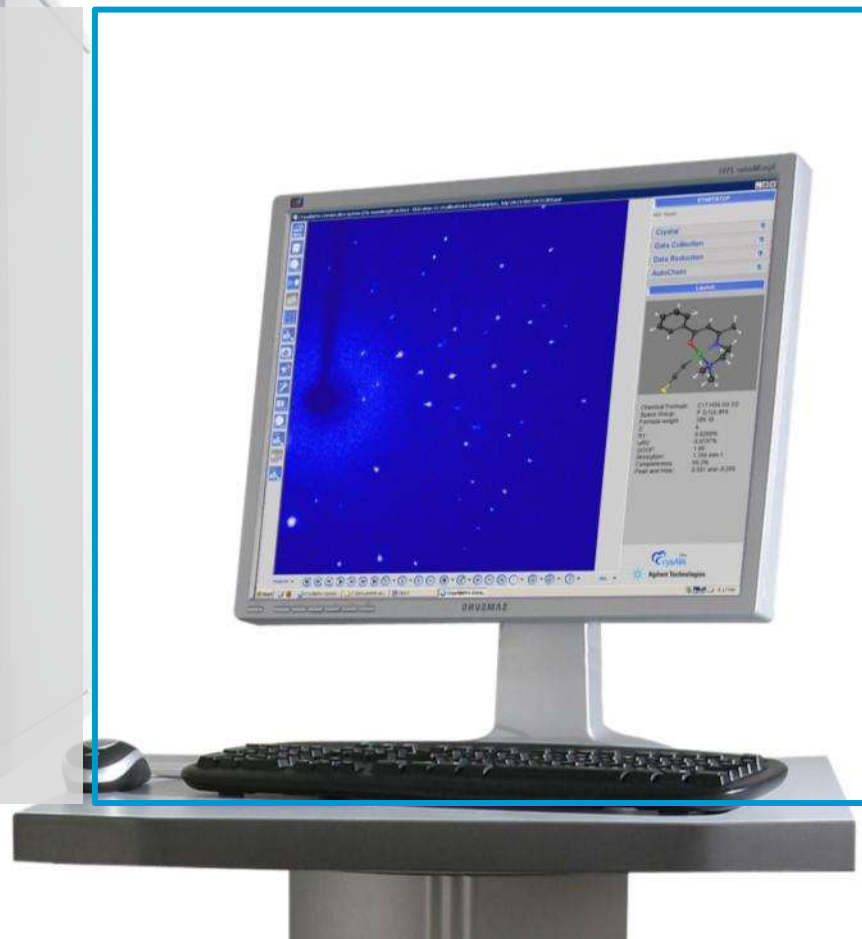


Méthodes de intégration: CrysAlis^{Pro}

Reciproque workshop -
Ambleteuse

Mathias Meyer
X-ray Group Software Manager

9th September 2014

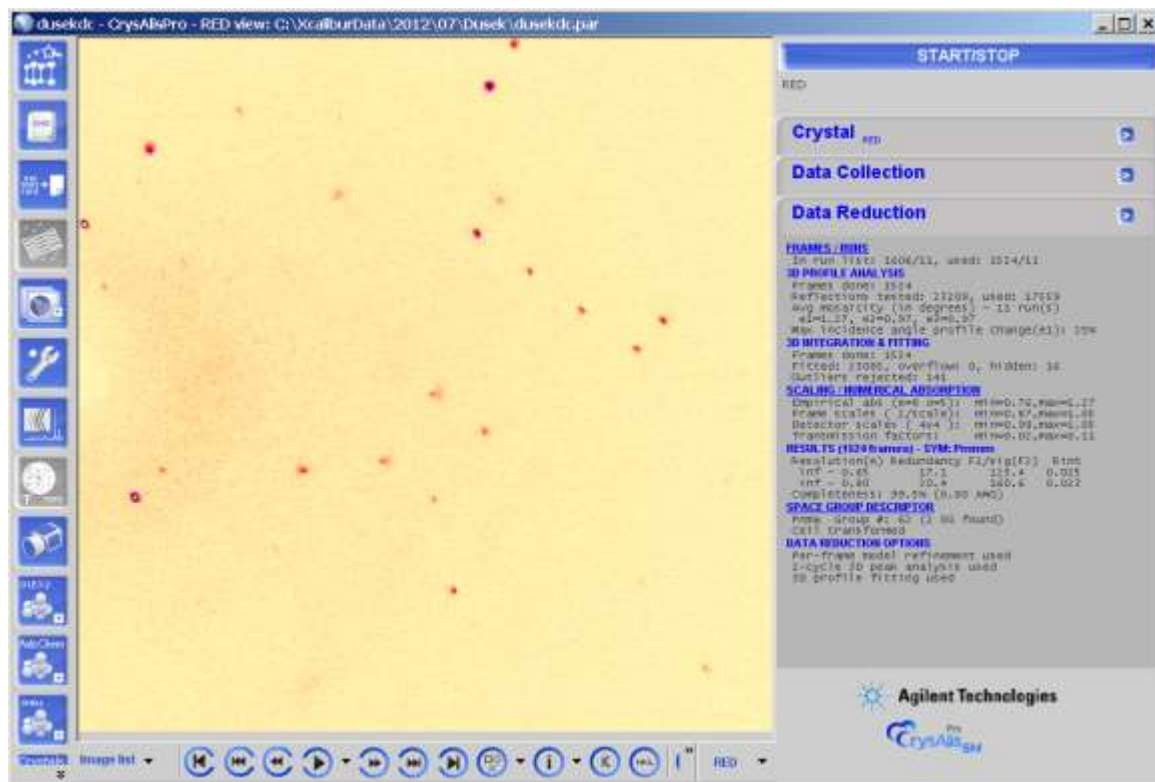
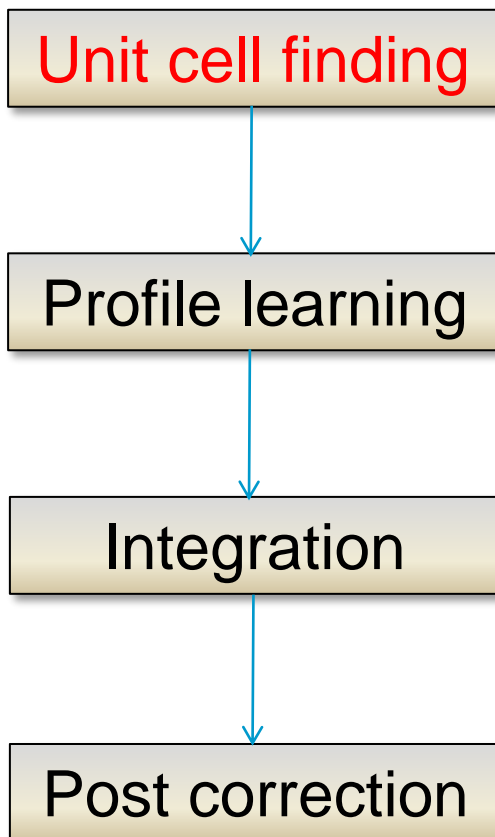


Agilent Technologies

Overview

- Unit cell finding
- Data reduction (Profile learning and integration)
- Post corrections
- Experimental workflow and strategy
- Twins
- Foreign formats
- Incommensurates
- Powders

Typical data reduction sequence



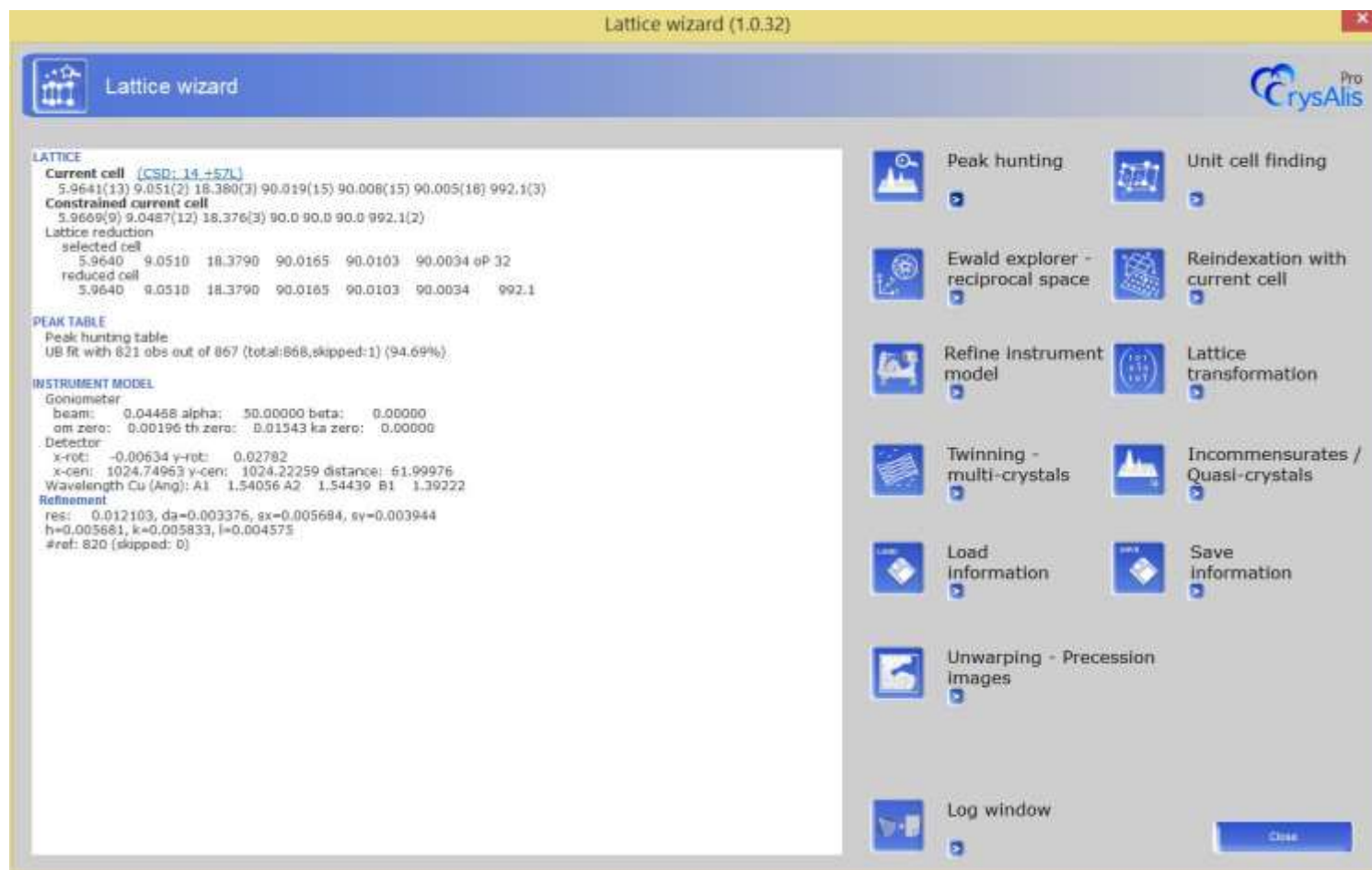
Unit cell finding

- Peak hunting
- Unit cell finding
 - Automatic
 - Manual
- Ewald^{Pro}
- Intelligent filtering
- Instrument model



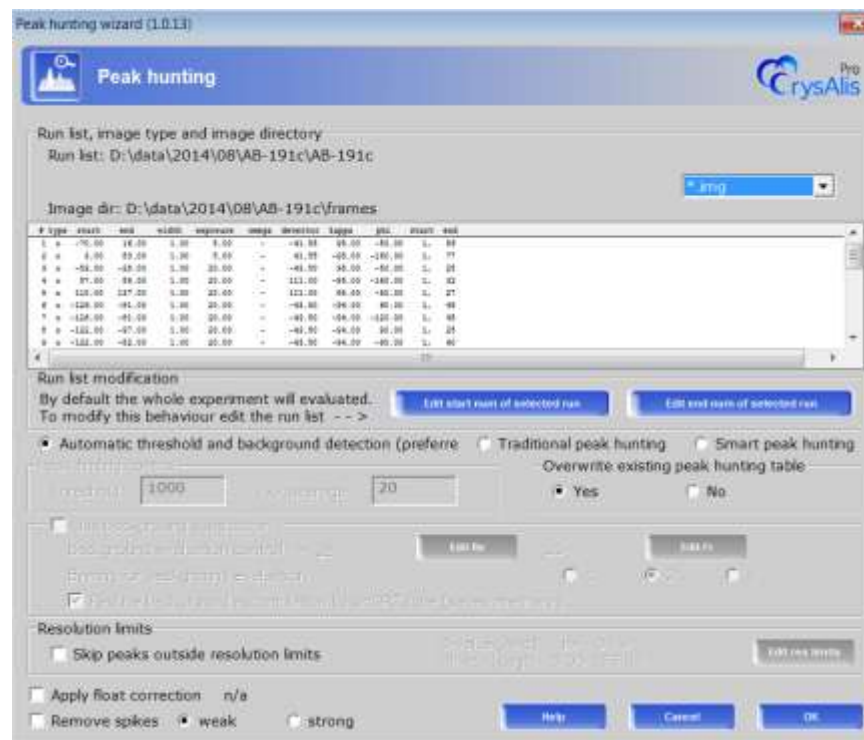
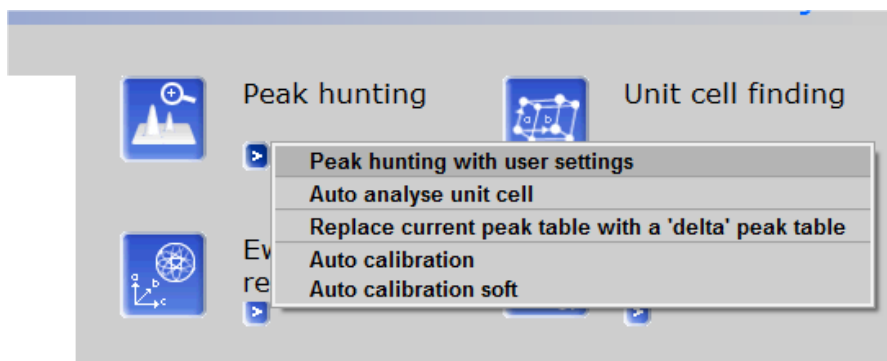
Unit cell finding

- Lattice wizard power tool



Unit cell finding – Peak hunting

- Defaults work well for 'normal' crystals and twins
- Super-structures may require tweak



Unit cell finding – Peak hunting

- Data filtering via peak table

Peak table editing (1.0.9)

Peak table editing

number	h	k	l	a	b	c	d	intensity	flag	prod. gao
1	1	-2	-4	-0.14117	-0.15870	0.12026	6.13288	406	i gl	1
2	1	-2	-4	-0.12061	-0.10194	0.13685	6.79225	4099	i gl	1
3	1	2	-4	-0.07890	0.07968	0.22759	6.13248	2236	i gl	1
4	1	0	-11	-0.06994	0.16215	0.69288	2.18746	370	i gl	1
5	0	4	-1	0.05813	0.20785	0.14949	8.81036	1241	i gl	1
6	1	-9	-12	-0.24939	-0.49358	0.87219	2.14052	485	i gl	1
7	1	-4	-6	-0.18796	-0.31272	0.18914	3.74901	318	i gl	1
8	1	1	-4	-0.09798	0.01389	0.20390	6.81002	9731	i gl	1
9	1	-1	-16	-0.19469	-0.53723	0.77185	1.78172	379	i gl	1
10	1	1	-16	-0.14609	-0.22119	0.81874	1.78246	438	i gl	1
11	1	4	-6	-0.09396	0.14933	0.37713	3.74909	490	i gl	1
12	1	8	-12	-0.06040	0.09670	0.70723	2.14811	802	i gl	1
13	-1	-9	-19	-0.13147	-0.60939	0.97466	1.63139	471	i gl	1
14	0	-7	-11	-0.10061	-0.61559	0.39762	2.03969	1492	i gl	1
15	1	-2	-6	-0.19469	-0.19933	0.23913	6.87600	1414	i gl	1
16	-1	9	-19	0.16239	0.22603	0.30269	1.63277	466	i gl	1
17	-1	-9	-11	-0.12971	-0.74911	0.37928	1.78908	486	i gl	1
18	0	-7	-9	-0.16924	-0.87653	0.29640	2.23940	448	i gl	1
19	0	-7	-10	-0.17171	-0.59696	0.34657	2.18734	598	i gl	1
20	0	-6	-4	-0.12103	-0.62171	0.06959	3.87519	530	i gl	1
21	0	-3	-16	-0.14929	-0.40974	0.74939	1.70933	621	i gl	1
22	1	-1	-6	-0.12975	-0.14007	0.25949	6.72107	1597	i gl	1
23	1	2	-6	-0.09302	0.03139	0.32907	6.85446	2049	i gl	1
24	0	7	-12	0.04819	0.16444	0.77709	1.93690	360	i gl	1
25	0	7	-11	0.05955	0.18241	0.72721	2.04929	3269	i gl	1
26	1	-2	-10	-0.18049	-0.27557	0.44107	2.79641	2520	i gl	1
27	1	-2	-8	-0.17079	-0.23994	0.33793	3.45966	2489	i gl	1
28	1	1	-6	-0.10923	-0.02972	0.30639	6.72223	1988	i gl	1
29	0	9	-16	-0.04049	-0.14361	0.59931	1.71031	399	i gl	1

Delete Up Rew

Edit Down Reject

hkl format
☒ integer ☐ fractional

Coordinates
☐ angles ☒ cartesian ☐ detector

Copy to clip Exit sorted

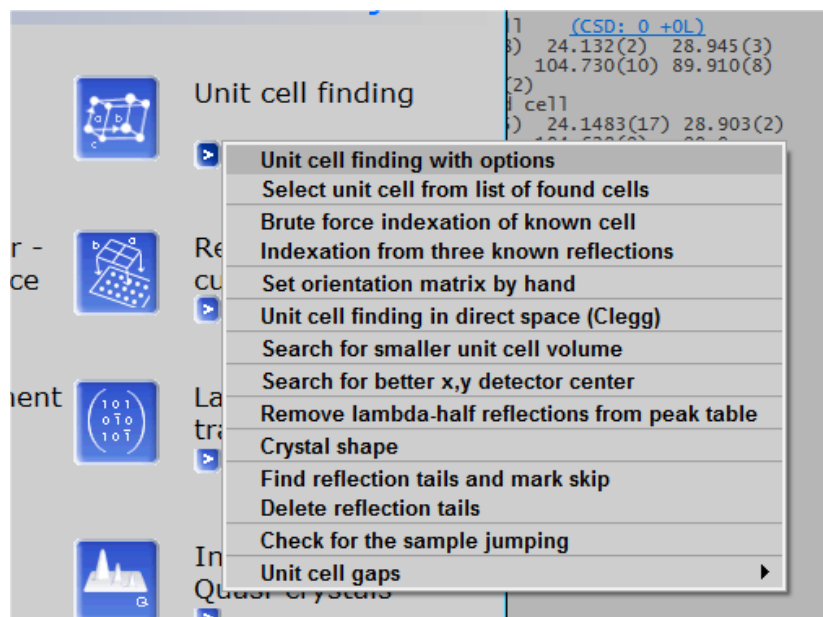
Help Exit

Ewald explorer - reciprocal space

Peak table editing
Peak table flags
Produce axial photo
Old ewald explorer

Re model

Unit cell finding – Automatic unit cell finding



Find cell...

Peak table

- ☒ Normal peak table
- ☐ Delta (differential) peak table

☐ Find center ☐ Use search box

Algorithm

- ☒ T-vector Dirax
- ☐ Stereographic

Sample type

- ☒ Single crystal
 - Unit cell
 - ☒ SM ☐ PX ☐ User
 - min: 2.0 max: 120.0
- ☐ Twin / multocrystal
 - # of components: 2 min: 2.0 max: 120.0

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.

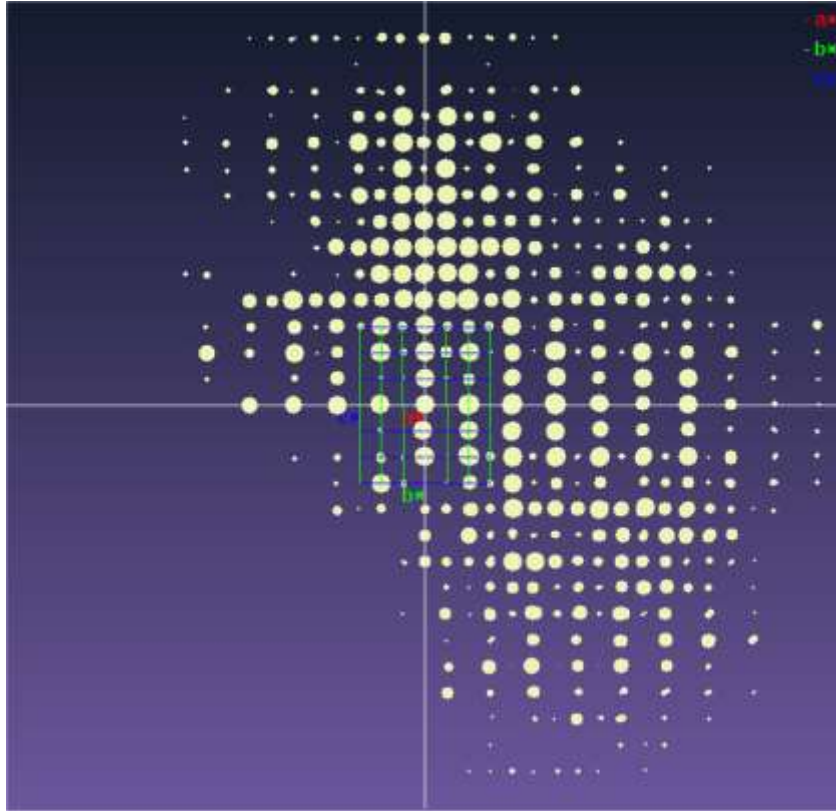
☐ Consider Bravais lattice type

☒ Force identical lattice for all components (uncheck for multi-crystal)

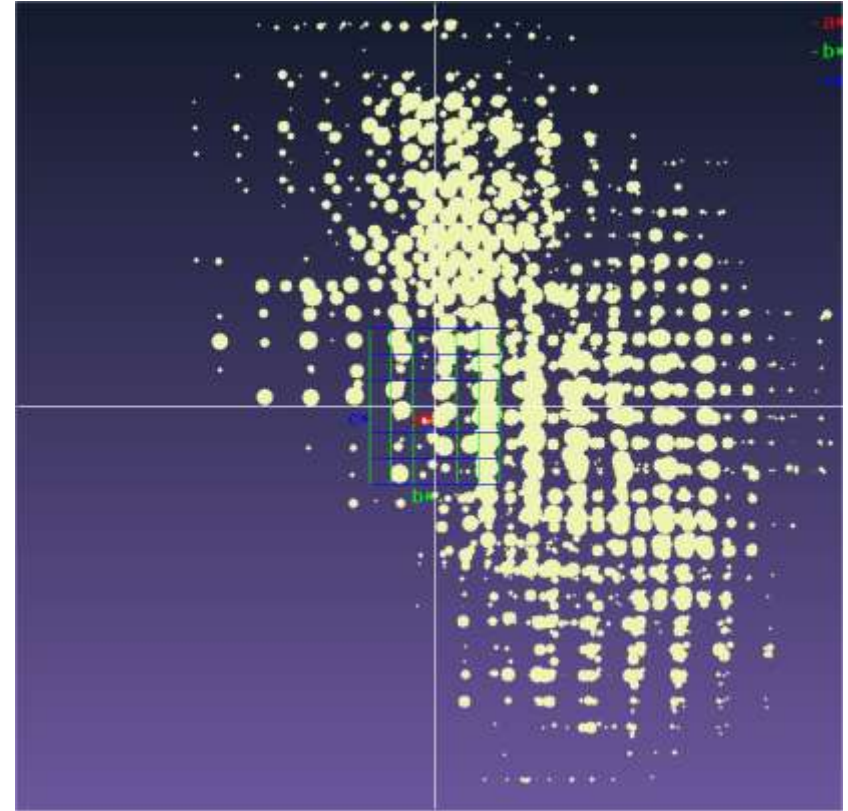
Known cell

☐ Search known cell 16.22 24.13 28.95 90.10 104.73 89.91

Unit cell finding – Influence of bad instrument model



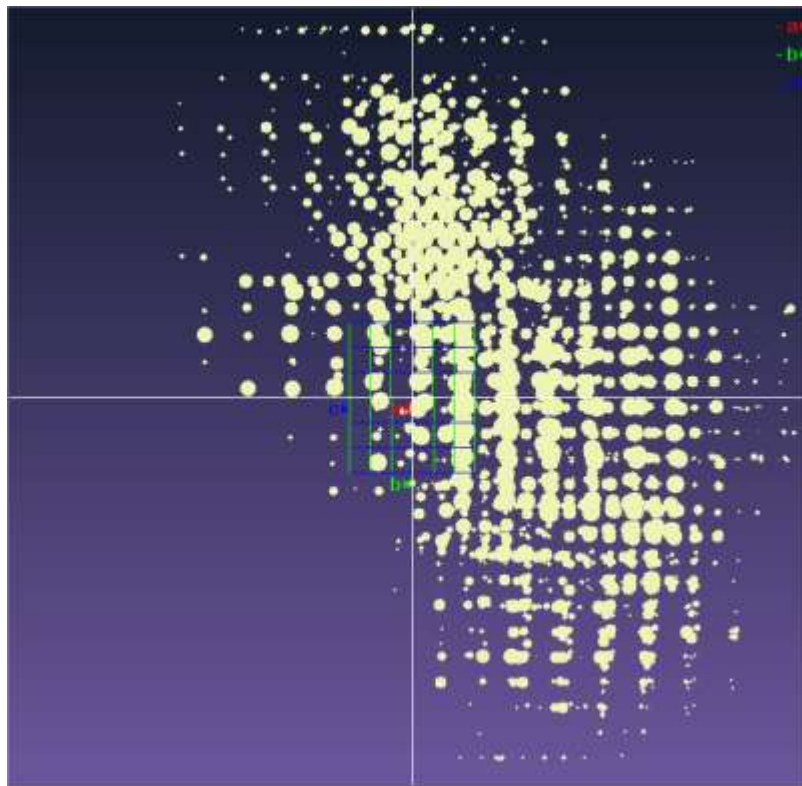
Good instrument model



Bad instrument model:

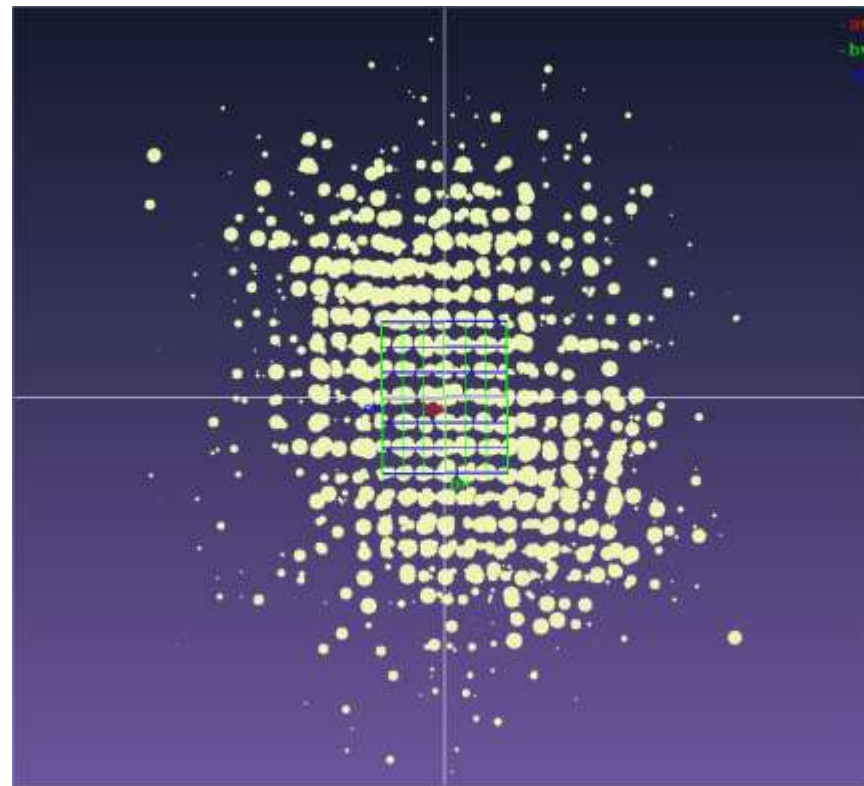
Beam center deviation 40 pix

Unit cell finding – Automatic unit cell finding



Bad instrument model:

Beam center deviation 40 pix

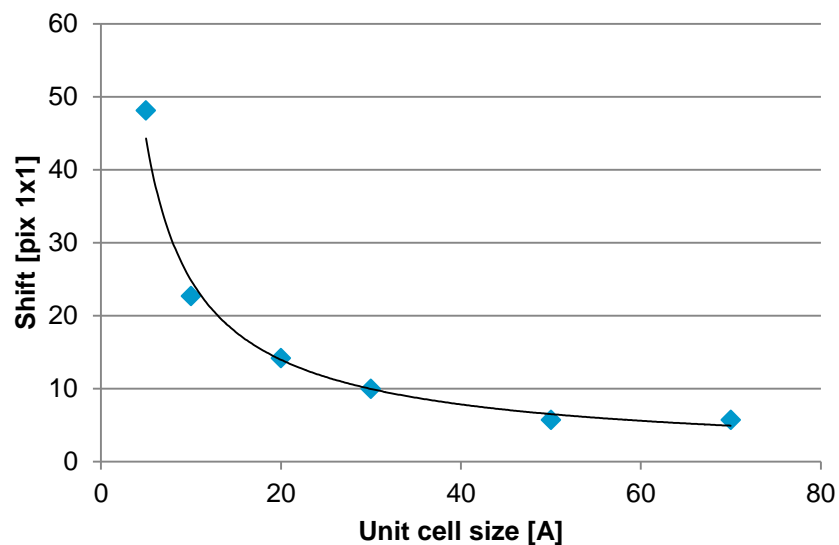


Bad instrument model:

Delta-peak table



Unit cell finding – Automatic unit cell finding



Peak table

☐ Normal peak table

☒ Delta (differential) peak table

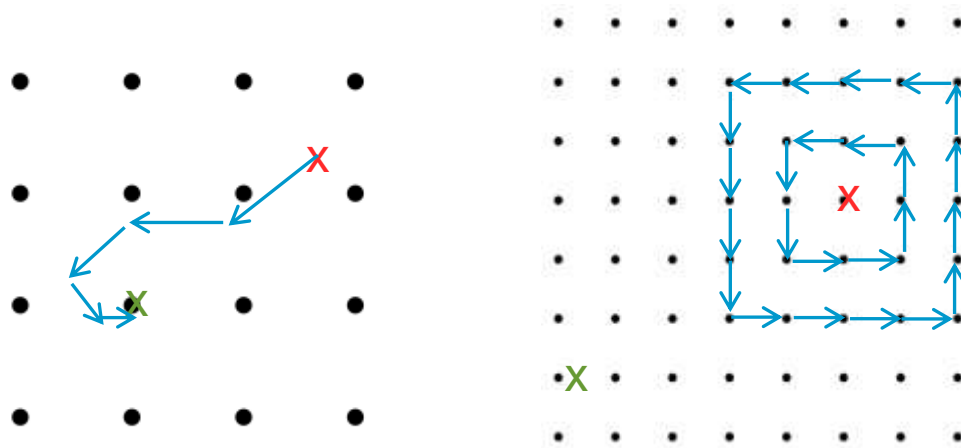
☒ Find center

☒ Use search box [Edit](#)

Algorithm

☒ T-vector Dirax

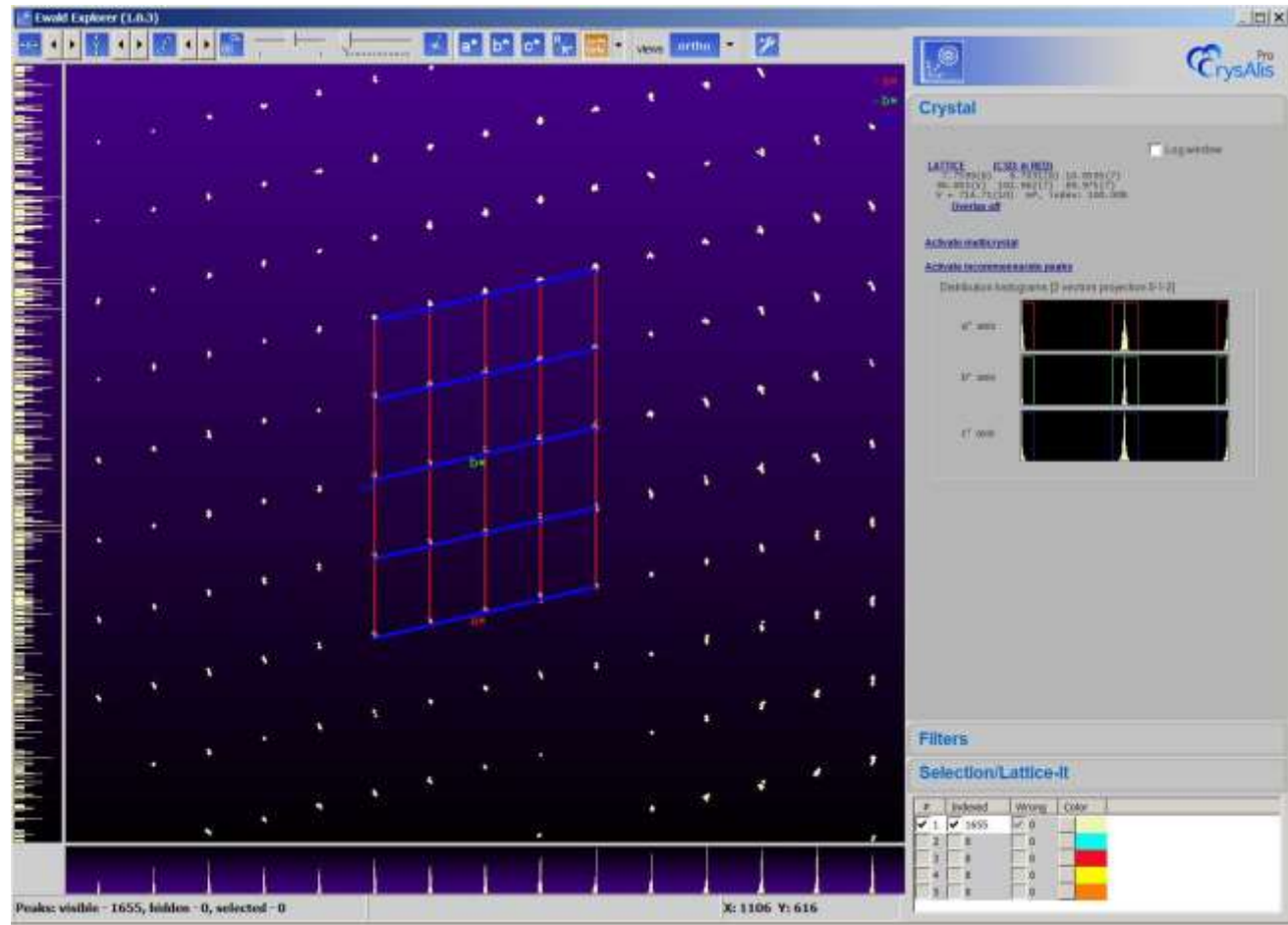
☐ Stereographic



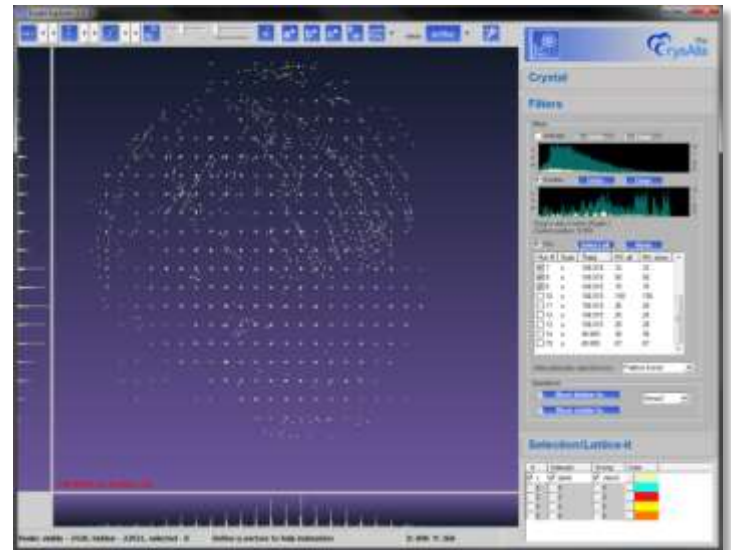
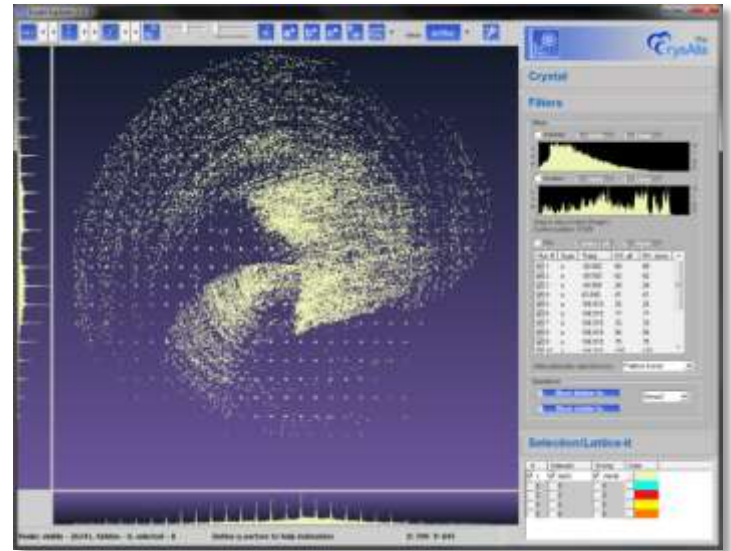
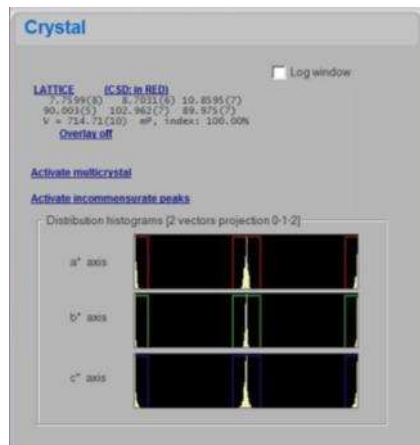
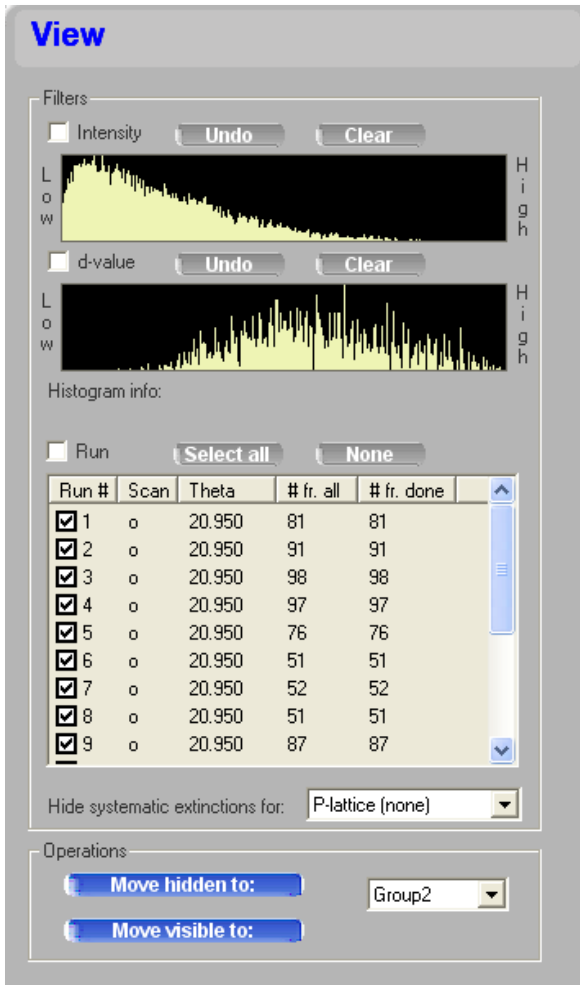
Power Tools

Ewald^{Pro}

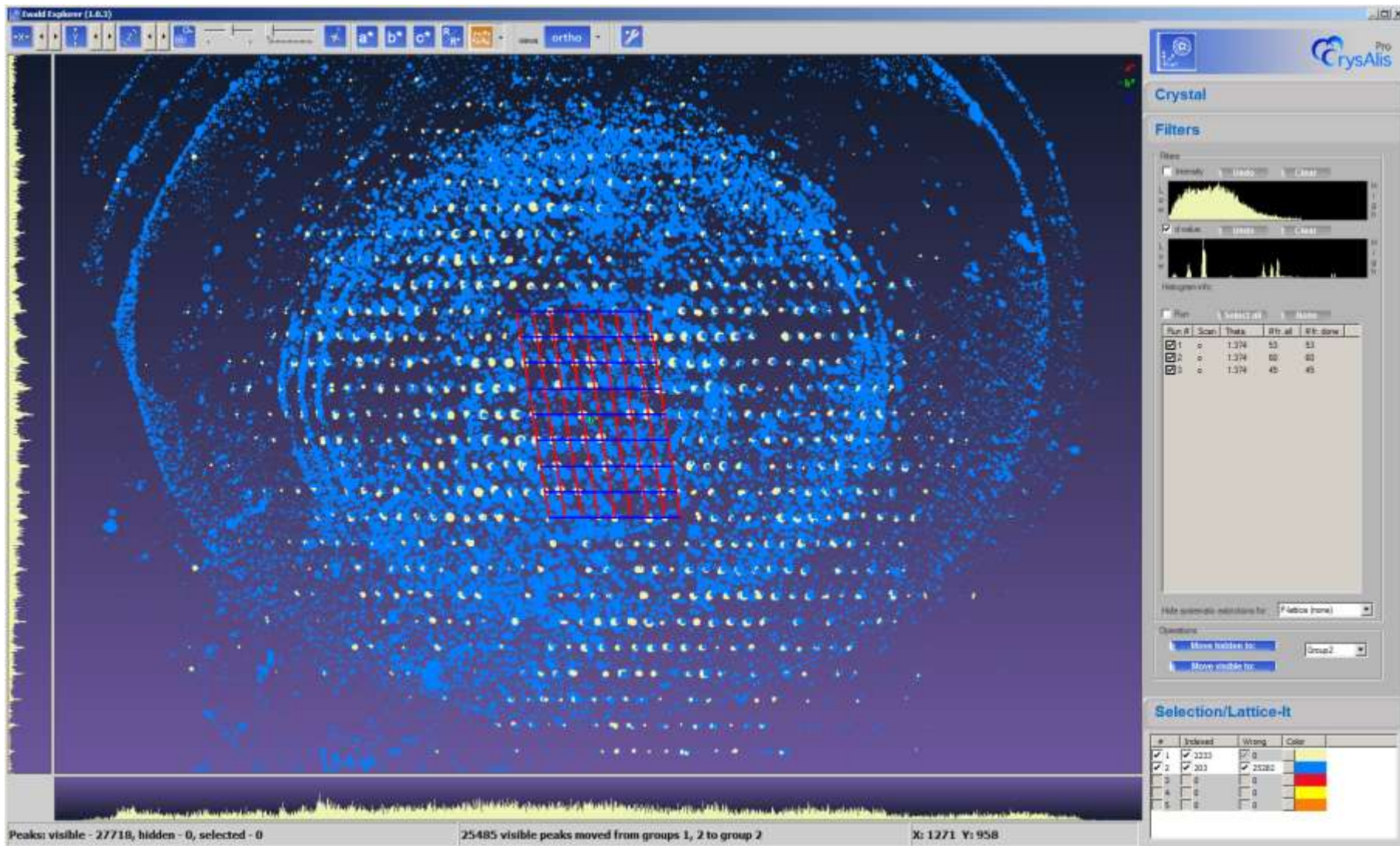
- User-friendly, powerful reciprocal lattice viewer
- Twin/Multi-lattice visualisation
- Drag filter tools – simple & intuitive
- Lattice^{IT} tool for defining periodicity
- Reflection grouping
- Incommensurate overlay tools



Ewald^{Pro} – Functional Tabs



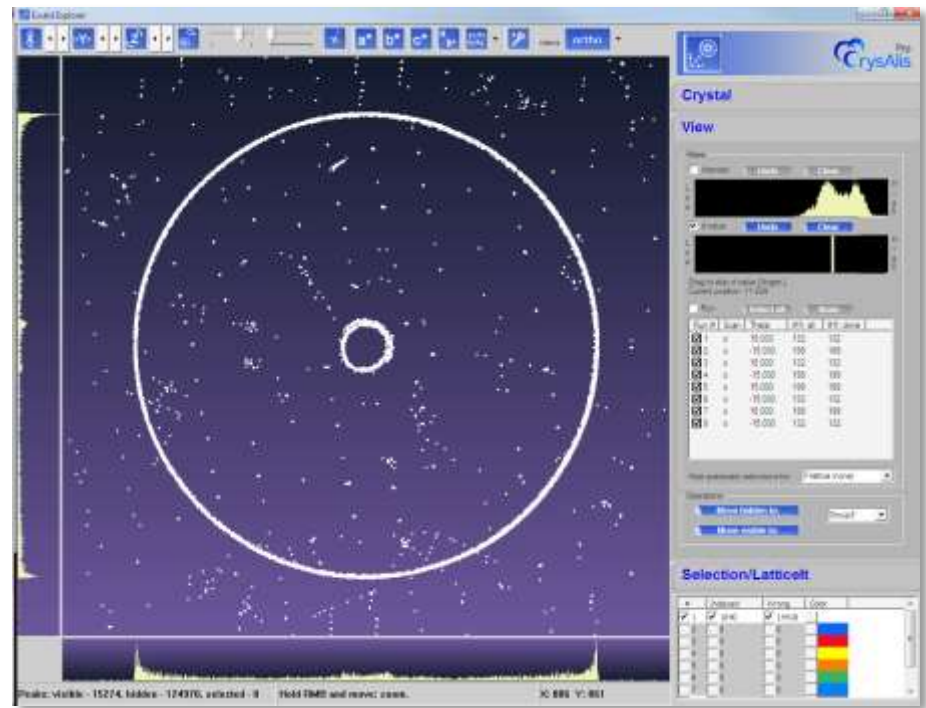
Ewald^{Pro} – Ice Ring Example



EwaldPro – Groups

Groups:

- Indexation quality
- Twin components
- Resolution range
- Incommensurates
- Specific features (weak/strong peaks, powder rings, etc.)



#	Indexed	Wrong	Color
✓ 1	✓ 1715	✓ 36	
2	0	0	
3	0	0	
4	0	0	
5	0	0	

#	UB1	UB2	Overl...	Wrong	Color
✓ 1	✓ 3619	✓ 0	✓ 0	✓ 0	
✓ 2	0	✓ 3860	✓ 0	0	
✓ 3	✓ 0	✓ 0	✓ 5539	✓ 0	
4	0	0	0	0	
✓ 5	✓ 0	✓ 0	0	✓ 254	

Ewald^{Pro} – Lattice^{It}



Selection/LatticeIt

Select all Invert selection Deselect all

Selection tools

☐ None ☐ Rubber band

☒ LatticeIt refine angle (with hints) ▾

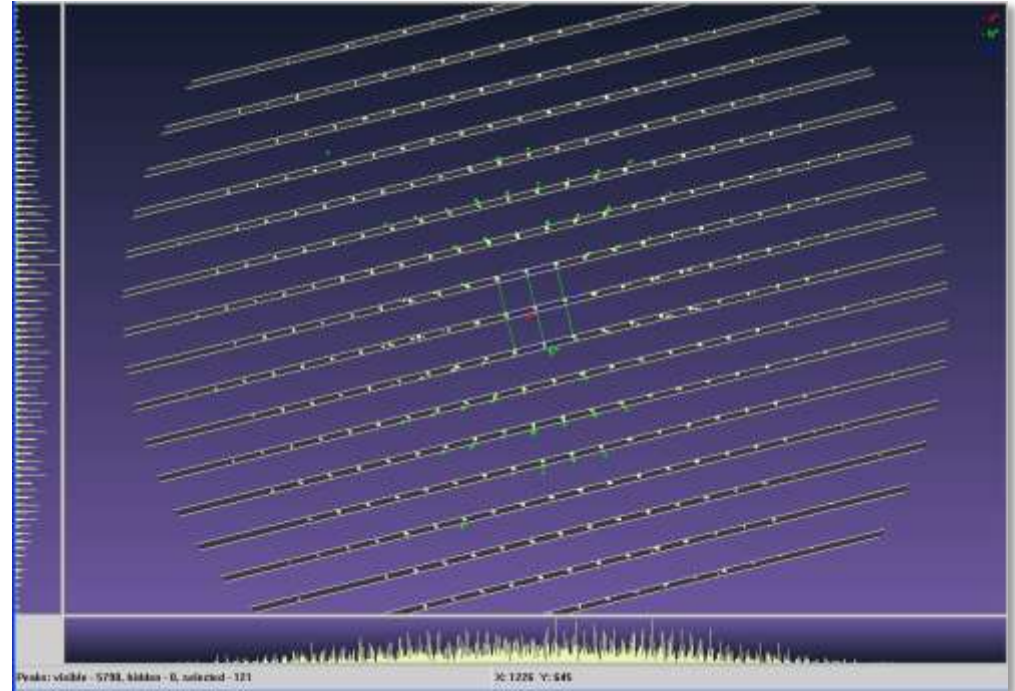
#	angle	period	width
<input checked="" type="checkbox"/> 1	13.8	49 pix	7.5 %

New Remove

Angle: ◀ ▶ Period: ◀ ▶ Width: ◀ ▶

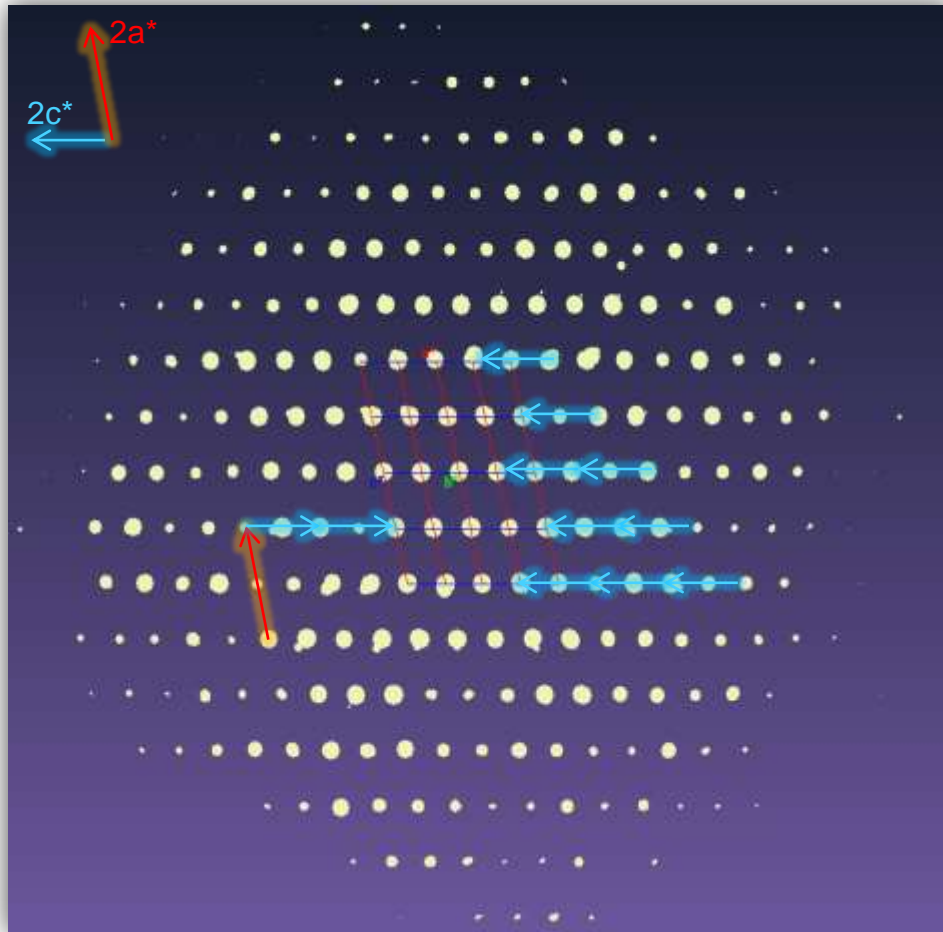
Operations

Move selected to: Group2 ▾



EwaldPro – new view mode: collapse peaks

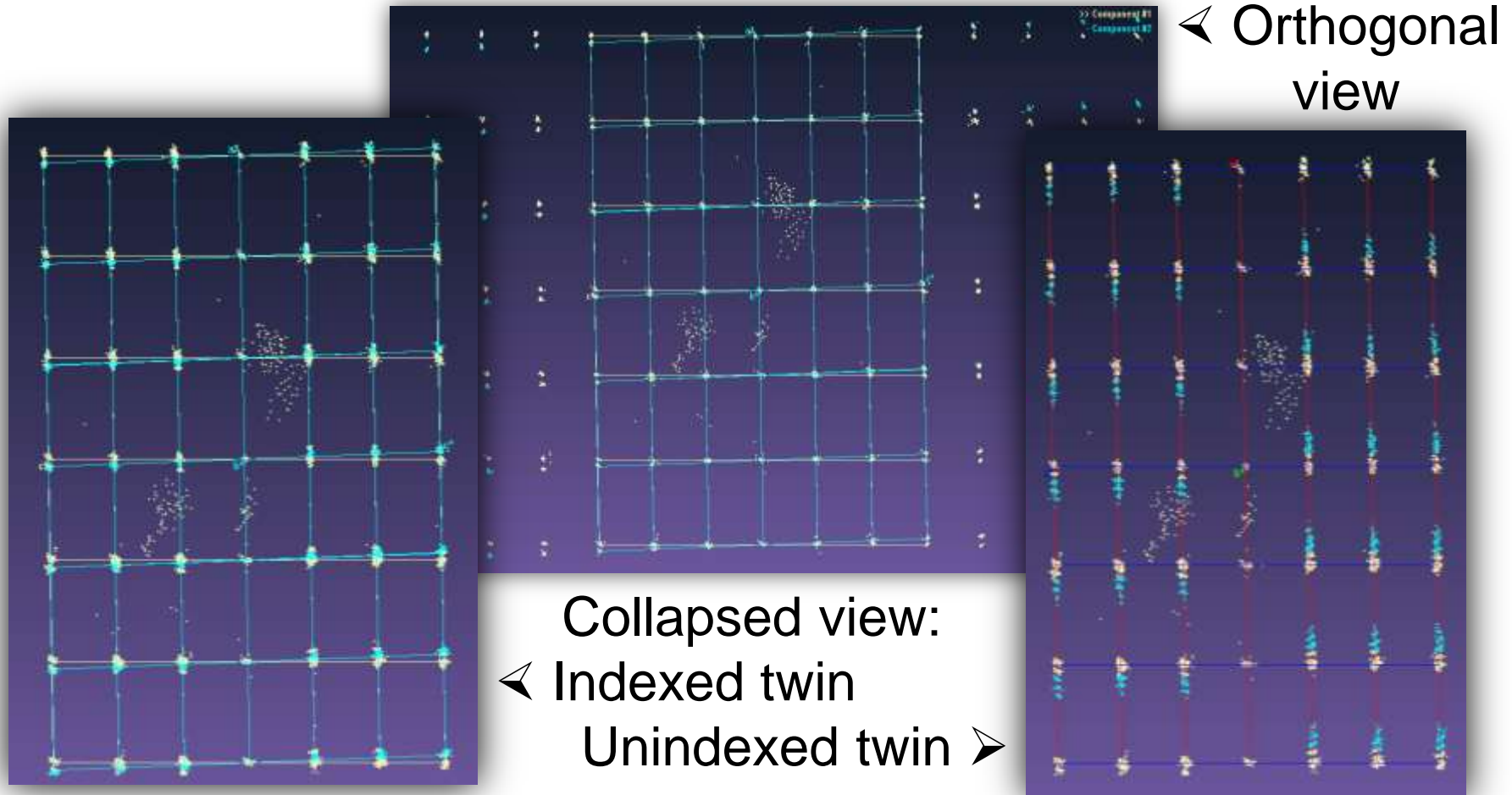
Collapse peak view – construction



1. Lattice vectors are multiplied by lattice overlay size
2. Subtract lattice vectors until given peak hits range

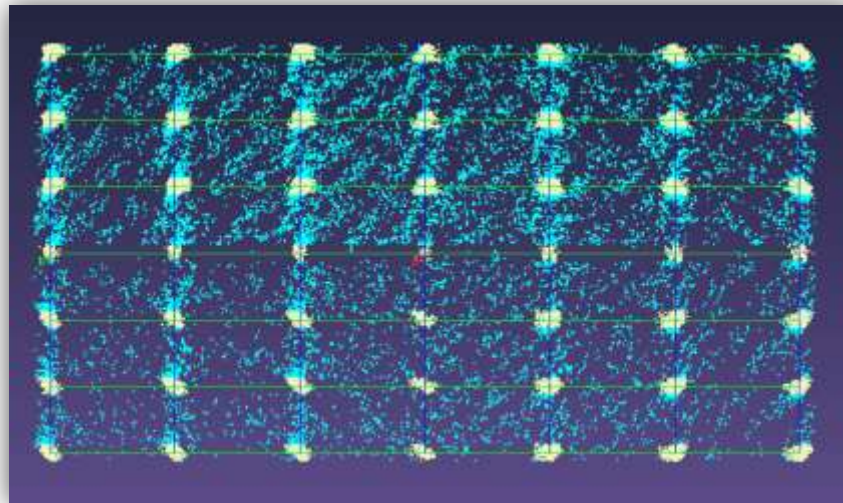
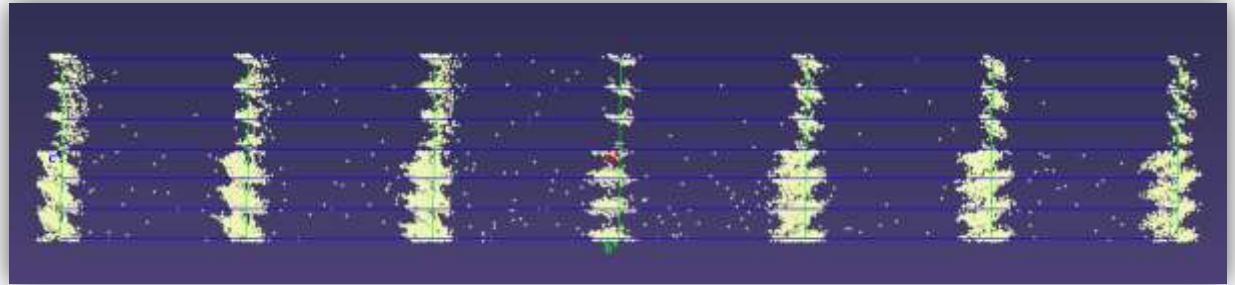
EwaldPro – new view mode: collapse peaks

