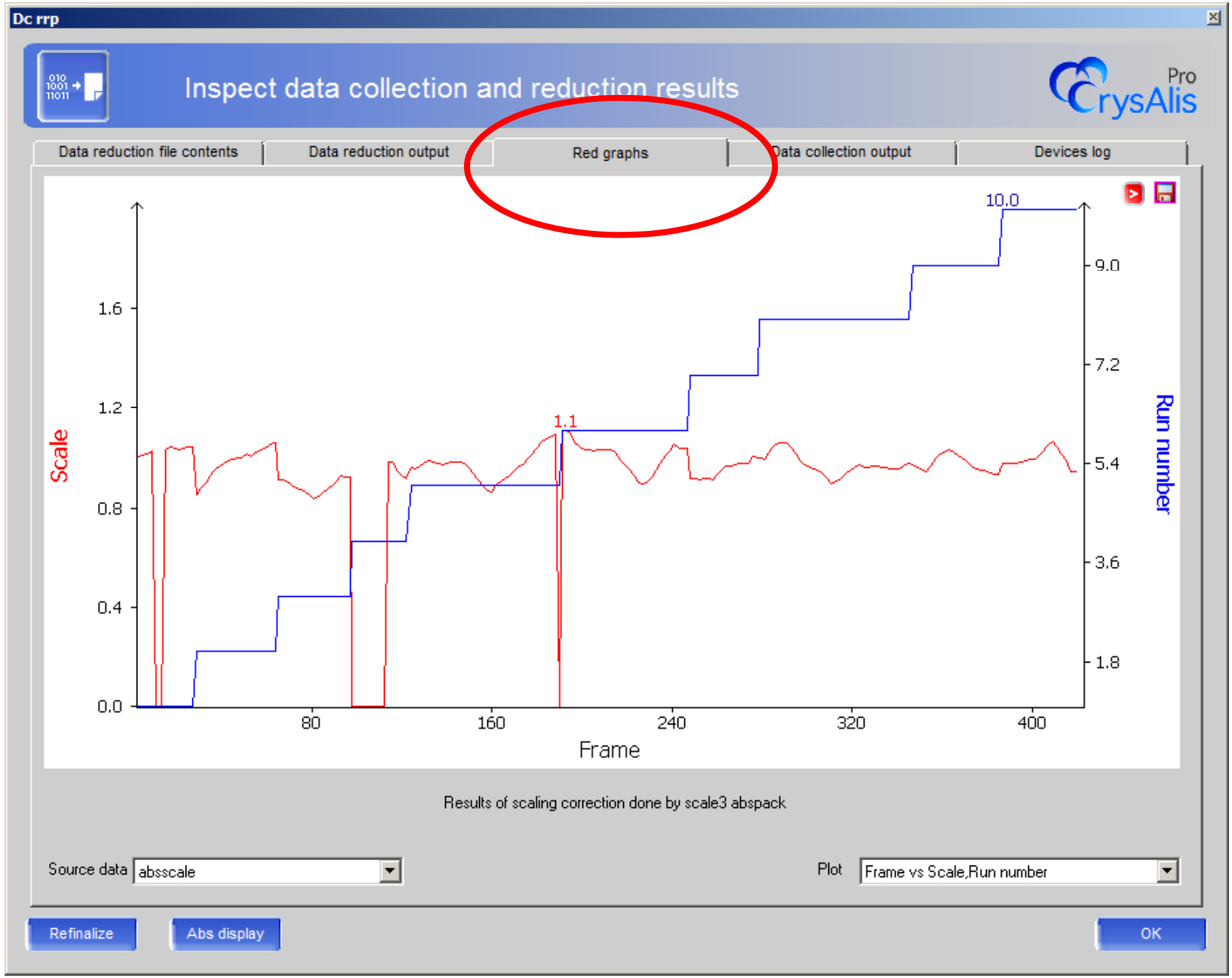
Data reduction file contents | Data reduction output | Red graphs | Data collection output | Devices log

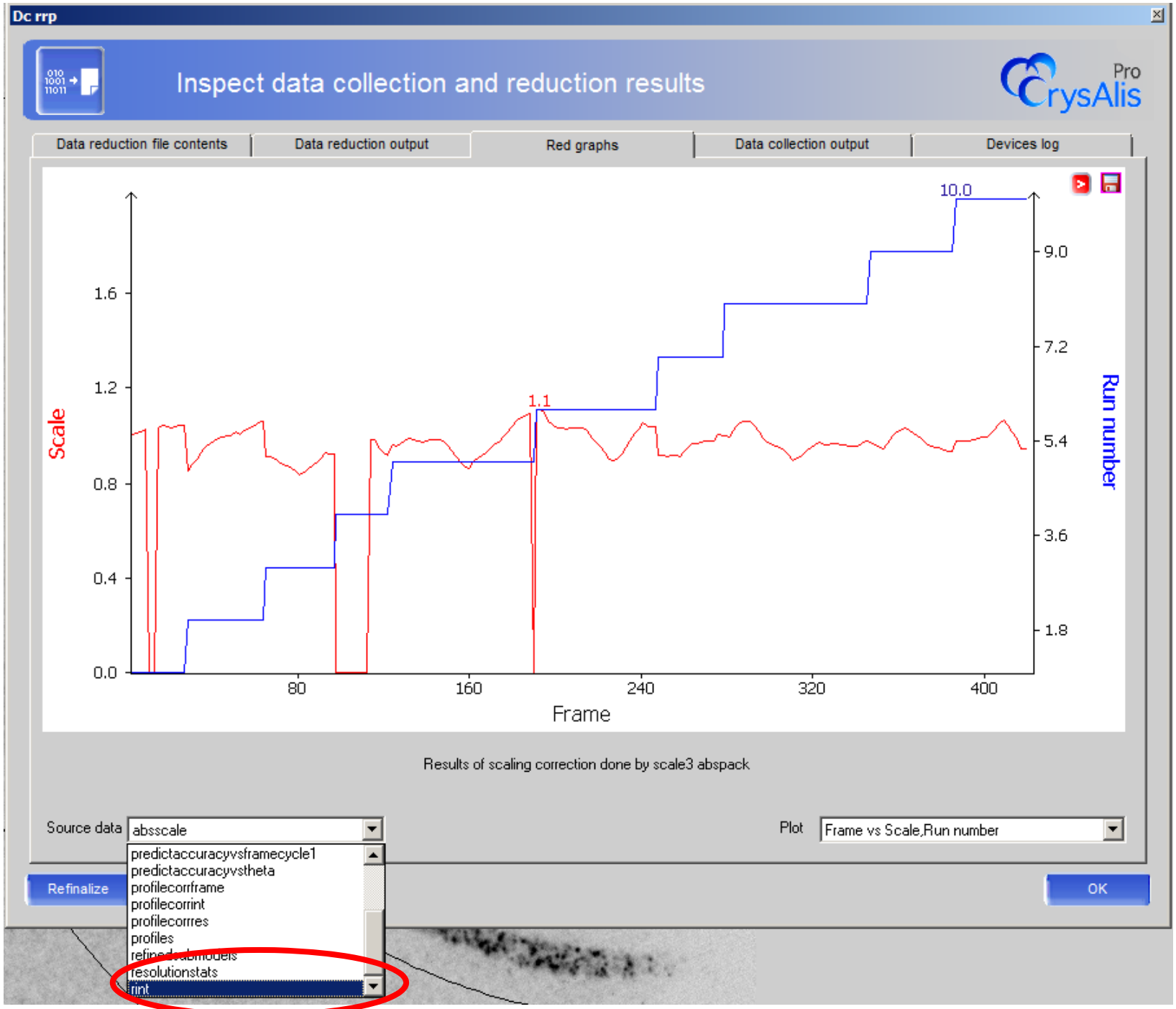
1.34-1.17	350	65	65	100.0	5.4	4376.16	16.47	0.115	0.046
1.17-1.07	269	65	65	100.0	4.1	3042.06	11.77	0.218	0.075
1.07-0.98	264	67	65	97.0	4.1	3034.99	10.47	0.359	0.089
0.98-0.92	266	68	66	97.1	4.0	2304.14	8.57	0.288	0.098
0.92-0.87	263	68	65	95.6	4.0	1477.23	5.79	0.531	0.146
0.87-0.83	204	67	65	97.0	3.1	1378.82	4.64	0.451	0.174
0.83-0.80	222	69	65	94.2	3.4	1378.41	4.73	0.549	0.173
0.80-0.75	125	123	70	56.9	1.8	1642.45	3.83	0.265	0.183
inf-0.75	2686	724	657	90.7	4.1	3770.95	15.49	0.179	0.066
inf-0.80	2566	603	589	97.7	4.4	3867.60	16.03	0.178	0.063

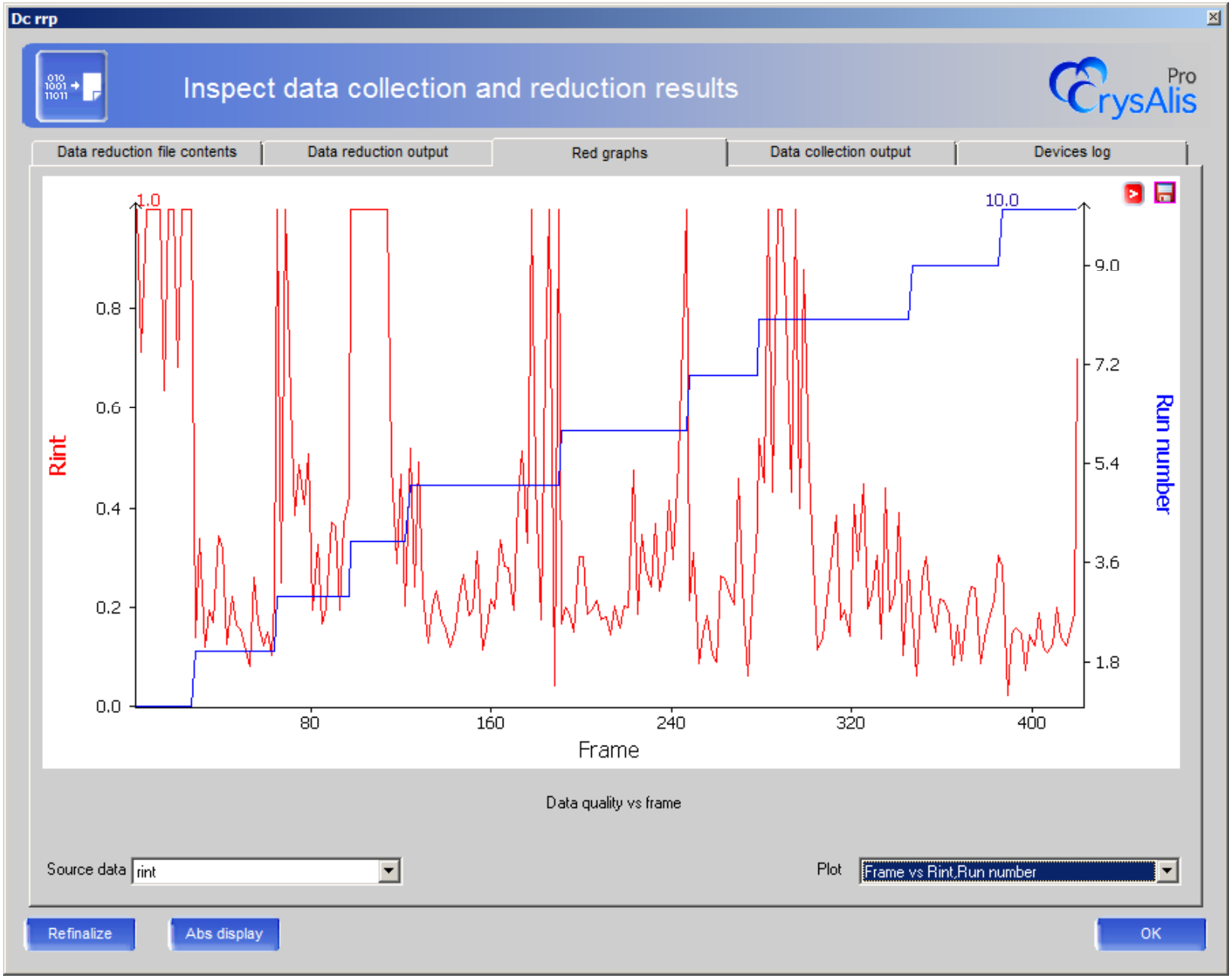
Statistics vs resolution (taking redundancy into account) - Laue group: P6/m (hex-c)

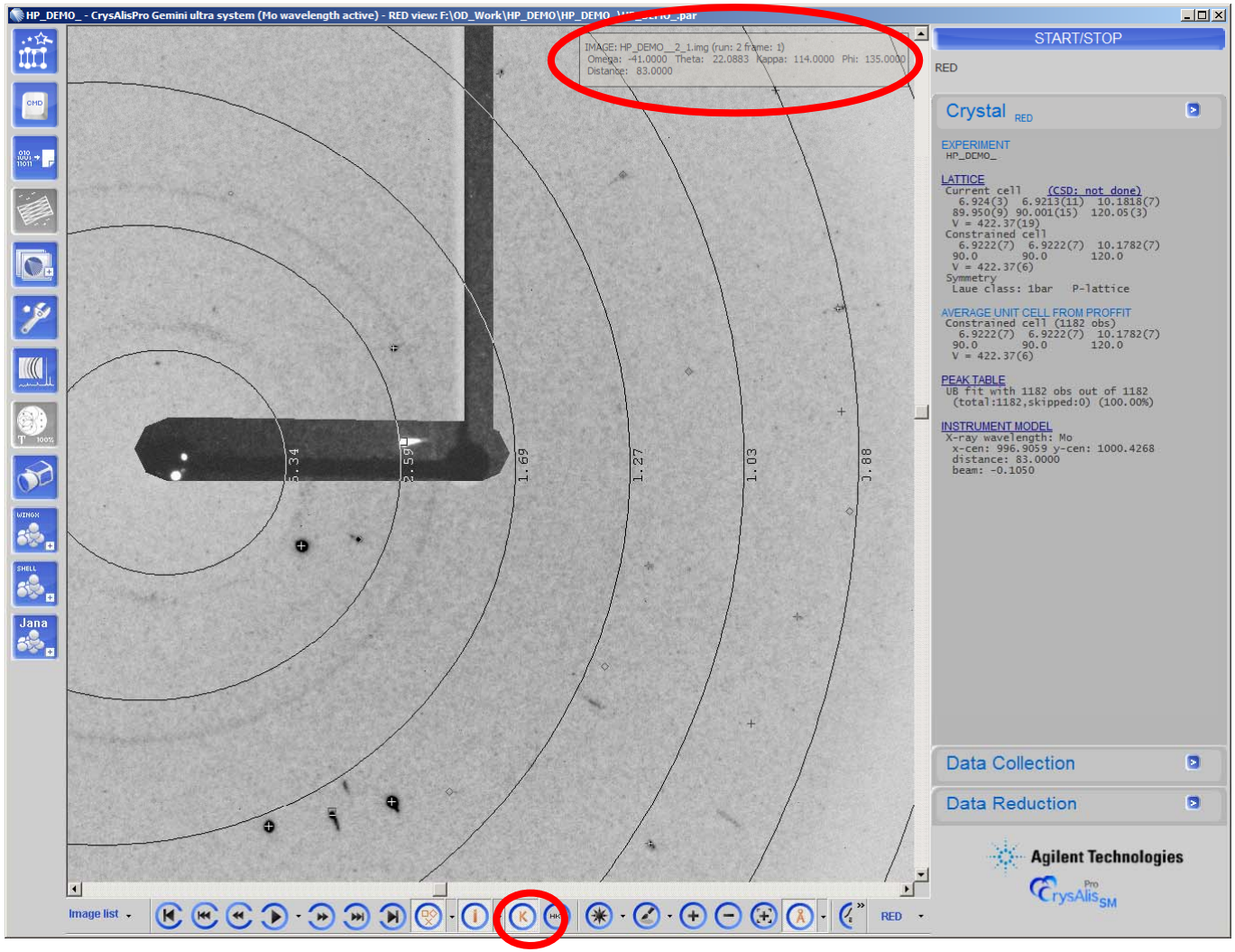
resolution(A)	# kept	# theory	# unique	% complete	average redundancy	mean F2	mean F2/sig(F2)	Rint	RsigmaB
inf-1.70	365	37	37	100.0	9.9	7439.89	46.34	0.070	0.017
1.69-1.33	383	37	37	100.0	10.4	6698.87	41.29	0.106	0.026
1.33-1.15	352	37	37	100.0	9.5	4567.71	21.00	0.112	0.031
1.14-1.04	302	37	37	100.0	8.2	2635.08	15.43	0.290	0.069
1.04-0.96	295	37	37	100.0	8.0	3244.61	15.07	0.390	0.066
0.96-0.91	244	37	37	100.0	6.6	1602.12	8.18	0.395	0.102
0.91-0.86	274	37	37	100.0	7.4	1429.96	8.00	0.588	0.119
0.86-0.82	205	37	37	100.0	5.5	1433.78	6.41	0.674	0.139
0.82-0.78	197	37	37	100.0	5.3	1736.34	7.15	0.449	0.130
0.78-0.75	69	48	39	81.3	1.8	986.38	2.72	0.589	0.270
inf-0.75	2686	381	372	97.6	7.2	3770.95	20.97	0.200	0.052
inf-0.80	2566	318	318	100.0	8.1	3867.60	21.72	0.199	0.048

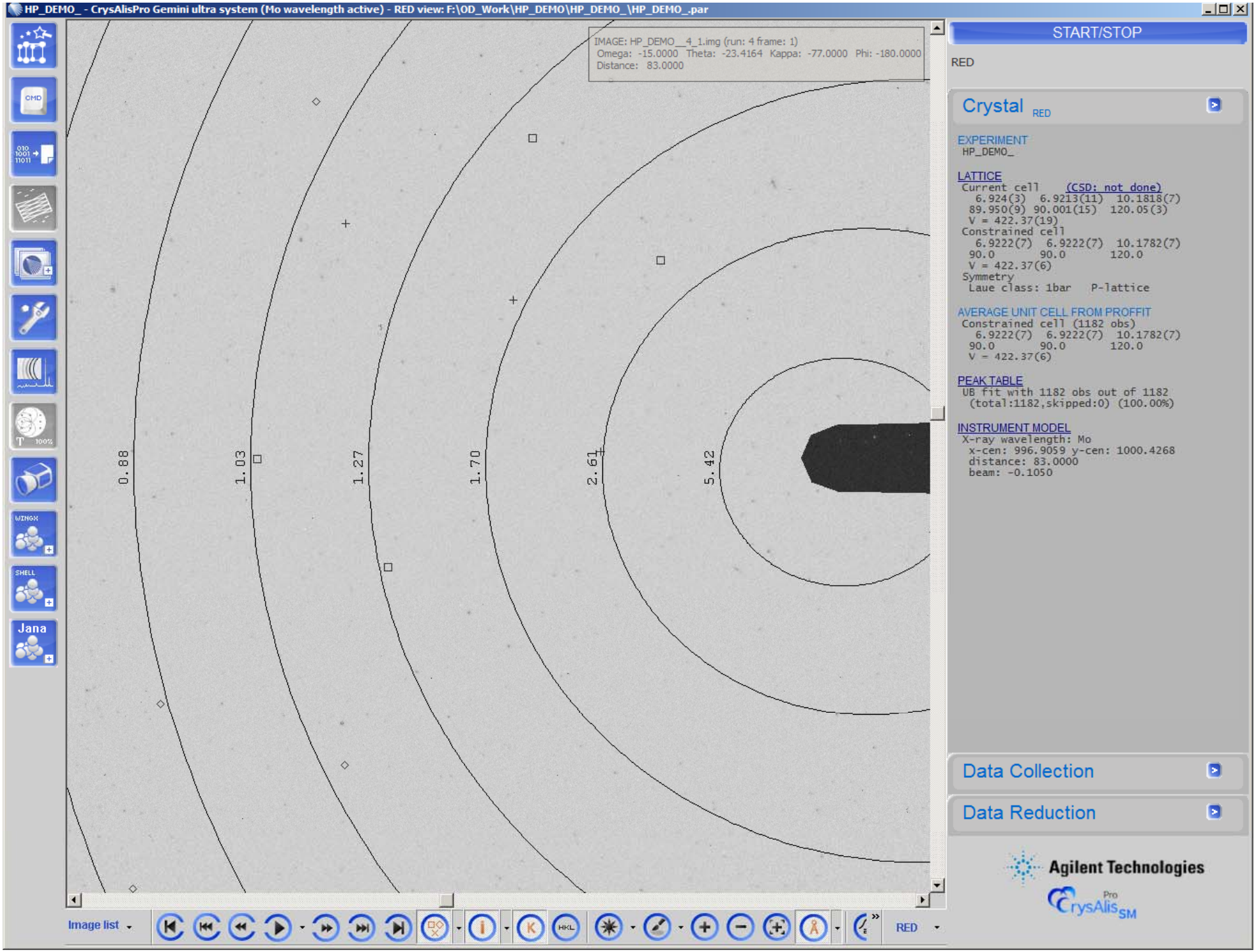
Refinalize | Abs display | OK


Dc rrp
Inspect data collection and reduction results
Pro
CrysAlis
Data reduction file contents
Data reduction output
Red graphs
Data collection output
Devices log
10.0
1.1
Scale
Run number
Frame
1.6
1.2
0.8
0.4
0.0
9.0
7.2
5.4
3.6
1.8
80
160
240
320
400
Results of scaling correction done by scale3 abspack
Source data
absscale
Plot
Frame vs Scale,Run number
Refinalize
Abs display
OK


Dc rrp
Inspect data collection and reduction results
CrysAlis Pro
Data reduction file contents
Data reduction output
Red graphs
Data collection output
Devices log
10.0
1.1
Scale
Run number
Frame
0.0
0.4
0.8
1.2
1.6
1.8
3.6
5.4
7.2
9.0
80
160
240
320
400
Results of scaling correction done by scale3 abspack
Source data
absscale
predictaccuracyvsframecycle1
predictaccuracyvstheta
profilecorrframe
profilecorrint
profilecorrres
profiles
resolutionstats
rint
Plot
Frame vs Scale,Run number
Refinalize
OK


Dc rrp
Inspect data collection and reduction results
CrysAlis Pro
Data reduction file contents
Data reduction output
Red graphs
Data collection output
Devices log
1.0
10.0
0.8
0.6
0.4
0.2
0.0
Rint
9.0
7.2
5.4
3.6
1.8
Run number
80
160
240
320
400
Frame
Data quality vs frame
Source data
rint
Plot
Frame vs Rint,Run number
Refinalize
Abs display
OK


HP_DEMO_ - CrysAlisPro Gemini ultra system (Mo wavelength active) - RED view: F:\OD_Work\HP_DEMO\HP_DEMO_
IMAGE: HP_DEMO__2_1.img (run: 2 frame: 1)
Omega: -41.0000 Theta: 22.0883 Kappa: 114.0000 Phi: 135.0000
Distance: 83.0000
5.34
2.59
1.69
1.27
1.03
0.88
START/STOP
RED
Crystal RED
EXPERIMENT
HP_DEMO_
LATTICE
Current cell (CSD: not done)
6.924(3) 6.9213(11) 10.1818(7)
89.950(9) 90.001(15) 120.05(3)
V = 422.37(19)
Constrained cell
6.9222(7) 6.9222(7) 10.1782(7)
90.0 90.0 120.0
V = 422.37(6)
Symmetry
Laue class: 1bar P-lattice
AVERAGE UNIT CELL FROM PROFFIT
Constrained cell (1182 obs)
6.9222(7) 6.9222(7) 10.1782(7)
90.0 90.0 120.0
V = 422.37(6)
PEAK TABLE
UB fit with 1182 obs out of 1182
(total:1182,skipped:0) (100.00%)
INSTRUMENT MODEL
X-ray wavelength: Mo
x-cen: 996.9059 y-cen: 1000.4268
distance: 83.0000
beam: -0.1050
Data Collection
Data Reduction
Agilent Technologies
CrysAlisPro SM
Image list
RED
Jana


HP_DEMO_ - CrysAlisPro Gemini ultra system (Mo wavelength active) - RED view: F:\OD_Work\HP_DEMO\HP_DEMO_\HP_DEMO_.par
IMAGE: HP_DEMO__4_1.img (run: 4 frame: 1)
Omega: -15.0000 Theta: -23.4164 Kappa: -77.0000 Phi: -180.0000
Distance: 83.0000
0.88
1.03
1.27
1.70
2.61
5.42
START/STOP
RED
Crystal RED
EXPERIMENT
HP_DEMO_
LATTICE
Current cell (CSD: not done)
6.924(3) 6.9213(11) 10.1818(7)
89.950(9) 90.001(15) 120.05(3)
V = 422.37(19)
Constrained cell
6.9222(7) 6.9222(7) 10.1782(7)
90.0 90.0 120.0
V = 422.37(6)
Symmetry
Laue class: 1bar P-lattice
AVERAGE UNIT CELL FROM PROFFIT
Constrained cell (1182 obs)
6.9222(7) 6.9222(7) 10.1782(7)
90.0 90.0 120.0
V = 422.37(6)
PEAK TABLE
UB fit with 1182 obs out of 1182
(total:1182,skipped:0) (100.00%)
INSTRUMENT MODEL
X-ray wavelength: Mo
x-cen: 996.9059 y-cen: 1000.4268
distance: 83.0000
beam: -0.1050
Data Collection
Data Reduction
Agilent Technologies
Pro CrysAlisSM
Image list
RED

Pre-Analysis by the user:

In this case of bad data:

Hint that something is not good from

(1) Looking through frames:

e.g. Run 4 has lots of non-exposed frames

(3) Automatic data reduction also shows bad RINT for run 4,

but also things are not good

(1) Introduction to *CrysAlisPro*

(2) General remarks

(3) Centre DAC

(4) Data collection

(5) Pre-analysis by user

(6) Best possible data reduction for high pressure data

Get good unit cell

Data reduction

Absorption correction

(1) Introduction to *CrysAlisPro*

(2) General remarks

(3) Centre DAC

(4) Data collection

(5) Pre-analysis by user

(6) Best possible data reduction for high pressure data

Get good unit cell

Data reduction

Absorption correction

Unit cell

Good crystal, good centering, good data collection

→ *CrysAlisPro* usually finds UB

What to do if this is not the case:

(1) New peak search with : exclude region of powder ring from gasket

(2) Delete strong diamond reflections

(3) Search for known cell

Unit cell

Good crystal, good centering, good data collection

→ *CrysAlisPro* usually finds UB

What to do if this is not the case:

(1) New peak search with : exclude region of powder ring from gasket

(2) Delete strong diamond reflections

(3) Search for known cell

Unit cell

Good crystal, good centering, good data collection

→ *CrysAlisPro* usually finds UB

UB still not found:

Dataset very likely not good enough, nevertheless further tricks to find UB

(4) In reciprocal space viewer:

Delete every reflection, that you know is not from the sample you are interested in

(5) Delete reflection list and hand pick reflections

Unit cell

Now, how does it work in the software:

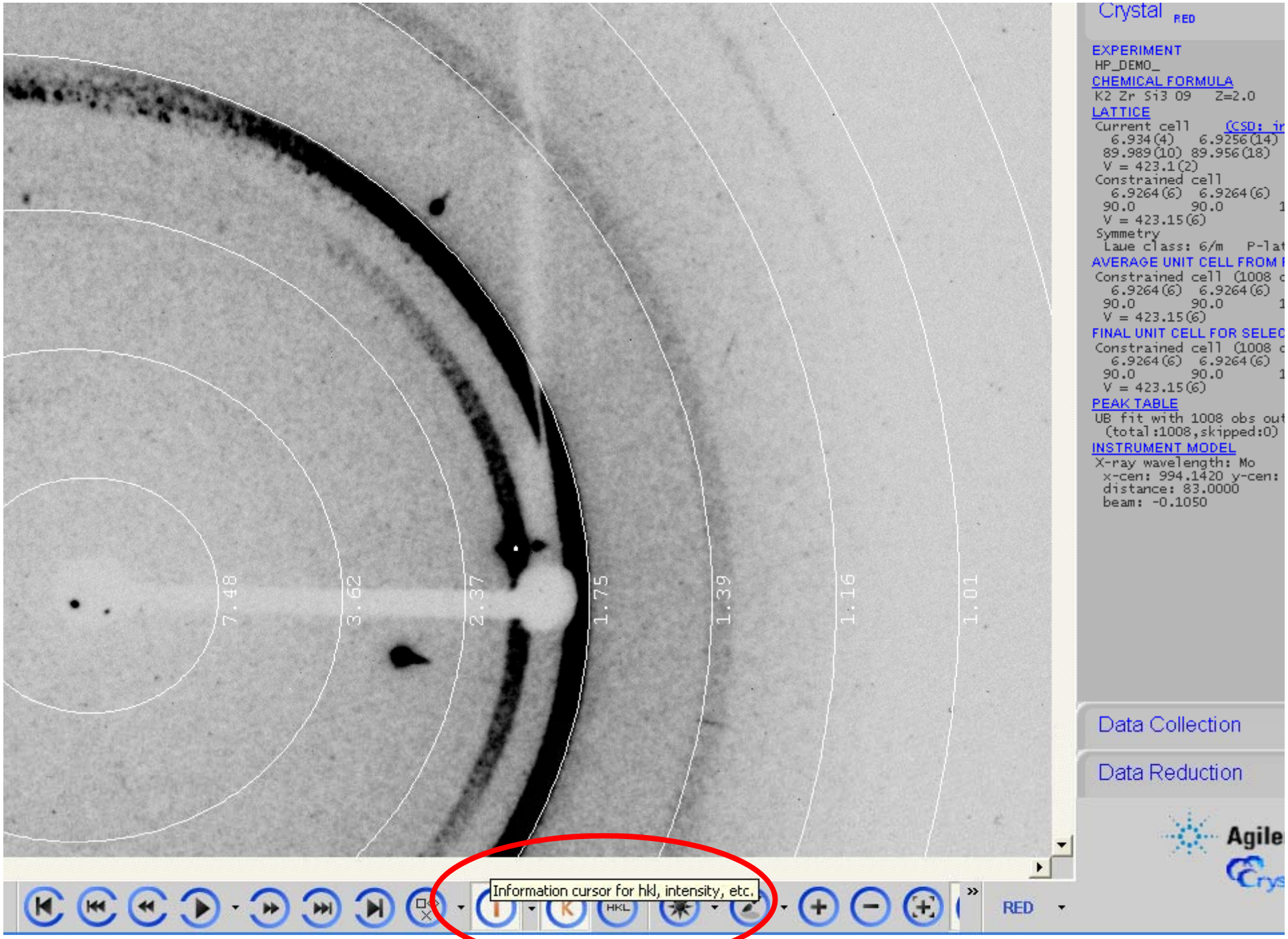
Unit cell

Now, how does it work in the software:

(1) New peak search with

(1) exclude region of powder ring from gasket

(2) reflections around beamstop


Crystal RED
EXPERIMENT
HP_DEMO_
CHEMICAL FORMULA
K2 Zr Si3 O9 Z=2.0
LATTICE
Current cell
6.934(4) 6.9256(14)
89.989(10) 89.956(18)
V = 423.1(2)
Constrained cell
6.9264(6) 6.9264(6)
90.0 90.0
V = 423.15(6)
Symmetry
Laue class: 6/m
AVERAGE UNIT CELL FROM
Constrained cell (1008
6.9264(6) 6.9264(6)
90.0 90.0
V = 423.15(6)
FINAL UNIT CELL FOR SELEC
Constrained cell (1008
6.9264(6) 6.9264(6)
90.0 90.0
V = 423.15(6)
PEAK TABLE
UB fit with 1008 obs
(total:1008,skipped:0)
INSTRUMENT MODEL
X-ray wavelength: Mo
x-cen: 994.1420 y-cen:
distance: 83.0000
beam: -0.1050
7.48
3.62
2.37
1.75
1.39
1.16
1.01
Data Collection
Data Reduction
Information cursor for hkl, intensity, etc.
HKL
RED

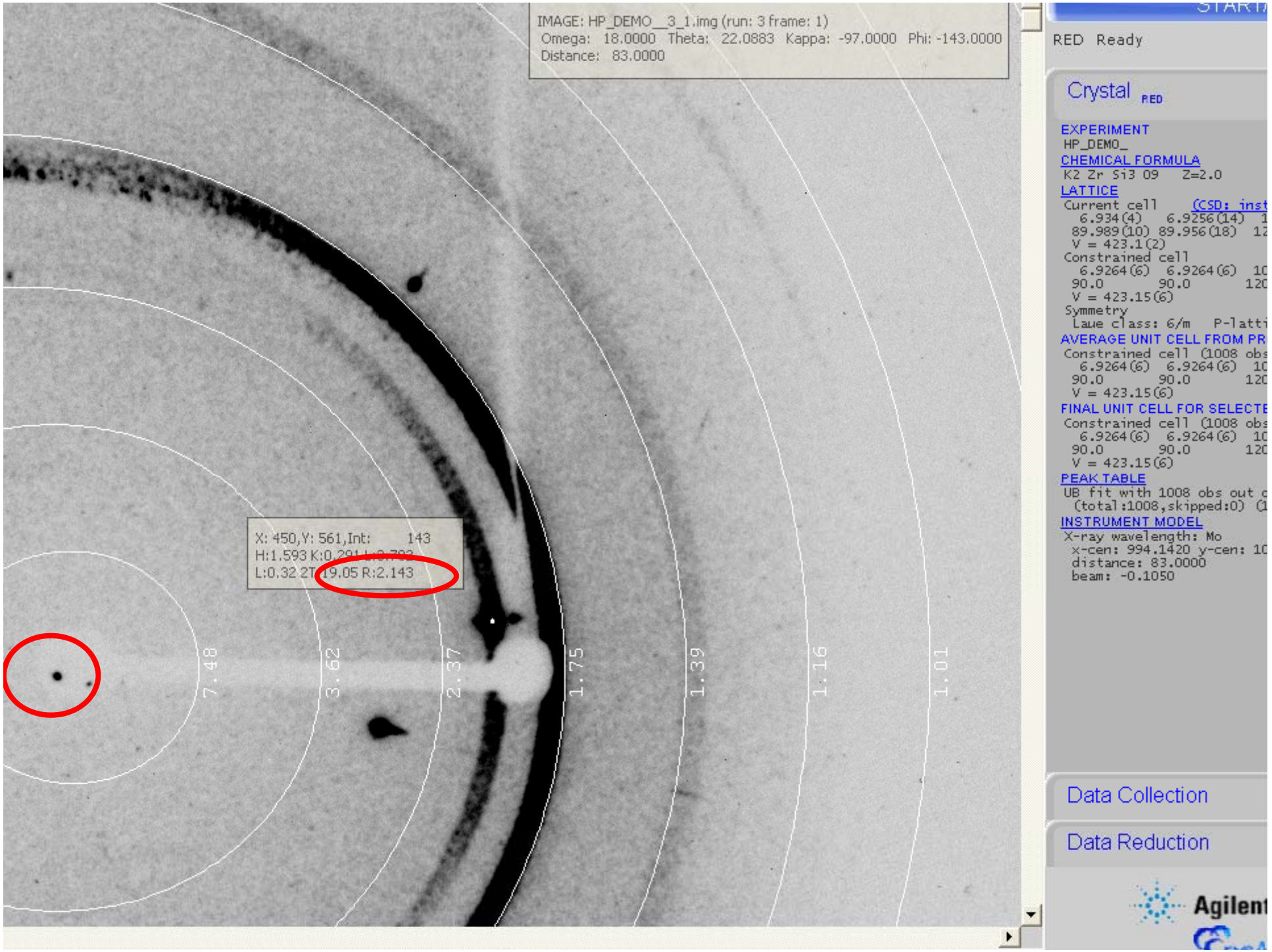


IMAGE: HP_DEMO__3_1.img (run: 3 frame: 1)
Omega: 18.0000 Theta: 22.0883 Kappa: -97.0000 Phi: -143.0000
Distance: 83.0000
X: 450,Y: 561,Int: 143
H:1.593 K:0.291
L:0.32 2T 19.05 R:2.143
7.48
3.62
2.37
1.75
1.39
1.16
1.01
RED Ready
Crystal RED
EXPERIMENT
HP_DEMO_
CHEMICAL FORMULA
K2 Zr Si3 O9 Z=2.0
LATTICE
Current cell (CSD: inst
6.934(4) 6.9256(14) 1
89.989(10) 89.956(18) 12
V = 423.1(2)
Constrained cell
6.9264(6) 6.9264(6) 10
90.0 90.0 120
V = 423.15(6)
Symmetry
Laue class: 6/m P-latti
AVERAGE UNIT CELL FROM PR
Constrained cell (1008 obs
6.9264(6) 6.9264(6) 10
90.0 90.0 120
V = 423.15(6)
FINAL UNIT CELL FOR SELECTE
Constrained cell (1008 obs
6.9264(6) 6.9264(6) 10
90.0 90.0 120
V = 423.15(6)
PEAK TABLE
UB fit with 1008 obs out o
(total:1008,skipped:0) (1
INSTRUMENT MODEL
X-ray wavelength: Mo
x-cen: 994.1420 y-cen: 10
distance: 83.0000
beam: -0.1050
Data Collection
Data Reduction
Agilent

Unit cell

Now, how does it work in the software:

1) New peak search with

(1) exclude region of powder ring from gasket

(2) reflections around beamstop

Either from commandline:

Command **um skipd dmax dmin** prevents peak searching between dmax and dmin

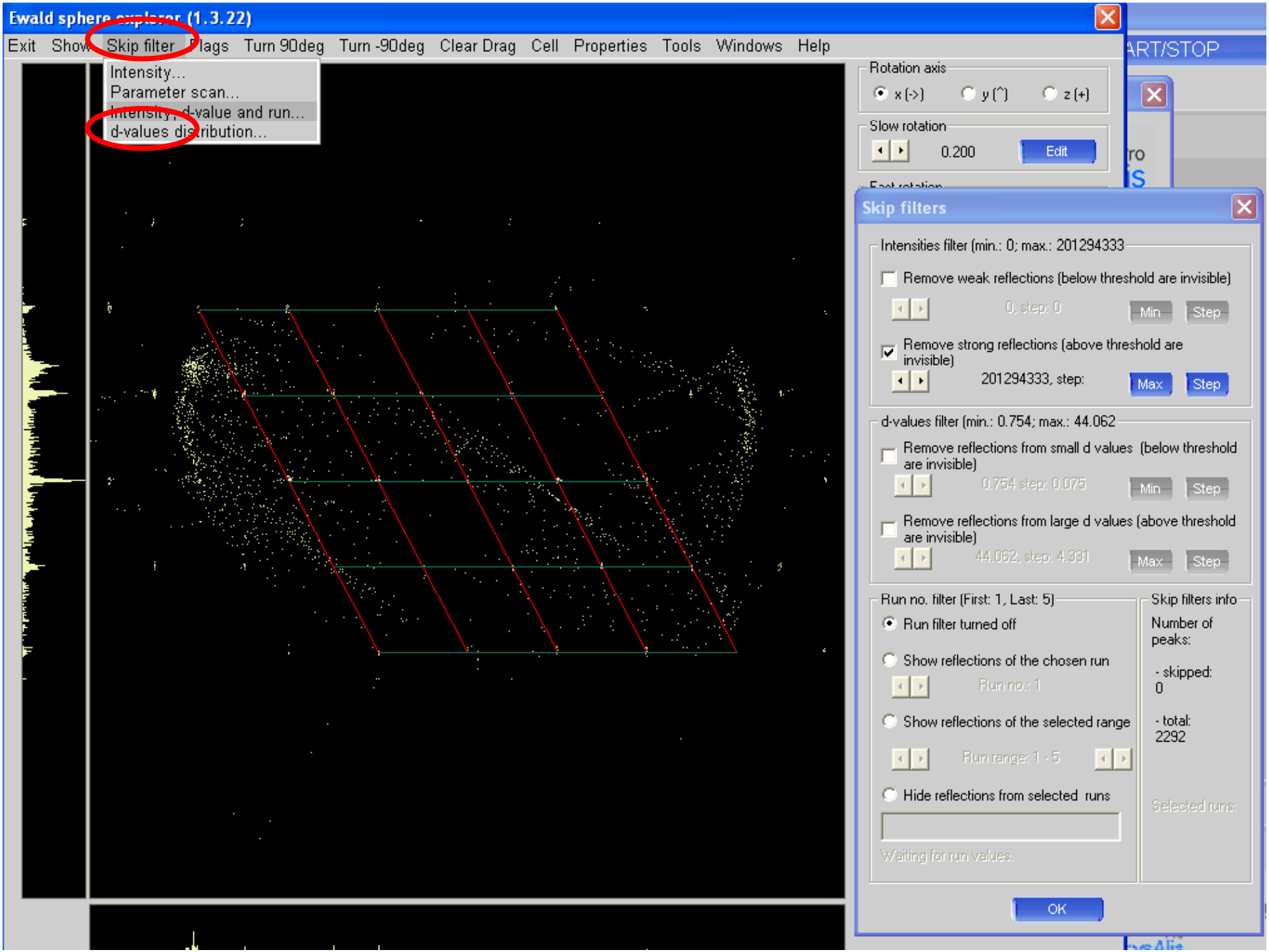
Reflection around beamstop:

skip region from 999.0 down to slightly longer than your unit-cell

um skipd 999.0 9.0

Set a skip region for strong powder lines from gasket material:

um skipd 2.15 1.75


Ewald sphere explorer (1.3.22)
Exit
Show
Skip filter
Flags
Turn 90deg
Turn -90deg
Clear Drag
Cell
Properties
Tools
Windows
Help
Intensity...
Parameter scan...
Intensity, d-value and run...
d-values distribution...
Rotation axis
x (->)
y (^)
z (+)
Slow rotation
0.200
Edit
Skip filters
Intensities filter (min.: 0; max.: 201294333
Remove weak reflections (below threshold are invisible)
0, step: 0
Min
Step
Remove strong reflections (above threshold are invisible)
201294333, step:
Max
Step
d-values filter (min.: 0.754; max.: 44.062
Remove reflections from small d values (below threshold are invisible)
0.754 step: 0.075
Min
Step
Remove reflections from large d values (above threshold are invisible)
44.062, step: 4.331
Max
Step
Run no. filter (First: 1, Last: 5)
Run filter turned off
Show reflections of the chosen run
Run no.: 1
Show reflections of the selected range
Run range: 1 - 5
Hide reflections from selected runs
Waiting for run values.
Skip filters info
Number of peaks:
- skipped:
0
- total:
2292
Selected runs:
OK