Collapse peak view – twin example



EwaldPro – new view mode: collapse peaks

Collapse peak view – other examples

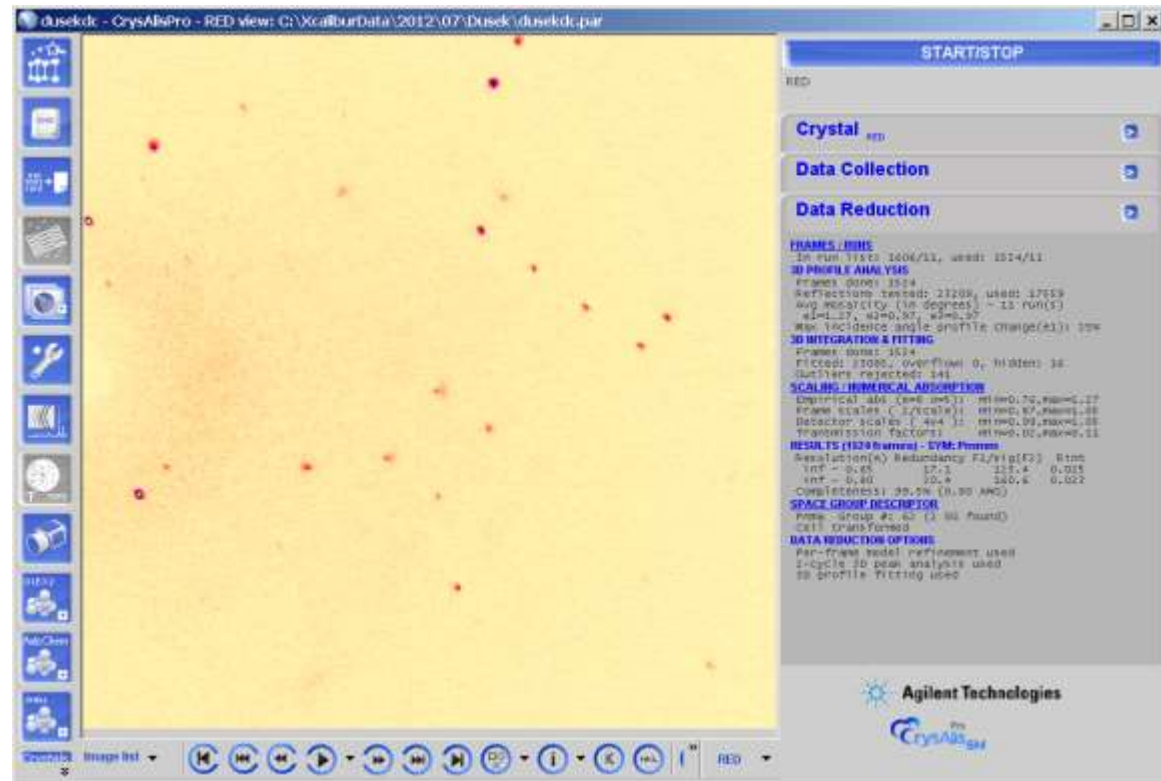
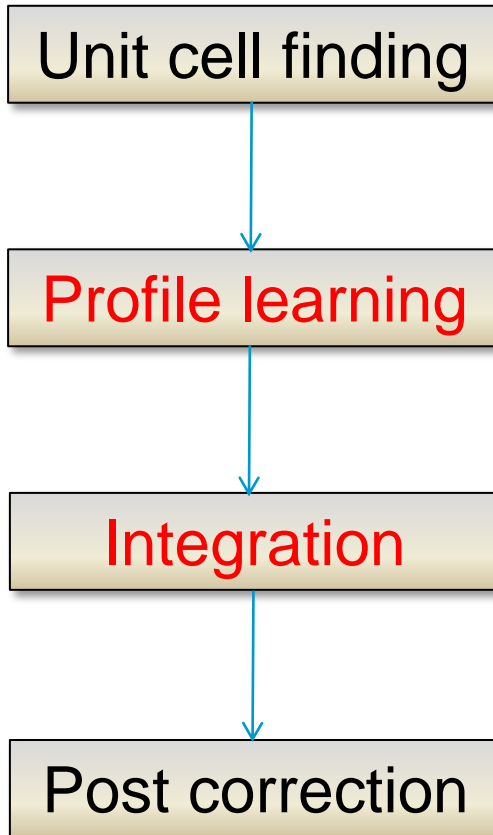


incommensurate

moving
crystal

multicrystal

Typical data reduction sequence



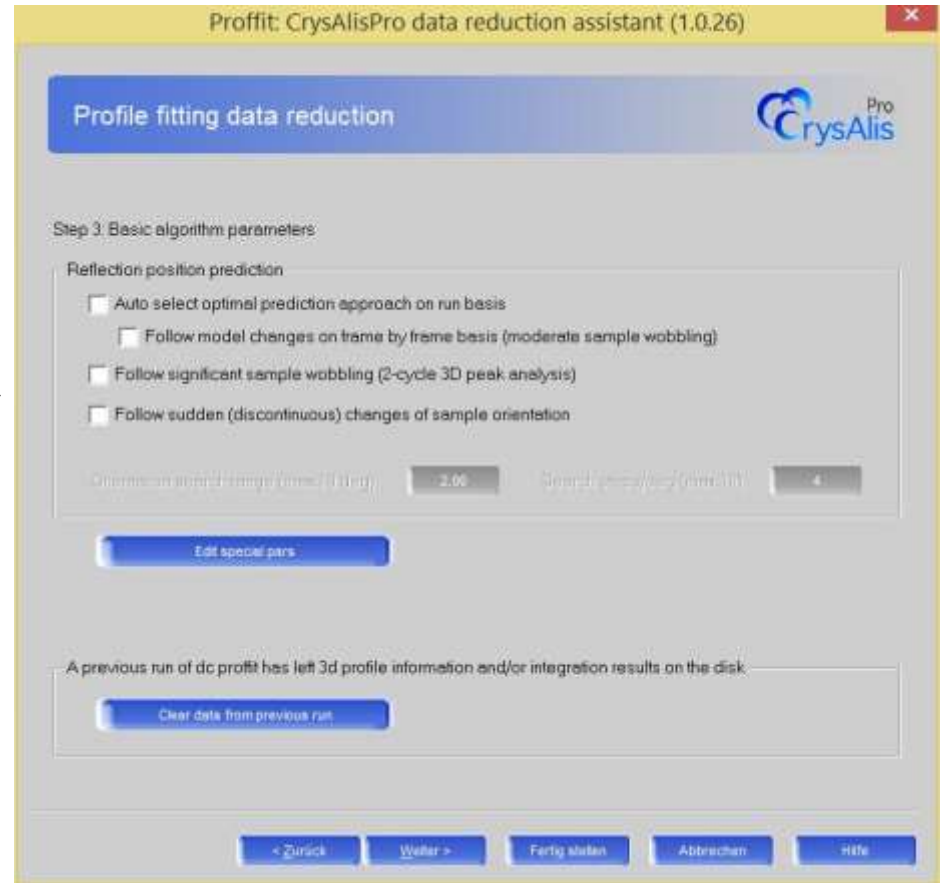
Data reduction wizard

- Collecting information
- Approaches of reflection finding/profile learning/integration
- Approaches to background



Data reduction wizard – options

- Model refinement options – for optimal prediction accuracy
- Special data reduction parameters



Model refinement options

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) Search steps/deg (max 10)

- Basic mode
- Single model for all data from one run
- Usually best choice for properly centered samples on a stiff holder

PROFFITPEAK

Profile analysis

PROFFITMAIN

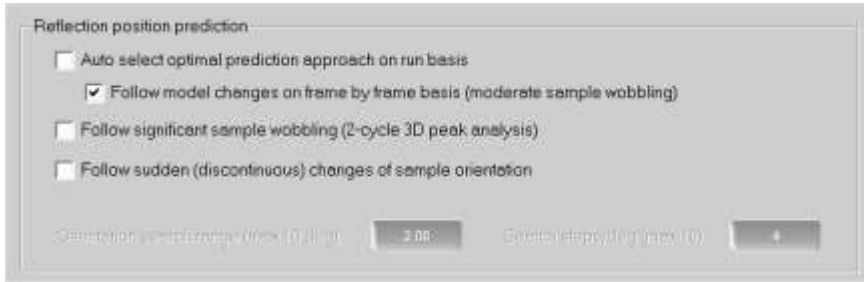
Cell/model
refinement

For every run

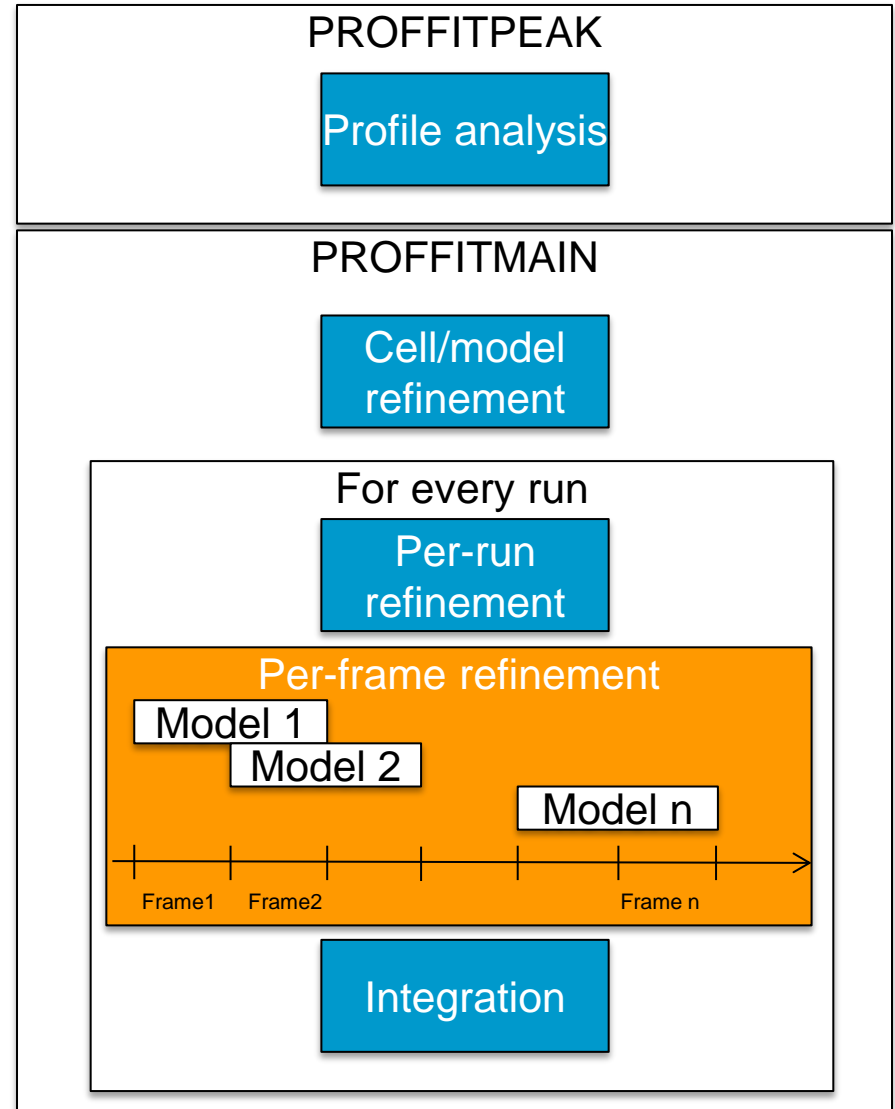
Per-run
refinement

Integration

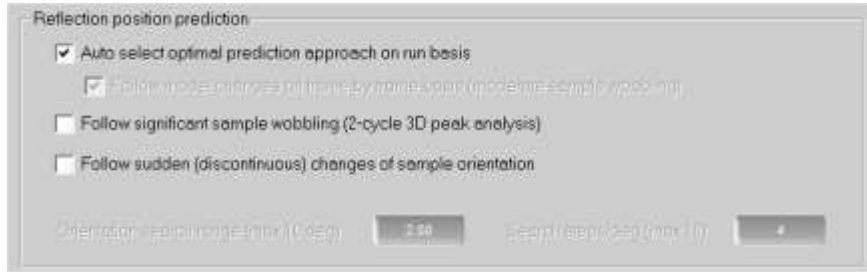
Model refinement options



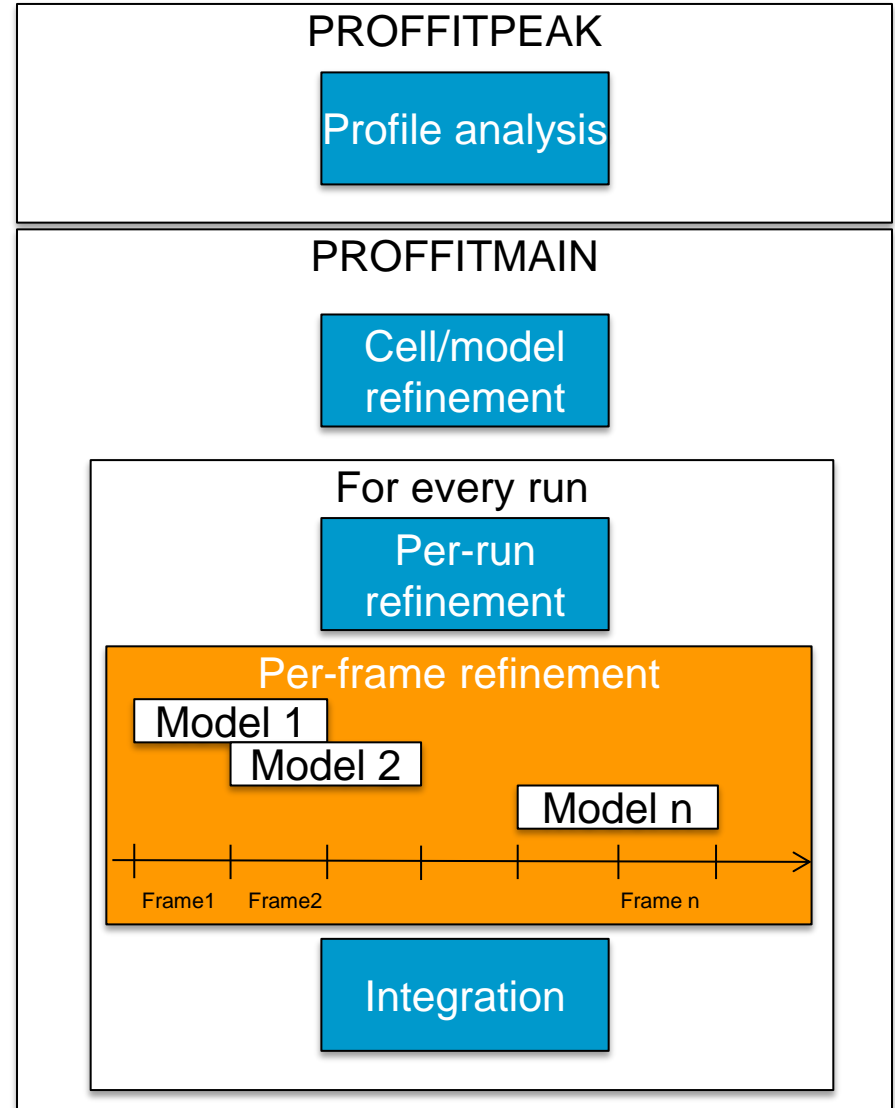
- „Per-frame” models
- Follows smooth crystal moving (“wobbling”) with respect to the beam/detector
- Use if the indexation after peak hunting is above 90%, but spots are not fully covered by masks during integration



Model refinement options



- Auto selects the optimal approach on run basis! Best default option



Model refinement options

Reflection position prediction

☒ Auto select optimal prediction approach on run basis

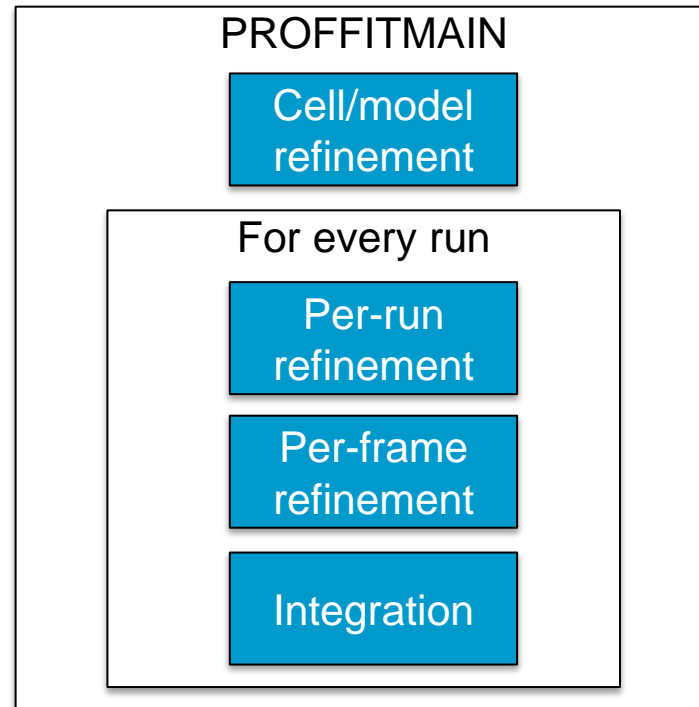
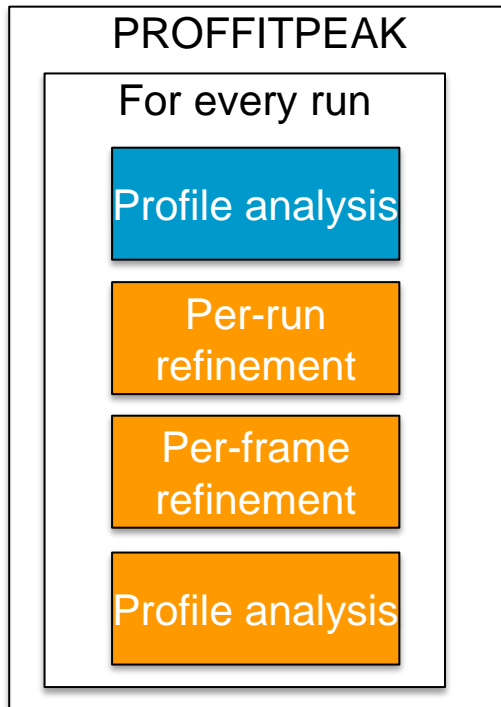
☒ Follow model changes on frame by frame basis (moderate sample wobbling)

☒ Follow significant sample wobbling (2-cycle 3D peak analysis)

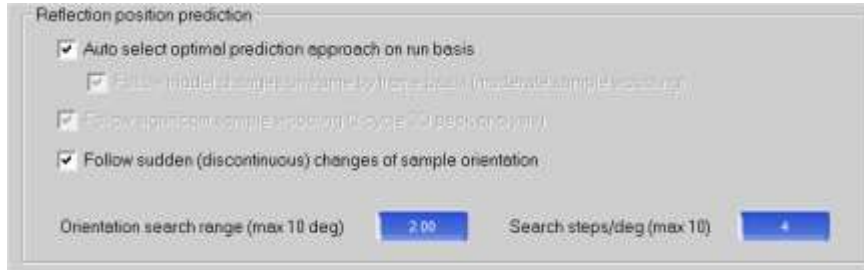
☐ Follow sudden (discontinuous) changes of sample orientation

Orientation search range (max 10 deg) Search steps/deg (max 10)

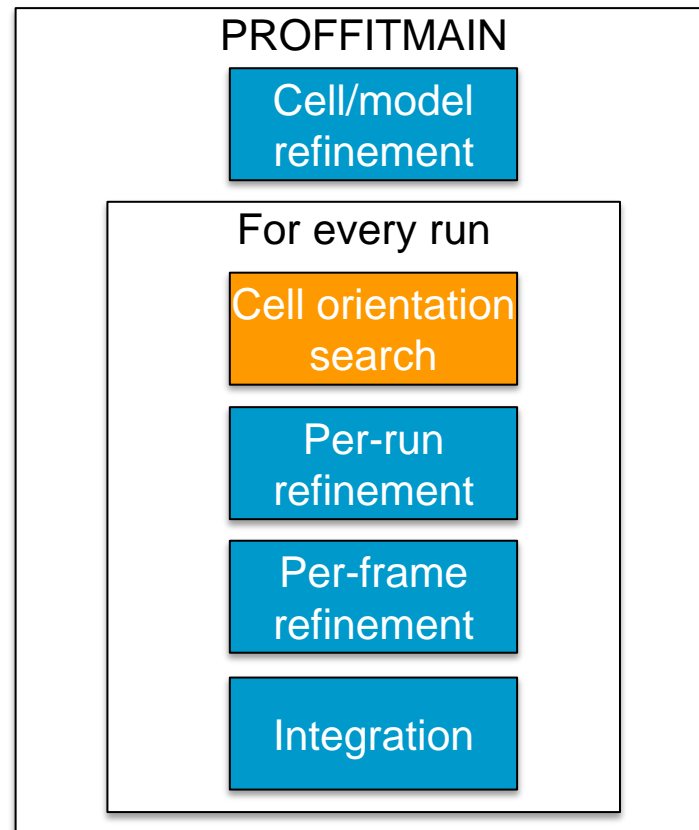
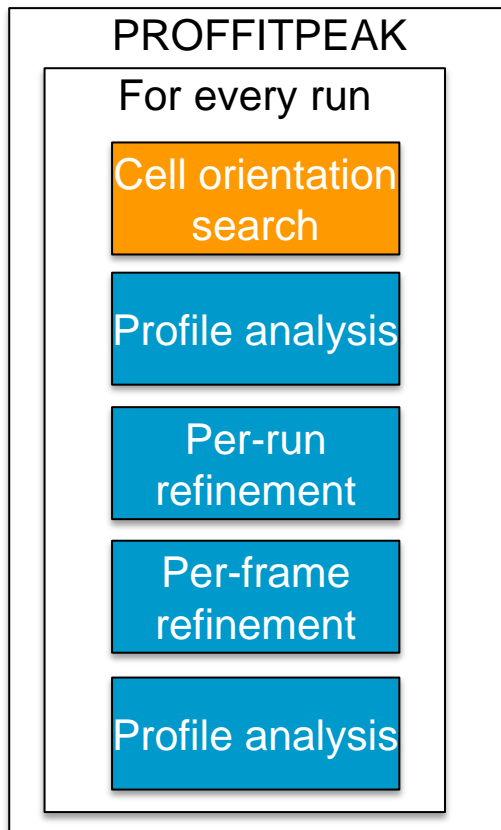
- 2-cycle PROFFITPEAK
- Covers more difficult “wobbling” cases



Model refinement options

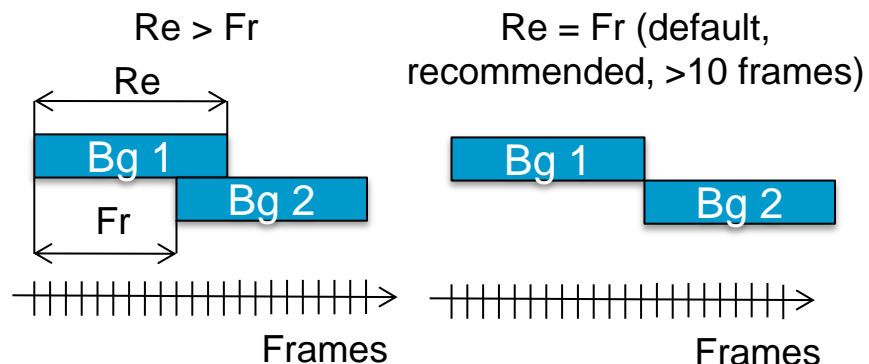


- Model refinement with matrix orientation search
- For discontinuous sample jumps (LT, flexible holder, not fixed properly, etc.)



Data reduction wizard – background

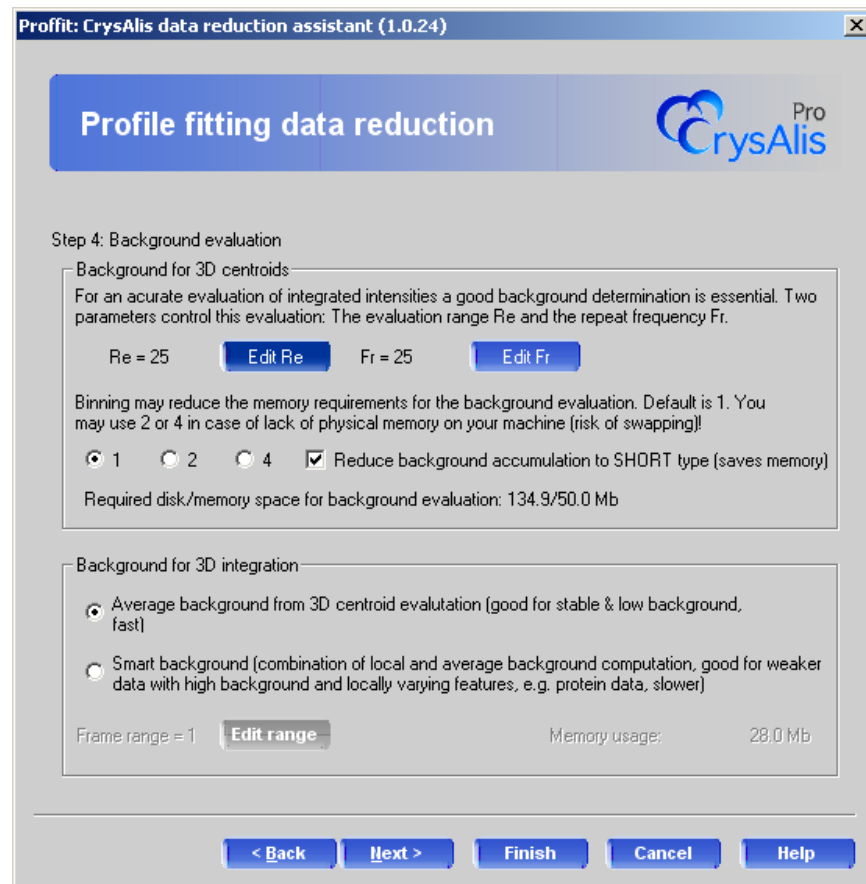
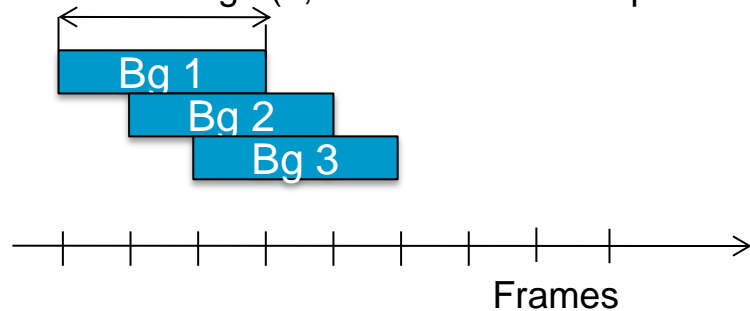
- Average background ($Re > 10$ frames)



- Smart background:

- local background recomputed on every frame and fitted individually for every spot

Frame range (1, 3 or 5 – more is impractical)



Optimal data – hints at data reduction time

- Check for experiment artifacts (empty frames, trips)
- Apply Bravais lattice where obvious
- Special pars
 - Use bad reflection filter
 - Use reduced profile size if overlapping
 - Incidence correction, prediction accuracy
- Smart background on high background data
- xx proffitloop



Special pars

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- 0.79
2theta (deg): 0.00-153.74

☒ Reject reflections with bad profiles (e.g. for HP data)

[I/sig > 10](#) & [Profile agreement < 0.8](#)

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

Extinction rules

No extinction rules specified [Show rules](#)

HINT: You can use DC EXTINGT to add extinction rules and DC CLEAREXTINCT to remove selected or all rules from the list

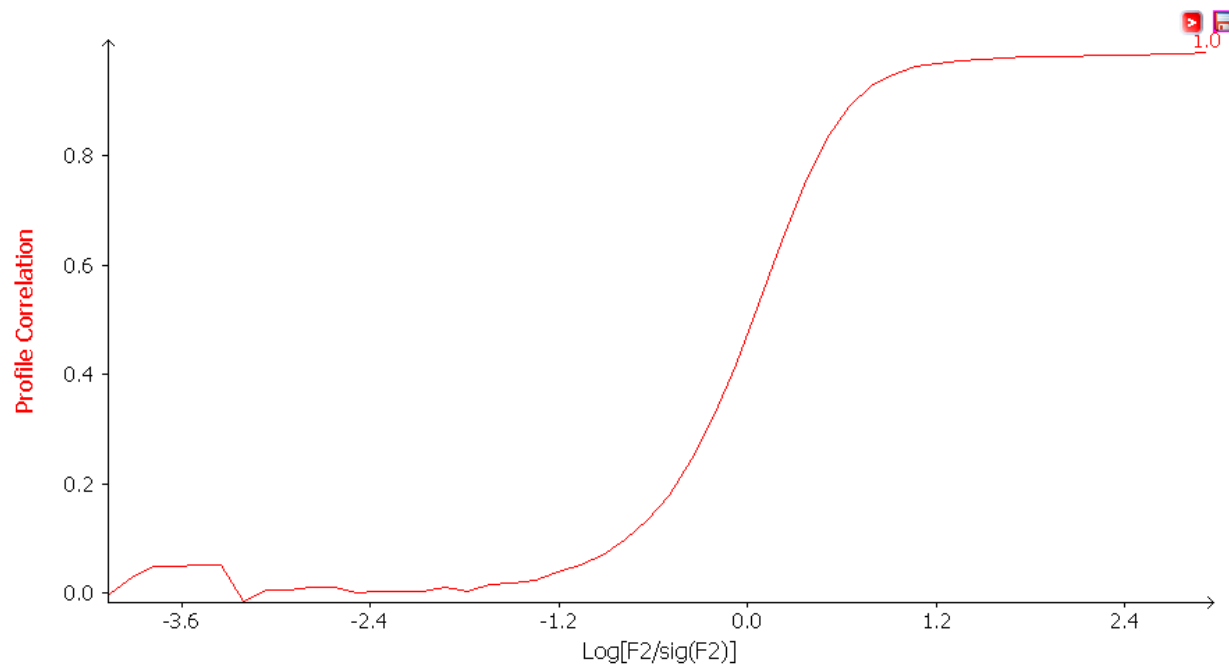
[OK](#) [Cancel](#)

3D profile fitting

- Distorted rotation method data is mapped to 'Kabsch-space'. Similar like XDS, but more complex
- Strong reflection data serve as reference profile. No shape assumption is made!
- All data is profile fitted to the 'nearby' reference profile. For strong data this means summation, for weak filtering
- You may choose to take less/more than 4sig of reference profile

Special pars- outliers

- Filter intruders by correlation coefficient.
- All strong reflections are self similar.