Unit cell

Now, how does it work in the software:

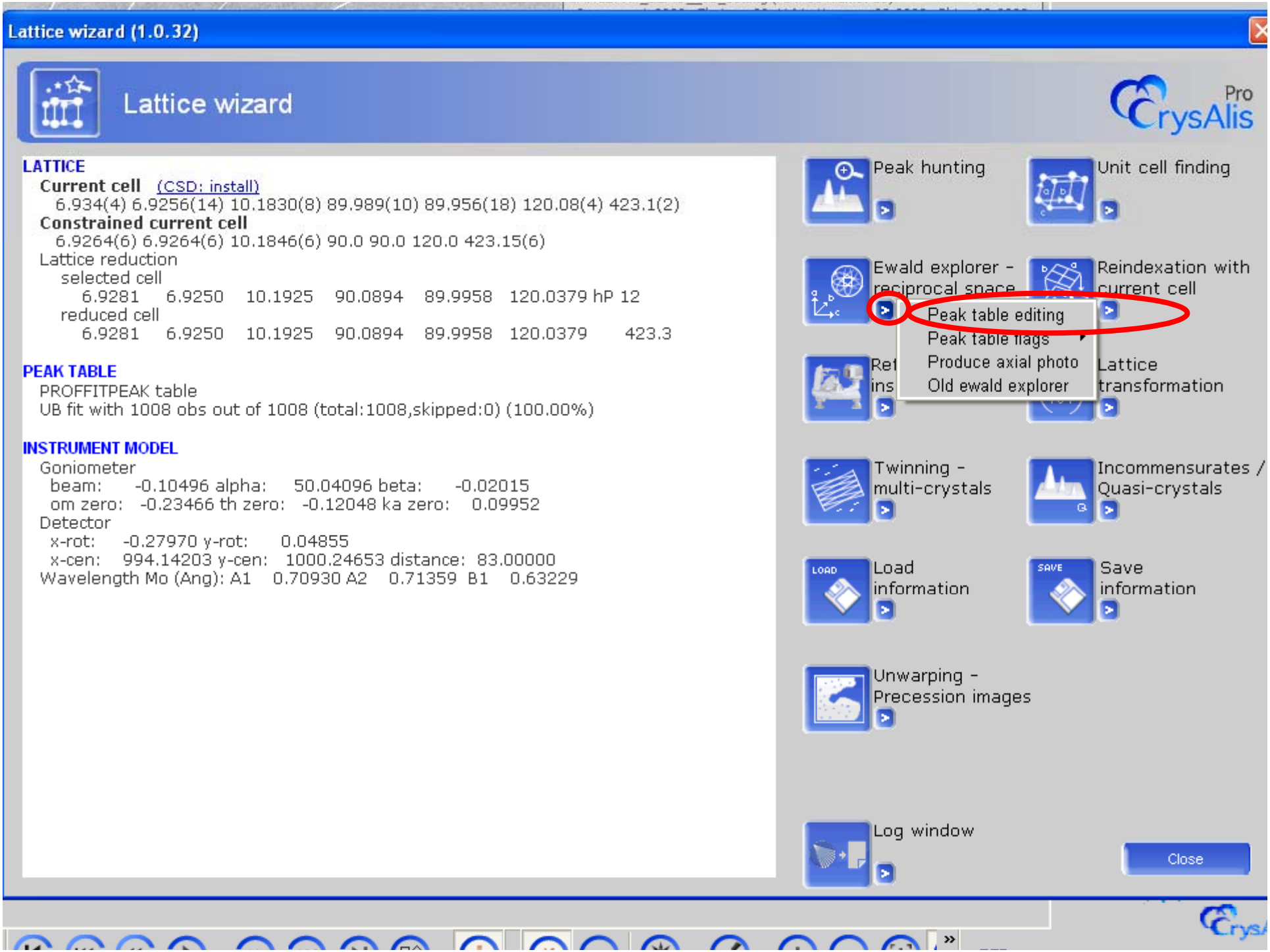
(2) Delete strong diamond reflections

Many ways to do that:

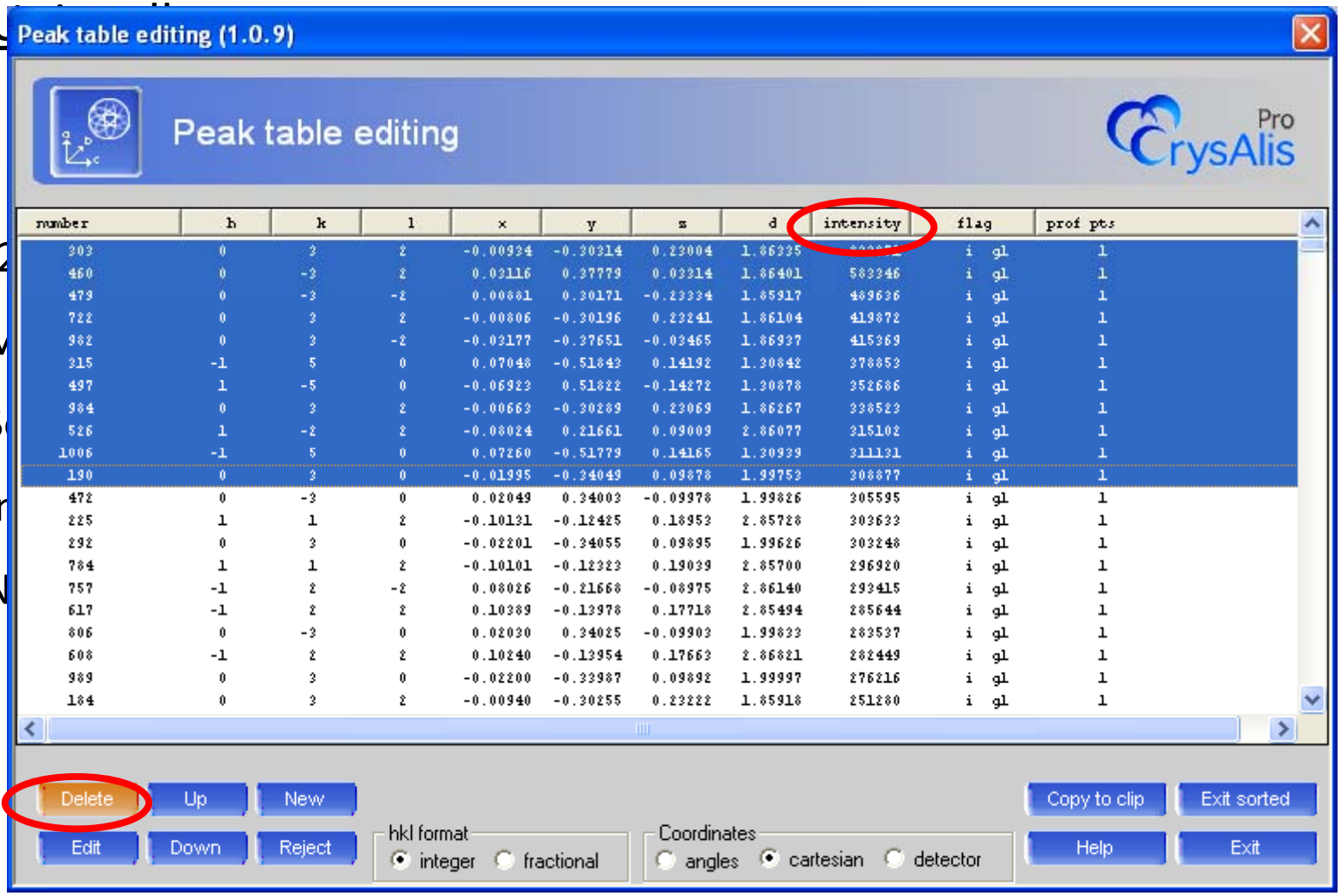
Sort list by intensity and delete strongest reflections

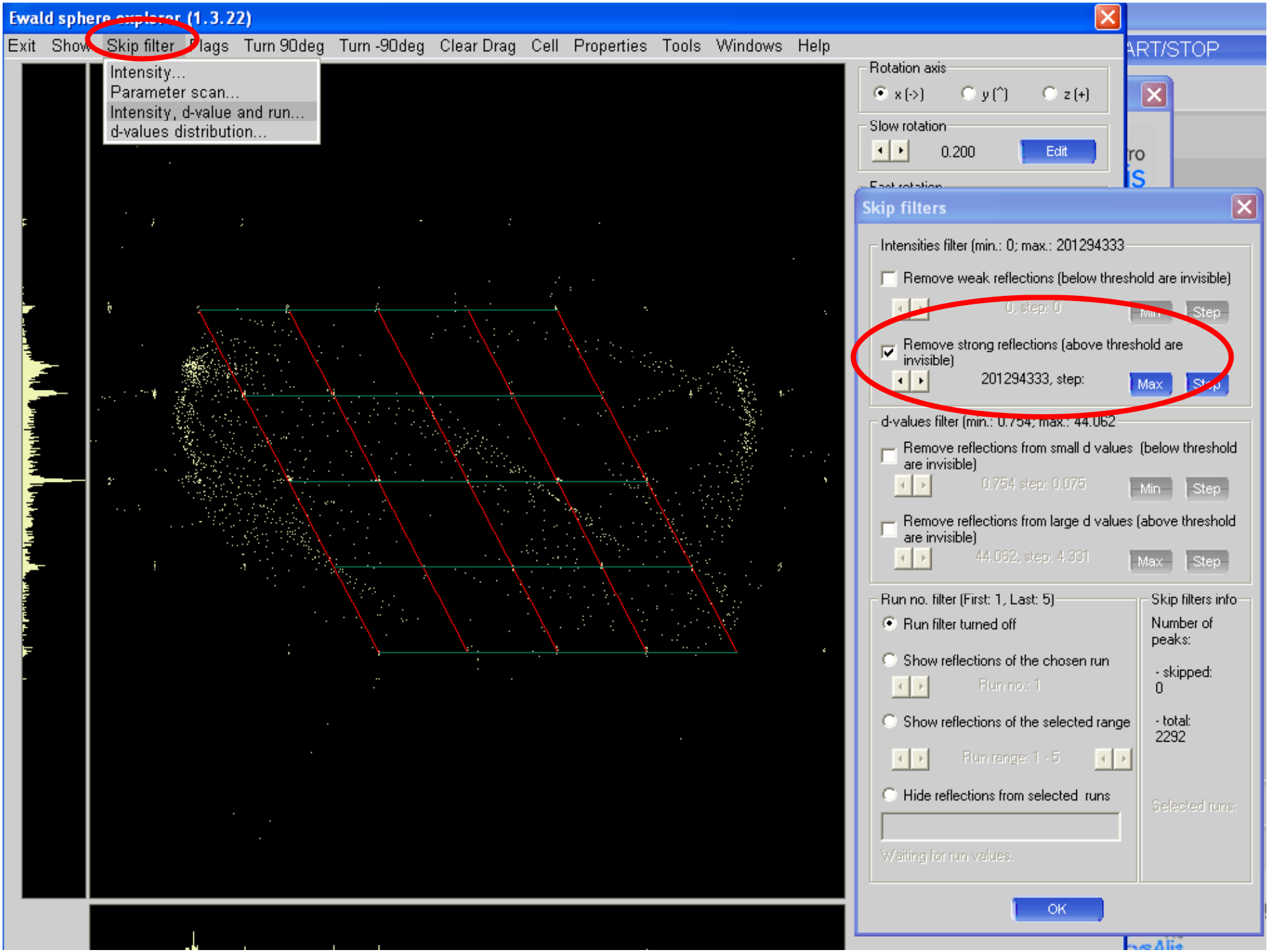
Intensity filter in Ewald explorer

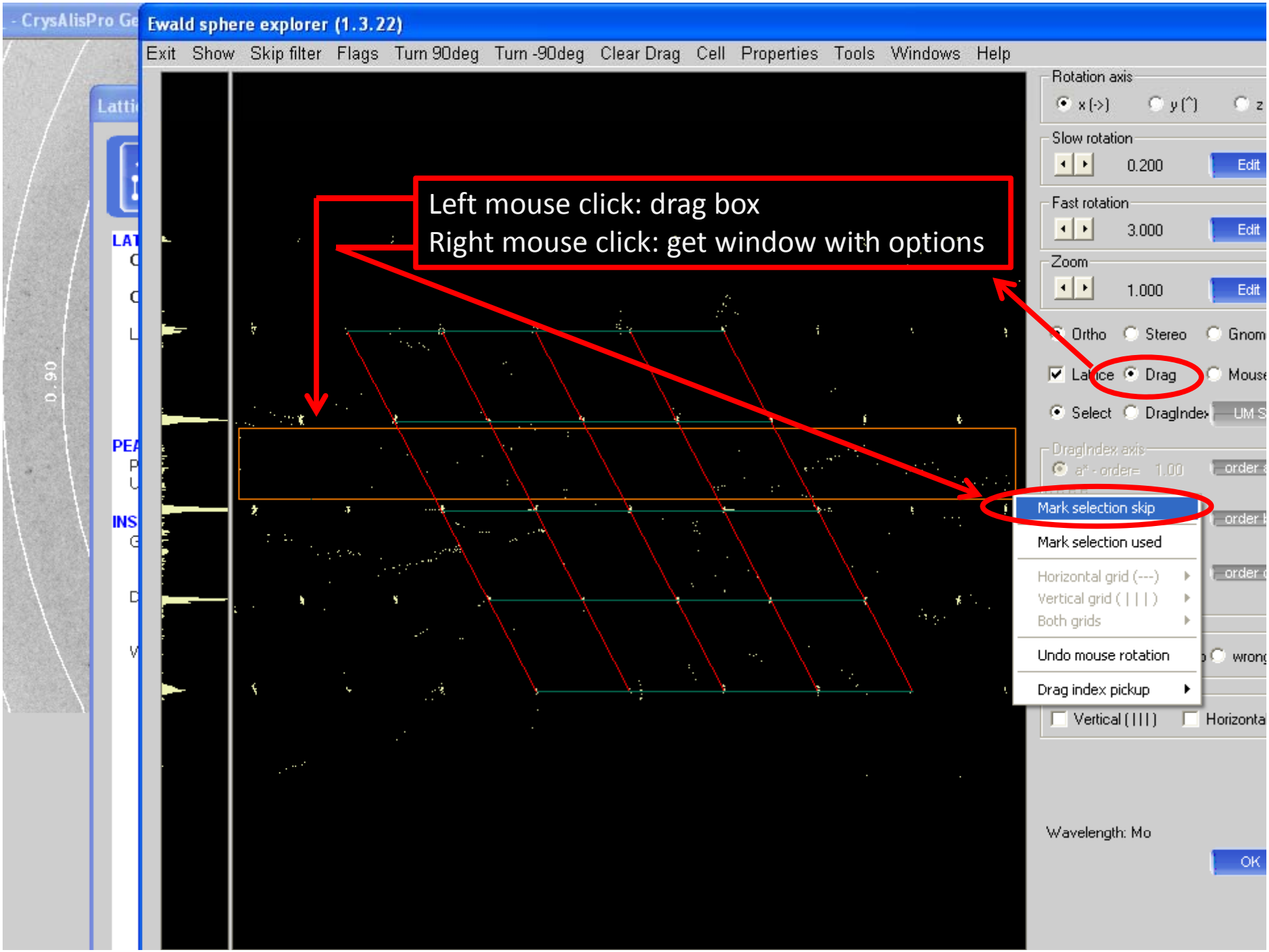
New Ewald explorer→ see OD talk

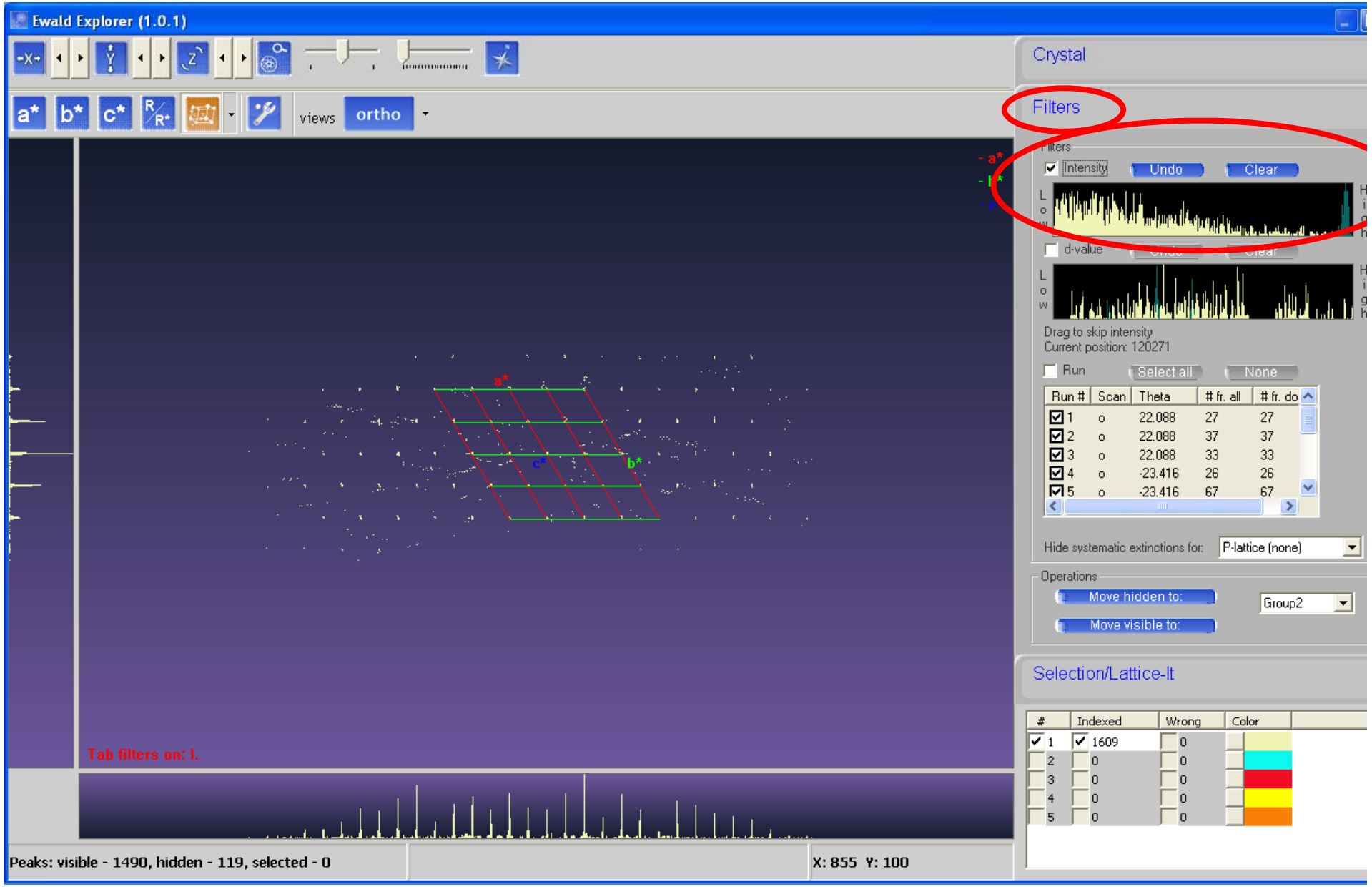

Lattice wizard (1.0.32)
Lattice wizard
Pro CrysAlis
LATTICE
Current cell (CSD: install)
6.934(4) 6.9256(14) 10.1830(8) 89.989(10) 89.956(18) 120.08(4) 423.1(2)
Constrained current cell
6.9264(6) 6.9264(6) 10.1846(6) 90.0 90.0 120.0 423.15(6)
Lattice reduction
selected cell
6.9281 6.9250 10.1925 90.0894 89.9958 120.0379 hP 12
reduced cell
6.9281 6.9250 10.1925 90.0894 89.9958 120.0379 423.3
PEAK TABLE
PROFFITPEAK table
UB fit with 1008 obs out of 1008 (total:1008,skipped:0) (100.00%)
INSTRUMENT MODEL
Goniometer
beam: -0.10496 alpha: 50.04096 beta: -0.02015
om zero: -0.23466 th zero: -0.12048 ka zero: 0.09952
Detector
x-rot: -0.27970 y-rot: 0.04855
x-cen: 994.14203 y-cen: 1000.24653 distance: 83.00000
Wavelength Mo (Ang): A1 0.70930 A2 0.71359 B1 0.63229
Peak hunting
Unit cell finding
Ewald explorer - reciprocal space
Reindexation with current cell
Peak table editing
Peak table flags
Produce axial photo
Old ewald explorer
Lattice transformation
Twinning - multi-crystals
Incommensurates / Quasi-crystals
LOAD
Load information
SAVE
Save information
Unwarping - Precession images
Log window
Close

1.261	022 diamond
1.075	113 diamond




Ewald sphere explorer (1.3.22)
Exit
Show
Skip filter
Flags
Turn 90deg
Turn -90deg
Clear Drag
Cell
Properties
Tools
Windows
Help
Intensity...
Parameter scan...
Intensity, d-value and run...
d-values distribution...
Rotation axis
x (->)
y (^)
z (+)
Slow rotation
0.200
Edit
Skip filters
Intensities filter (min.: 0; max.: 201294333
Remove weak reflections (below threshold are invisible)
Step
Remove strong reflections (above threshold are invisible)
201294333, step:
Max
Step
d-values filter (min.: 0.754; max.: 44.062
Remove reflections from small d values (below threshold are invisible)
0.754 step: 0.075
Min
Step
Remove reflections from large d values (above threshold are invisible)
44.062, step: 4.331
Max
Step
Run no. filter (First: 1, Last: 5)
Run filter turned off
Show reflections of the chosen run
Run no.: 1
Show reflections of the selected range
Run range: 1 - 5
Hide reflections from selected runs
Waiting for run values.
Skip filters info
Number of peaks:
- skipped:
0
- total:
2292
Selected runs:
OK

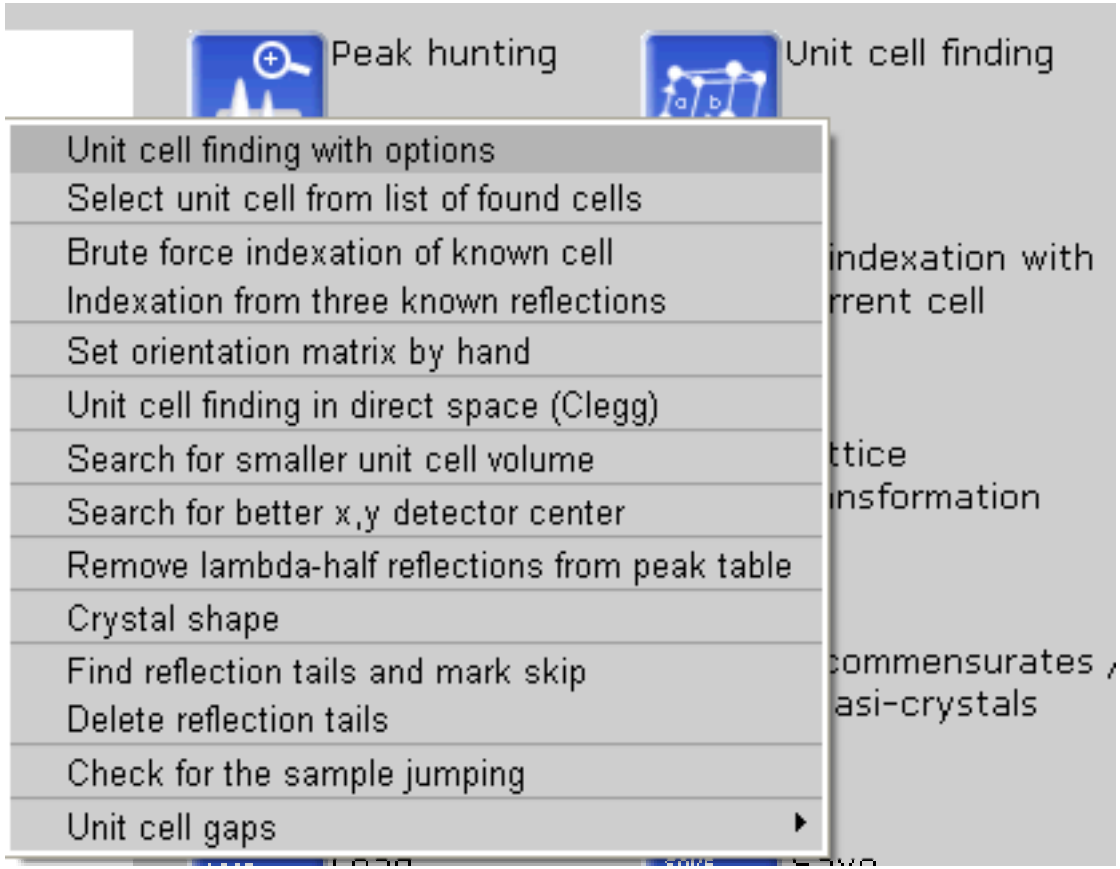

- CrysAlisPro Ge
Ewald sphere explorer (1.3.22)
Exit Show Skip filter Flags Turn 90deg Turn -90deg Clear Drag Cell Properties Tools Windows Help
Left mouse click: drag box
Right mouse click: get window with options
Rotation axis
x (->) y (^) z
Slow rotation
0.200
Edit
Fast rotation
3.000
Edit
Zoom
1.000
Edit
Ortho Stereo Gnom
Lattice Drag Mouse
Select DragIndex
DragIndex axis
Mark selection skip
Mark selection used
Horizontal grid (---)
Vertical grid (| | |)
Both grids
Undo mouse rotation
Drag index pickup
Vertical (|||)
Wavelength: Mo
OK


Ewald Explorer (1.0.1)
a*
b*
c*
views
ortho
Crystal
Filters
Intensity
Undo
Clear
d-value
Drag to skip intensity
Current position: 120271
Run
Select all
None
Run # Scan Theta # fr. all # fr. do
1 o 22.088 27 27
2 o 22.088 37 37
3 o 22.088 33 33
4 o -23.416 26 26
5 o -23.416 67 67
Hide systematic extinctions for:
P-lattice (none)
Operations
Move hidden to:
Move visible to:
Group2
Selection/Lattice-It
Indexed Wrong Color
1 1609 0
2 0 0
3 0 0
4 0 0
5 0 0
Tab filters on: I.
Peaks: visible - 1490, hidden - 119, selected - 0
X: 855 Y: 100

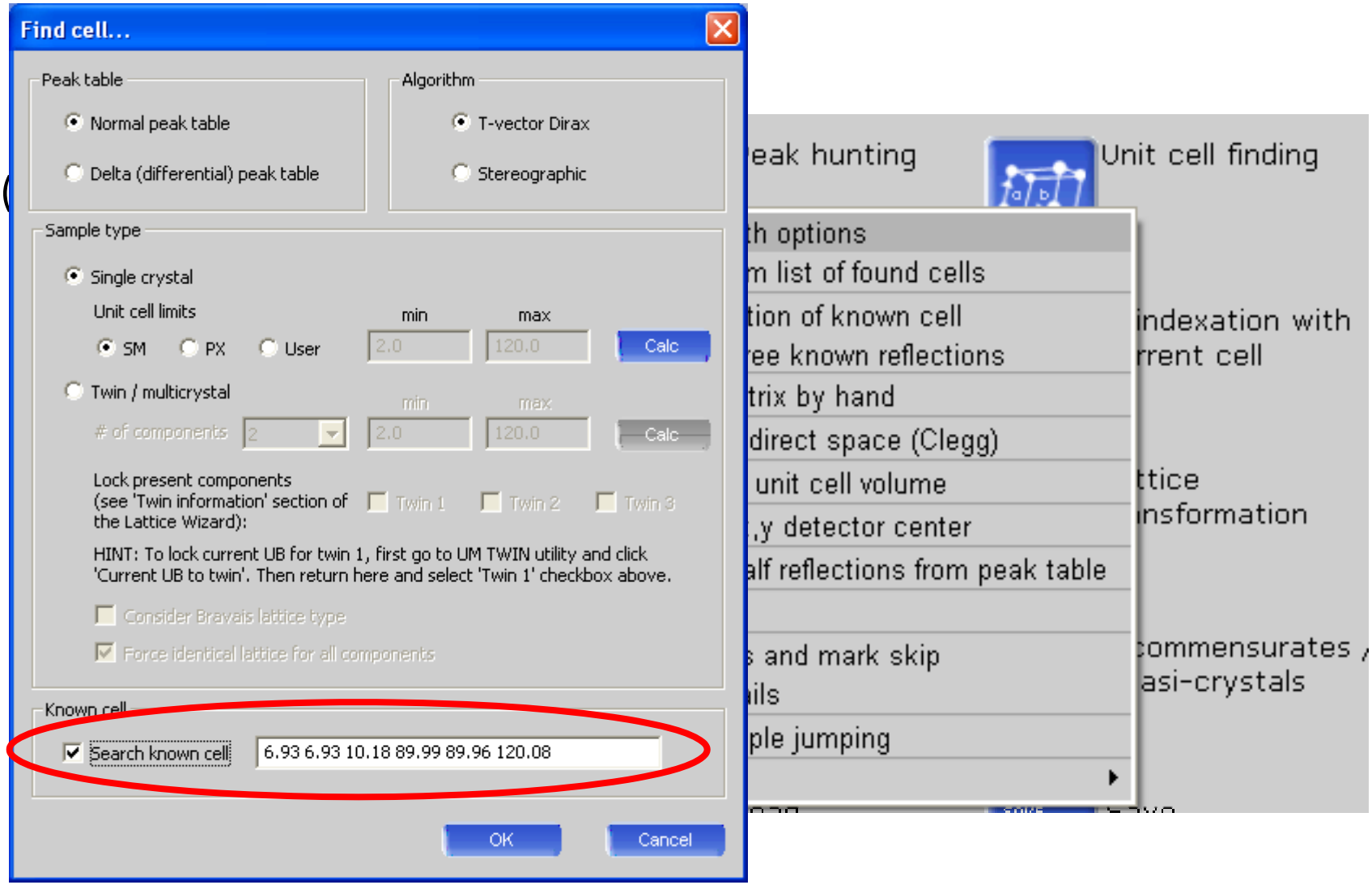
Unit cell

Now, how does it work in the software:

(3) Search for known cell



Unit cell


Find cell...
Peak table
Normal peak table
Delta (differential) peak table
Algorithm
T-vector Dirax
Stereographic
Sample type
Single crystal
Unit cell limits
min
max
SM
PX
User
2.0
120.0
Calc
Twin / multicrystal
# of components
Lock present components
(see 'Twin information' section of
the Lattice Wizard):
Twin 1
Twin 2
Twin 3
HINT: To lock current UB for twin 1, first go to UM TWIN utility and click
'Current UB to twin'. Then return here and select 'Twin 1' checkbox above.
Consider Bravais lattice type
Force identical lattice for all components
Known cell
Search known cell
6.93 6.93 10.18 89.99 89.96 120.08
OK
Cancel
eak hunting
Unit cell finding
th options
m list of found cells
tion of known cell
ee known reflections
trix by hand
direct space (Clegg)
unit cell volume
,y detector center
alf reflections from peak table
s and mark skip
ils
ple jumping
indexation with
rrent cell
ttice
nsformation
commensurates
asi-crystals

Unit cell

Good crystal, good centering, good data collection

→ *CrysAlisPro* usually finds UB

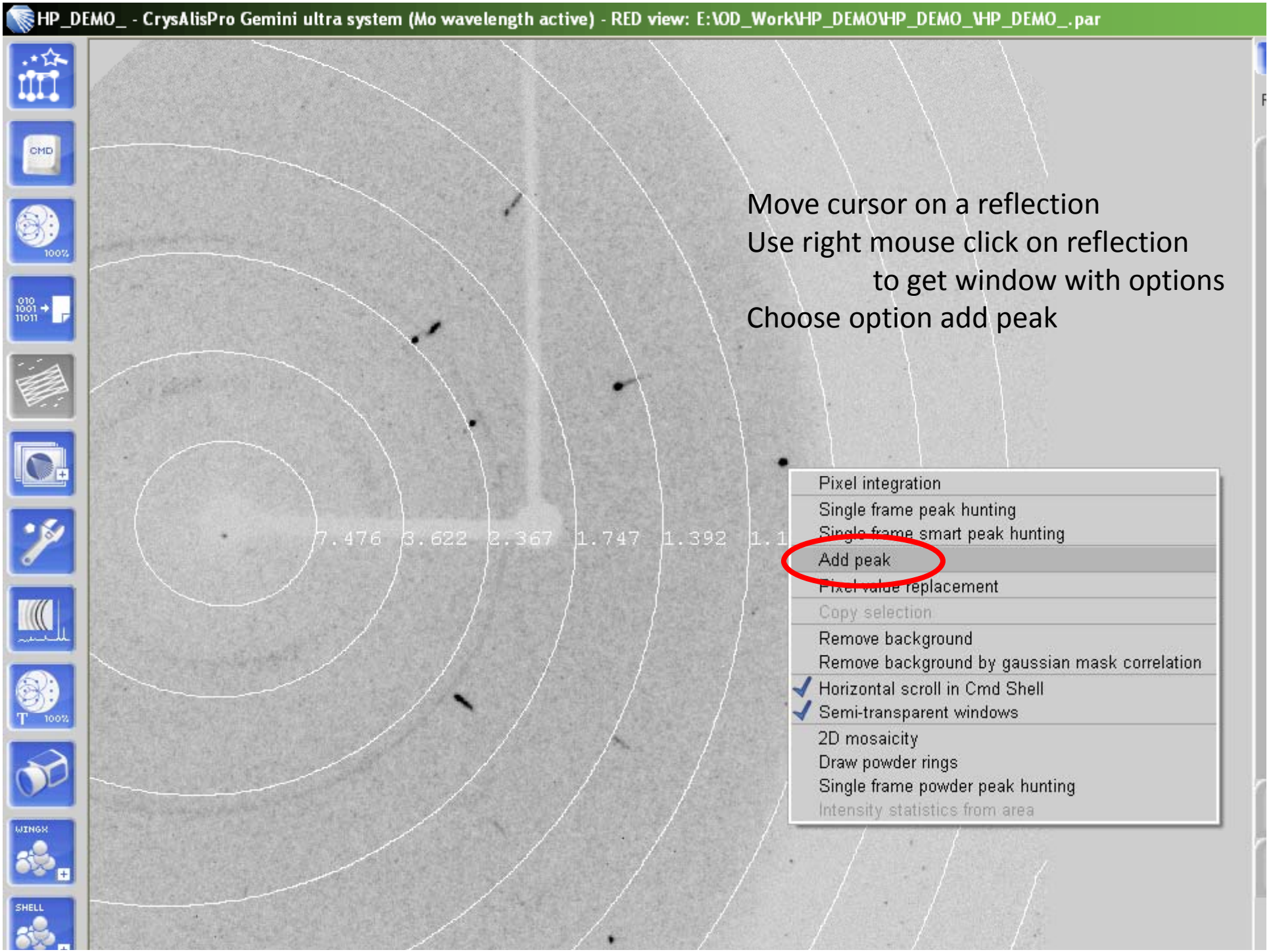
UB still not found:

Dataset very likely not good enough, nevertheless further tricks to find UB

(4) In reciprocal space viewer:

Delete every reflection, that you know is not from the sample you are interested in

(5) Delete reflection list and hand pick reflections


HP_DEMO_ - CrysAlisPro Gemini ultra system (Mo wavelength active) - RED view: E:\OD_Work\HP_DEMO\HP_DEMO_\HP_DEMO_.par
Move cursor on a reflection
Use right mouse click on reflection
to get window with options
Choose option add peak
7.476
3.622
2.367
1.747
1.392
Pixel integration
Single frame peak hunting
Single frame smart peak hunting
Add peak
Pixel value replacement
Copy selection
Remove background
Remove background by gaussian mask correlation
Horizontal scroll in Cmd Shell
Semi-transparent windows
2D mosaicity
Draw powder rings
Single frame powder peak hunting
Intensity statistics from area

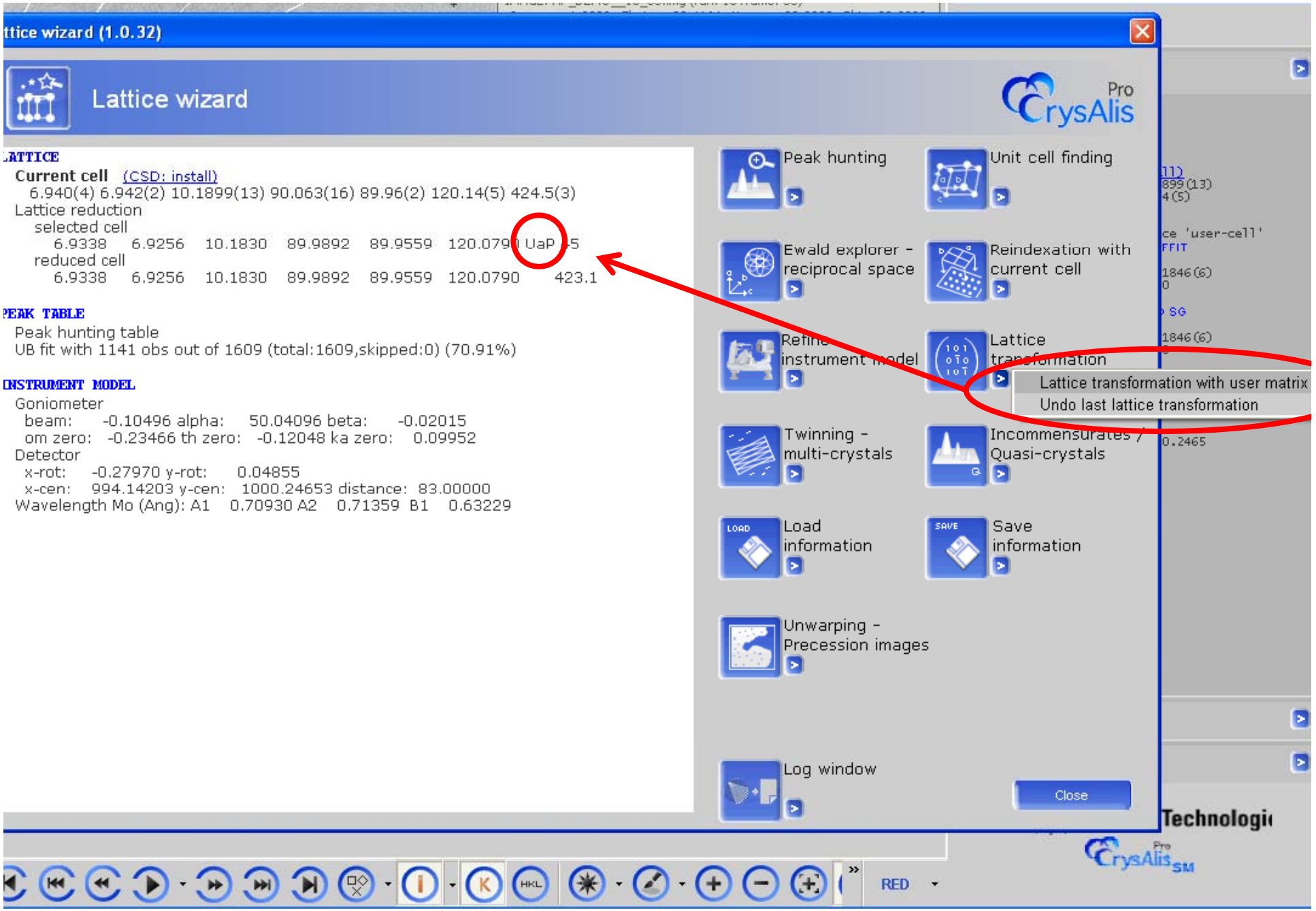
Unit cell

Cell should now be found

If problems occur: make sure this cell is used during data reduction

Transform cell with Unit-Transformation 100/010/001

→ *CrysAlisPro* uses **u**sed-defined UB


ttice wizard (1.0.32)
Lattice wizard
Pro CrysAlis
LATTICE
Current cell (CSD: install)
6.940(4) 6.942(2) 10.1899(13) 90.063(16) 89.96(2) 120.14(5) 424.5(3)
Lattice reduction
selected cell
6.9338 6.9256 10.1830 89.9892 89.9559 120.0790 UaP -5
reduced cell
6.9338 6.9256 10.1830 89.9892 89.9559 120.0790 423.1
PEAK TABLE
Peak hunting table
UB fit with 1141 obs out of 1609 (total:1609,skipped:0) (70.91%)
INSTRUMENT MODEL
Goniometer
beam: -0.10496 alpha: 50.04096 beta: -0.02015
om zero: -0.23466 th zero: -0.12048 ka zero: 0.09952
Detector
x-rot: -0.27970 y-rot: 0.04855
x-cen: 994.14203 y-cen: 1000.24653 distance: 83.00000
Wavelength Mo (Ang): A1 0.70930 A2 0.71359 B1 0.63229
Peak hunting
Unit cell finding
Ewald explorer - reciprocal space
Reindexation with current cell
Refine instrument model
Lattice transformation
Lattice transformation with user matrix
Undo last lattice transformation
Twinning - multi-crystals
Incommensurates / Quasi-crystals
LOAD
Load information
SAVE
Save information
Unwarping - Precession images
Log window
Close
RED