Special pars - incidence

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

Profile size analysis (per incidence angle)

Incidence Average profile size - assuming Gaussian shape (in degrees)					
angle (deg)	# of peaks	sig_e1 (stddev)	sig_e2 (stddev)	sig_e3 (stddev)	
-----	-----	-----	-----	-----	
0-12.6	769	1.639 (0.304)	1.430 (0.233)	1.757 (0.880)	
12.7-18.1	769	1.624 (0.354)	1.430 (0.242)	1.649 (0.875)	
18.1-22.5	769	1.624 (0.357)	1.431 (0.258)	1.572 (0.828)	
22.5-26.2	769	1.594 (0.369)	1.414 (0.258)	1.542 (0.839)	
26.2-29.9	769	1.627 (0.372)	1.410 (0.282)	1.440 (0.736)	
29.9-32.8	769	1.630 (0.364)	1.382 (0.255)	1.391 (0.719)	
32.8-35.8	769	1.594 (0.341)	1.341 (0.267)	1.392 (0.708)	
35.8-38.9	769	1.632 (0.349)	1.337 (0.266)	1.315 (0.594)	
38.9-41.9	769	1.638 (0.330)	1.294 (0.269)	1.303 (0.659)	
41.9-51.6	769	1.618 (0.323)	1.221 (0.270)	1.188 (0.610)	
-----	-----	-----	-----	-----	
0-51.6	7690	1.622 (0.347)	1.369 (0.269)	1.455 (0.769)	

Fitted profile normalization line parameters

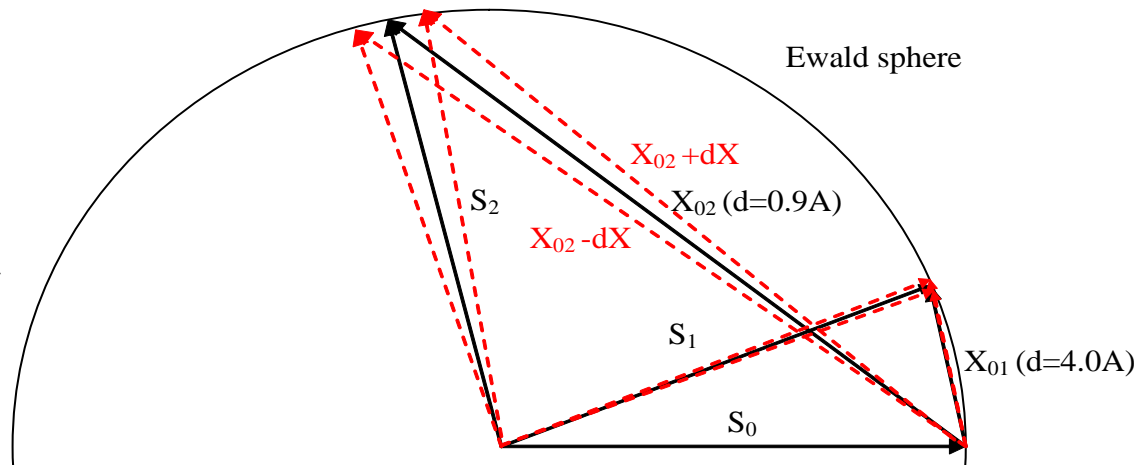
e1 dimension: a=0.0022 b=0.99

e2 dimension: a=-0.0019 b=1.08

e3 dimension: a=-0.0136 b=1.46

Prediction uncertainty – problem

- Problem lies in spot prediction uncertainty
- Higher inaccuracy at high theta



For profile size of 0.8 deg integration mask size is 8 pixels at incidence angle 0 deg (Atlas detector, 2x2 binning, at 55 mm)

Detector theta (deg)	Std dev of misprediction (deg)	Std dev of misprediction (pix)	Max misprediction (pix)
11	0.014	0.13	0.78
24	0.02	0.2	1.2
40	0.025	0.25	1.5
111	0.04 – 0.08	0.4 – 0.8	2.4 – 4.8

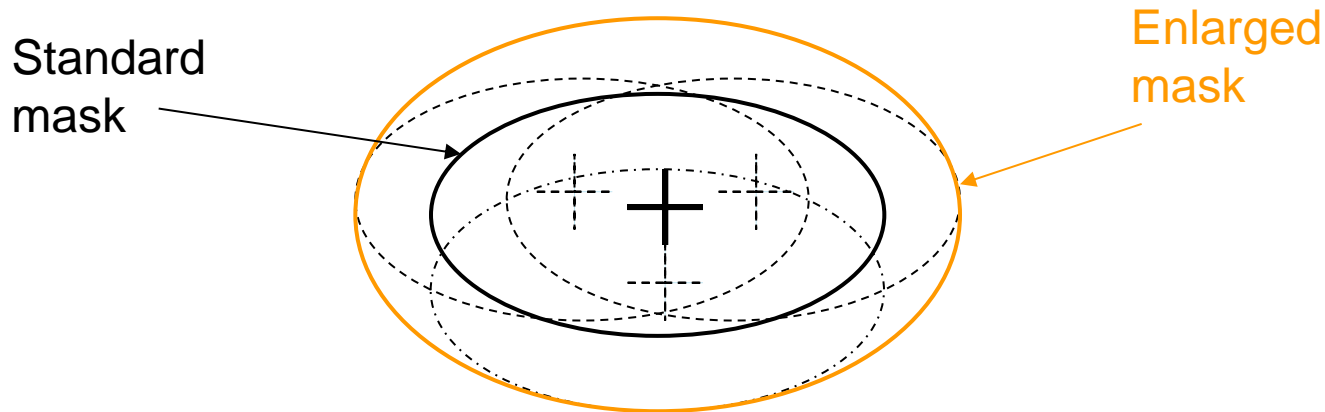


Prediction uncertainty – solution

- Estimate prediction uncertainty at given theta

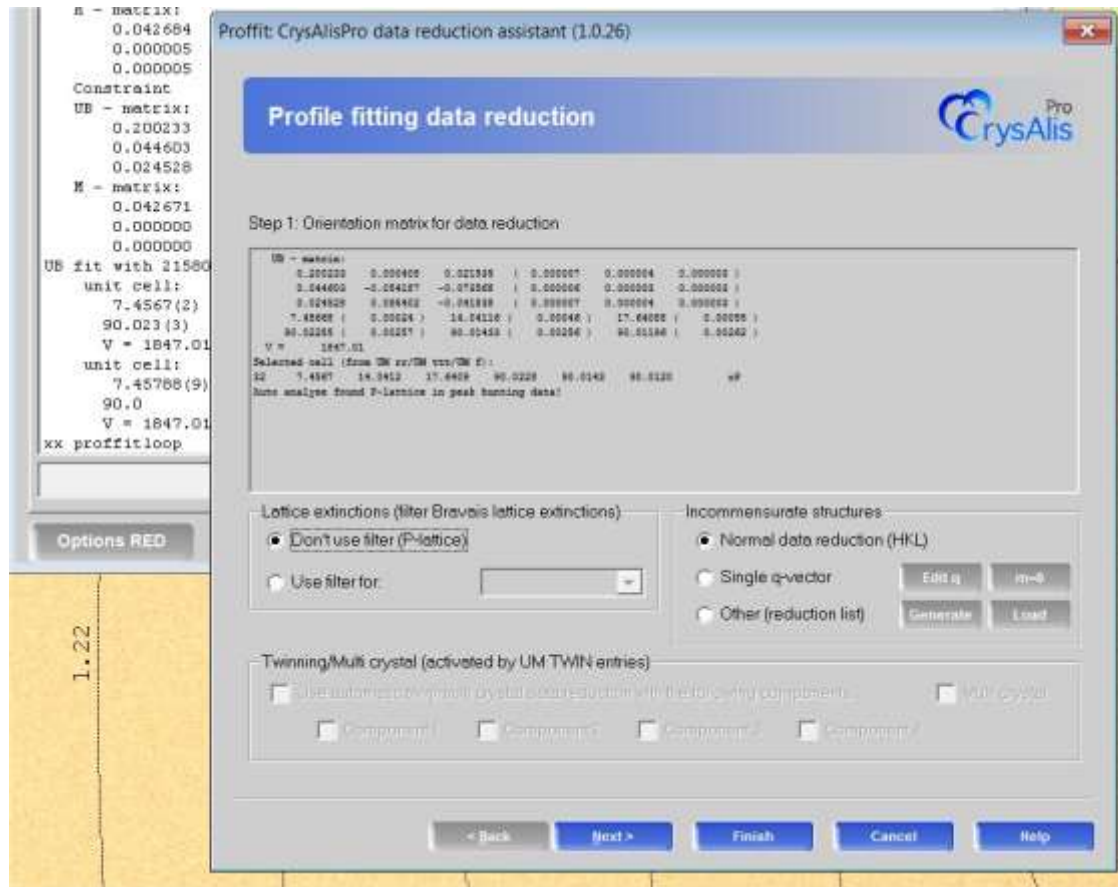
Prediction accuracy statistics (in degrees)				
Resolution	# of peaks	Average prediction error (in degrees)		
		delta_e1 (stddev)	delta_e2 (stddev)	delta_e3 (stddev)
1.35-1.22	82	0.002 (0.020)	0.001 (0.013)	0.003 (0.170)
1.22-1.14	82	-0.002 (0.025)	0.009 (0.016)	0.020 (0.165)
1.14-1.08	82	0.003 (0.024)	0.004 (0.020)	0.020 (0.175)
1.08-1.04	82	-0.012 (0.034)	-0.002 (0.021)	-0.026 (0.153)
1.04-0.99	82	0.001 (0.026)	-0.003 (0.021)	-0.018 (0.153)
0.99-0.95	82	-0.007 (0.033)	-0.005 (0.030)	-0.004 (0.147)
0.95-0.91	82	-0.005 (0.038)	-0.009 (0.029)	-0.001 (0.132)
0.91-0.87	82	-0.008 (0.038)	-0.015 (0.041)	-0.002 (0.127)
0.87-0.84	82	0.006 (0.038)	-0.012 (0.041)	-0.016 (0.106)
0.84-0.80	73	0.005 (0.051)	0.017 (0.087)	0.002 (0.103)
1.35-0.80	811	-0.002 (0.034)	-0.002 (0.038)	-0.002 (0.146)

- Enlarge integration mask according to prediction uncertainty

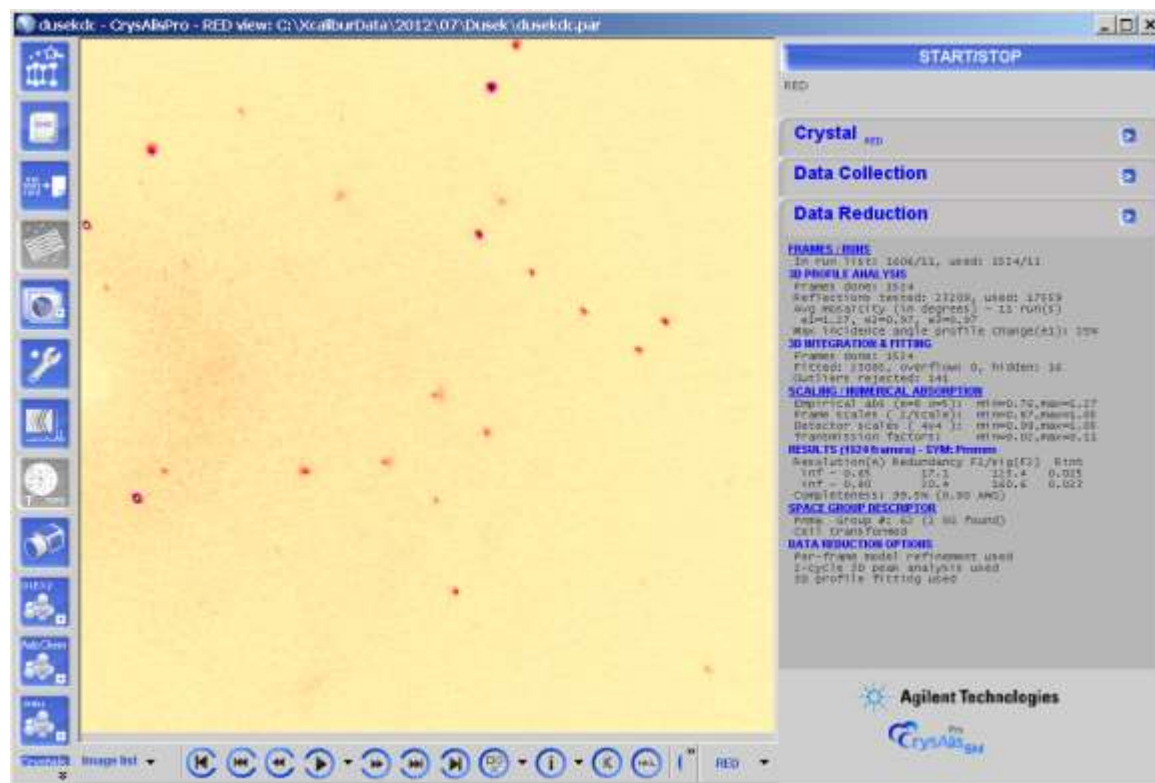
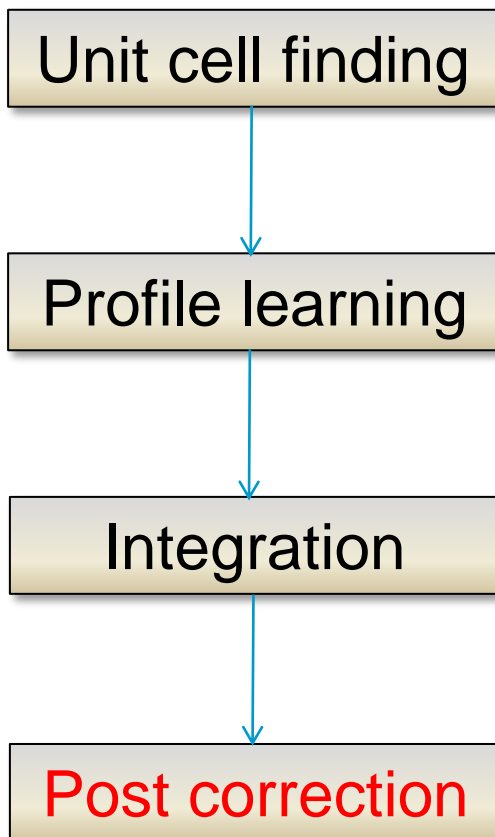


Automatic multi scenario data processing

- xx proffitloop
- Some 32+ processing combinations



Typical data reduction sequence



Data reduction (reflection integration) vs. data finalization

- Integration results land in: *.rrpprof
- Finalization results are *.rrpprof transformed to hkl and cif files.



Post correction motivation

- Frame information to HKL file information
- Improve I/σ of redundant information
- Reduce 'systematic effects'

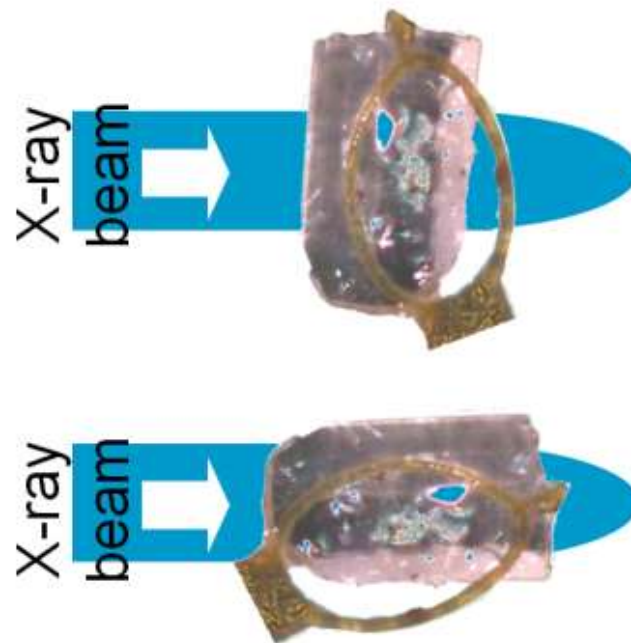
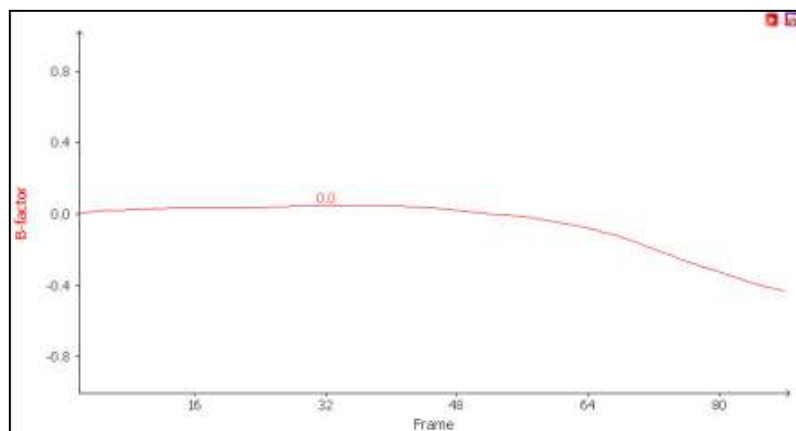
Note:

Post corrections can only correct observed data!



Systematic effects/Noise sources

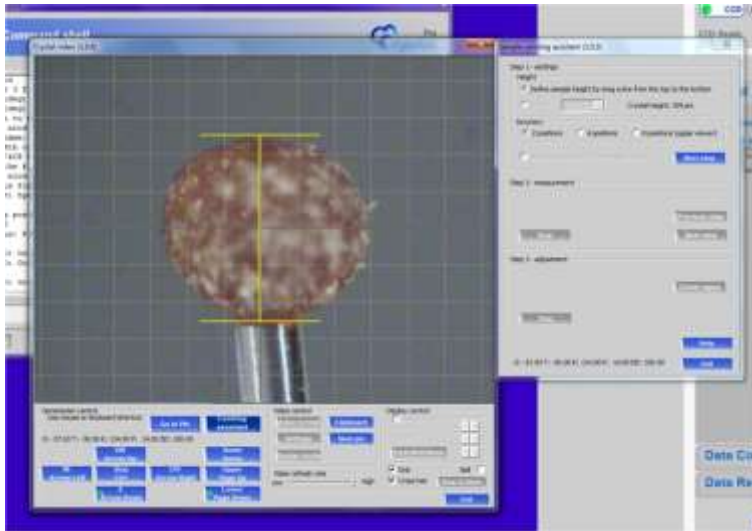
- Absorption (sample/holder)
- Beam illumination effects
- Sample centering
- Sample decay
- Experiment effects: cryo, generator
- Mounting technique



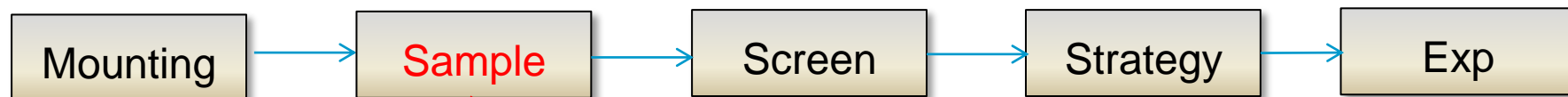
Data acquisition sequence: correction requisites



- CrysAlis^{Pro} 'Centering assistant' reduces centering error for anisotropic samples
- Height estimator 'remembered' for automatic shape generator
- Movie registers sample shape for later evaluation (triggered at experiment time)



Data acquisition sequence: correction requisites



- Sample description
- Chemical compound

Mu-calculator (1.0.3): Absorption coefficient in mm⁻¹

Cell and wavelength:
6.40880 8.37274 11.27700 88.886 18.881 88.894 887.9
Mo-radiation

Z:

Chemical formula: (e.g. C11 H18 O12)
Repeat (if slow elements, separate elements by space)

Formula:
[element(s)]
O=16.00(15.68); Se=4.80(18.32); Co=8.88(65.03)
Formula wt: 1676.10 Molar wt: 17.89
Density: 4.86 g cm⁻³ 1.83
ρ(Se): 732.80 Å/mol 23.71 Non-relativ: 21.71

my(mn-1) 17.89430 ☐ Editing

Edit crystal sample description (1.0.3)

Colour and description

Qualifier	Intensity	Colour
Sample colour: <input type="text" value="metallic"/>	<input type="text" value="light"/>	<input type="text" value="white"/>

Sample shape:

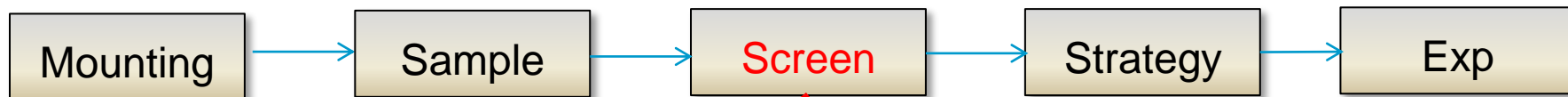
Radius (mm): Length (mm):

Size (mm)

Min	Mid	Max
<input type="text" value="0.166"/>	<input type="text" value="0.293"/>	<input type="text" value="0.350"/>

NOTE: Crystal size is computed based on defined shape.

Data acquisition sequence: correction requisites



- CrysAlis^{Pro} screening tool gives unit cell in <10sec
- Selection of sample with proper diffraction power and sample quality

SM Screening

Screening

Mount Screening >

PEAKS
UB fit with 70 obs out of 70 (100.0%)

UNIT CELL (CSD: 14-4371)
PG: mm orthorhombic P
5.97(2) 9.05(2) 18.356(17)
90.02(13) 90.26(15) 90.2(2)
V = 992(5)

QUALITY

Resolution(A)	N	I/sig	I/sig _o
inf - 1.23	91	24.0	26.5
1.28- 1.23(last)	10	12.9	14.2

Well diffracting sample

Diff. limit: beyond 1.23 (theta res. limit) for I/sig=2.0
Mosaicity: e1=1.2, e2=1.2, e3=2.0 (deg), Iso= 1.49 (deg)

Experiment - Complete data for publication

Name: exp_209

Detector=52.0mm, Res. = 0.837Ang, I/sig.=15.0, width=1.0deg, Movie, cryo off, Strategy: Complete data (default mode), Exposure: 1.0s 4.0s

Exposure time: 1.0 s

Start Pre-Exp. (5 min) Edit

Goniometer

Omega	Theta	Kappa	Phi	Distance
20.0	-35.0	0.0	0.0	52.0

Generator

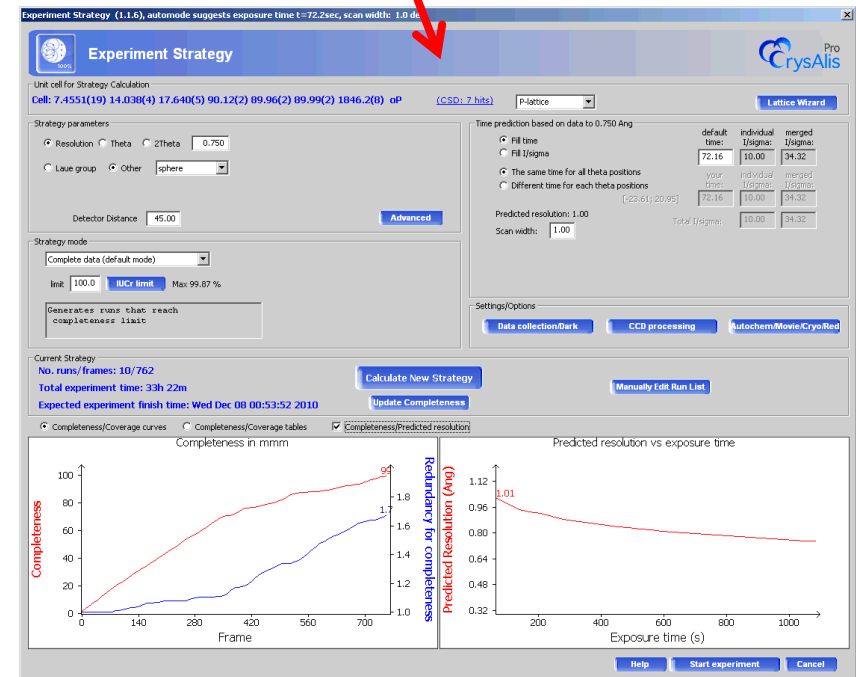
kV	mA
50.0	0.80



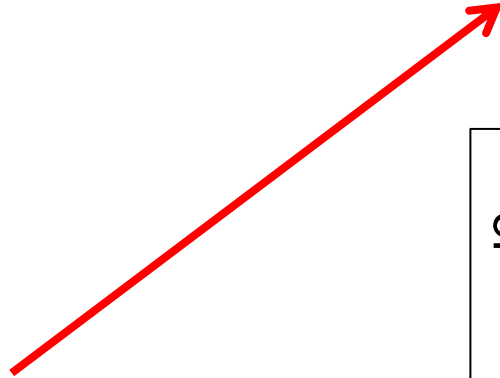
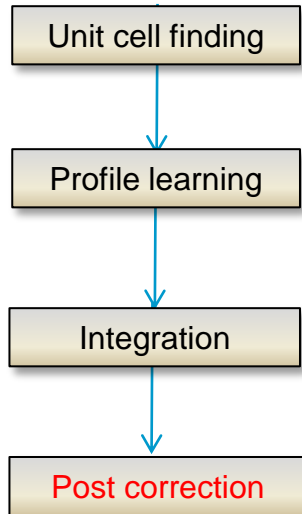
Data acquisition sequence: correction requisites