(1) Introduction to *CrysAlisPro*

(2) General remarks

(3) Centre DAC

(4) Data collection

(5) Pre-analysis by user

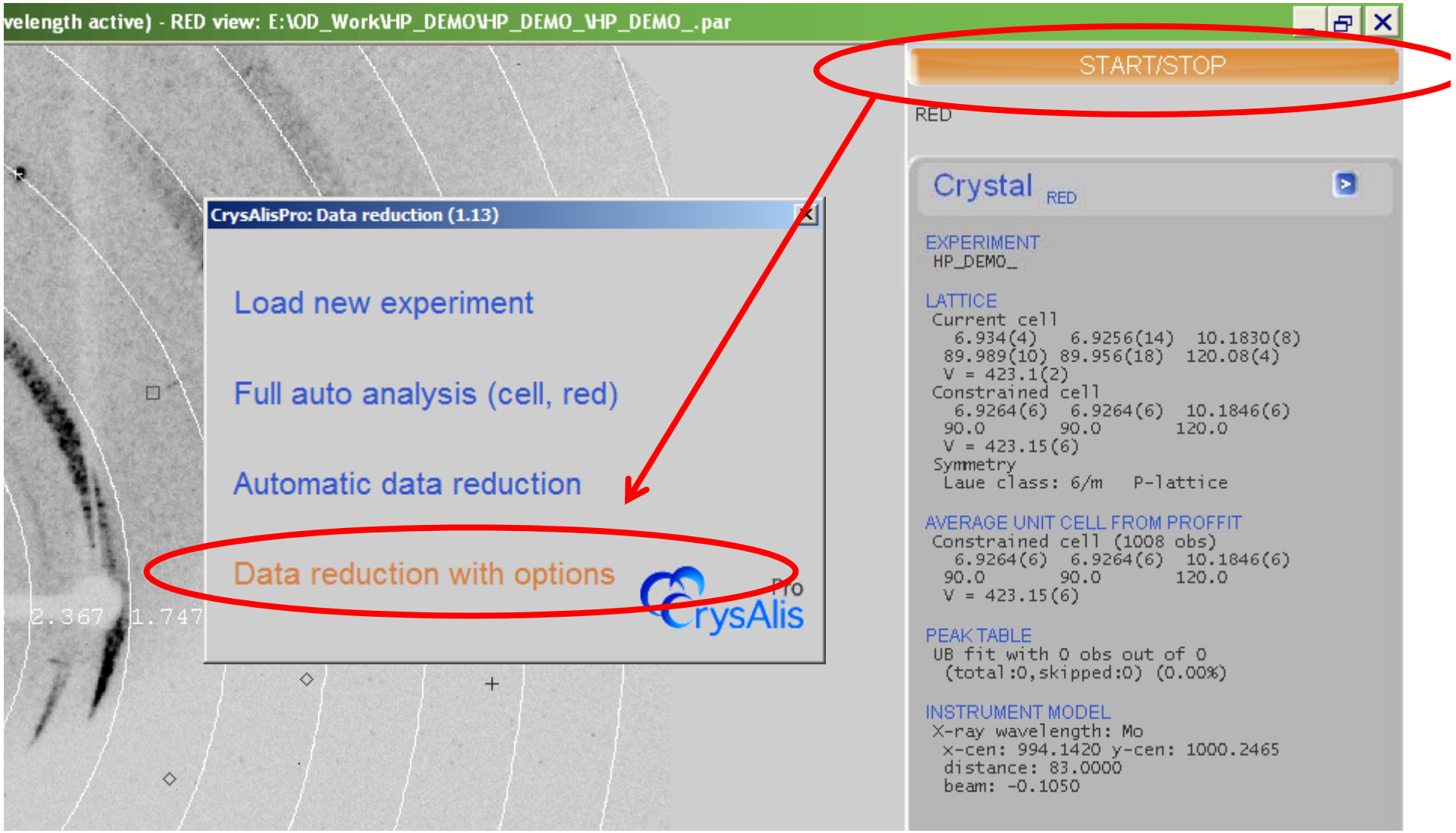
(6) Best possible data reduction for high pressure data

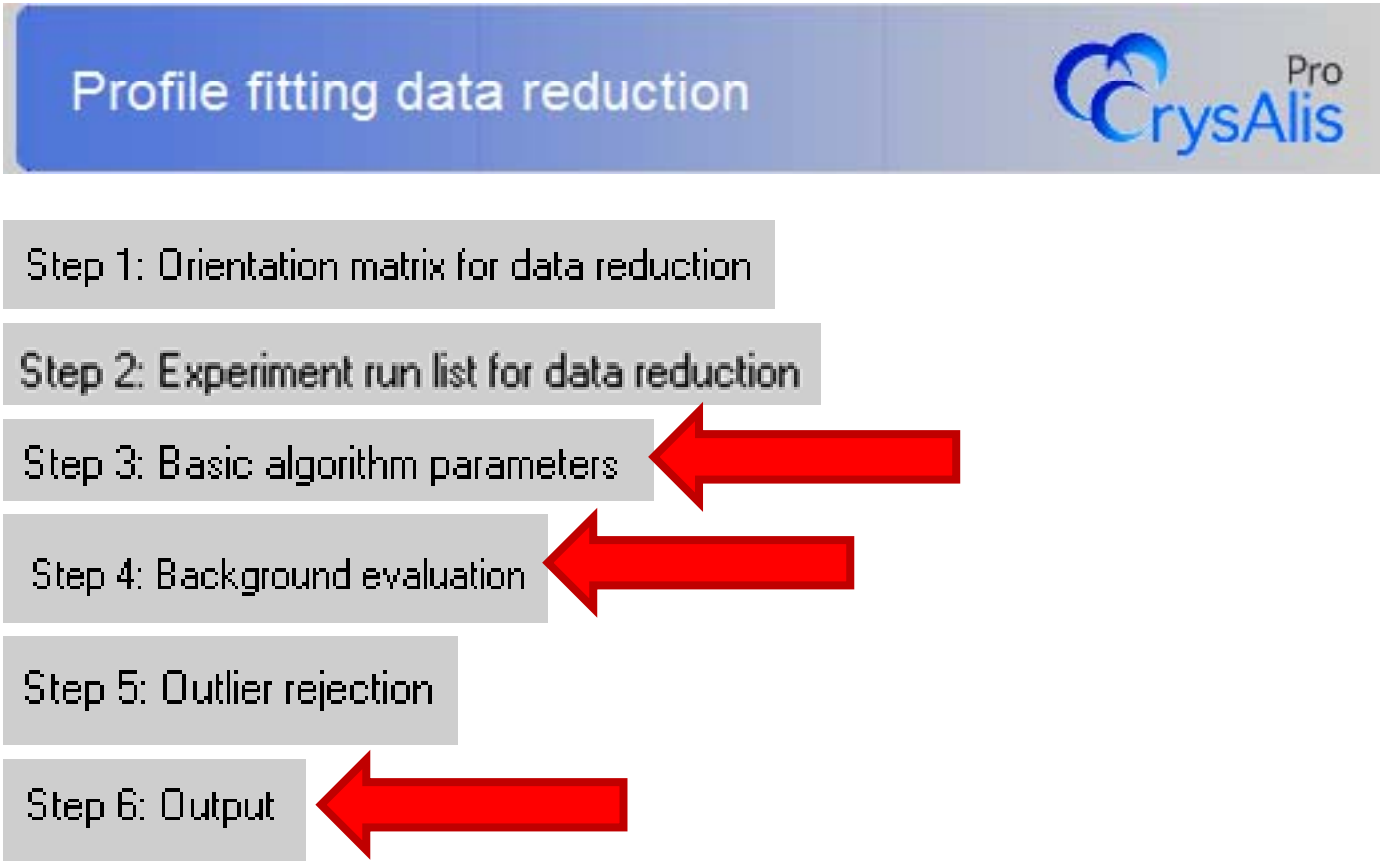
Get good unit cell

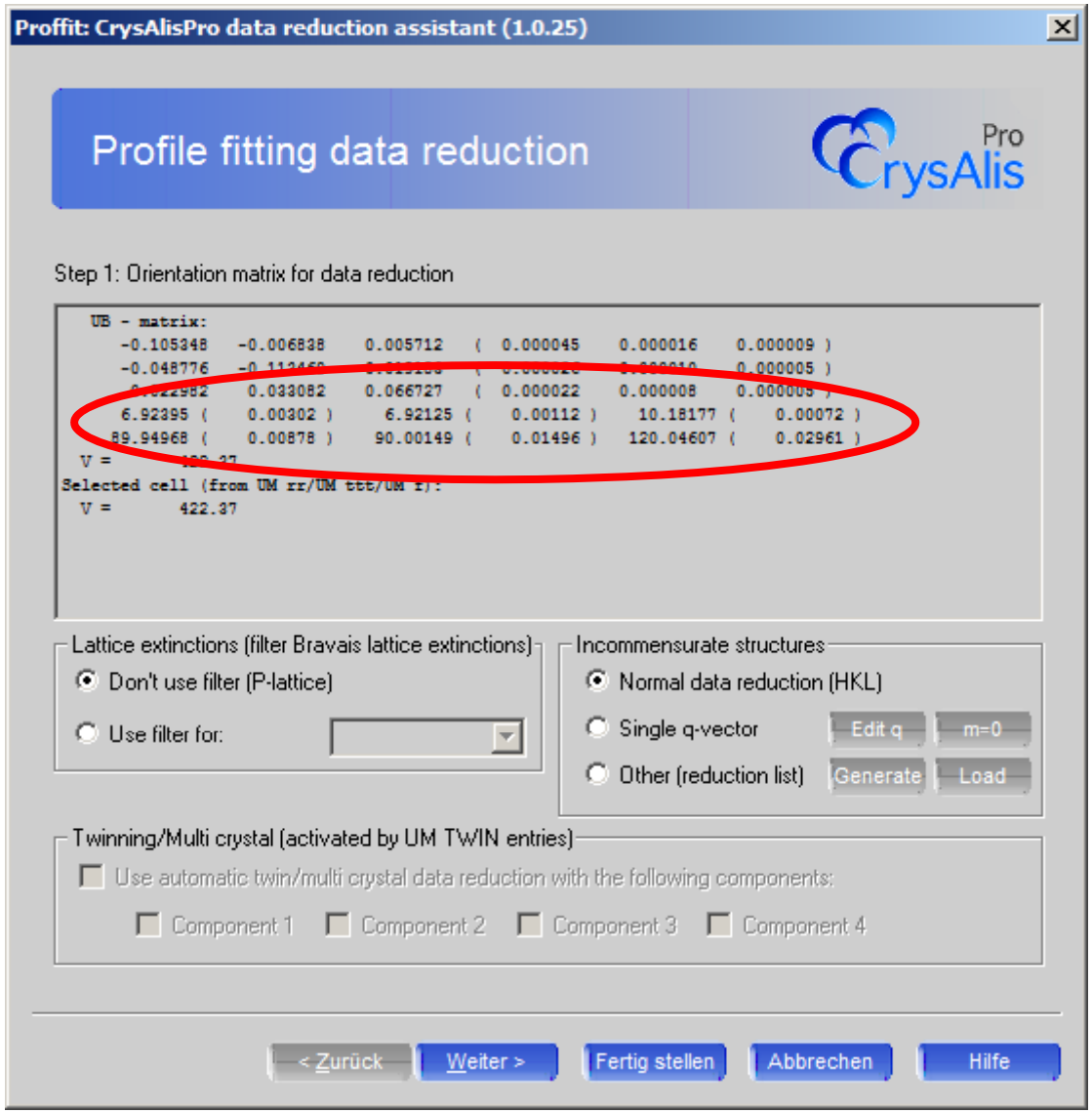
Data reduction

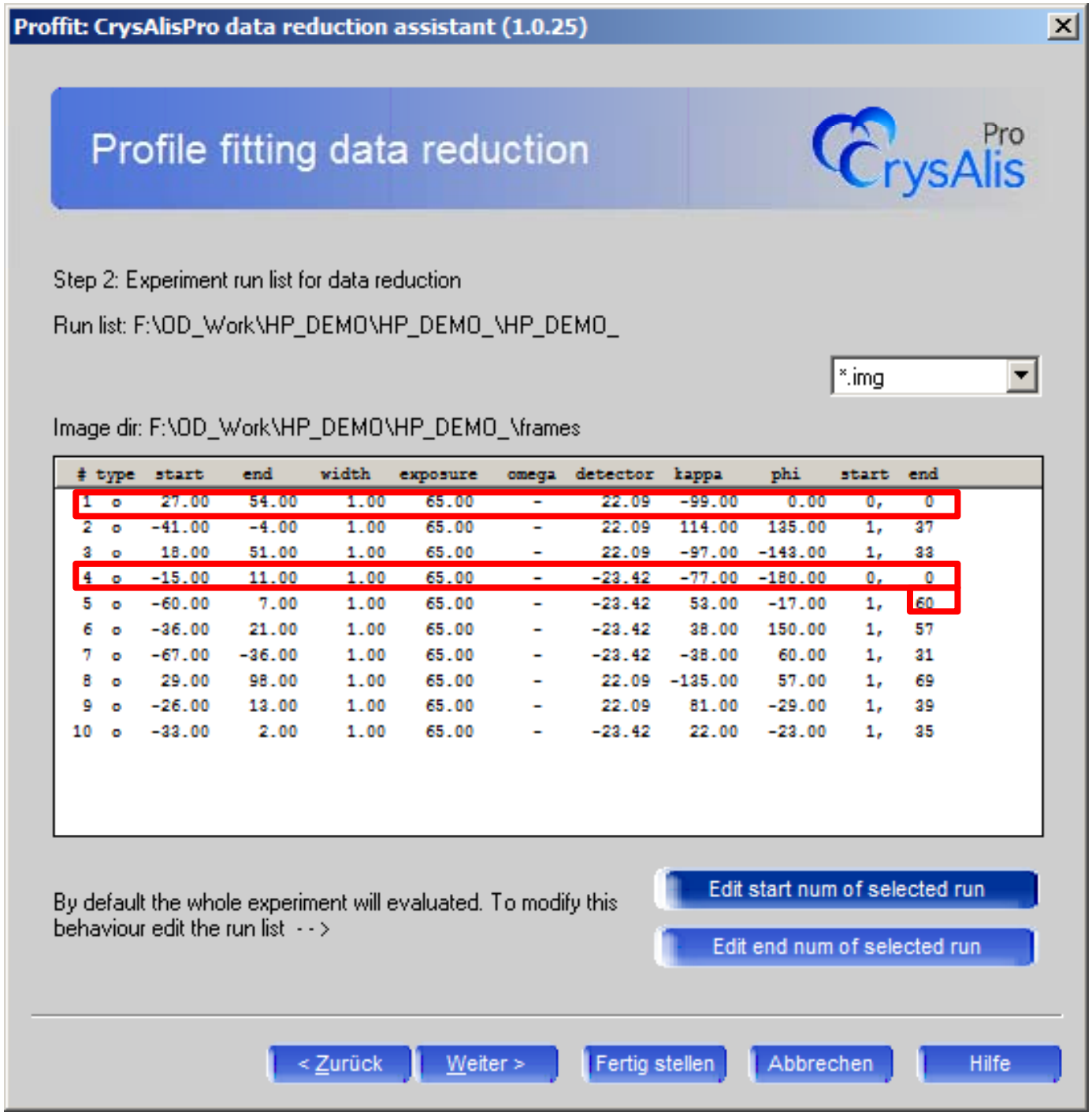
Absorption correction

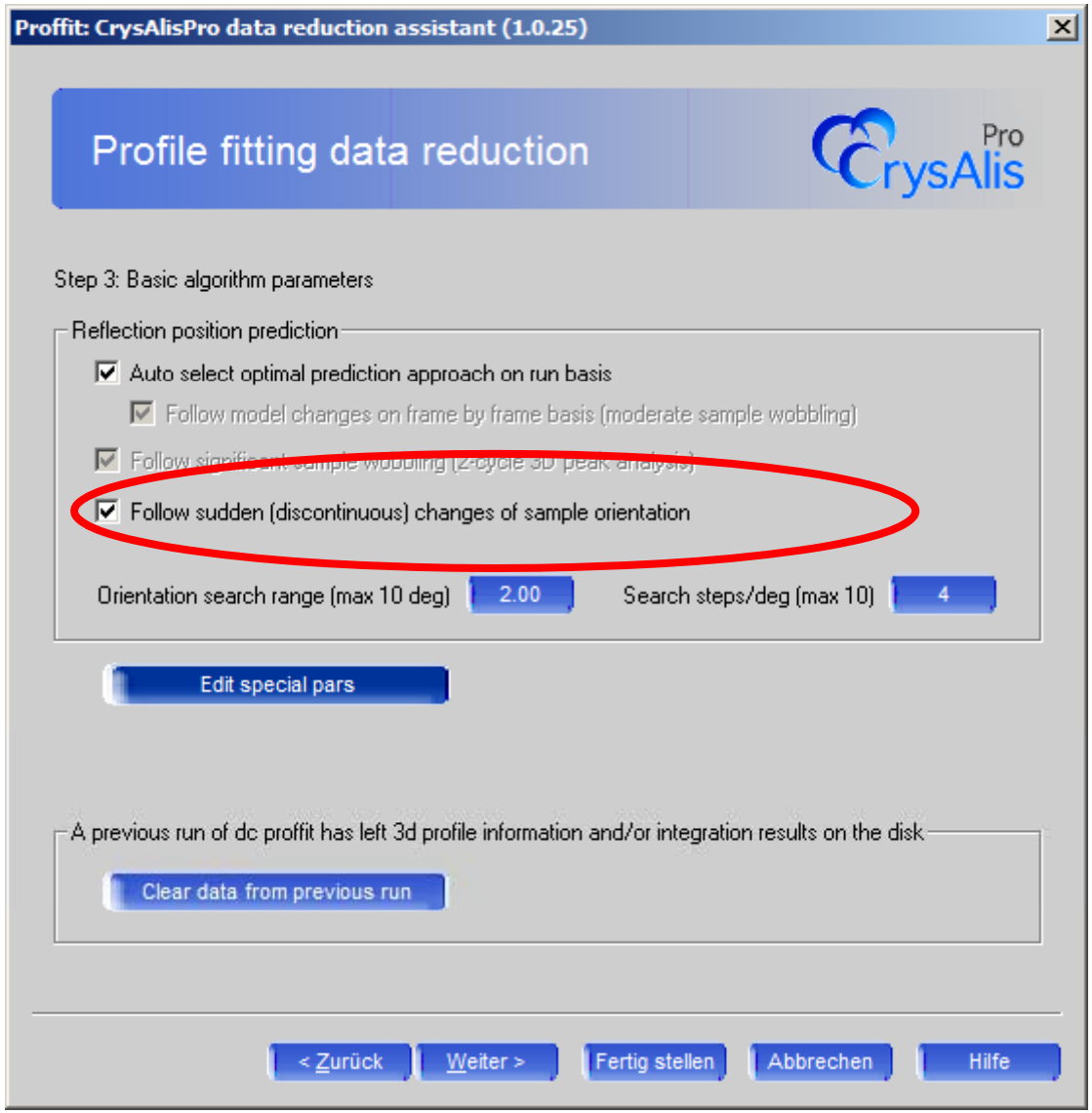
So now: best possible data reduction for high pressure data