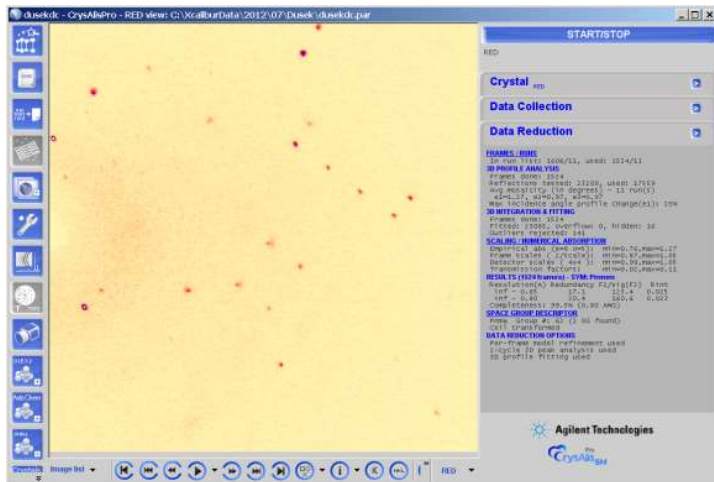
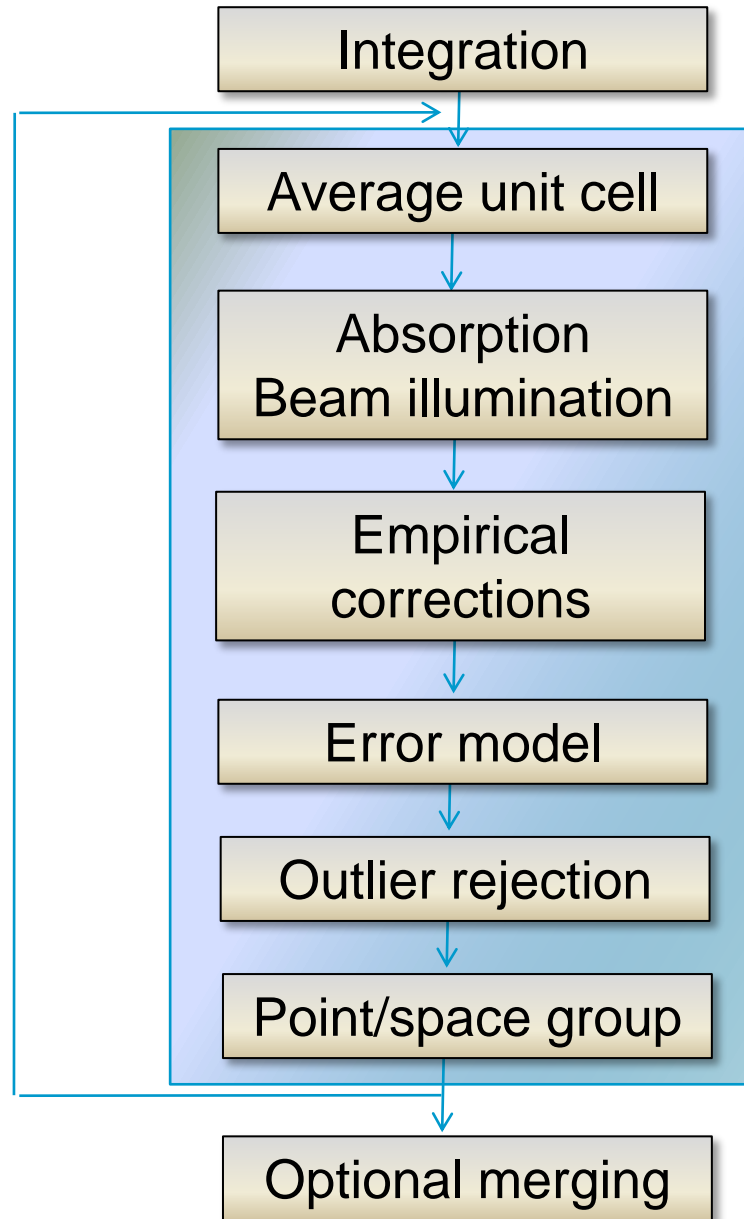
- Based on unit cell, Laue symmetry and chemical constraints the proper strategy is computed
- I/σ calculator helps to find proper 'detectivity level' for the sample
- Target redundancy can be set



Post correction layout

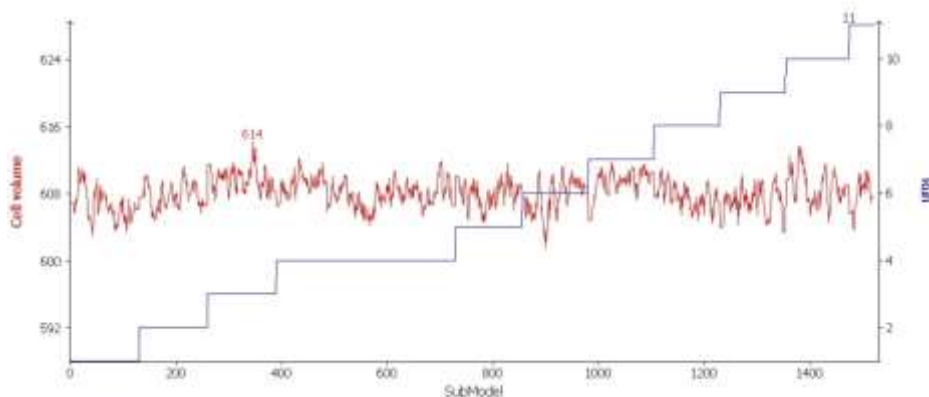
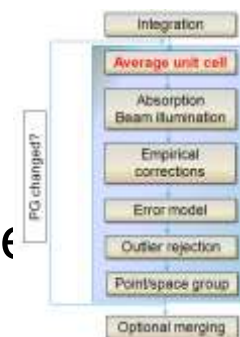


PG changed?



Average unit cell from all data

- Post integration: most accurate model description available
- 3D profiles known
- All experimental aberrations are corrected for



Crystal RED

EXPERIMENT
dusekdc

CHEMICAL FORMULA
O16 Se4 Cs8 Z=1.0

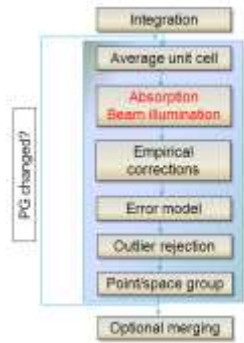
LATTICE
Current cell (CSD: 0 hits)
6.43868(15) 8.3727(2) 11.2770(3)
89.996(2) 90.003(2) 89.994(2)
V = 607.94(2)
Constrained cell
6.43748(11) 8.37306(9) 11.27867(12)
90.0 90.0 90.0
V = 607.936(14)
Symmetry
Laue class: 2/m2/m2/m P-lattice

AVERAGE UNIT CELL FROM PROFFIT
Constrained cell (17521 obs)
6.43748(11) 8.37305(9) 11.27867(12)
90.0 90.0 90.0
V = 607.936(14)

FINAL UNIT CELL FOR SELECTED SG
Constrained cell (17521 obs)
8.37306(9) 6.43748(11) 11.27867(12)
90.0 90.0 90.0
V = 607.936(14)

'Face-based': Absorption, beam illumination

- Clark & Reid analytical formulation (allows shape optimization)¹
- Gaussian grid, numerical integration²
- Beam illumination
- High Pressure: Gasket shadowing, diamond correction³



Note:

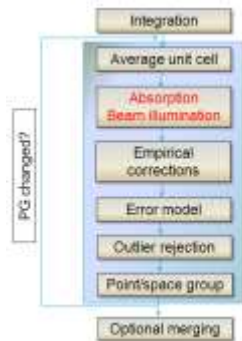
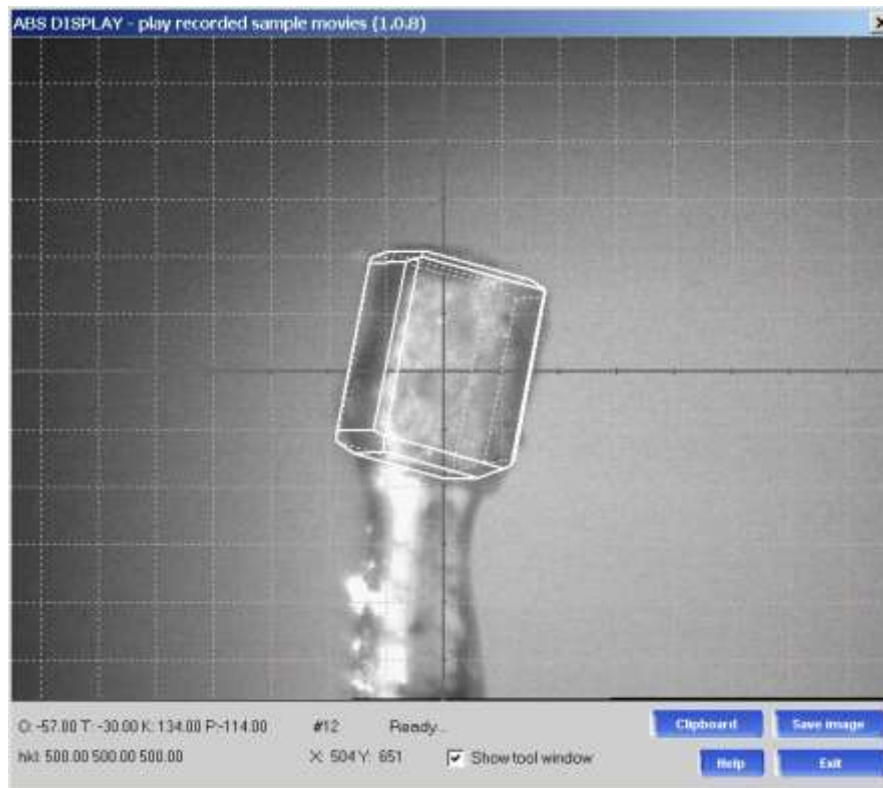
Shape description and chemical composition have to be known!

¹Clark, R. C. & Reid, J. S. (1995). Acta Cryst. A51, 887-897; ²Busing, W. R. & Levy, H. A. (1957). Acta Cryst. 10, 180-182;

³Angel, R. J. (2004). J. Appl. Cryst.. 37, 486-492

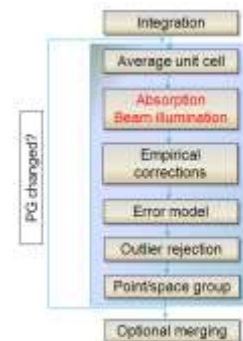
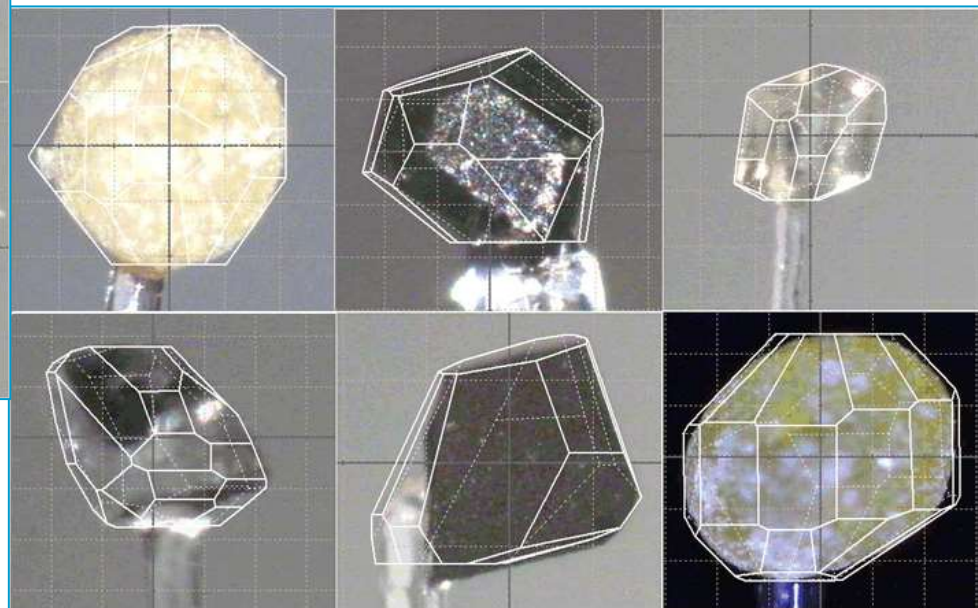
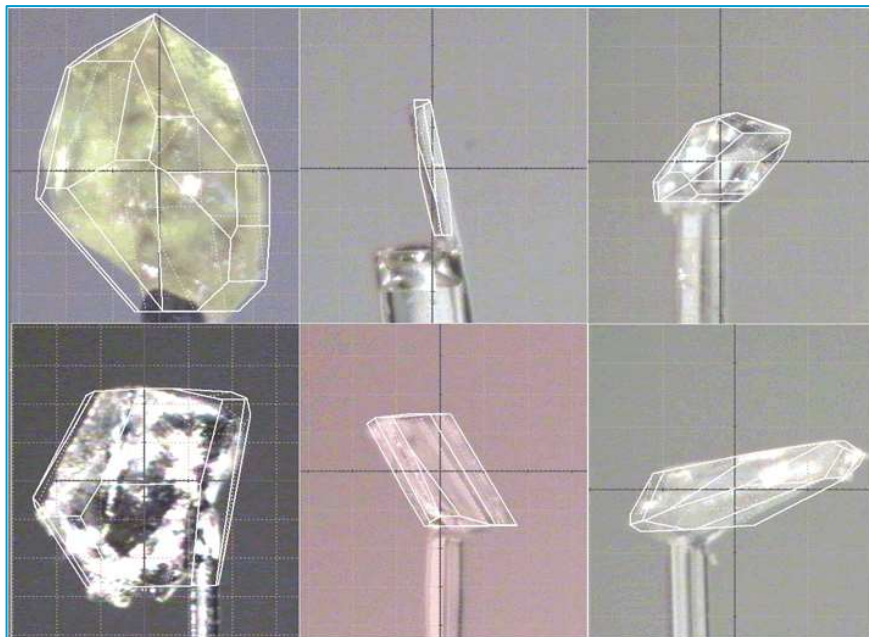


'Face-based': Absorption, beam illumination

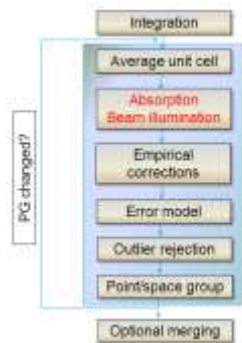


Shape building takes time... Automatic?

- Auto shape



'Face-based': Absorption, beam illumination



Type of absorption correction selector

☒ Analytical absorption correction after Clark & Reid

☐ Gaussian grid absorption correction (Numerical integration)

Grid dimensions:

x: 10 y: 10 z: 10 ☒ Equal in all directions

Gaussian grid orientation:

☒ Crystal system

☐ Longest diagonal

☐ Longest edge

Beam profile correction

☐ Apply beam profile correction

☒ Graphite monochromator (Enhance) - 1D horizontal gaussian

Collimator size: ☐ 0.2 mm ☐ 0.5 mm ☐ 0.6 mm ☒ 1.0 mm

☐ Mirror (Nova, Nova Ultra) - 2D gaussian

☐ Flat beam

Beam size - FWHM for Gaussian profile (mm):

0.50 Edit horizontal beam dia

0.50 Edit vertical beam dia

OK Cancel

Type of absorption correction selector

☐ Analytical absorption correction after Clark & Reid

☒ Gaussian grid absorption correction (Numerical integration)

Grid dimensions:

x: 10 y: 10 z: 10 ☒ Equal in all directions

Gaussian grid orientation:

☒ Crystal system

☐ Longest diagonal

☐ Longest edge

Beam profile correction

☐ Apply beam profile correction

☒ Graphite monochromator (Enhance) - 1D horizontal gaussian

Collimator size: ☐ 0.2 mm ☐ 0.5 mm ☐ 0.6 mm ☒ 1.0 mm

☐ Mirror (Nova, Nova Ultra) - 2D gaussian

☐ Flat beam

Beam size - FWHM for Gaussian profile (mm):

0.50 Edit horizontal beam dia

0.50 Edit vertical beam dia

OK Cancel

¹Clark, R. C. & Reid, J. S. (1995). Acta Cryst. A51, 887-897; ²Busing, W. R. & Levy, H. A. (1957). Acta Cryst. 10, 180-182

'Face-based': Absorption, beam illumination

Type of absorption correction selector

☐ Analytical absorption correction after Clark & Reid

☒ Gaussian grid absorption correction (Numerical integration)

Grid dimensions

x: 10 y: 10 z: 10 ☒ Equal in all directions

Gaussian grid orientation

☒ Crystal system

☐ Longest diagonal

☐ Longest edge

Beam profile correction

☒ Apply beam profile correction

☐ Graphite monochromator (Enhance) - 1D horizontal gaussian

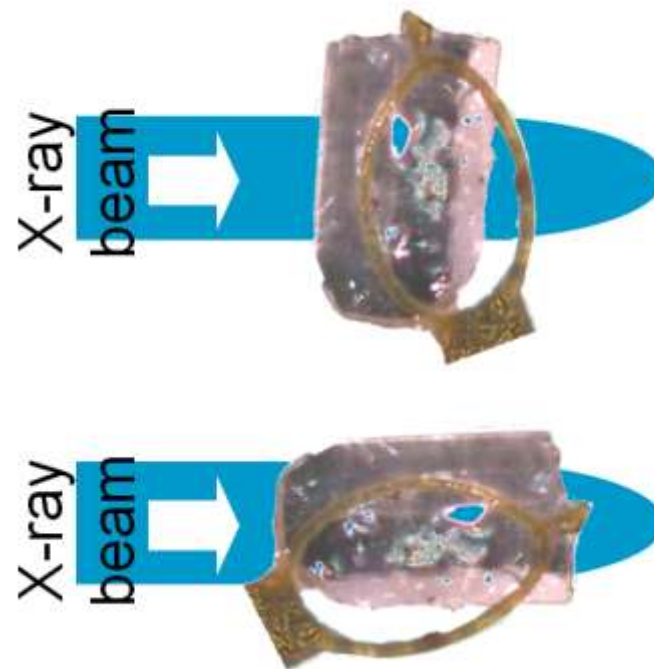
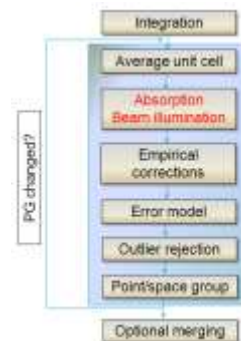
Collimator size: ☐ 0.3 mm ☐ 0.5 mm ☐ 0.8 mm ☒ 1.0 mm

☒ Mirrors (Nova, Mova, Ultra) - 2D gaussian

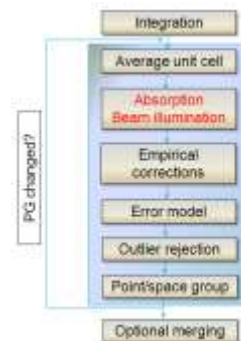
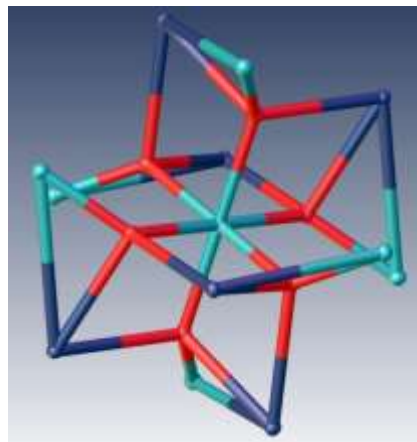
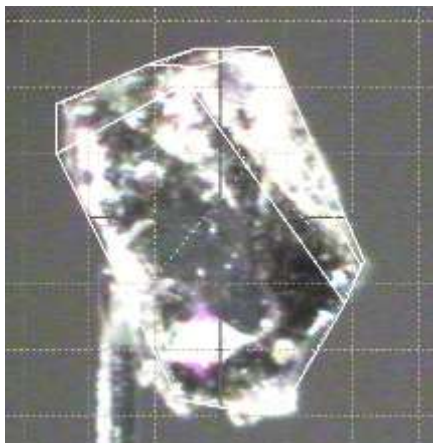
☐ Flat beam

Beam size - FWHM for Gaussian profile (mm): 0.50

0.50



'Face-based': Absorption, beam illumination

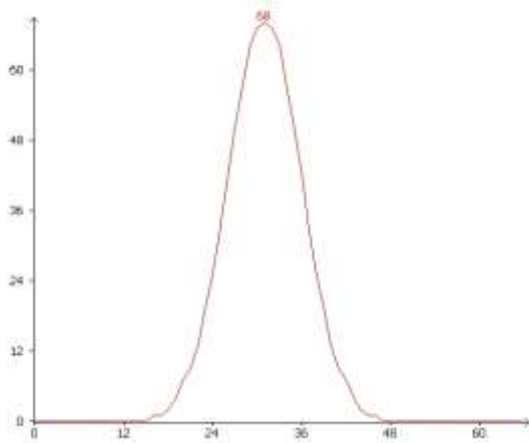


yttrium_mo – inorganic ($\text{Y}_3\text{Al}_5\text{O}_{12}$),
Mo, $\mu=20.3\text{mm}^{-1}$, redundancy=30 (Ia-3d) , Nova micro-source

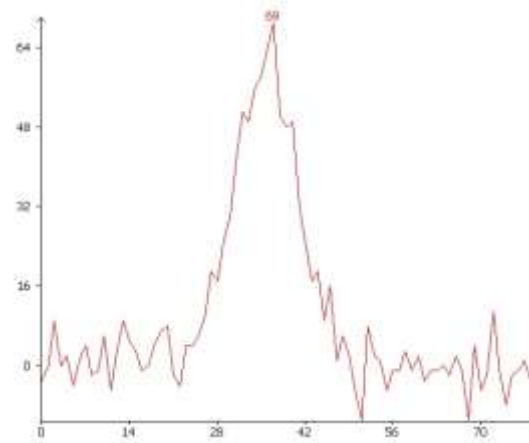
	R_{int}	I/σ	R1
No correction	50%	7.8	8.9%
Absorption + beam illumination	20%	9.8	3.4%



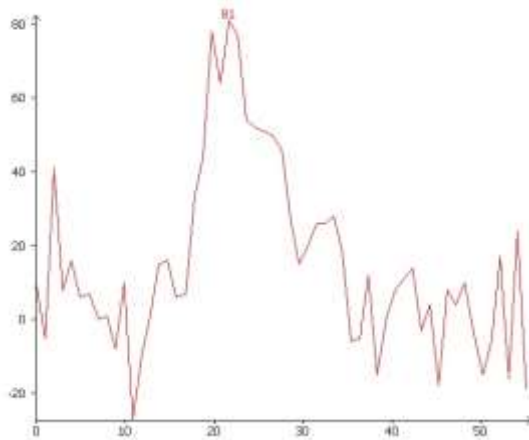
Post corrections can only correct observed data!



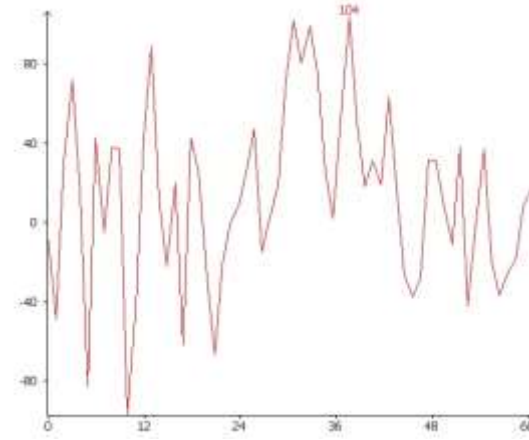
No noise - excellent



Low noise - good



Medium noise - ok



High noise – hopeless!

Strategy dilemma: redundancy vs. time

Experiment Strategy

Unit cell for Strategy Calculation
Cell: 7.4551(19) 14.038(4) 17.640(5) 90.12(2) 89.96(2) 89.99(2) 1846.2(8) oP (CSD: 7 hits)

Strategy parameters
☒ Resolution ☐ Theta ☐ 2Theta 0.750
☐ Laue group ☒ Other sphere
 Detector Distance 45.00

Time prediction based on data to 0.750 Ang
☒ Fill time
☐ Fill I/sigma
☒ The same time for all theta positions
☐ Different time for each theta positions [~23.61; 20.95]
 Predicted resolution: 1.00
 Scan width: 1.00

default time:	individual I/sigma:	merged I/sigma:
72.16	10.00	34.32
your time:	individual I/sigma:	merged I/sigma:
72.16	10.00	34.32
Total I/sigma:	10.00	34.32

Strategy mode
 Complete data (default mode)
 limit 100.0 IUCr limit Max 99.87 %
 Generates runs that reach completeness limit

Settings/Options
 Data collection/Dark CCD processing Autochem/Movie/Cryo/Red

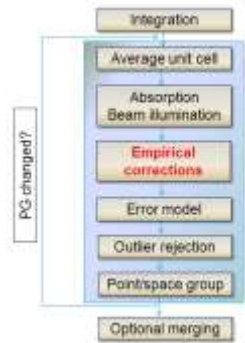
Current Strategy
 No. runs/frames: 10/762
 Total experiment time: 33h 22m
 Expected experiment finish time: Wed Dec 08 00:53:52 2010

☒ Completeness/Coverage curves ☐ Completeness/Coverage tables ☒ Completeness/Predicted resolution

Buttons: Calculate New Strategy, Update Completeness, Manually Edit Run List, Help, Start experiment, Cancel

Empirical correction: logic

- Scaling
- Empirical absorption based on spherical harmonics
- Decay
- Detector sensitivity



$$I_{\text{corr},1} = C_1 I_1 \quad \Delta_1 = I_{\text{aver}} - I_{\text{corr},1}$$

$$I_{\text{corr},2} = C_2 I_2 \quad \Delta_2 = I_{\text{aver}} - I_{\text{corr},2}$$



$$I_{\text{corr},n} = C_n I_n \quad \Delta_n = I_{\text{aver}} - I_{\text{corr},n}$$

$$I_{\text{aver}} = \sum I_{\text{corr_prev},i}$$

Note:
Redundancy is the key ingredient!

Play modes

- Scales
- Empirical
- Decay A, B
- Sensitivity

Scale3 abspack (1.0.7) - Refinement of scales, emp. absorption and sensitivity

hkl-file: D:\Data\protein\remeasure\run1.hkl

Output dir D:\Data\protein\remeasure

Symmetry settings

☐ Use Friedel mates as equivalent even for noncentrosymmetric SG

LS refinement control

SigCut:

☐ Exclude strongest unique reflections (along with all symmetric equivalents)

Frame scaling

☒ Automatic frame scale assignment ☒ Apply frame scaling

How many frames have a common scale?

Variation restraint (ESD):

☒ Reject frame scales < & >

B-factor/A-factor refinement

☒ Refine B-factors

☐ Refine A-factors

Restraint

Empirical absorption correction

☒ Automatic parameter selection for absorption correction

Max even order: Max odd order: 44 parameters

☐ Absorption correction before frame scaling (recommended for strong absorbers)

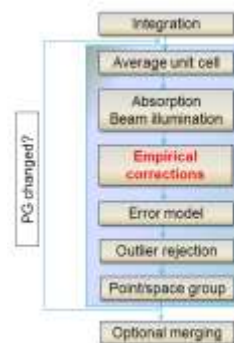
Detector area scaling

How many detector area regions?