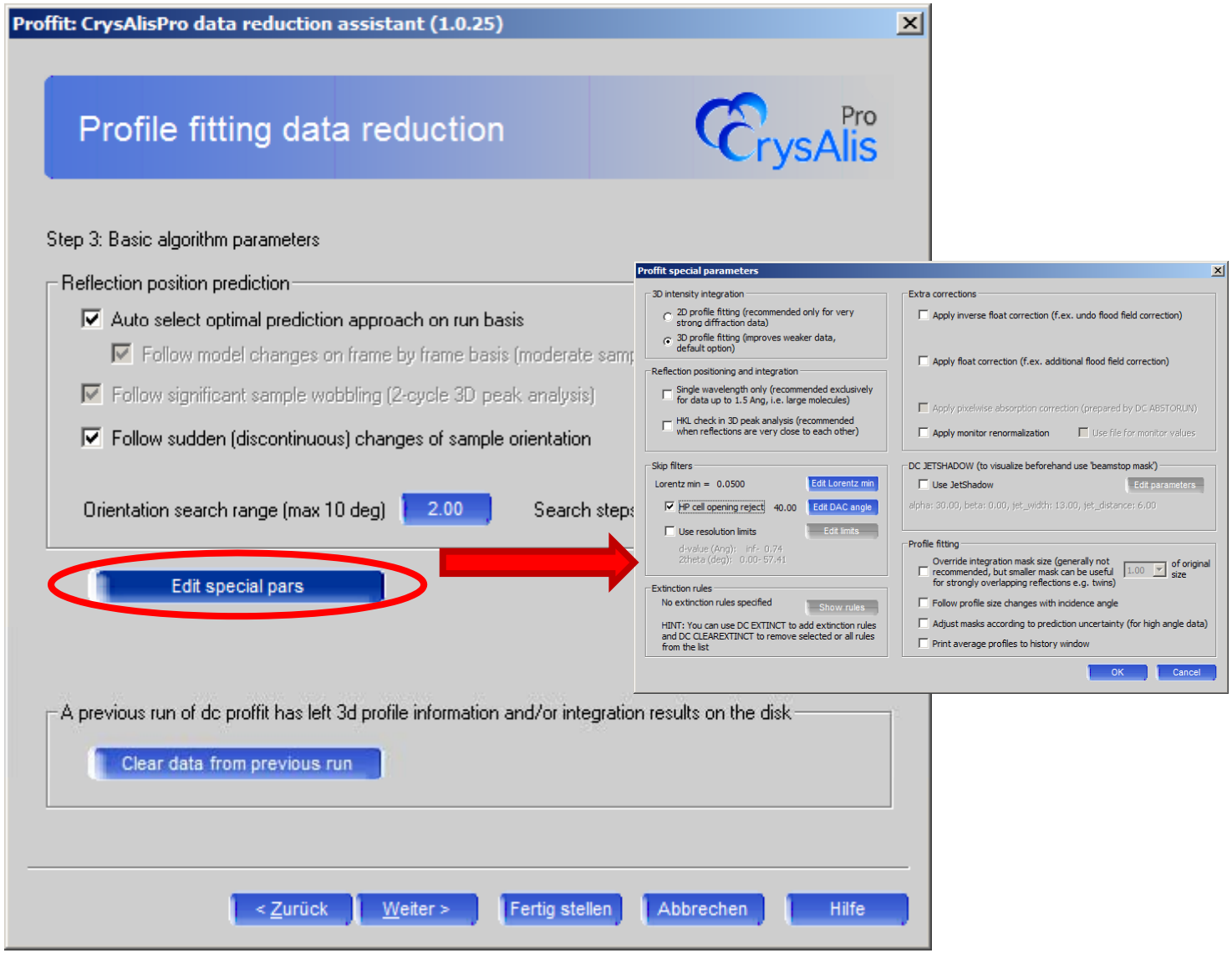


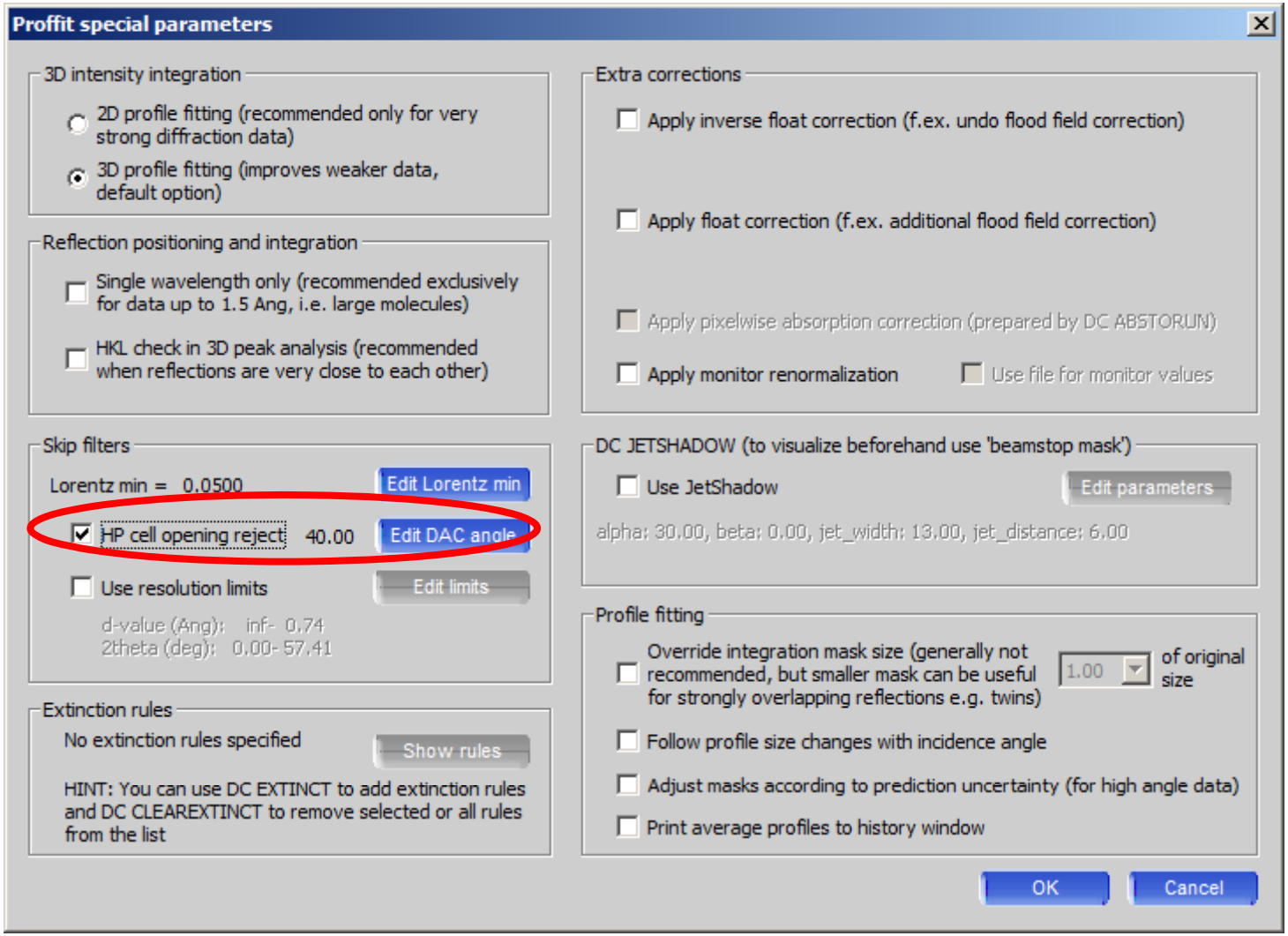

Profile fitting data reduction
Pro
CrysAlis
Step 1: Orientation matrix for data reduction
Step 2: Experiment run list for data reduction
Step 3: Basic algorithm parameters
Step 4: Background evaluation
Step 5: Outlier rejection
Step 6: Output

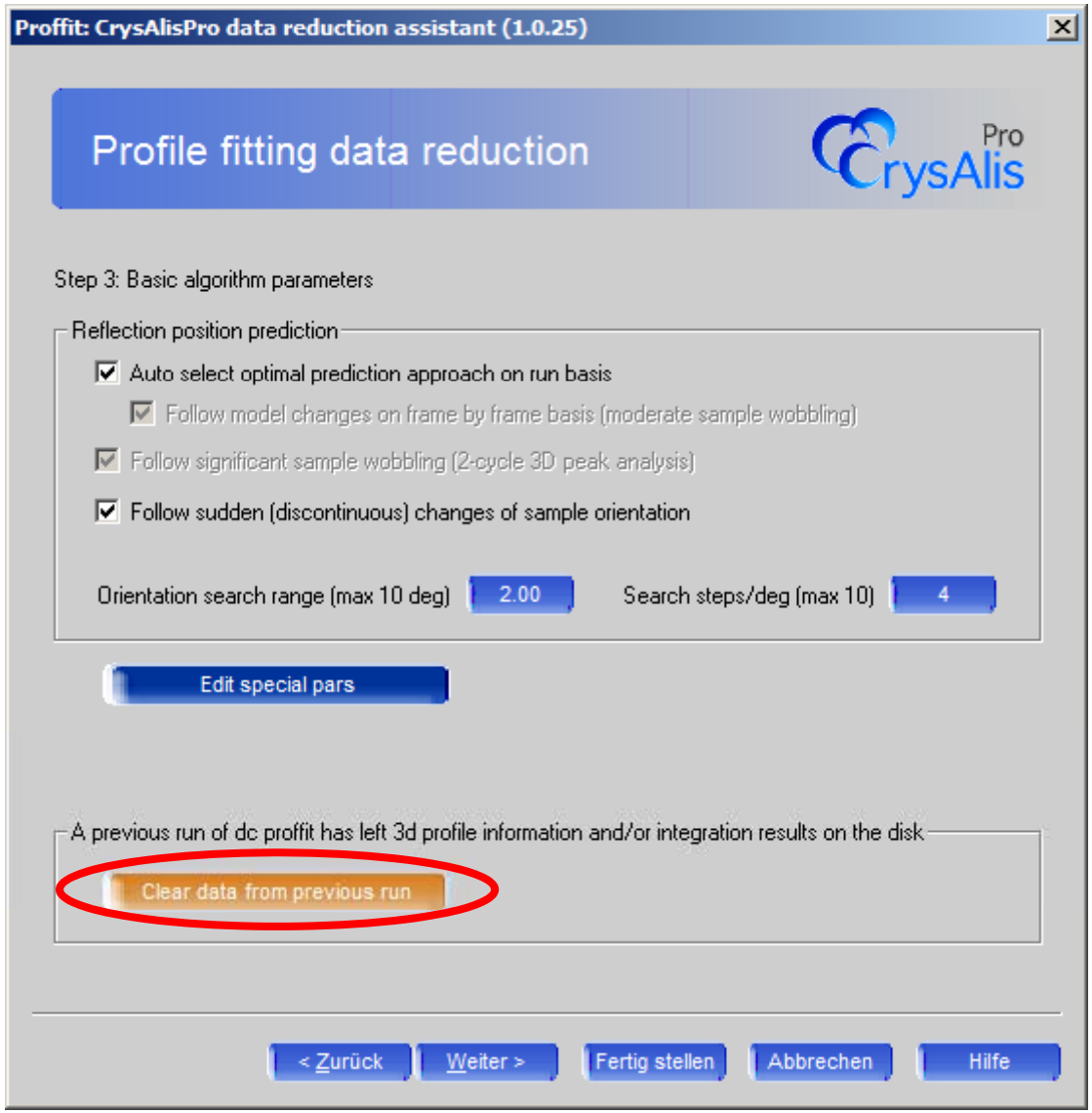

Proffit: CrysAlisPro data reduction assistant (1.0.25)
Profile fitting data reduction
CrysAlis Pro
Step 1: Orientation matrix for data reduction
UB - matrix:
-0.105348 -0.006838 0.005712 (0.000045 0.000016 0.000009)
-0.048776 (0.000005)
0.033082 0.066727 (0.000022 0.000008 0.000005)
6.92395 (0.00302) 6.92125 (0.00112) 10.18177 (0.00072)
89.94968 (0.00878) 90.00149 (0.01496) 120.04607 (0.02961)
Selected cell (from UM rr/UM ttt/UM t):
V = 422.37
Lattice extinctions (filter Bravais lattice extinctions)
Don't use filter (P-lattice)
Use filter for:
Incommensurate structures
Normal data reduction (HKL)
Single q-vector
Edit q
m=0
Other (reduction list)
Generate
Load
Twinning/Multi crystal (activated by UM TWIN entries)
Use automatic twin/multi crystal data reduction with the following components:
Component 1
Component 2
Component 3
Component 4
< Zurück
Weiter >
Fertig stellen
Abbrechen
Hilfe

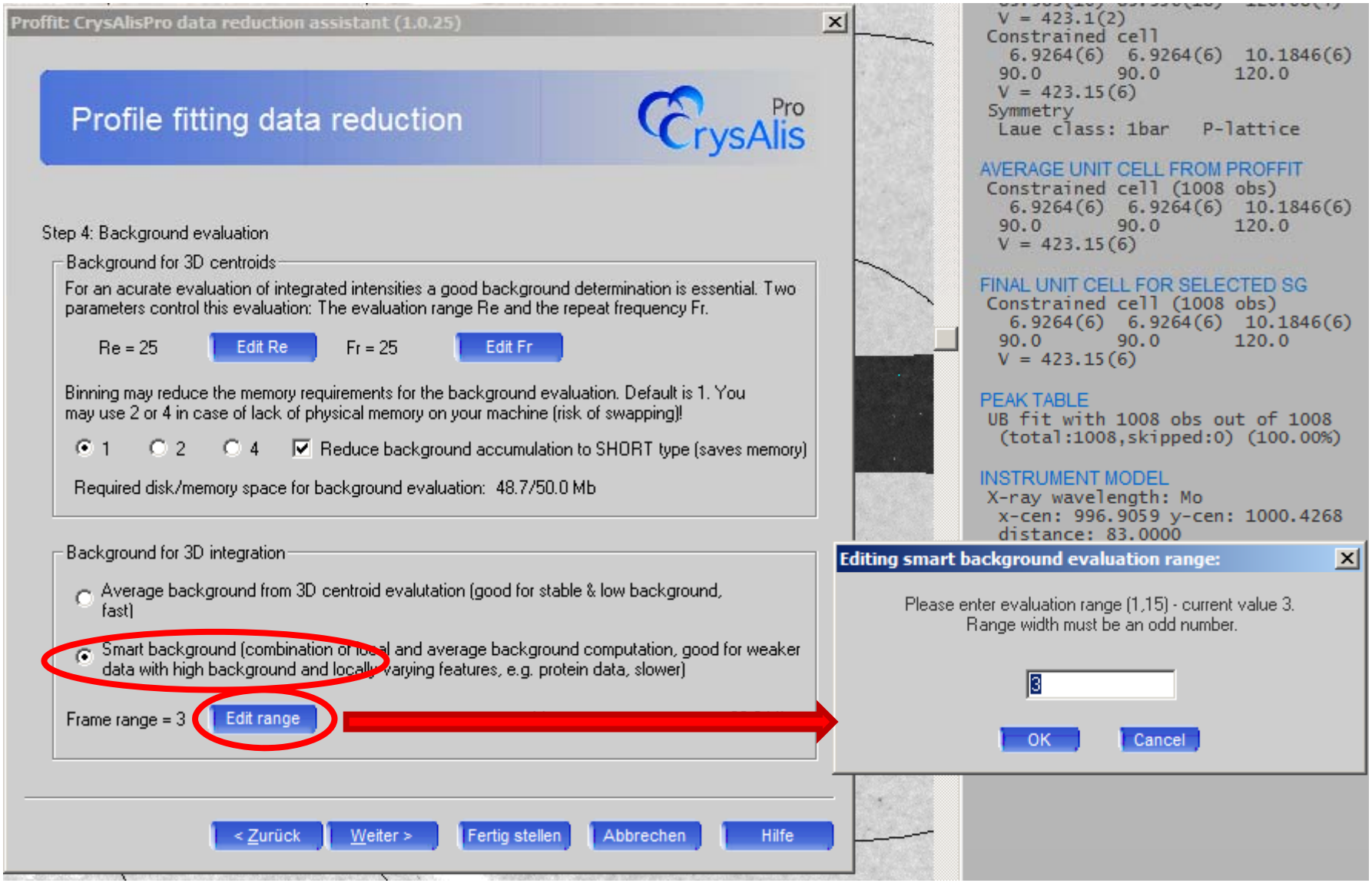

Proffit: CrysAlisPro data reduction assistant (1.0.25)
Profile fitting data reduction
CrysAlis Pro
Step 2: Experiment run list for data reduction
Run list: F:\OD_Work\HP_DEMO\HP_DEMO_\HP_DEMO_
*.img
Image dir: F:\OD_Work\HP_DEMO\HP_DEMO_\frames
type start end width exposure omega detector kappa phi start end
1 o 27.00 54.00 1.00 65.00 - 22.09 -99.00 0.00 0, 0
2 o -41.00 -4.00 1.00 65.00 - 22.09 114.00 135.00 1, 37
3 o 18.00 51.00 1.00 65.00 - 22.09 -97.00 -143.00 1, 33
4 o -15.00 11.00 1.00 65.00 - -23.42 -77.00 -180.00 0, 0
5 o -60.00 7.00 1.00 65.00 - -23.42 53.00 -17.00 1, 60
6 o -36.00 21.00 1.00 65.00 - -23.42 38.00 150.00 1, 57
7 o -67.00 -36.00 1.00 65.00 - -23.42 -38.00 60.00 1, 31
8 o 29.00 98.00 1.00 65.00 - 22.09 -135.00 57.00 1, 69
9 o -26.00 13.00 1.00 65.00 - 22.09 81.00 -29.00 1, 39
10 o -33.00 2.00 1.00 65.00 - -23.42 22.00 -23.00 1, 35
By default the whole experiment will evaluated. To modify this behaviour edit the run list - - >
Edit start num of selected run
Edit end num of selected run
< Zurück
Weiter >
Fertig stellen
Abbrechen
Hilfe

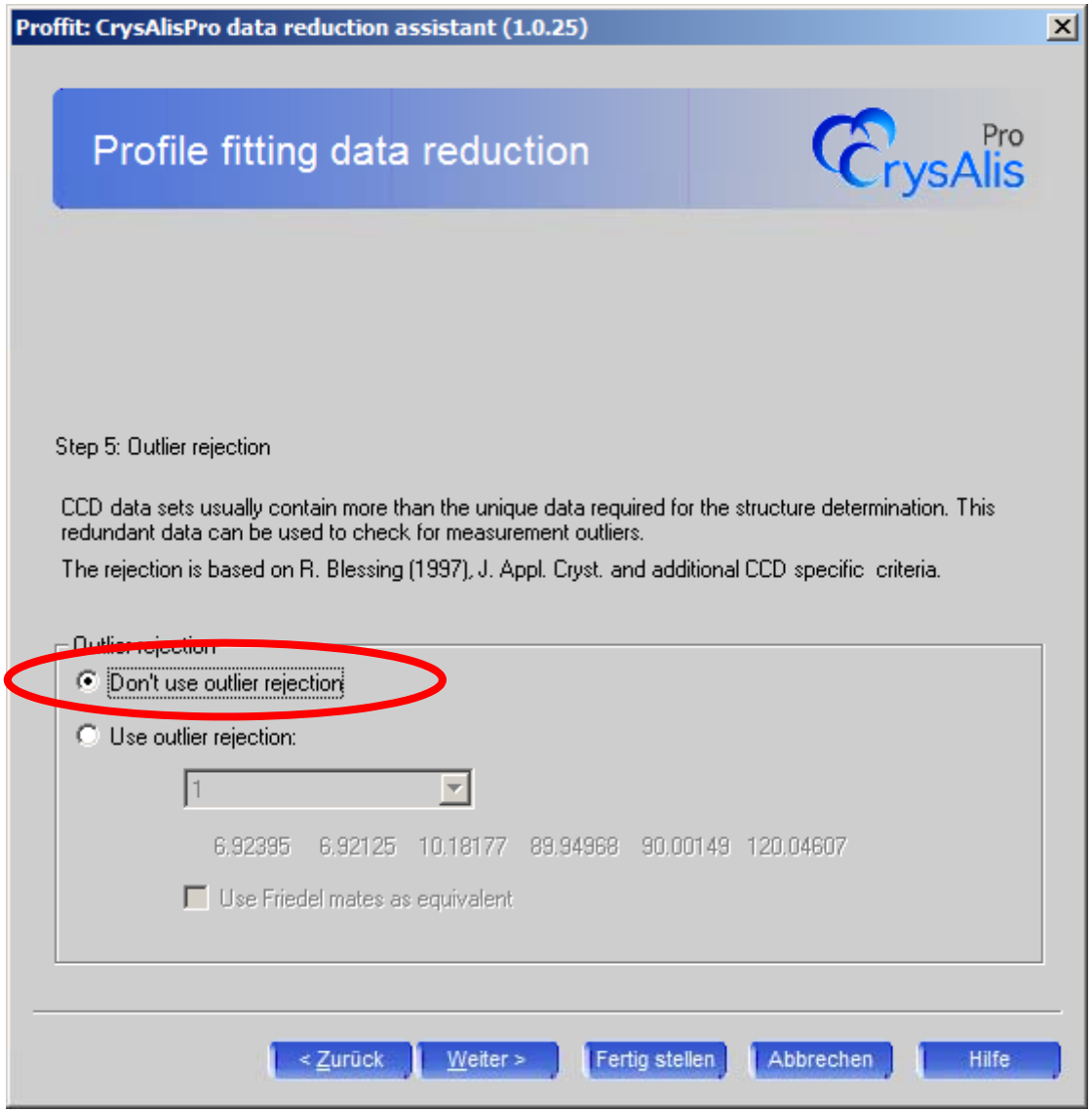

Proffit: CrysAlisPro data reduction assistant (1.0.25)
Profile fitting data reduction
Pro
CrysAlis
Step 3: Basic algorithm parameters
Reflection position prediction
Auto select optimal prediction approach on run basis
Follow model changes on frame by frame basis (moderate sample wobbling)
Follow sudden (discontinuous) changes of sample orientation
Orientation search range (max 10 deg)
2.00
Search steps/deg (max 10)
4
Edit special pars
A previous run of dc proffit has left 3d profile information and/or integration results on the disk
Clear data from previous run
< Zurück
Weiter >
Fertig stellen
Abbrechen
Hilfe