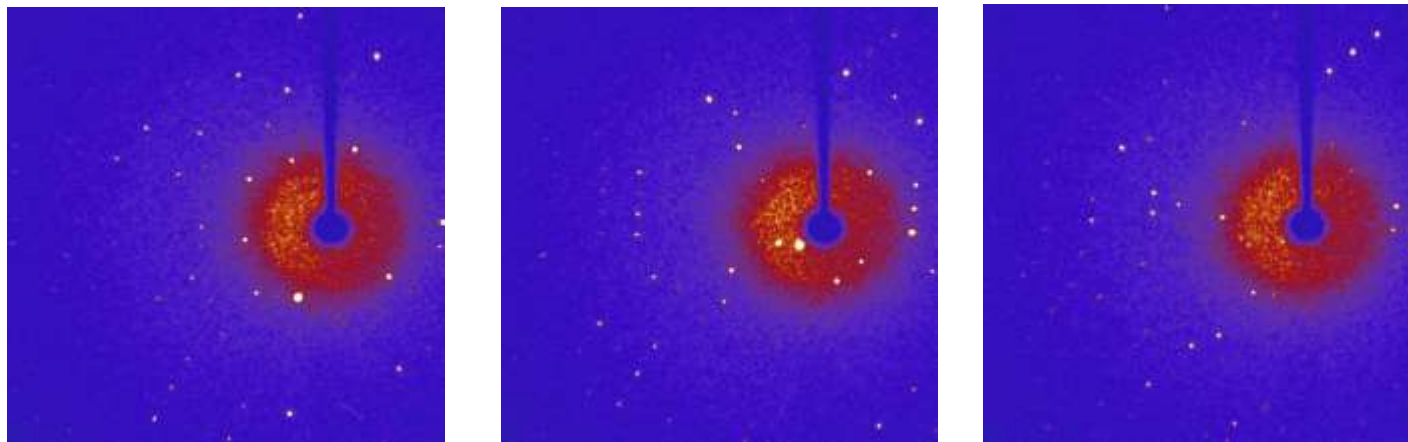
☐ Apply detector correction

Variation restraint (ESD):

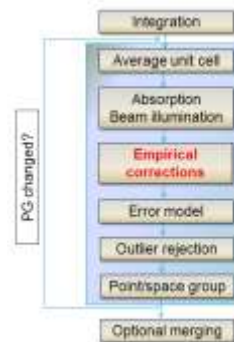
Buttons: Help OK Cancel



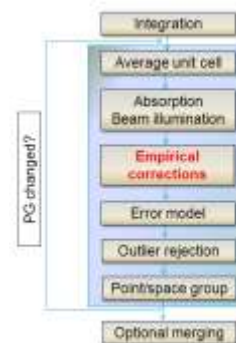
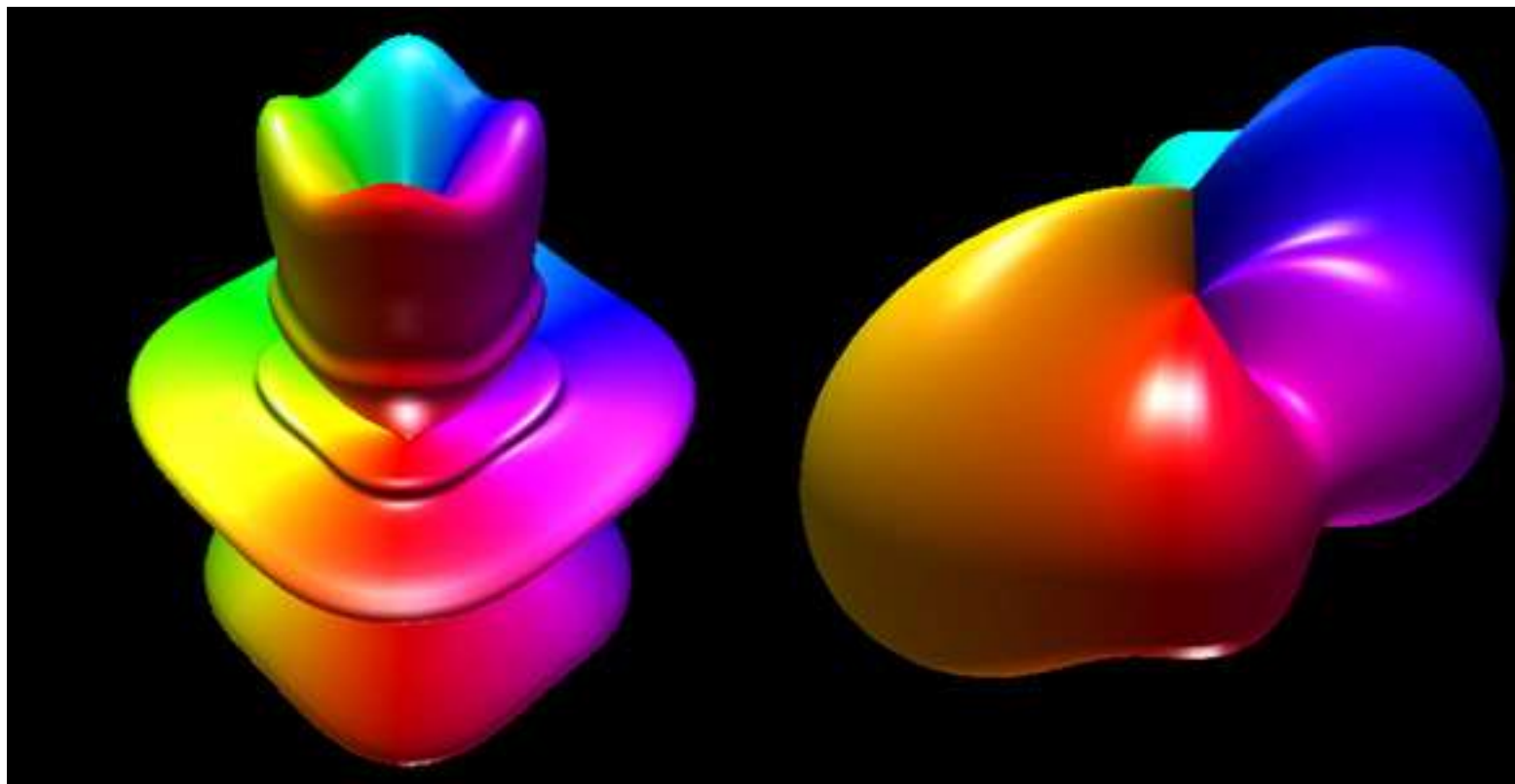
Play modes: scales



Batch

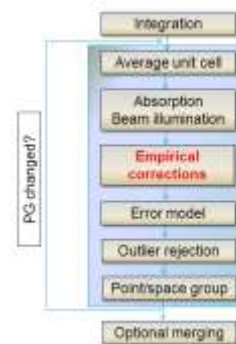
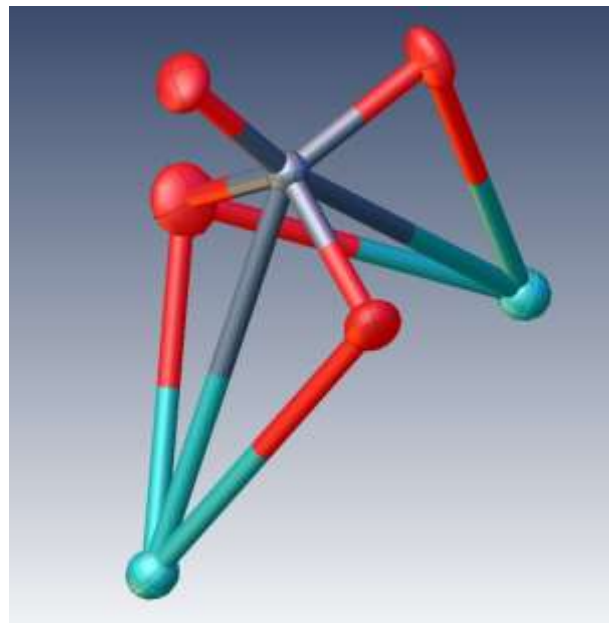
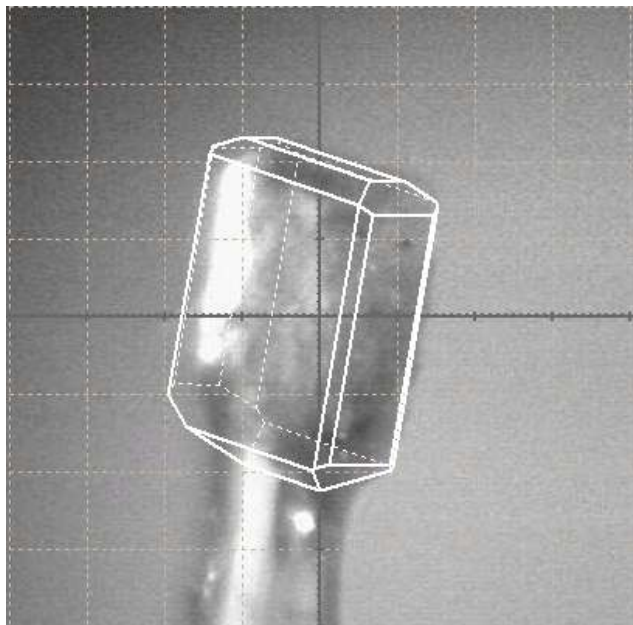


Play modes: Spherical harmonics



<http://paulbourke.net/geometry/sphericalh/> Blessing, R.H. (1995). Acta Cryst. A51, 33-38

Empirical vs. Numeric Absorption



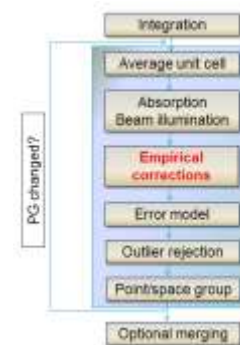
dusekdc (Michal Dusek) – inorganic (Cs_2SeO_4),
Mo, $\mu=17.9\text{mm}^{-1}$, redundancy = 4.5 ($\text{Pna}2_1$)

	R_{int}	I/σ	R1
No correction	14.4%	18	8.3%
Empirical	4%	20.4	3.3%

Empirical vs Numeric Absorption

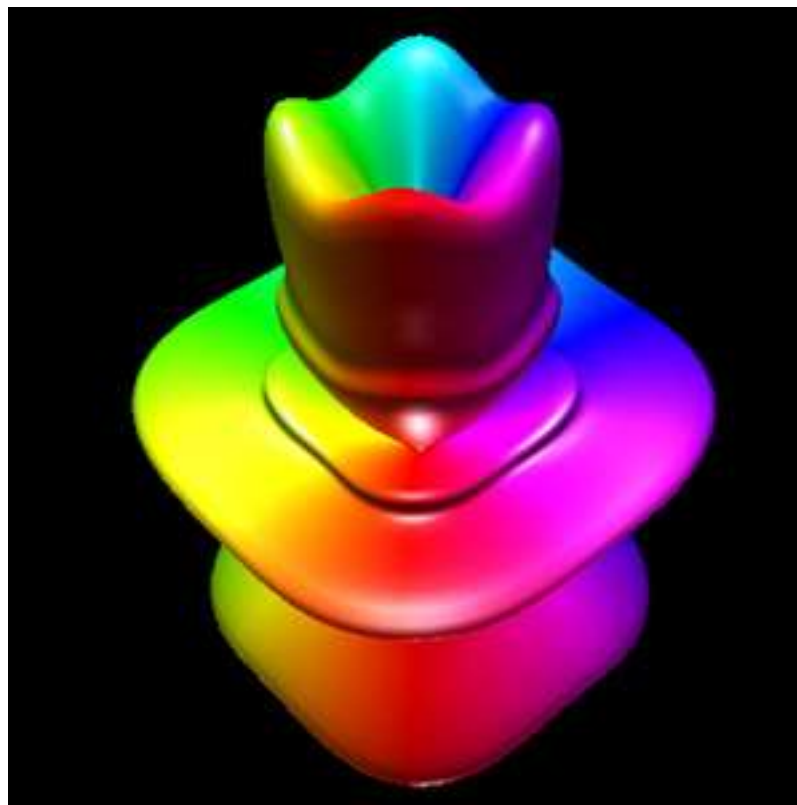
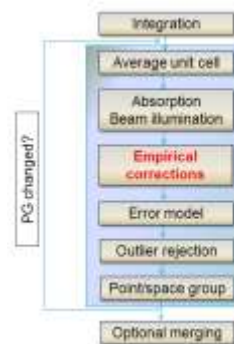
dusekdc (Michal Dusek) – inorganic (Cs_2SeO_4),
Mo, $\mu=17.9\text{mm}^{-1}$, redundancy = 4.5 ($\text{Pna}2_1$)

	R_{int}	I/σ	R1
No correction	14.4%	18	8.3%
Empirical	4%	20.4	3.3%
Numeric (faces)	2.6%	25	1.92%
Both	1.85%	28	1.81%



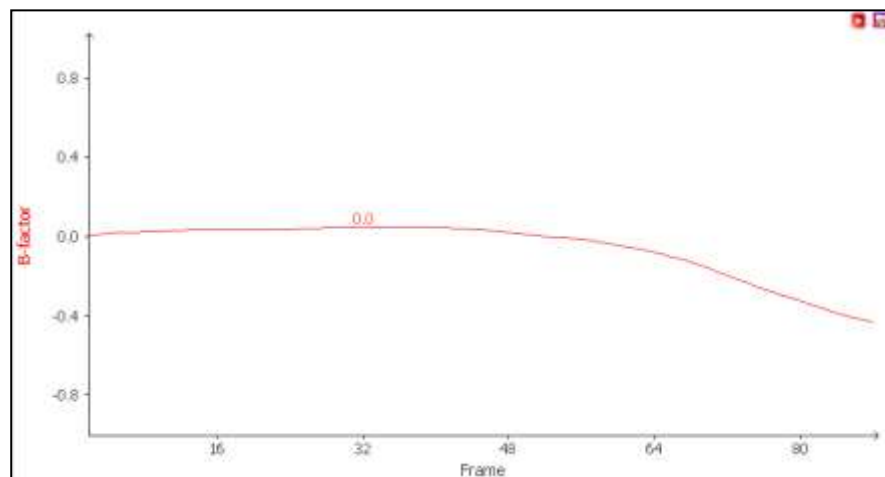
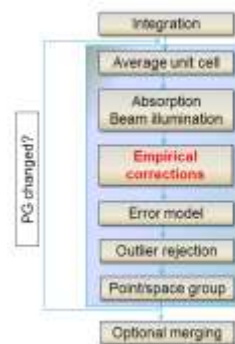
Why?

IMAGE: dusekdc_1_77.img (run: 1 frame: 77)
Omega: 71.0000 Theta: 33.0000 Kappa: 80.0000 Phi: 0.0000

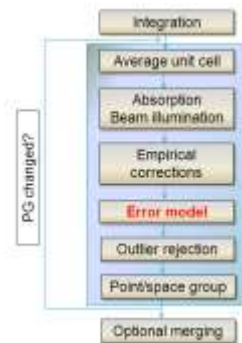


Play modes: Decay and detector sensitivity

$$K_B = e^{-2 \left[B_i \left(\frac{\sin \theta}{\lambda} \right)^2 + A_i \left(\frac{\sin \theta}{\lambda} \right) \right]}$$



Error model fitting



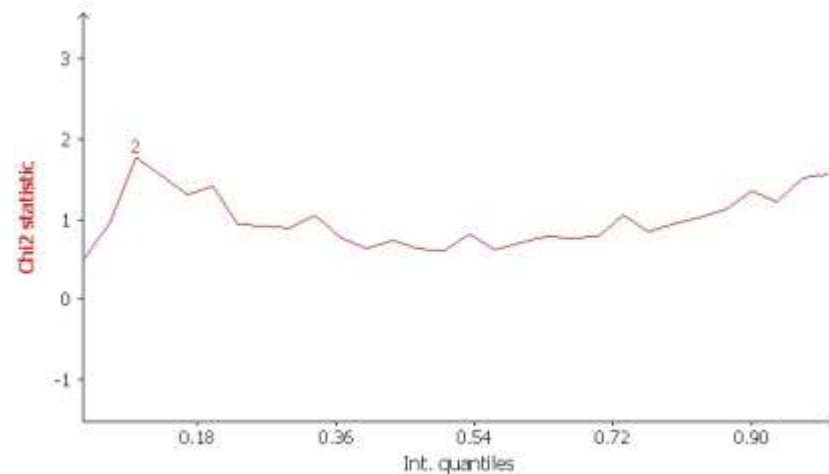
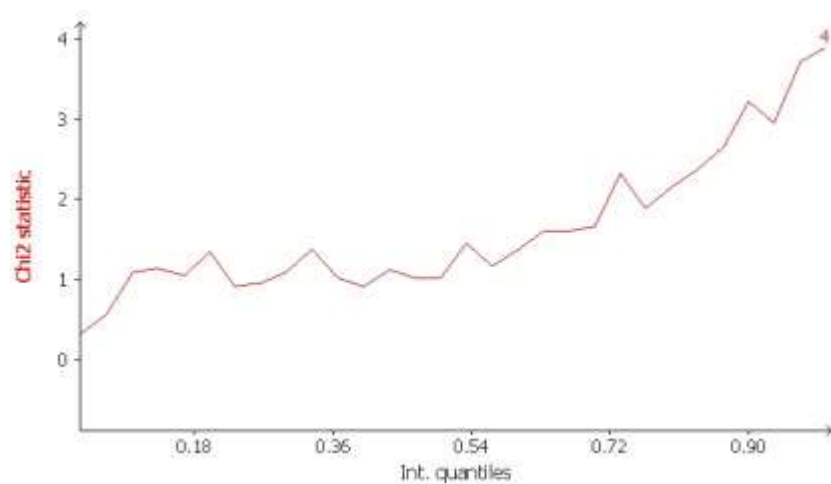
Error model options

Select error model

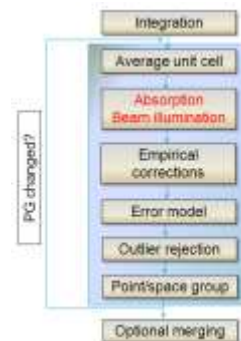
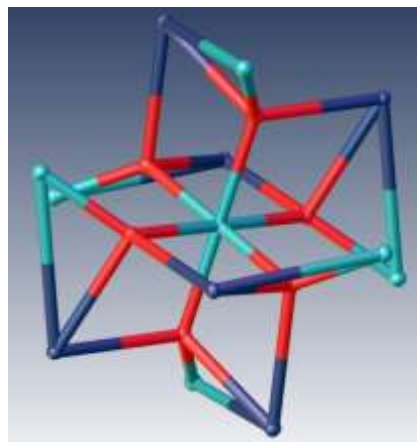
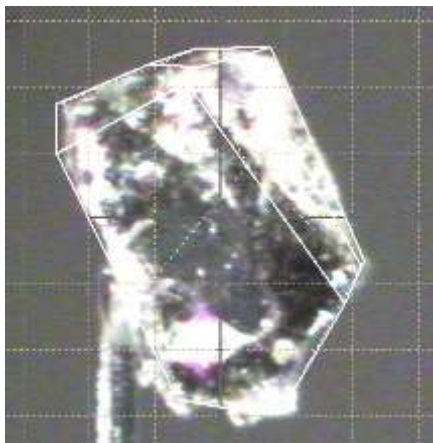
- ☐ Machine error only
- ☐ Scale+ machine error (SADABS like)
- ☐ Gain for signal counts + machine error
- ☐ Common gain for signal and background counts + machine error
- ☒ Separate gains for signal and background counts + machine error

☐ Recycle ABSPACK after error model estimation

OK Cancel



All corrections together...



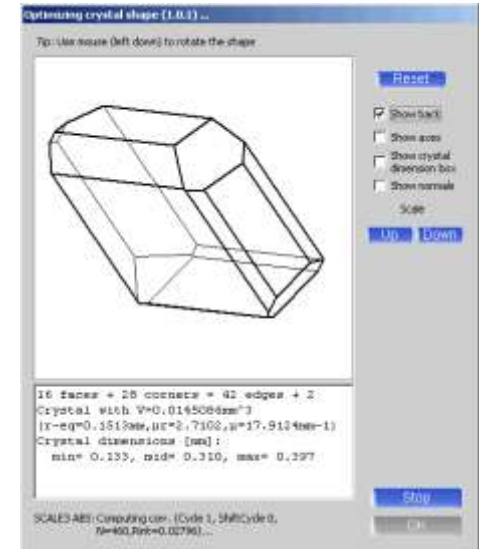
yttrium_mo – inorganic ($\text{Y}_3\text{Al}_5\text{O}_{12}$),
 Mo, $\mu=20.3\text{mm}^{-1}$, redundancy=30 (la-3d) , Nova micro-source

	R_{int}	I/σ	R1
No correction	50%	7.8	8.9%
Empirical	35%	8.5	7%
Absorption + beam illumination	20%	9.8	3.4%
Abs. + beam ill. + empirical	13.6%	11.6	2.4%

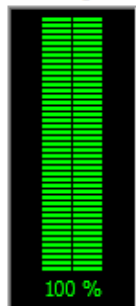


Multi – core

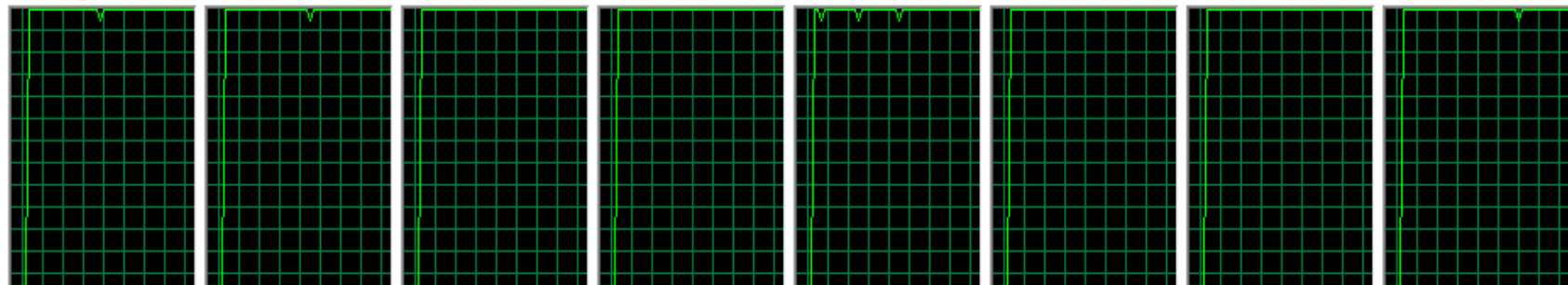
- All absorption and beam illumination computation
- All empirical corrections
- Auto shape generation
- Shape optimizer
- All matrix operations for Least squares
- What a blessing...



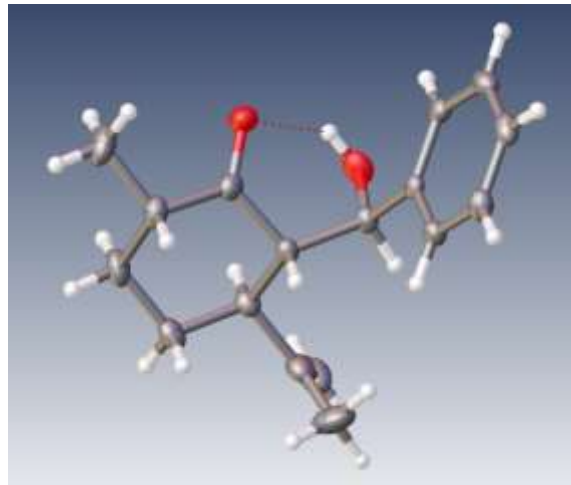
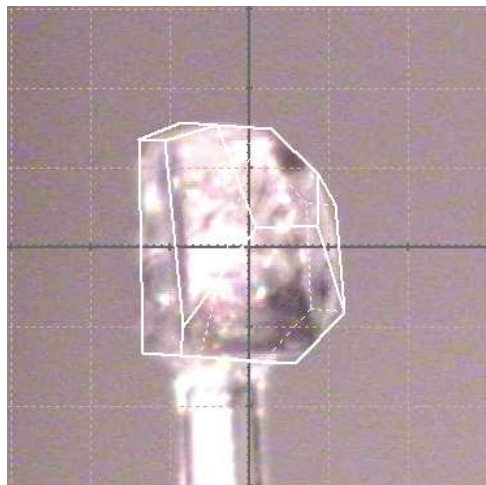
CPU Usage



CPU Usage History



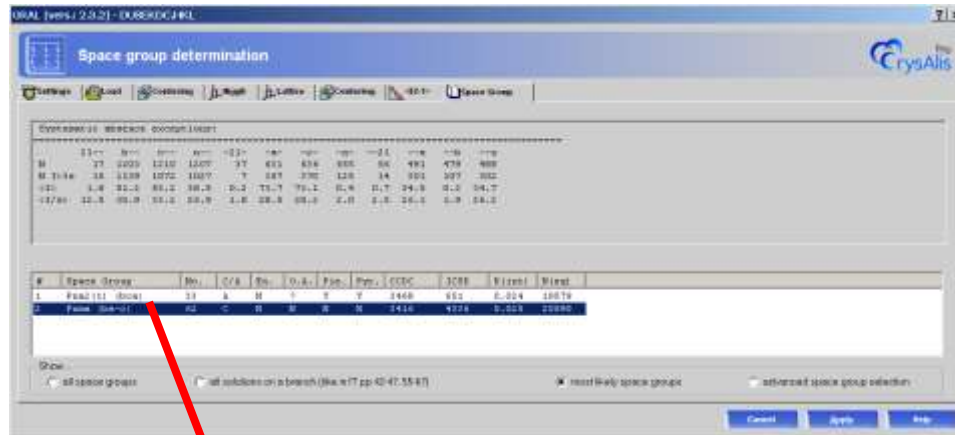
A very normal Cu data set



Schott (Peter Jones) – organic (C₁₇ H₂₂ O₂),
Cu, $\mu=0.58\text{mm}^{-1}$, redundancy=7.4 (P2₁2₁2₁)

	R _{int}	I/ σ	R1
No correction	10%	23	3.73%
Empirical	2%	34	2.93%
Empirical + Numeric	2%	34	2.93%

Proper space group for empirical corrections

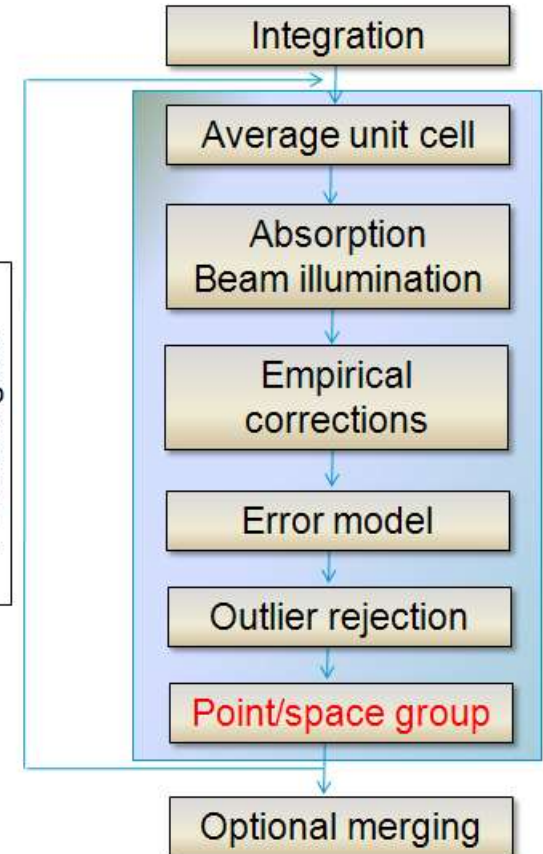


#	Space Group	No.	C/A	En.	O.A.	Pie.	Pyr.	CCDC	ICSD	R(int)
1	Pna2(1) (bca)	33	A	N	?	Y	Y	3468	651	0.024
2	Pnma (ba-c)	62	C	N	N	N	N	3416	4336	0.025

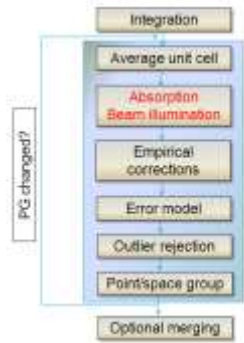
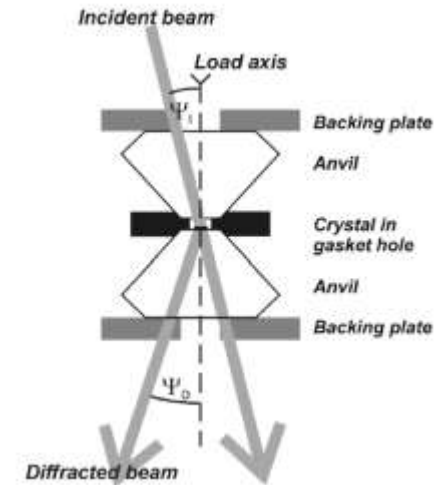
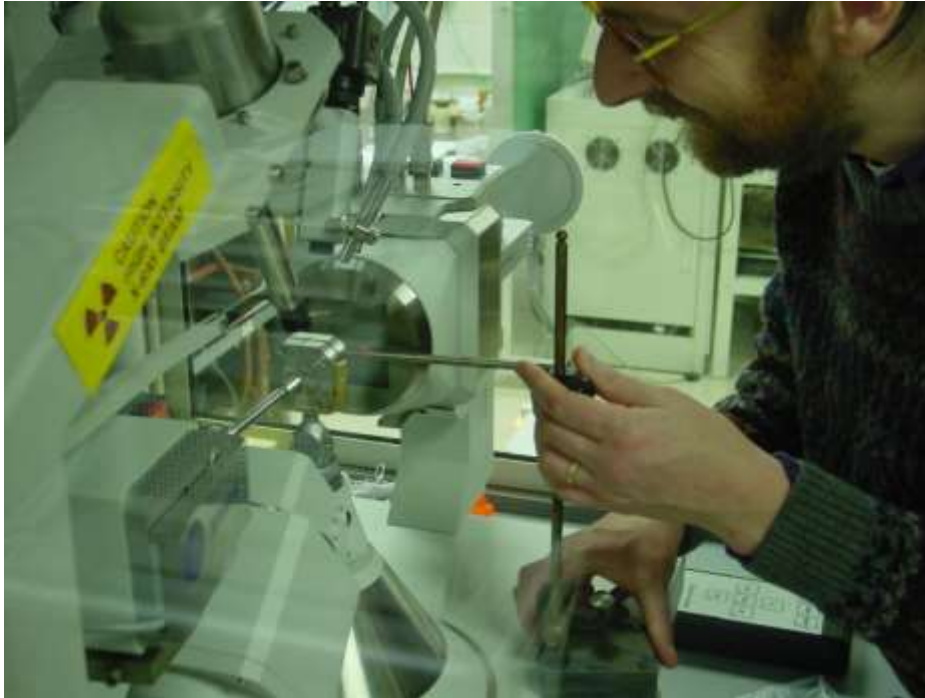
Show ...

☐ all space groups
 ☐ all solutions on a branch (like in IT pp 42-47, 55-67)

PG changed?



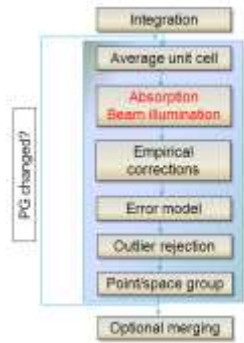
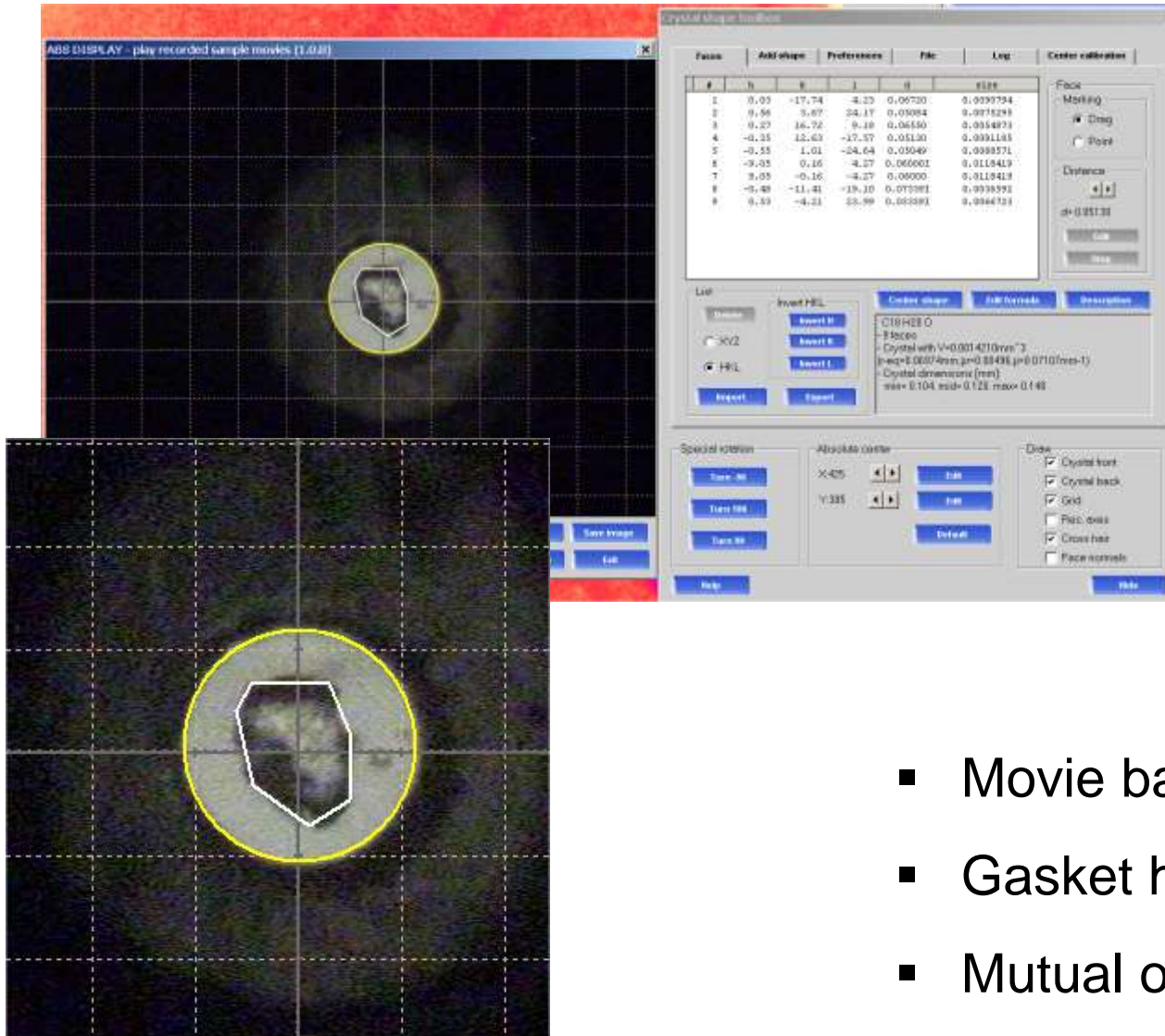
'Face-based': Diamond anvil cell correction



- Absorption
- Gasket shadowing
- Backing plates

Angel, R. J. (2004). J. Appl. Cryst.. 37, 486-492

'Face-based': Diamond anvil cell correction



- Movie based sample shape
- Gasket hole
- Mutual offsets



'Face-based': Diamond anvil cell correction

Absorb parameter dialog (1.0.2)

Disclaimer: CrysAlisPro offers an interface to Ross Angle's HP cell correction program 'absorb'. Agilent is not responsible for the functioning of absorb and cannot guarantee the relevance, completeness or accuracy of this program.

☒ Automatic generation of absorb input file

Hint: The sample shape and gasket information are derived from 'abs display' and can be modified there.

High pressure cell type: **TYPE 1**

HP cell opening angle 'sw a' = 40.0 **Edit**

Gasket:

Material: **USER** Material mu [mm⁻¹] = 21.0 **Edit**

Gasket thickness and radius [microns] = 300.0 300.0 **Edit**

Diamonds:

☒ Two anvils assumed identical ☐ Two different anvils (1: incident, 2: diffracted)

Anvil 1/2 thickness [mm] = 1.05 **Edit**

Backing plate

☐ Brakes ☒ Two plates assumed identical ☐ Two different plates (1: incident, 2: diffracted)

Plate thickness [mm] (at exp) (at atm) = 1.0 0.2 **Edit**

Shape option (Warning: shape should be defined in abs display)

☒ Open shape ☐ Closed shape with faces ☐ Closed shape with corners

☐ Manual generation of absorb input file

TITLE text

'Absorb input file generated by CrysAlisPro'

DAC TYPE 1

CELL	6.2947	12.8011	17.6345	90.00	97.53	90.00
DBL	0.1116136159	-0.0004161223	-0.0018188952			
DBL	0.0196677318	0.0090013450	0.0396351101			

Saving

Load **Save** ☒ Current experiment ☐ System

Help **Absorb Help** **OK** **Cancel**

Type of absorption correction selector

☐ Analytical absorption correction after Clark & Reid

☐ Gaussian grid absorption correction (Numerical integration)

Grid dimensions: **100** **100** **100** **Edit grid dimensions**

Gaussian grid correction:

☒ Crystal system ☐ Variable refractive ☐ Variable density

Beam profile correction:

☐ Apply beam profile correction:

☒ Adaptive window profile (5xwindow) - 2D (circular) gaussian

Grid factor size: ☐ 0.3 mm ☐ 0.5 mm ☐ 0.8 mm ☐ 0.0 mm

☐ Window (1: incident, 2: diffracted) - 2D gaussian

☐ Flatbeam

Beam size - EMM for Gaussian profile (mm): **0.00** **Edit horizontal beam size**

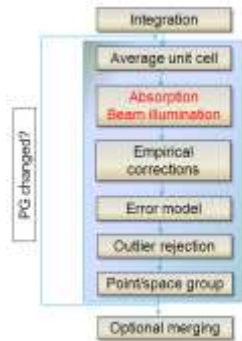
0.00 **Edit vertical beam size**

☒ High pressure cell correction (based on external program 'absorb' by Ross Angle)

High pressure parameters

Absorb settings saved in mm (at_exp) **Edit HP absorb pars**

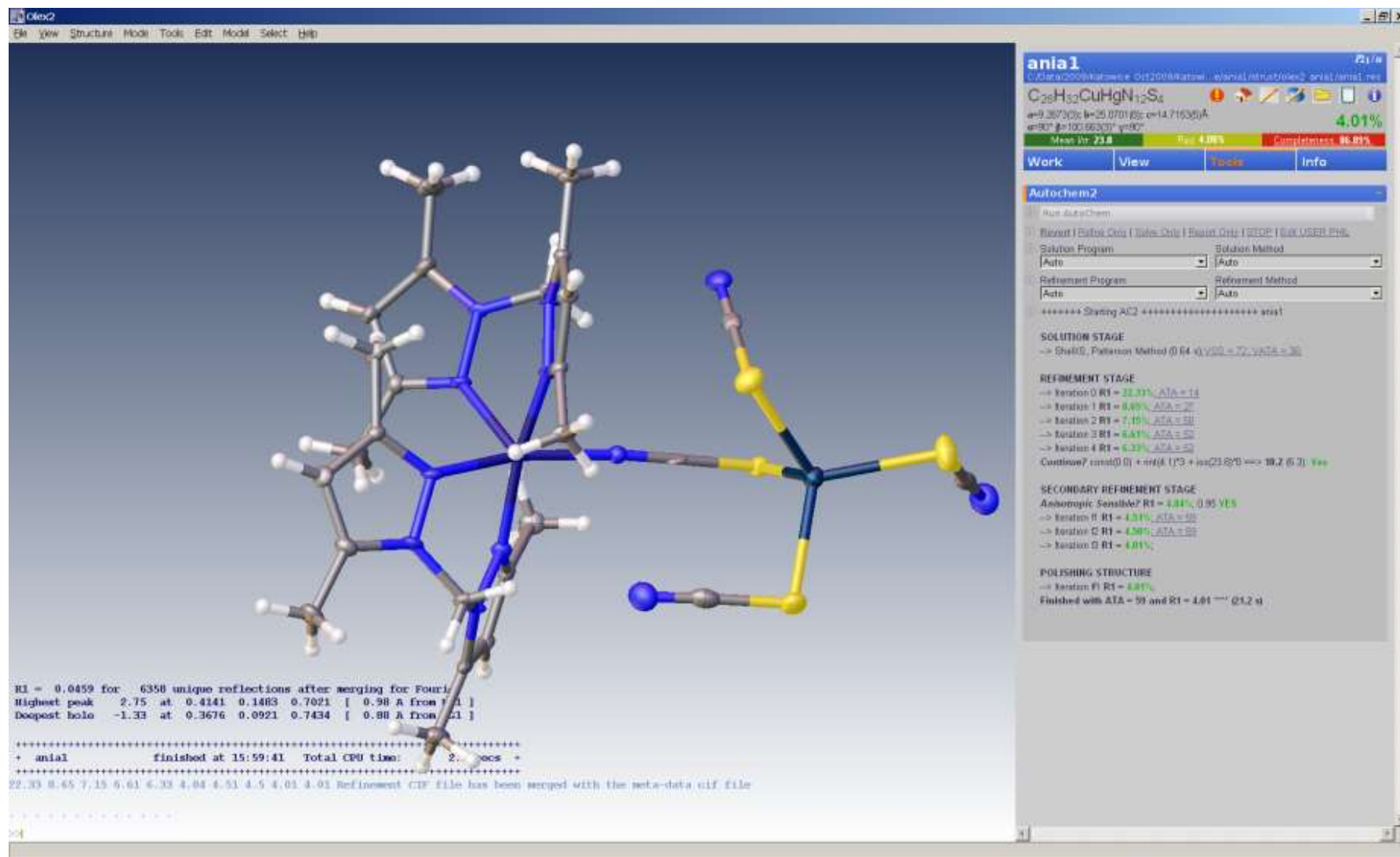
OK **Cancel**



- Automatically generated input files for Absorb 7.0
- Fully integrated to workflow



Integration with external programs



Summary - corrections

- Post corrections for area detector are instrumental for good data quality
- Redundancy enables most of those correction (Red>3 good start)
- We can only correct observed data! Use right exposure and technology (source, detector)
- For absorbing samples ($\mu > 5\text{mm}^{-1}$) numeric face based corrections are very necessary. Take the effort!
- For samples with $\mu < 5\text{mm}^{-1}$ generally empirical corrections are sufficient! Anisotropic sample may require beam illumination correction, especially with micro-sources!
- Use modern mounting technology!
- CrysAlis^{Pro} workflow integration helps to get most from corrections

Data finalization – optimal data

- Problems with...
- Experiment
- Unit cell
- Data reduction
- Finalization
- Pseudo symmetry, twinning, incommensurate



Approach a data set...

- Inspect executive tab
- Warning signs:
 - Run list incomplete
 - High mosaicity
 - Scaling unusual
 - I/sig low; low redundancy
 - SG issues

```
Data Reduction
FRAMES / RUNS
In run list: 402/7, used: 340/6

3D PROFILE ANALYSIS
Frames done: 340
Reflections tested: 2178, used: 1568
Avg mosaicity (in degrees) - 6 run(s)
e1=1.10, e2=1.11, e3=1.31
Max incidence angle profile change(e3): 11%

3D INTEGRATION & FITTING
Frames done: 340
Fitted: 2231, overflow: 0, hidden: 15
Outliers rejected: 1

FINALIZATION INPUT FILE
Filename: mm

FINALIZATION OUTPUT HKL FILE
Filename: mm

SCALING / NUMERICAL ABSORPTION
Empirical abs (e=2 o=0): min=0.99,max=1.01
Frame scales ( 1/scale): min=0.97,max=1.05
Friedel pairs treated as equivalent

RESULTS (340 frames) - SYM: Pmmm
Resolution(A) Redundancy F2/sig(F2) Rint
inf - 0.80 1.9 26.7 0.030
inf - 0.84 2.0 27.9 0.030
Completeness: 95.8% (0.84 ANG)
Anom compl.: 83.6% (P222)

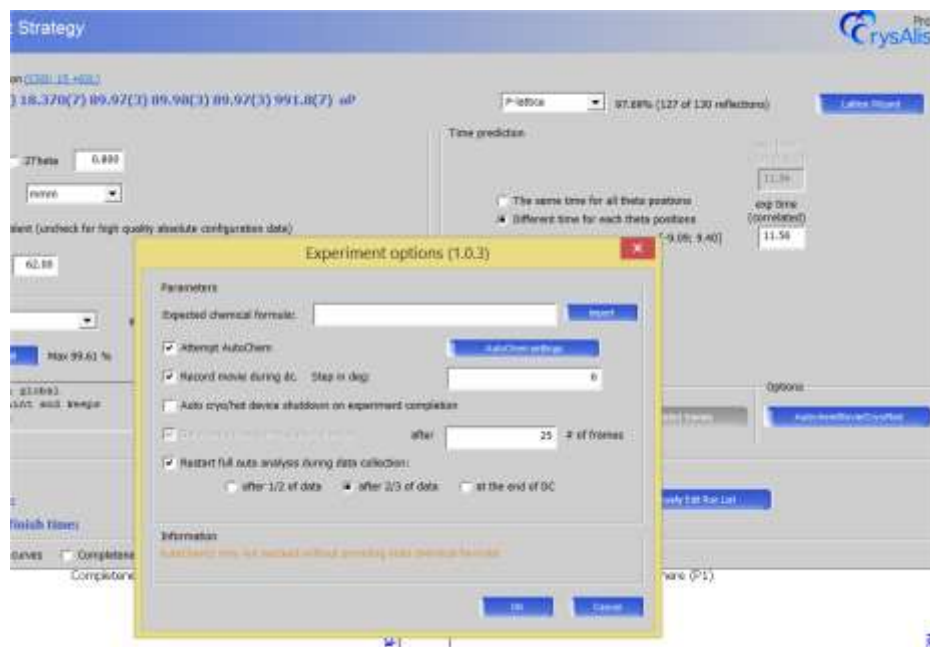
SPACE GROUP DESCRIPTOR
P2(1)2(1)2(1) Group #: 19 (3 SG found)
no data coverage: h00, 0k0,

DATA REDUCTION OPTIONS
Per-frame model refinement used
2-cycle 3D peak analysis used
3D profile fitting used
```




Approach a data set...

- Run 'Full auto analysis' on all data
- Concurrent may get stuck



CrysAlisPro: Data reduction (1.13)

- Load new experiment
- Full auto analysis (cell, red)
- Automatic data reduction
- Data reduction with options



Approach a data set...

- Inspect frames:
 - Low/high background
 - Diffuse scattering, split reflections, twin
 - Empty frames, strange frames
- Inspect movie:
 - Sample mounting



Optimal data – hints at experiment time

- Centering/Sample choice/holder/amount of oil
- Low T: de-ice runs
- Absorption: make movie 1-6 deg
- Concurrent data red re-start
- Cu – Mo choice
- Collect redundant data
- Rather reduce scan width than increase dd
- Check your diffraction limit



Optimal data – hints at unit cell finding time

- Check for non-indexing reflections
 - Garbage (ice rings, powder), twin, sample jump
 - Re-run refine model
- Ewald
 - Use filters (intensity, lattice type) and groups
 - Use intensity view
- Check chemical formula unit cell consistency

Optimal data – hints at data reduction time

- Check for experiment artifacts (empty frames, trips)
- Apply Bravais lattice where obvious
- Special pars
 - Use bad reflection filter
 - Use reduced profile size if overlapping
 - Incidence correction, prediction accuracy
- Smart background on high background data
- xx proffitloop

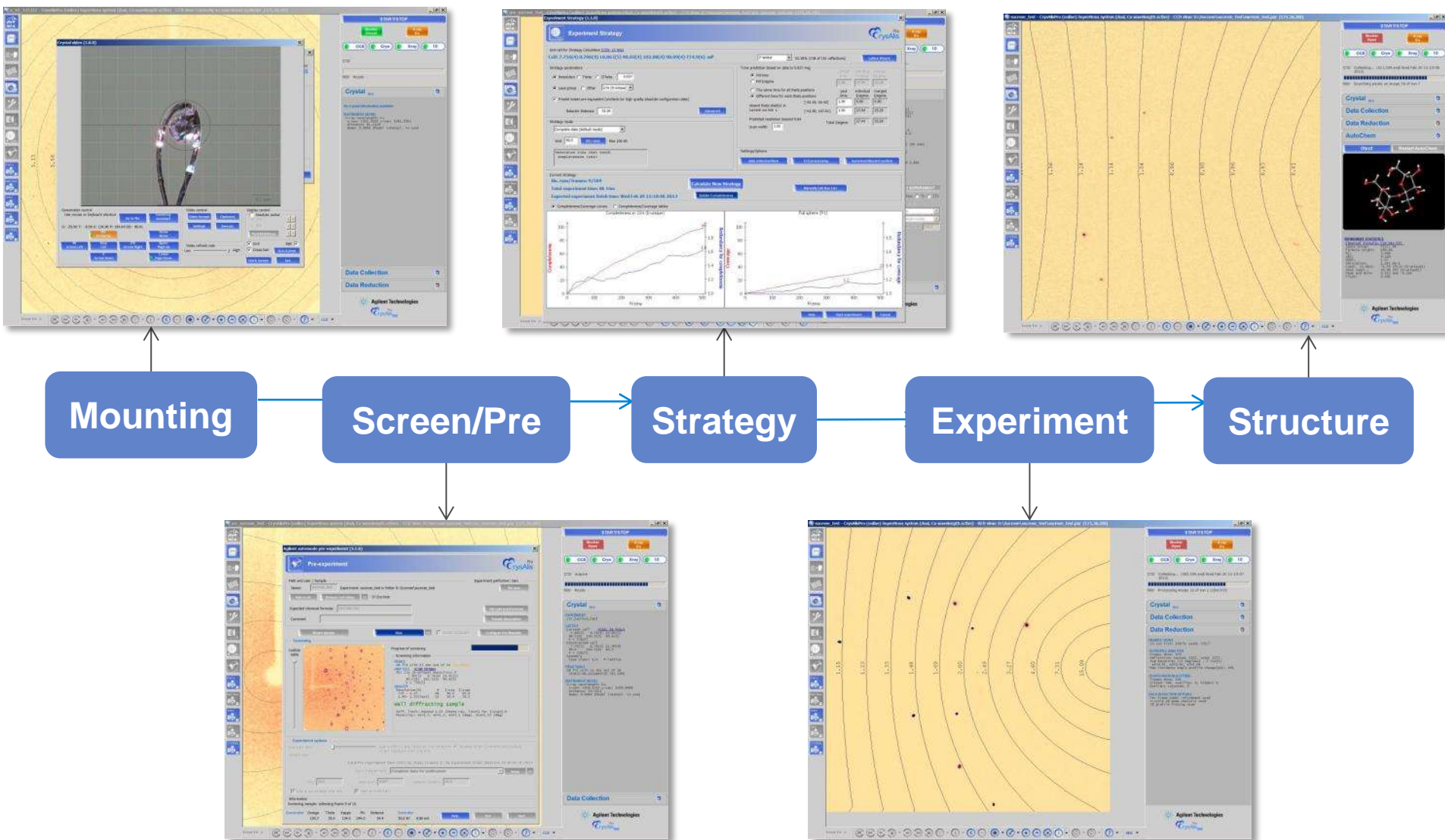


Optimal data – hints at data finalization time

- Hand set empirical parameters
- Use shape based absorption correction
- Apply filters carefully (e.g. Rint)
- Interactively decide space group
- Remove unnecessary data via d-value filter

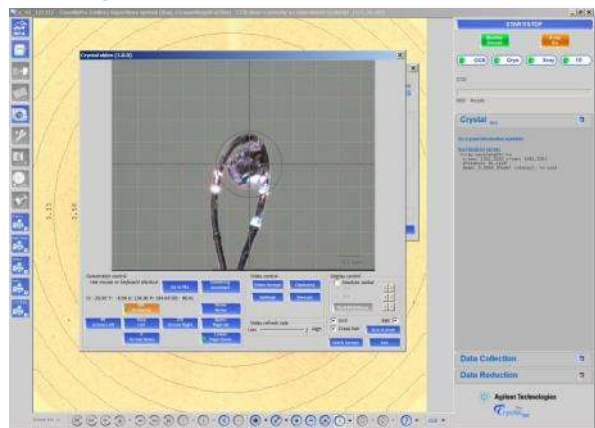


Typical Work-Flow



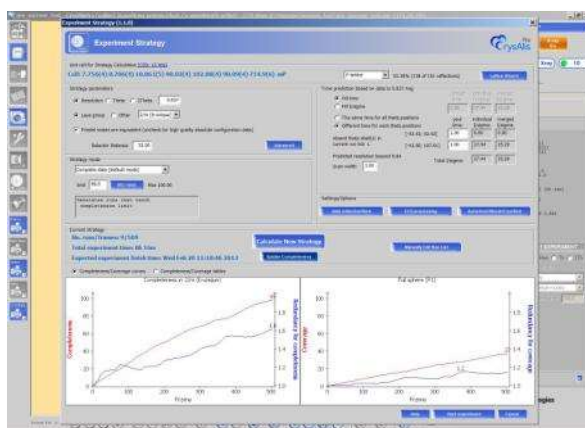
Typical Work-Flow

Enabled
by IMS



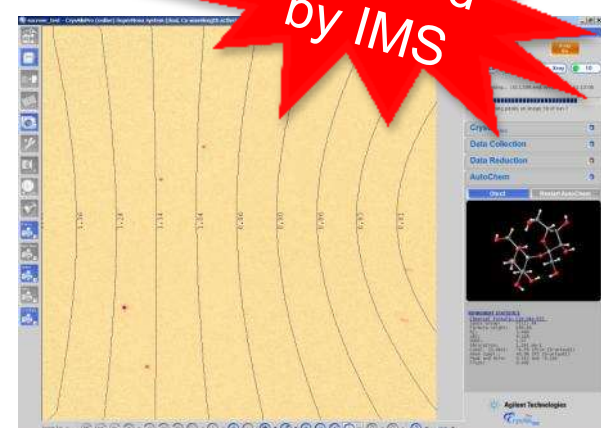
Mounting

<1min



Strategy

<30s

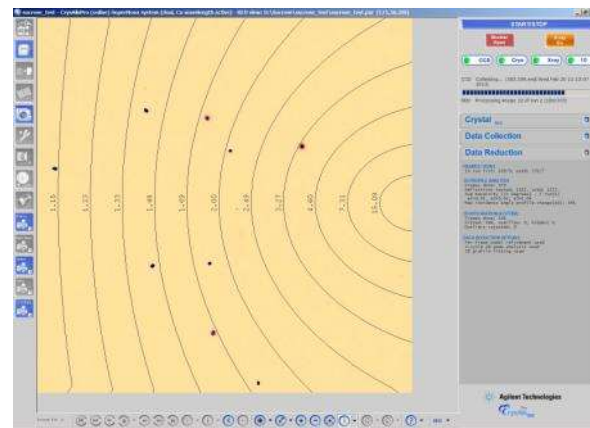
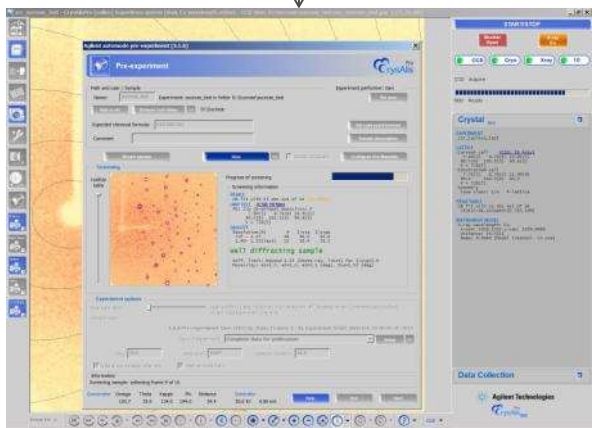


Experiment

15min-3d

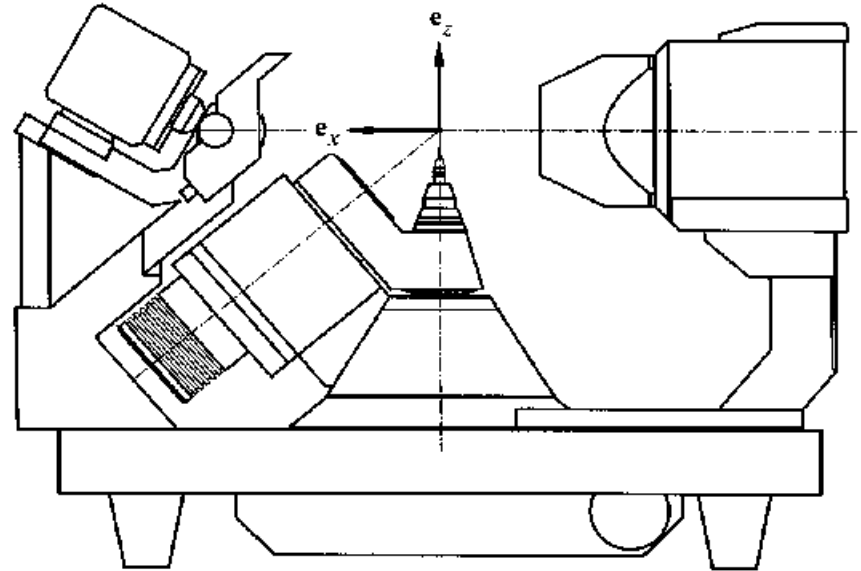
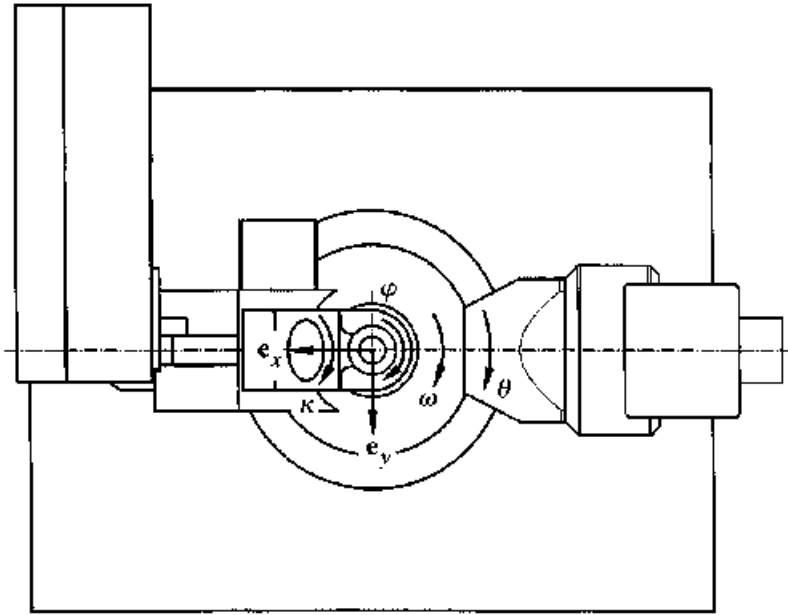
Structure

concurrent

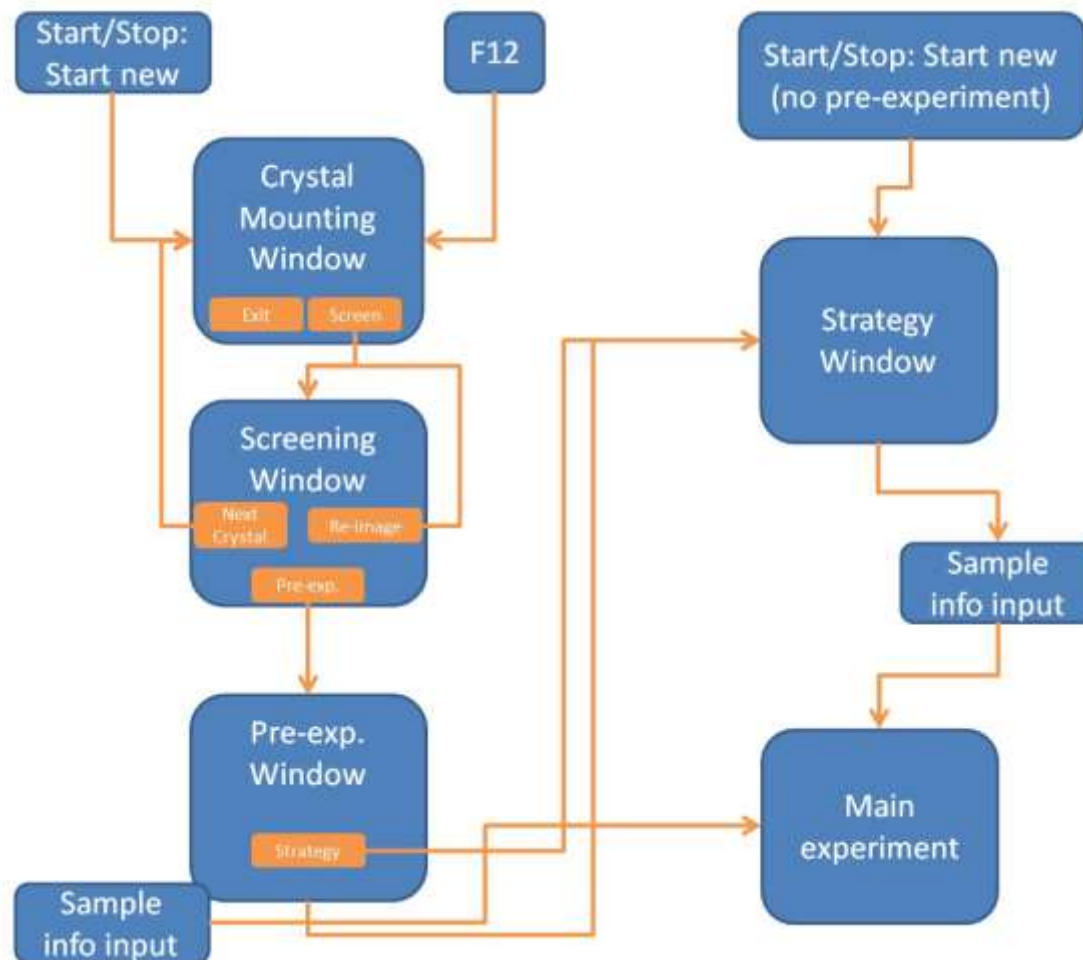


Agilent Technologies

Excursion - instrument



Simplified 3-Step Pre-Experiment Work-Flow



Tab Based Screening 1



SM Screening

Screening

Mount

Screening

>

PEAKS
 UB fit with 70 obs out of 70 (100.0%)
UNIT CELL [\(CSD: 14+437L\)](#)
 PG: mm orthorhombic P
 5.97(2) 9.05(2) 18.356(17)
 90.02(13) 90.26(15) 90.2(2)
 V = 992(5)