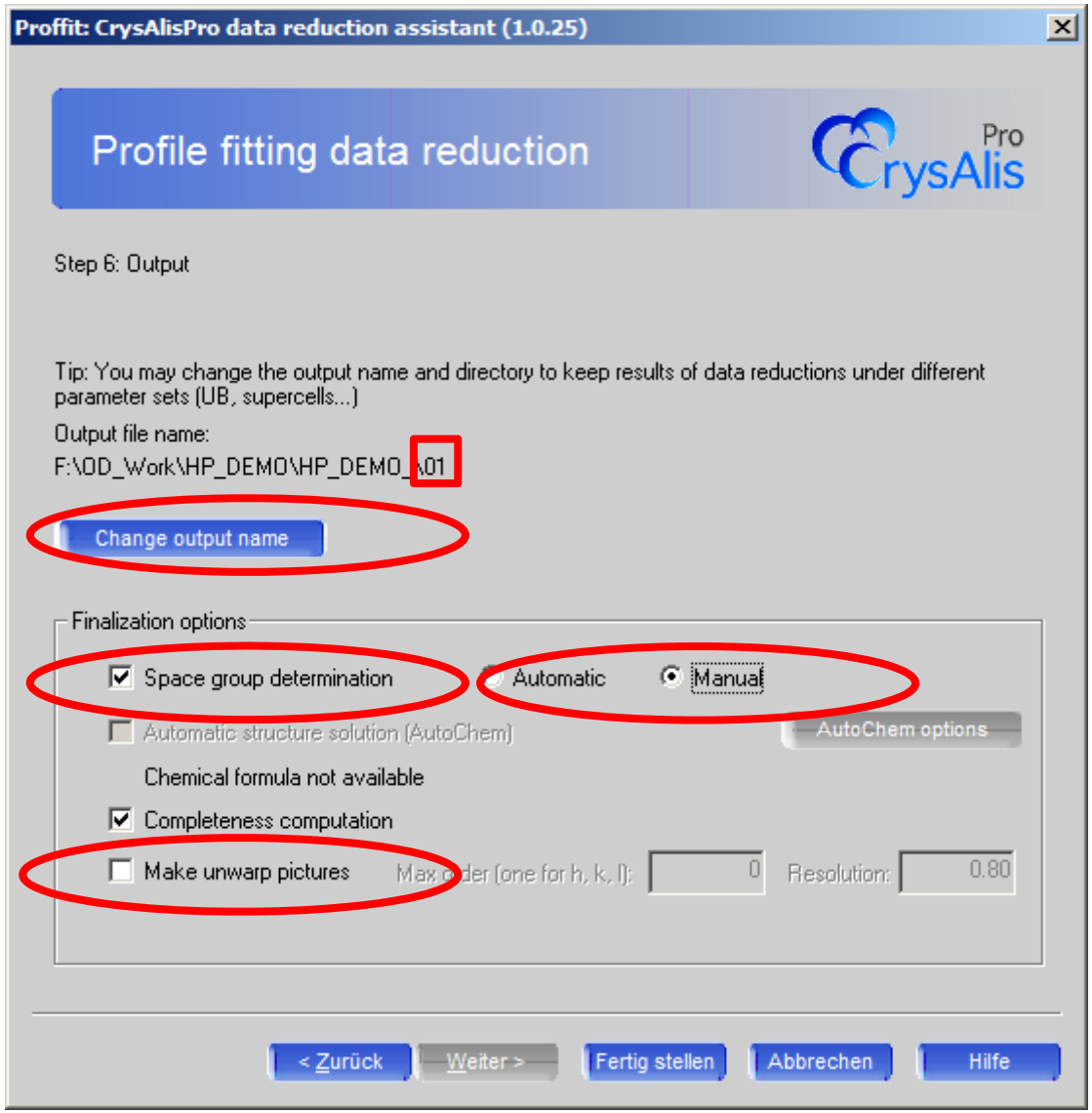

Proffit: CrysAlisPro data reduction assistant (1.0.25)
Profile fitting data reduction
Pro
CrysAlis
Step 3: Basic algorithm parameters
Reflection position prediction
Auto select optimal prediction approach on run basis
Follow model changes on frame by frame basis (moderate samp
Follow significant sample wobbling (2-cycle 3D peak analysis)
Follow sudden (discontinuous) changes of sample orientation
Orientation search range (max 10 deg)
2.00
Search steps
Edit special pars
A previous run of dc proffit has left 3d profile information and/or integration results on the disk
Clear data from previous run
< Zurück
Weiter >
Fertig stellen
Abbrechen
Hilfe
Proffit special parameters
3D intensity integration
2D profile fitting (recommended only for very strong diffraction data)
3D profile fitting (improves weaker data, default option)
Reflection positioning and integration
Single wavelength only (recommended exclusively for data up to 1.5 Ang, i.e. large molecules)
HKL check in 3D peak analysis (recommended when reflections are very close to each other)
Skip filters
Lorentz min = 0.0500
Edit Lorentz min
HP cell opening reject
40.00
Edit DAC angle
Use resolution limits
Edit limits
d-value (Ang): inf- 0.74
2theta (deg): 0.00- 57.41
Extinction rules
No extinction rules specified
Show rules
HINT: You can use DC EXTINCT to add extinction rules and DC CLEAREXTINCT to remove selected or all rules from the list
Extra corrections
Apply inverse float correction (f.ex. undo flood field correction)
Apply float correction (f.ex. additional flood field correction)
Apply pixelwise absorption correction (prepared by DC ABSTORUN)
Apply monitor renormalization
Use file for monitor values
DC JETSHADOW (to visualize beforehand use 'beamstop mask')
Use JetShadow
Edit parameters
alpha: 30.00, beta: 0.00, jet_width: 13.00, jet_distance: 6.00
Profile fitting
Override integration mask size (generally not recommended, but smaller mask can be useful for strongly overlapping reflections e.g. twins)
1.00
of original size
Follow profile size changes with incidence angle
Adjust masks according to prediction uncertainty (for high angle data)
Print average profiles to history window
OK
Cancel


Proffit special parameters
3D intensity integration
2D profile fitting (recommended only for very strong diffraction data)
3D profile fitting (improves weaker data, default option)
Reflection positioning and integration
Single wavelength only (recommended exclusively for data up to 1.5 Ang, i.e. large molecules)
HKL check in 3D peak analysis (recommended when reflections are very close to each other)
Skip filters
Lorentz min = 0.0500
Edit Lorentz min
HP cell opening reject 40.00
Edit DAC angle
Use resolution limits
Edit limits
d-value (Ang): inf- 0.74
2theta (deg): 0.00- 57.41
Extinction rules
No extinction rules specified
Show rules
HINT: You can use DC EXTINCT to add extinction rules and DC CLEAREXTINCT to remove selected or all rules from the list
Extra corrections
Apply inverse float correction (f.ex. undo flood field correction)
Apply float correction (f.ex. additional flood field correction)
Apply pixelwise absorption correction (prepared by DC ABSTORUN)
Apply monitor renormalization
Use file for monitor values
DC JETSHADOW (to visualize beforehand use 'beamstop mask')
Use JetShadow
Edit parameters
alpha: 30.00, beta: 0.00, jet_width: 13.00, jet_distance: 6.00
Profile fitting
Override integration mask size (generally not recommended, but smaller mask can be useful for strongly overlapping reflections e.g. twins)
1.00
of original size
Follow profile size changes with incidence angle
Adjust masks according to prediction uncertainty (for high angle data)
Print average profiles to history window
OK
Cancel


Proffit: CrysAlisPro data reduction assistant (1.0.25)
Profile fitting data reduction
Pro
CrysAlis
Step 3: Basic algorithm parameters
Reflection position prediction
Auto select optimal prediction approach on run basis
Follow model changes on frame by frame basis (moderate sample wobbling)
Follow significant sample wobbling (2-cycle 3D peak analysis)
Follow sudden (discontinuous) changes of sample orientation
Orientation search range (max 10 deg)
2.00
Search steps/deg (max 10)
4
Edit special pars
A previous run of dc proffit has left 3d profile information and/or integration results on the disk
Clear data from previous run
< Zurück
Weiter >
Fertig stellen
Abbrechen
Hilfe


Proffit: CrysAlisPro data reduction assistant (1.0.25)
Profile fitting data reduction
Pro CrysAlis
Step 4: Background evaluation
Background for 3D centroids
For an acurate evaluation of integrated intensities a good background determination is essential. Two parameters control this evaluation: The evaluation range Re and the repeat frequency Fr.
Re = 25
Edit Re
Fr = 25
Edit Fr
Binning may reduce the memory requirements for the background evaluation. Default is 1. You may use 2 or 4 in case of lack of physical memory on your machine (risk of swapping)!
1
2
4
Reduce background accumulation to SHORT type (saves memory)
Required disk/memory space for background evaluation: 48.7/50.0 Mb
Background for 3D integration
Average background from 3D centroid evalutation (good for stable & low background, fast)
Smart background (combination of local and average background computation, good for weaker data with high background and locally varying features, e.g. protein data, slower)
Frame range = 3
Edit range
< Zurück
Weiter >
Fertig stellen
Abbrechen
Hilfe
V = 423.1(2)
Constrained cell
6.9264(6) 6.9264(6) 10.1846(6)
90.0 90.0 120.0
V = 423.15(6)
Symmetry
Laue class: 1bar P-lattice
AVERAGE UNIT CELL FROM PROFFIT
Constrained cell (1008 obs)
6.9264(6) 6.9264(6) 10.1846(6)
90.0 90.0 120.0
V = 423.15(6)
FINAL UNIT CELL FOR SELECTED SG
Constrained cell (1008 obs)
6.9264(6) 6.9264(6) 10.1846(6)
90.0 90.0 120.0
V = 423.15(6)
PEAK TABLE
UB fit with 1008 obs out of 1008
(total:1008,skipped:0) (100.00%)
INSTRUMENT MODEL
X-ray wavelength: Mo
x-cen: 996.9059 y-cen: 1000.4268
distance: 83.0000
Editing smart background evaluation range:
Please enter evaluation range (1,15) - current value 3.
Range width must be an odd number.
3
OK
Cancel


Proffit: CrysAlisPro data reduction assistant (1.0.25)
Profile fitting data reduction
Pro
CrysAlis
Step 5: Outlier rejection
CCD data sets usually contain more than the unique data required for the structure determination. This redundant data can be used to check for measurement outliers.
The rejection is based on R. Blessing (1997), J. Appl. Cryst. and additional CCD specific criteria.
Outlier rejection
Don't use outlier rejection
Use outlier rejection:
1
6.92395 6.92125 10.18177 89.94968 90.00149 120.04607
Use Friedel mates as equivalent
< Zurück
Weiter >
Fertig stellen
Abbrechen
Hilfe


Proffit: CrysAlisPro data reduction assistant (1.0.25)
Profile fitting data reduction
Pro
CrysAlis
Step 6: Output
Tip: You may change the output name and directory to keep results of data reductions under different parameter sets (UB, supercells...)
Output file name:
F:\OD_Work\HP_DEMO\HP_DEMO_\01
Change output name
Finalization options
Space group determination
Automatic
Manual
Automatic structure solution (AutoChem)
AutoChem options
Chemical formula not available
Completeness computation
Make unwarp pictures
Max order (one for h, k, l):
0
Resolution:
0.80
< Zurück
Weiter >
Fertig stellen
Abbrechen
Hilfe

Dc rrp

Inspect data collection and reduction results

CrysAlis Pro

Data reduction file contents | Data reduction output | Red graphs | Data collection output | Devices log

1.27-1.11	238	238	29	8.2	4134.71	9.62	0.051	0.063	0.067
1.11-1.03	204	200	29	6.9	2870.75	7.44	0.100	0.112	0.093
1.03-0.95	207	207	29	7.1	3588.85	7.80	0.064	0.077	0.090
0.95-0.90	160	160	29	5.5	1760.28	4.97	0.086	0.103	0.137
0.90-0.85	153	153	29	5.3	1900.35	4.77	0.080	0.091	0.142
0.85-0.80	146	146	29	5.0	2089.86	4.95	0.090	0.100	0.147
0.80-0.76	72	72	33	2.2	2164.78	4.80	0.090	0.101	0.149
inf-0.76	1954	1948	294	6.6	4568.93	11.20	0.053	0.065	0.062
inf-0.80	1895	1889	266	7.1	4638.78	11.39	0.052	0.064	0.061

Statistics vs resolution (taking redundancy into account) - point group symmetry: P6/m

resolution(A)	# kept	# theory	# unique	% complete	average redundancy	mean F2	mean F2/sig(F2)	Rint	RsigmaB
inf-1.93	267	29	29	100.0	9.2	7801.31	70.75	0.032	0.010
1.89-1.48	259	29	29	100.0	8.9	8523.08	63.74	0.044	0.015
1.45-1.27	246	31	29	93.5	8.5	5184.41	39.92	0.047	0.019
1.27-1.11	238	34	29	85.3	8.2	4134.71	31.74	0.051	0.027
1.11-1.03	200	31	29	93.5	6.9	2870.75	21.31	0.100	0.040
1.03-0.95	207	39	29	74.4	7.1	3588.85	23.12	0.064	0.038
0.95-0.90	160	35	29	82.9	5.5	1760.28	12.45	0.086	0.063
0.90-0.85	153	41	29	70.7	5.3	1900.35	11.41	0.080	0.065
0.85-0.80	146	44	29	65.9	5.0	2089.86	11.89	0.090	0.069
0.80-0.76	72	58	33	56.9	2.2	2164.78	8.78	0.090	0.095
inf-0.76	1948	371	294	79.2	6.6	4568.93	34.87	0.053	0.030
inf-0.80	1889	318	266	83.6	7.1	4638.78	35.68	0.052	0.028

Data reduction ended at Wed Aug 01 12:00:23 2012

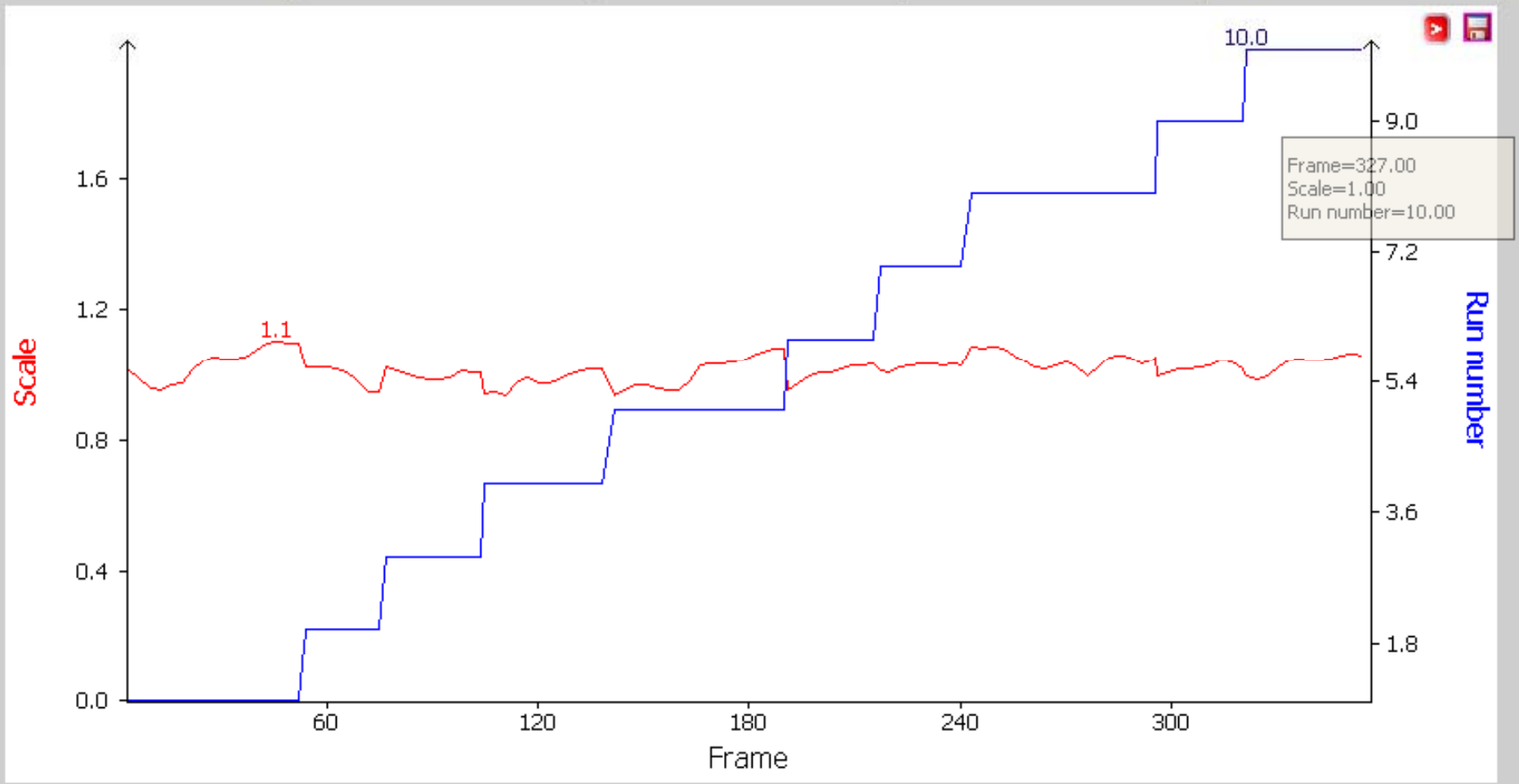
Refinalize | Abs display | 03 | OK

Dc rrp

Inspect data collection and reduction results

CrysAlis Pro

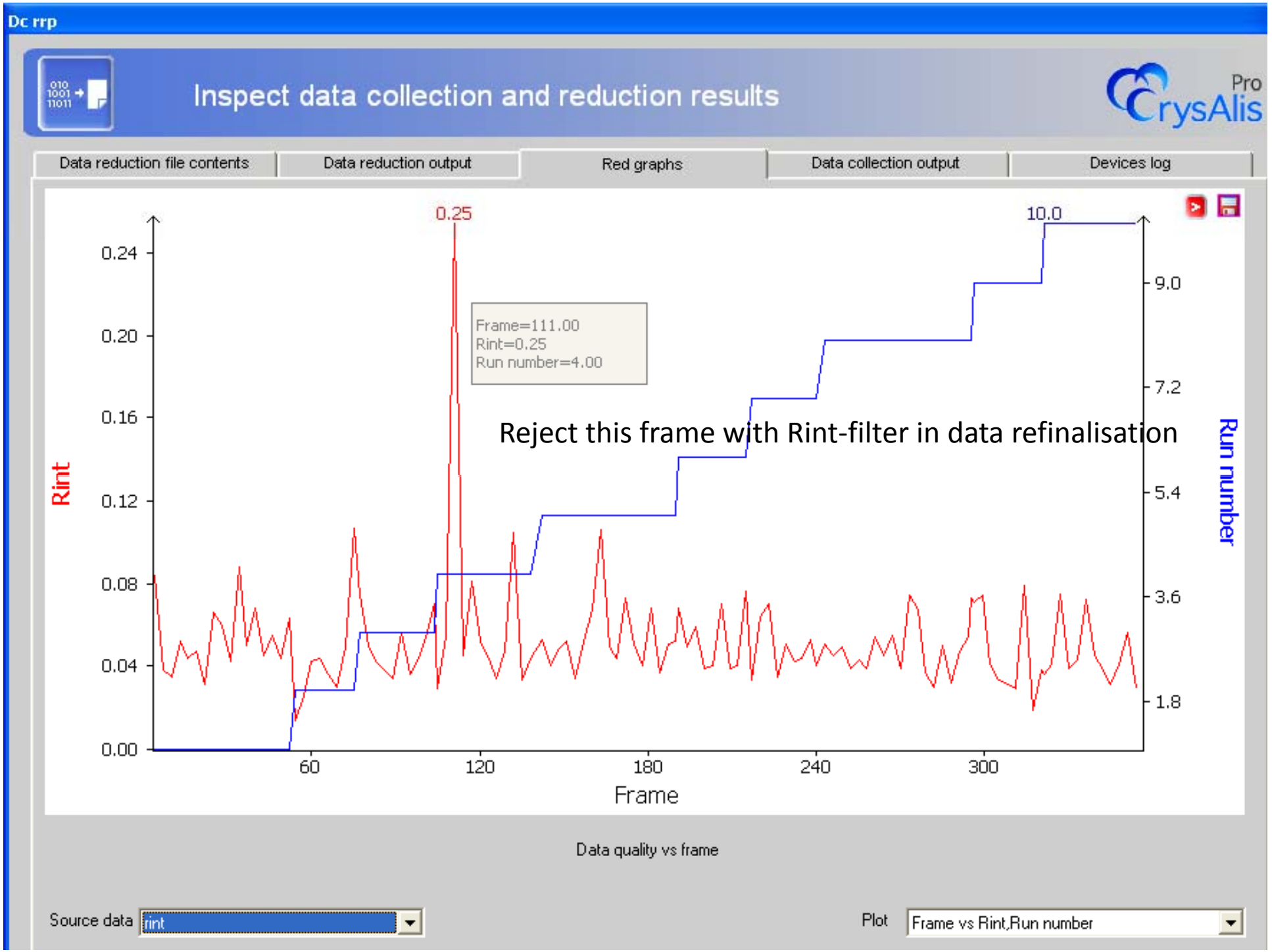
Data reduction file contents | Data reduction output | Red graphs | Data collection output | Devices log



Results of scaling correction done by scale3 abspack

Source data: absscale

Plot: Frame vs Scale,Run number


Dc rrp
Inspect data collection and reduction results
Pro
CrysAlis
Data reduction file contents
Data reduction output
Red graphs
Data collection output
Devices log
0.25
10.0
Frame=111.00
Rint=0.25
Run number=4.00
Reject this frame with Rint-filter in data refinalisation
Rint
Run number
Frame
0.24
0.20
0.16
0.12
0.08
0.04
0.00
9.0
7.2
5.4
3.6
1.8
60
120
180
240
300
Data quality vs frame
Source data
rint
Plot
Frame vs Rint,Run number

Dc rrp

Inspect data collection and reduction results

CrysAlis Pro

Data reduction file contents | Data reduction output | Red graphs | Data collection output | Devices log

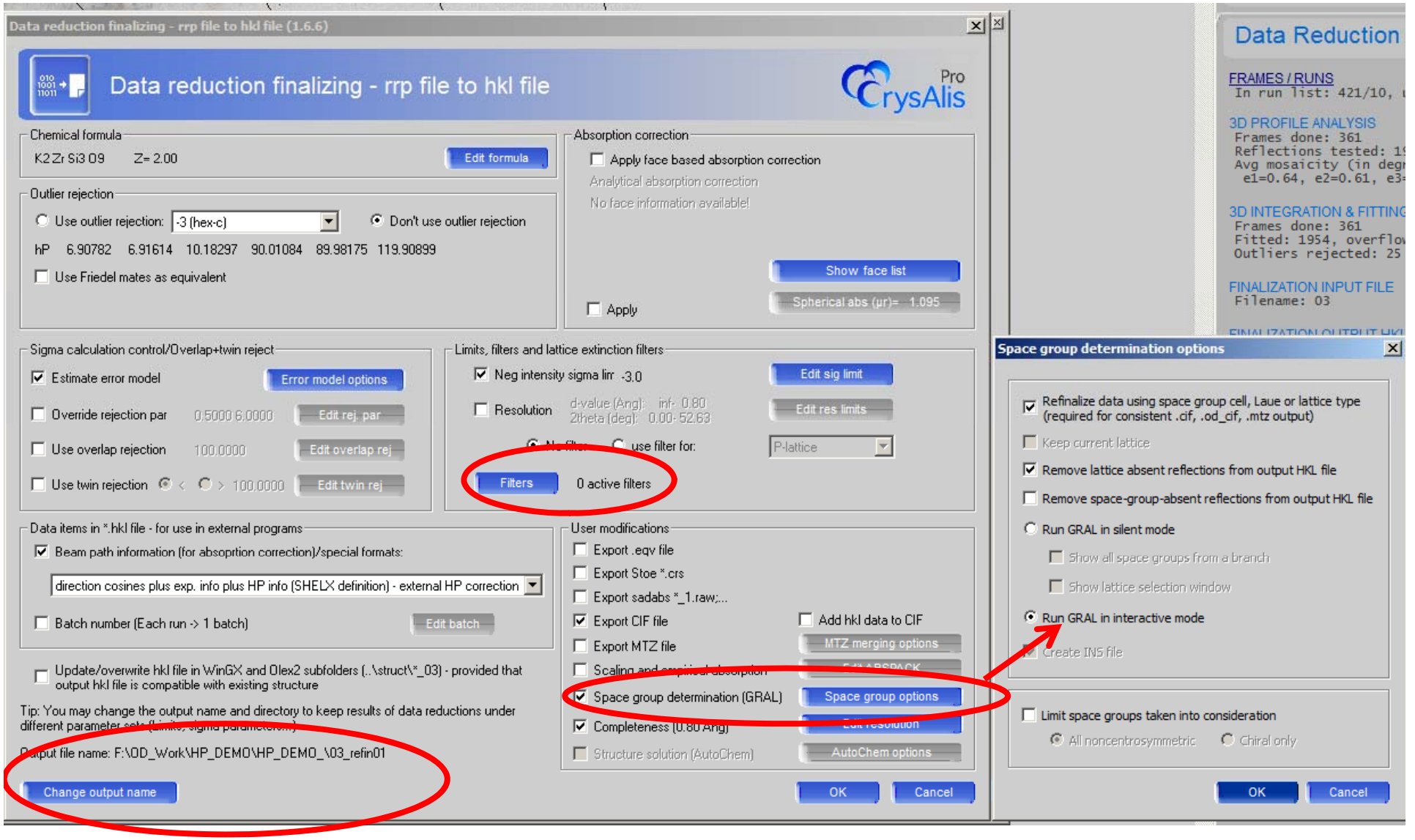
1.27-1.11	238	238	29		8.2	4134.71	9.62	0.051	0.063	0.067
1.11-1.03	204	200	29		6.9	2870.75	7.44	0.100	0.112	0.093
1.03-0.95	207	207	29		7.1	3588.85	7.80	0.064	0.077	0.090
0.95-0.90	160	160	29		5.5	1760.28	4.97	0.086	0.103	0.137
0.90-0.85	153	153	29		5.3	1900.35	4.77	0.080	0.091	0.142
0.85-0.80	146	146	29		5.0	2089.86	4.95	0.090	0.100	0.147
0.80-0.76	72	72	33		2.2	2164.78	4.80	0.090	0.101	0.149
inf-0.76	1954	1948	294		6.6	4568.93	11.20	0.053	0.065	0.062
inf-0.80	1895	1889	266		7.1	4638.78	11.39	0.052	0.064	0.061

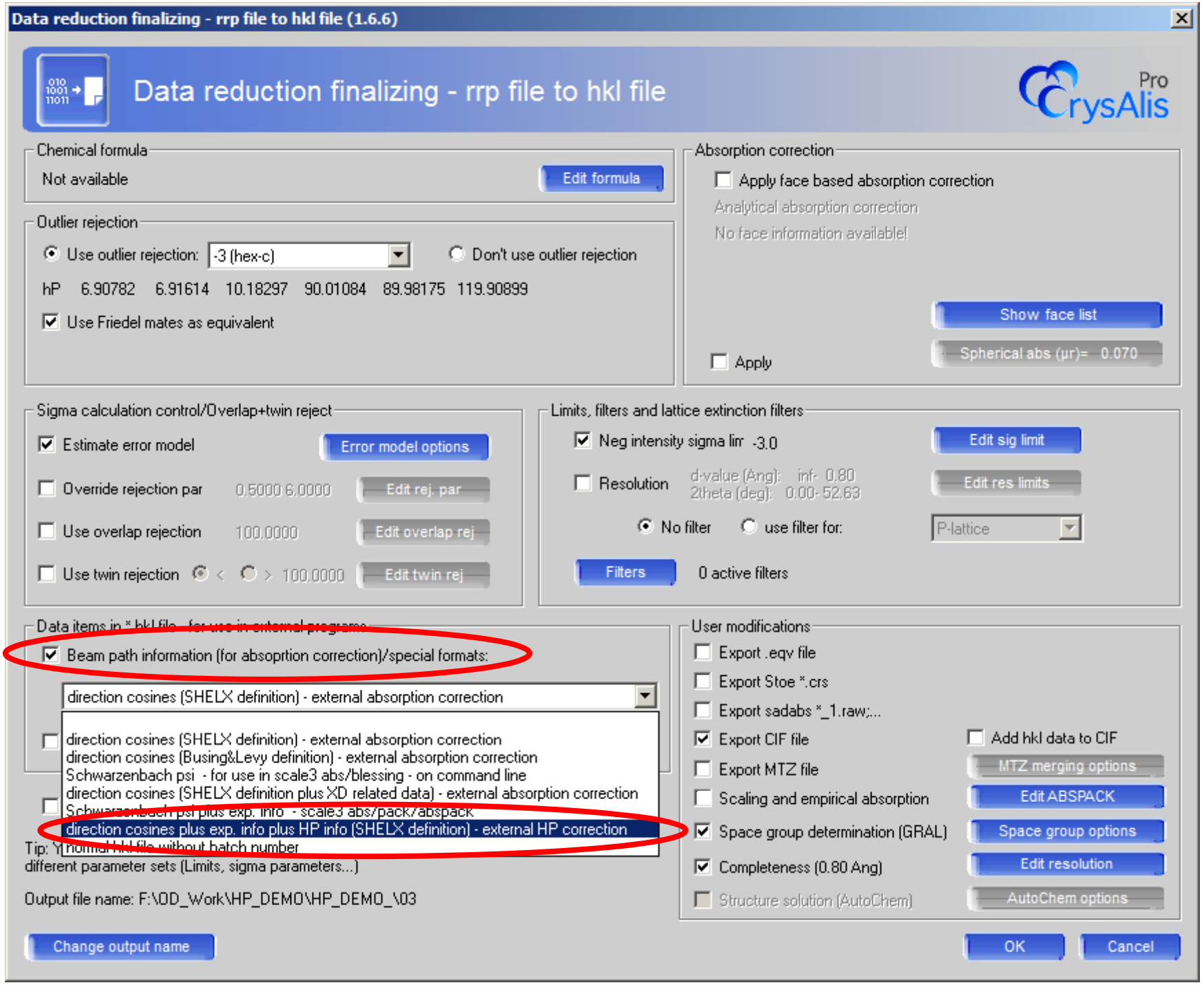
Statistics vs resolution (taking redundancy into account) - point group symmetry: P6/m

resolution(A)	# kept	# theory	# unique	% complete	average redundancy	mean F2	mean F2/sig(F2)	Rint	RsigmaB
inf-1.93	267	29	29	100.0	9.2	7801.31	70.75	0.032	0.010
1.89-1.48	259	29	29	100.0	8.9	8523.08	63.74	0.044	0.015
1.45-1.27	246	31	29	93.5	8.5	5184.41	39.92	0.047	0.019
1.27-1.11	238	34	29	85.3	8.2	4134.71	31.74	0.051	0.027
1.11-1.03	200	31	29	93.5	6.9	2870.75	21.31	0.100	0.040
1.03-0.95	207	39	29	74.4	7.1	3588.85	23.12	0.064	0.038
0.95-0.90	160	35	29	82.9	5.5	1760.28	12.45	0.086	0.063
0.90-0.85	153	41	29	70.7	5.3	1900.35	11.41	0.080	0.065
0.85-0.80	146	44	29	65.9	5.0	2089.86	11.89	0.090	0.069
0.80-0.76	72	58	33	56.9	2.2	2164.78	8.78	0.090	0.095
inf-0.76	1948	371	294	79.2	6.6	4568.93	34.87	0.053	0.030
inf-0.80	1889	318	266	83.6	7.1	4638.78	35.68	0.052	0.028

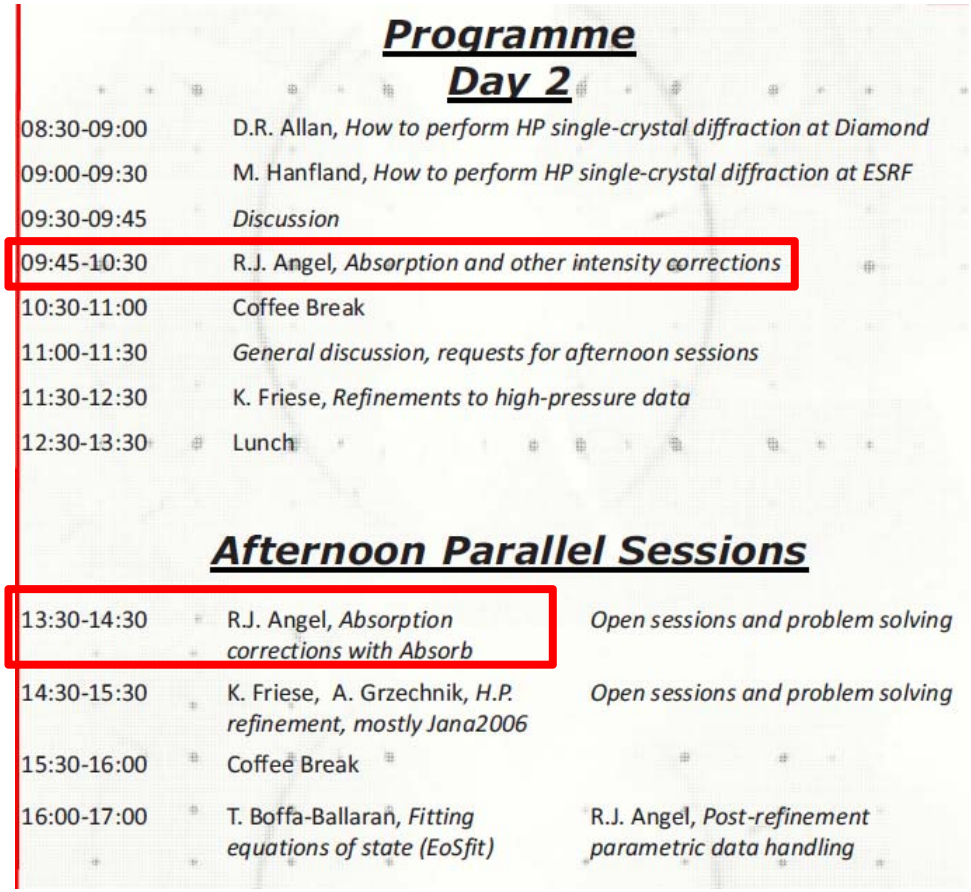
Data reduction ended at Wed Aug 01 12:00:23 2012

Refinalize | Abs display | 03 | OK


Data reduction finalizing - rrp file to hkl file (1.6.6)
Data reduction finalizing - rrp file to hkl file
Pro CrysAlis
Chemical formula
K2 Zr Si3 O9 Z= 2.00
Edit formula
Outlier rejection
Use outlier rejection: -3 (hex-c)
Don't use outlier rejection
hP 6.90782 6.91614 10.18297 90.01084 89.98175 119.90899
Use Friedel mates as equivalent
Absorption correction
Apply face based absorption correction
Analytical absorption correction
No face information available!
Show face list
Apply
Spherical abs (μr)= 1.095
Sigma calculation control/Overlap+twin reject
Estimate error model
Error model options
Override rejection par 0.5000 6.0000
Edit rej. par
Use overlap rejection 100.0000
Edit overlap rej
Use twin rejection < > 100.0000
Edit twin rej
Limits, filters and lattice extinction filters
Neg intensity sigma lim -3.0
Edit sig limit
Resolution d-value (Ang): inf- 0.80
2theta (deg): 0.00- 52.63
Edit res limits
use filter for:
P-lattice
Filters
0 active filters
Data items in *.hkl file - for use in external programs
Beam path information (for absoprtion correction)/special formats:
direction cosines plus exp. info plus HP info (SHELX definition) - external HP correction
Batch number (Each run -> 1 batch)
Edit batch
Update/overwrite hkl file in WinGX and Olex2 subfolders (..\struct*_03) - provided that output hkl file is compatible with existing structure
Tip: You may change the output name and directory to keep results of data reductions under different parameter sets (limits, sigma parameters...)
Output file name: F:\OD_Work\HP_DEMO\HP_DEMO_\03_refin01
Change output name
User modifications
Export .eqv file
Export Stoe *.crs
Export sadabs *_1.raw;...
Export CIF file
Add hkl data to CIF
Export MTZ file
MTZ merging options
Space group determination (GRAL)
Space group options
Completeness (0.80 Ang)
Structure solution (AutoChem)
AutoChem options
OK
Cancel
Data Reduction
FRAMES / RUNS
In run list: 421/10,
3D PROFILE ANALYSIS
Frames done: 361
Reflections tested: 1
Avg mosaicity (in deg
e1=0.64, e2=0.61, e3
3D INTEGRATION & FITTING
Frames done: 361
Fitted: 1954, overflo
Outliers rejected: 25
FINALIZATION INPUT FILE
Filename: 03
Space group determination options
Refinalize data using space group cell, Laue or lattice type (required for consistent .cif, .od_cif, .mtz output)
Keep current lattice
Remove lattice absent reflections from output HKL file
Remove space-group-absent reflections from output HKL file
Run GRAL in silent mode
Show all space groups from a branch
Show lattice selection window
Run GRAL in interactive mode
Create INS file
Limit space groups taken into consideration
All noncentrosymmetric
Chiral only
OK
Cancel


Data reduction finalizing - rrp file to hkl file (1.6.6)
Data reduction finalizing - rrp file to hkl file
Pro
CrysAlis
Chemical formula
Not available
Edit formula
Absorption correction
Apply face based absorption correction
Analytical absorption correction
No face information available!
Show face list
Spherical abs (μr)= 0.070
Apply
Outlier rejection
Use outlier rejection: -3 (hex-c)
Don't use outlier rejection
hP 6.90782 6.91614 10.18297 90.01084 89.98175 119.90899
Use Friedel mates as equivalent
Sigma calculation control/Overlap+twin reject
Estimate error model
Error model options
Override rejection par 0.5000 6.0000
Edit rej. par
Use overlap rejection 100.0000
Edit overlap rej
Use twin rejection < > 100.0000
Edit twin rej
Limits, filters and lattice extinction filters
Neg intensity sigma lim -3.0
Edit sig limit
Resolution d-value (Ang): inf- 0.80
2theta (deg): 0.00- 52.63
Edit res limits
No filter
use filter for:
P-lattice
Filters
0 active filters
Data items in *.hkl file - for use in external programs
Beam path information (for absoprtion correction)/special formats:
direction cosines (SHELX definition) - external absorption correction
direction cosines (SHELX definition) - external absorption correction
direction cosines (Busing&Levy definition) - external absorption correction
Schwarzenbach psi - for use in scale3 abs/blessing - on command line
direction cosines (SHELX definition plus XD related data) - external absorption correction
Schwarzenbach psi plus exp. info - scale3 abs/pack/abspack
direction cosines plus exp. info plus HP info (SHELX definition) - external HP correction
normal hkl file without batch number
Tip: Y
different parameter sets (Limits, sigma parameters...)
Output file name: F:\OD_Work\HP_DEMO\HP_DEMO_\03
User modifications
Export .eqv file
Export Stoe *.crs
Export sadabs *_1.raw;...
Export CIF file
Add hkl data to CIF
Export MTZ file
MTZ merging options
Scaling and empirical absorption
Edit ABSPACK
Space group determination (GRAL)
Space group options
Completeness (0.80 Ang)
Edit resolution
Structure solution (AutoChem)
AutoChem options
Change output name
OK
Cancel

Absorption correction:



Programme
Day 2

08:30-09:00	D.R. Allan, *How to perform HP single-crystal diffraction at Diamond*
09:00-09:30	M. Hanfland, *How to perform HP single-crystal diffraction at ESRF*
09:30-09:45	*Discussion*
09:45-10:30	R.J. Angel, *Absorption and other intensity corrections*
10:30-11:00	Coffee Break
11:00-11:30	*General discussion, requests for afternoon sessions*
11:30-12:30	K. Friese, *Refinements to high-pressure data*
12:30-13:30	Lunch

Afternoon Parallel Sessions

13:30-14:30	R.J. Angel, *Absorption corrections with Absorb*	*Open sessions and problem solving*
14:30-15:30	K. Friese, A. Grzechnik, *H.P. refinement, mostly Jana2006*	*Open sessions and problem solving*
15:30-16:00	Coffee Break	
16:00-17:00	T. Boffa-Ballaran, *Fitting equations of state (EoSfit)*	R.J. Angel, *Post-refinement parametric data handling*

Next version of *CrysAlisPro*

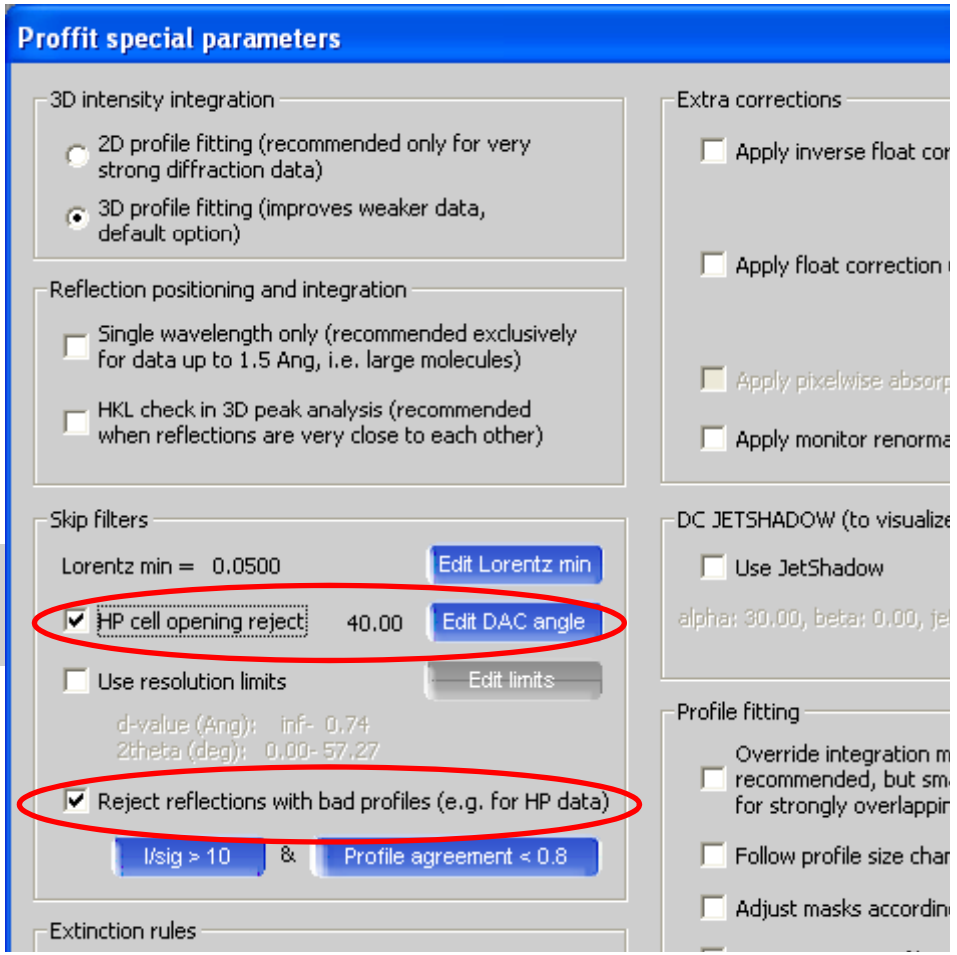
Call Program Absorb from *CrysAlisPro* directly → Mathias Meyer

New feature in *CrysAlisPro*:

Reject reflections with bad reflection profile:
If diamond reflection lies on a sample reflection

and

Reject defined regions
diamond reflections and
diamond tails
From peak list



Questions?

ASK colleges with experience in high pressure data integration

X-ray Diffraction Forum:

http://www.agilentxrdforum.com/

http://www.agilentxrdforum.com/yaf_login.aspx?returnurl=%2fdefault.aspx