QUALITY

Resolution(A)	N	I/sig	I/sig0
inf - 1.23	91	24.0	26.5
1.28- 1.23(last)	10	12.9	14.2

Well diffracting sample

Diff. limit: beyond 1.23 (theta res. limit) for I/sig=2.0
 Mosaicity: e1=1.2, e2=1.2, e3=2.0 (deg), Iso= 1.49 (deg)

Experiment - Complete data for publication

Name:

Detector=52.0mm, Res. = 0.837Ang, I/sig.=15.0, width=1.0deg, Movie, cryo off, Strategy: Complete data (default mode), Exposure: 1.0s 4.0s

Exposure time:

Start Pre-Exp. (5 min)

Edit

Goniometer

Omega	Theta	Kappa	Phi	Distance
20.0	-35.0	0.0	0.0	52.0

Generator

kV	mA
50.0	0.80



Tab Based Screening 2

Edit SM options

Lattice finding / Data reduction

- ☐ Use first delta peak table to attempt cell finding in auto-analysis
- ☐ Smart background during reflection integration
- ☐ Single wavelength data red
- ☐ HKL check in 3D peak analysis during data red
- ☒ 3rd cycle in 3D peak analysis
- ☒ Compute completeness after data red
- ☒ MT2 export data red for use in CCP4
- ☒ Run space group determination (GRAM) after data red
- ☐ Limit space groups taken into consideration

☐ Use prediction uncertainty for integration mask size adjustment

☒ Data reduction during data collection after 25 # of frames

☐ Restart full auto analysis during data collection

☐ Launch shape generation after movie

☒ Enable fast UB search during SM-screen [up to peaks] 400

Data collection / Strategy

- ☒ Use tab-based SM screening
- ☐ Use connectivity over fast for strategy
- ☐ Favour less runs in red alg

☐ XDS and/or ☐ Denzo and/or ☐ MOSFLM export during data collection

400 Max autodecode exposure time (sec)

Overlap computation:

- ☒ Complex approach: table with 9 cases: dd vs scan width
- ☐ Simplified approach: 1 case: current dd vs scan width
- ☐ Skip strategy reference frames (if data are time-triggered)

100.0 Default completeness goal (%)

2.0 1/sig criterion for max. resolution prediction

OC JETSHADOW (to visualize beforehand use beamstop mask)

☐ Use JetShadow

OK

SM Screening

Screening

Mount **Screening** >

PEAKS

UB fit with 70 obs out of 70 (100.0%)

UNIT CELL (CSD: 14+437L)

PG: mmm orthorhombic P

5.97(2) 9.05(2) 18.356(17)

90.02(13) 90.26(15) 90.2(2)

V = 992(5)

QUALITY

Resolution(A)	N	I/sig	I/sig0
inf - 1.23	91	24.0	26.5
1.28- 1.23(last)	10	12.9	14.2

Well diffracting sample

Diff. limit: beyond 1.23 (theta res. limit) for I/sig=2.0

Mosaicity: e1=1.2, e2=1.2, e3=2.0 (deg), Iso=1.49 (deg)

Experiment - Complete data for publication

Name: exp_209

Detector=52.0mm, Res. = 0.837Ang, I/sig=15.0, width=1.0deg, Movie, cryo off, Strategy: Complete data (default mode), Exposure: 1.0s 4.0s

Exposure time: 1.0 s

Start Pre-Exp. (5 min) **Edit**

Goniometer

Omega	Theta	Kappa	Phi	Distance
20.0	-35.0	0.0	0.0	52.0

Generator

KV	mA
50.0	0.80

Screening options (1.0.0)

Screening position

Align: theta: 135.0 Max res: 1.288

Pre-align to current position: ☐ 20 ☐ 40 ☐ 80

Exposure time: 2.0 Dark is waiting

Features of screening

Frames used: 3 frame(s) ☐ Read unit cell

Hardware settings

Generate

Information

Reset defaults **Cancel** **OK & Screen** **OK**

Agilent fast screening options (1.0.0)

Pre-experiment

Path and user / Sample

Name: Experiment: in folder

Get user

Cancel folder **Browse next folder** C:\WorkData

Expected chemical formula: **Get Last used formula**

Comments: **Sample description**

Experiment options

Exposure time: Detector distance=15.0mm, Resolution = 0.830Ang, I/sig=15.0, Scan width=1.0deg, Movie on, cryo shutdown off, Strategy mode: Complete data (default mode), Exposure time: 5.0s

Total Pre-experiment Time: 0:03, No. Runs/Frames: 3/15, Pre-experiment Finish: Mon Jul 01 09:48:40 2013

Type of experiment: **Quick look (atom connectivity)** **Setup**

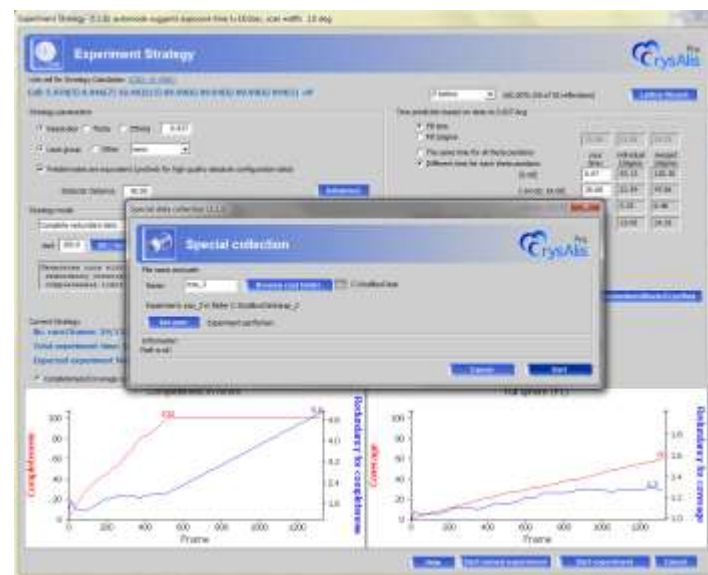
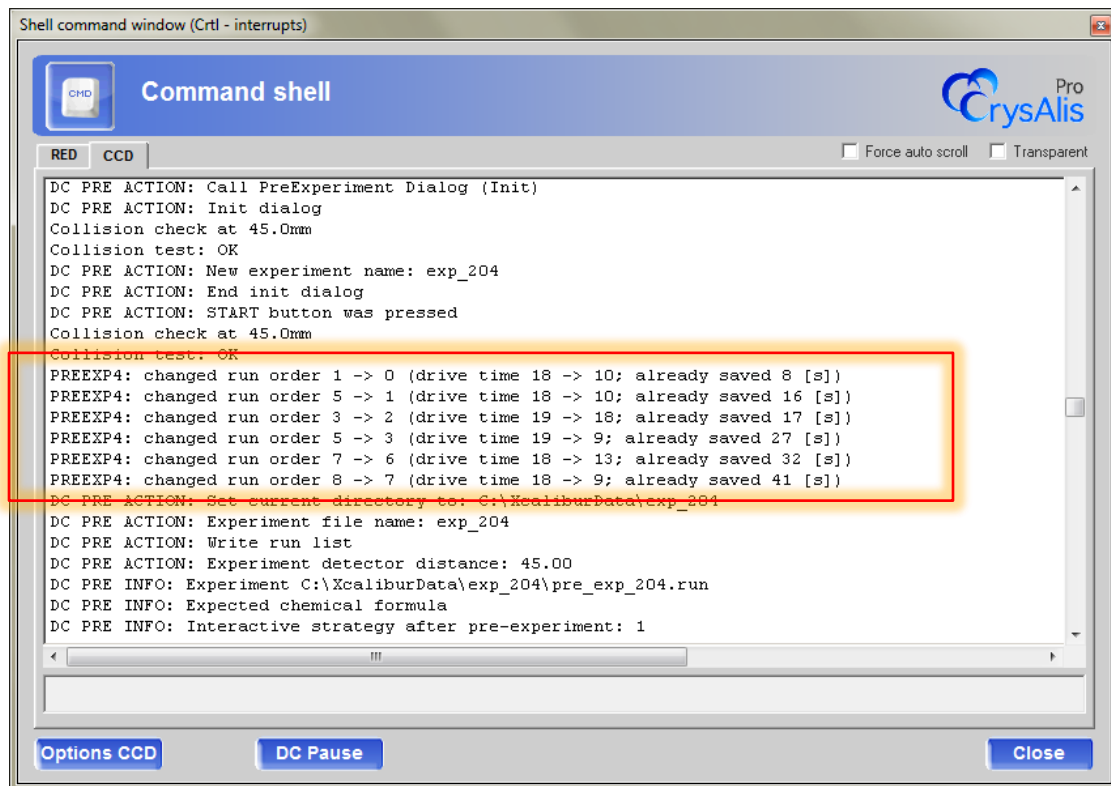
☒ Interactive strategy after pre ☒ Attempt AutoChem

Information

Help **Exit** **Start**



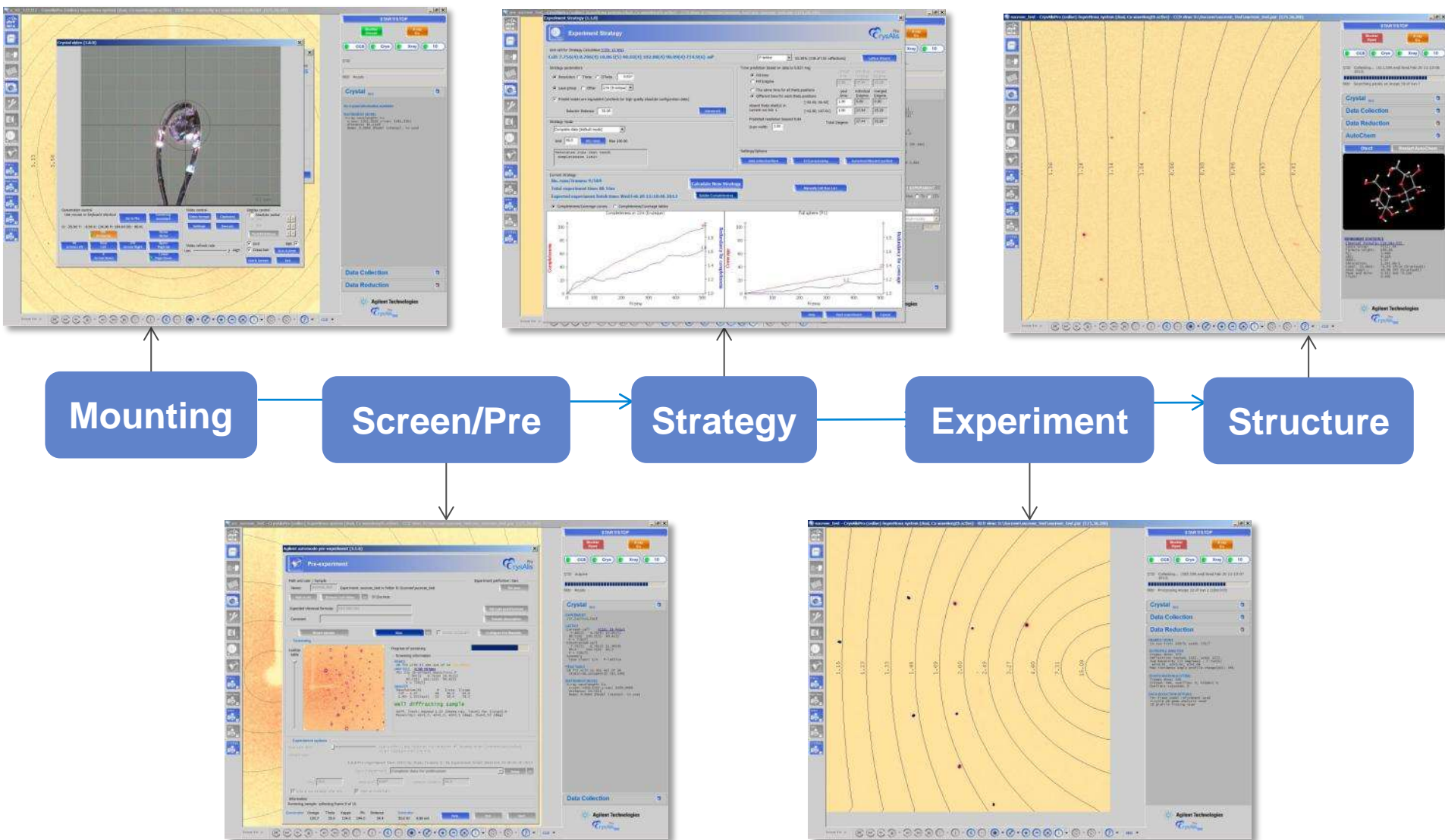
Tab Based Screening 3



Screening

- Pre-experiment automatically exposure time scaled!
- Screens only look at low theta
- Rapid turn around to next sample

Typical Work-Flow



Pre-experiment

- Careful experiment planning = same theta range as final experiment: $\text{Mo} = 0.8$; $\text{Cu} = 0.837$
- Experiment scaling works well up to factor 20.
- What resolution should I use?
 - Standard: defaults work well
 - Absolute structure determination: The highest you can get!
 - Connectivity only: 1.0 is fine

Excursion: I/σ

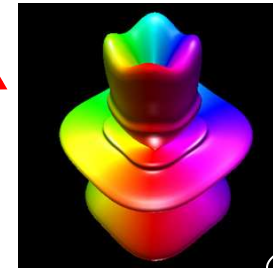


HW – instrumentation

$$\frac{I}{\sigma}$$



Procedure

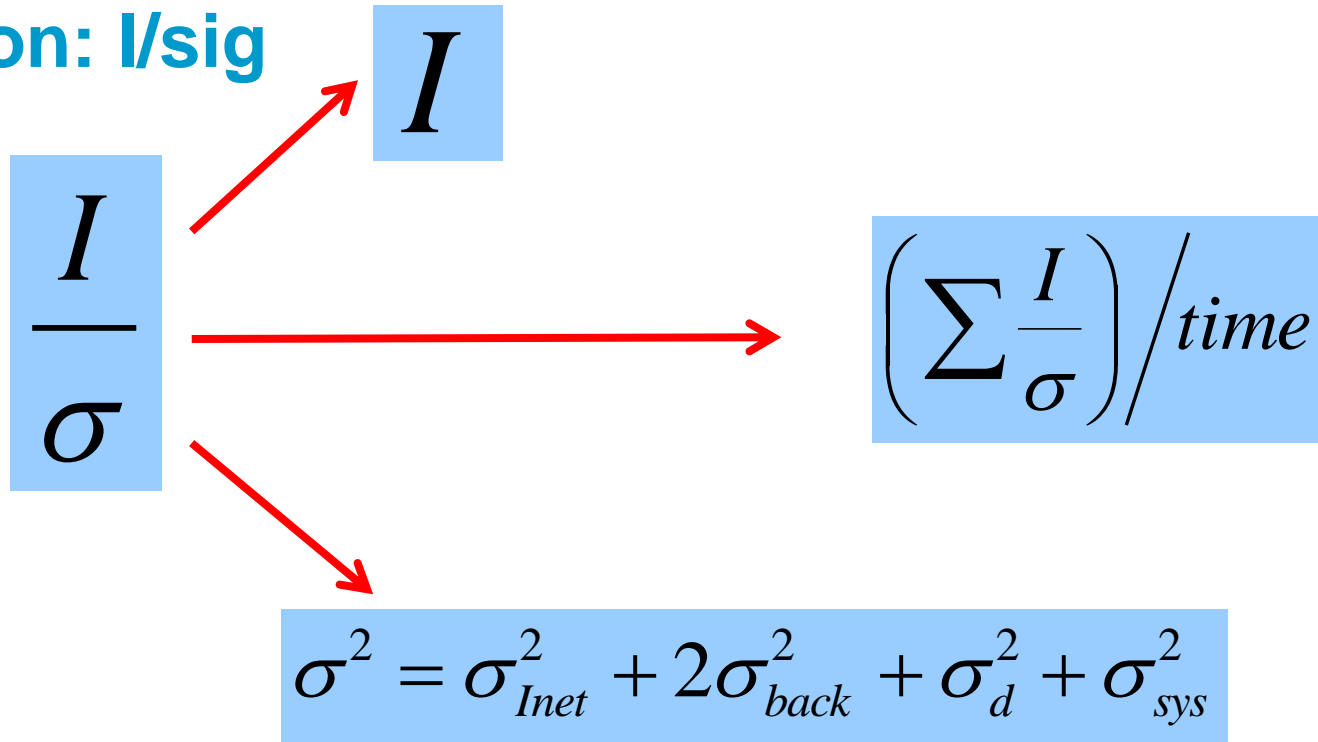


Data reduction
Corrections

- X-ray diffraction experiment are governed by Poisson statistics: I ; $\sigma(I) = \sqrt{I}$
- Rule of thumb: Inverse of I/σ is your R1



Excursion: I/sig



- Influence I: Time, bigger xx, X-ray source (wl, power, efficiency), sample temperature, redundancy
- Influence sig: less background, sample = beam, correlation



Pre-experiment tab

- Information on
- Unit cell
- Quality
- Diffraction limit

Pre-experiment

CELL (CSD: 15 +61L)
orthorhombic P (32) 97.7%(127/130)
5.965(2) 9.051(4) 18.370(7)
89.97(3) 89.98(3) 89.97(3)
V = 991.8(7)

INTENSITY STATISTICS (FULLS) 0.80A
N=336 min=0.80A max=3.86A
I/sig=9.1 obs=65.8% I/sig=13.3
time for I/sig=15.0= 13.5

MOSAICITY
e1=1.06, e2=1.19, e3=1.37 (deg)
#partials used=104

DIFFRACTION LIMIT
0.89A (pre)

☒ Run strategy after pre START EXPERIMENT

Target resolution: 0.800

Ang ☒ Res ☐ Th ☐ 2Th

Target I/sig: 15.00

I/sig (0.1-1000)

Experiment type:

Use Laue symmetry

Strategy type

Complete redundant data

Redundancy

5.0

Comp. limit

100.0

Strategy

- Two basic approaches: 'Fixed strategy' vs. 'Symmetry adapted strategy'
- 'Symmetry adapted strategy' requires sample knowledge:
 - Unit cell and orientation
 - Laue/point symmetry
 - Diffraction power
 - Sample mosaicity

Type of experiments

- Fastest possible
 - 'Complete data' (exploit symmetry)
 - Geometric objects (quadrant, hemisphere, full sphere)
- Charge density/absolute structure
 - Target redundancy
- Absorption correction
 - complete data + geometric objects
- Twin/multi crystal
 - Complete on all components



The strategy computation question

user constraints

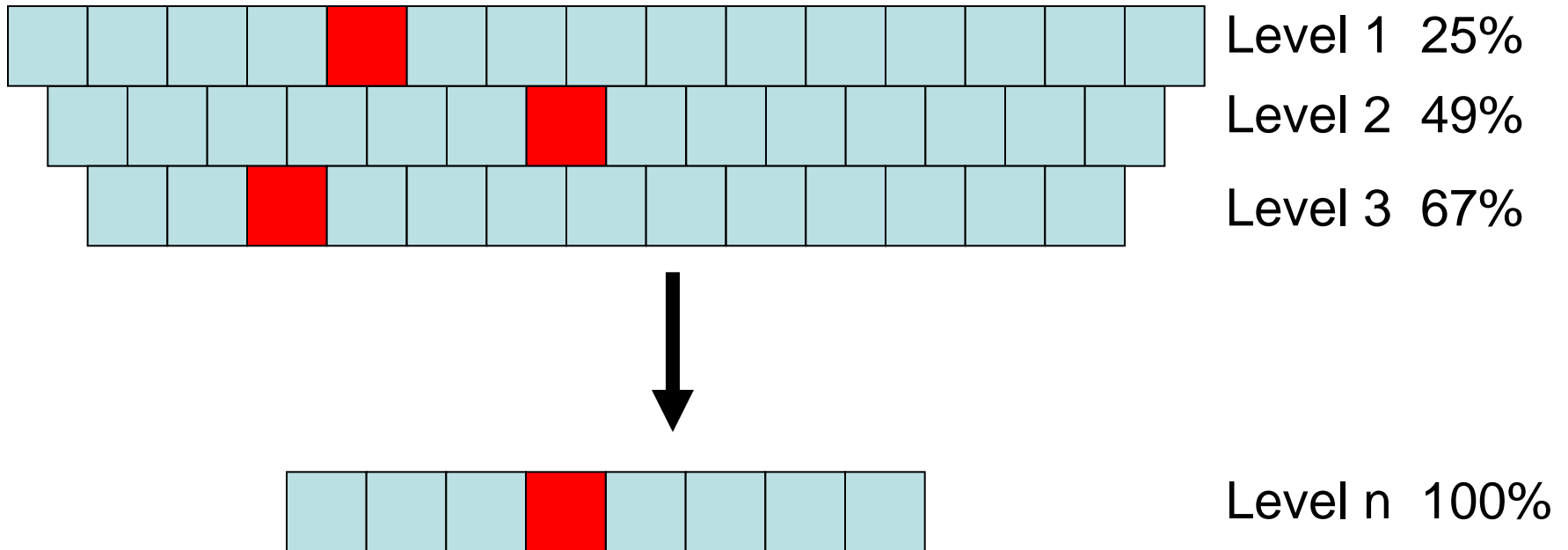


distance, detector,
collisions

Pool of runs

Typically 150-2000

The strategy computation question 2



What is efficient?

runs: 7, frames: 520
theta settings: 2; 25.31; 25.93, max resolution: 0.734

Laue: 2/m; Friedel off, Target 0.800Ang

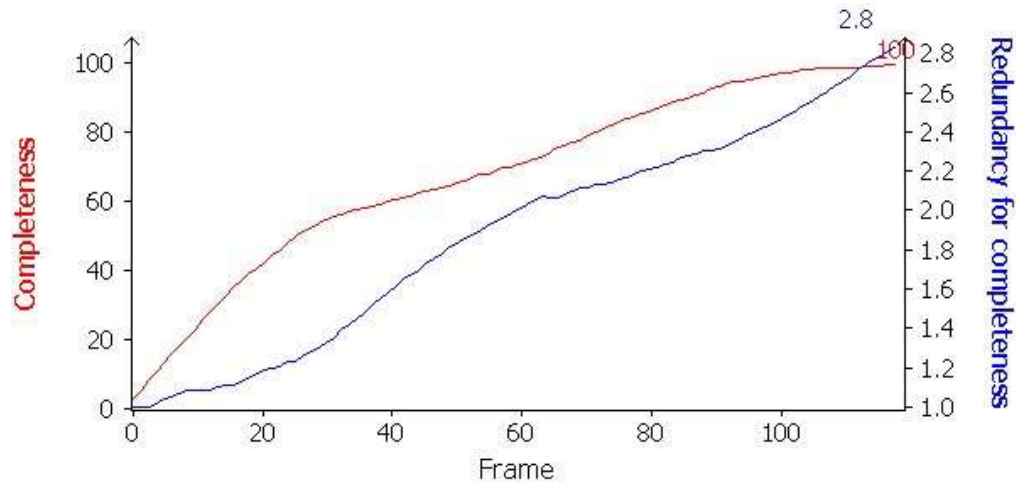
Completeness (under Laue symmetry)						Coverage (under P1)					
Res		#Data	#Theory	%	Redundancy	#Total	#Data	#Theory	%	Redundancy	#Total
18.50-	1.74	409	415	98.55%	2.7	1108	644	828	77.78%	1.7	1123
1.74-	1.37	415	415	100.00%	2.6	1084	637	828	76.93%	1.7	1077
1.37-	1.19	415	415	100.00%	2.3	942	593	828	71.62%	1.6	946
1.19-	1.09	415	415	100.00%	2.0	850	585	828	70.65%	1.4	845
1.09-	1.01	415	415	100.00%	1.9	776	530	828	64.01%	1.5	775
1.01-	0.95	415	415	100.00%	1.7	719	546	828	65.94%	1.3	719
0.95-	0.90	415	415	100.00%	1.6	680	522	828	63.04%	1.3	677
0.90-	0.86	415	415	100.00%	1.5	623	500	828	60.39%	1.2	624
0.86-	0.83	415	415	100.00%	1.5	619	507	828	61.23%	1.2	617
0.83-	0.80	417	417	100.00%	1.4	576	496	830	59.76%	1.2	574
<hr/>											
18.50-	0.80	4146	4152	99.86%	1.9	7977	5560	8282	67.13%	1.4	7977

Rule of thumb:

“When requiring 100% completeness the minimum achievable redundancy is about 2”

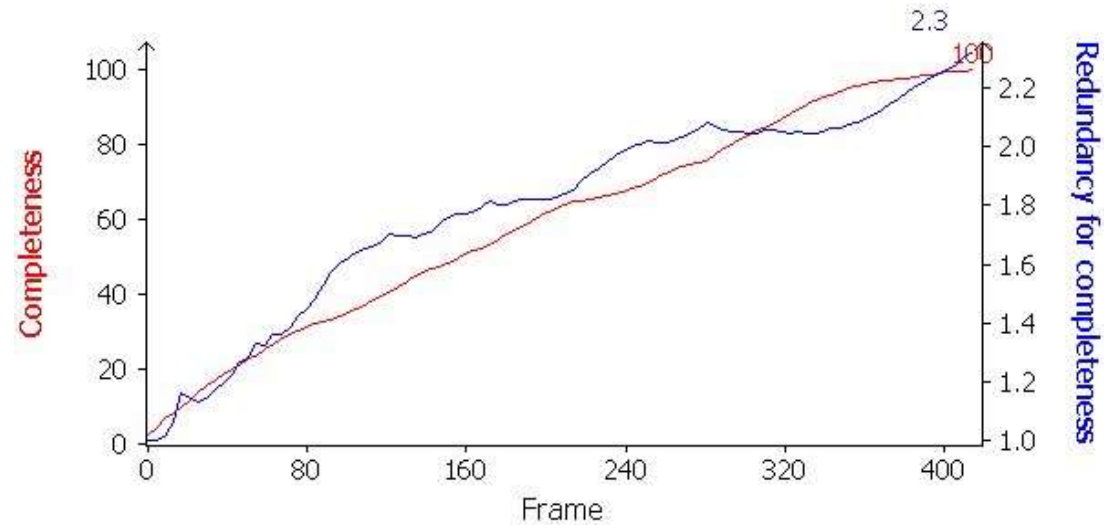


The rotation method



Mo

Cu



Rule of thumb:

“At similar conditions Cu experiments take 4-5 times more frames than Mo”



Detector size



Detector relative size	Unique speed	Observation speed
Eos 1	1	1
Atlas 2.4	1.3 – 1.6	1.6 – 1.8
Titan 3,7	1.4 – 1.8	2.0 – 2.2

Detector distance

Distance	Frames	Total time	Disk space
50 mm	167	1h 29m	180Mb
100 mm	378	3h 19m	400MB

Price:
2 x distance =
2 x frames

Sapphire, res =0.8, complete data

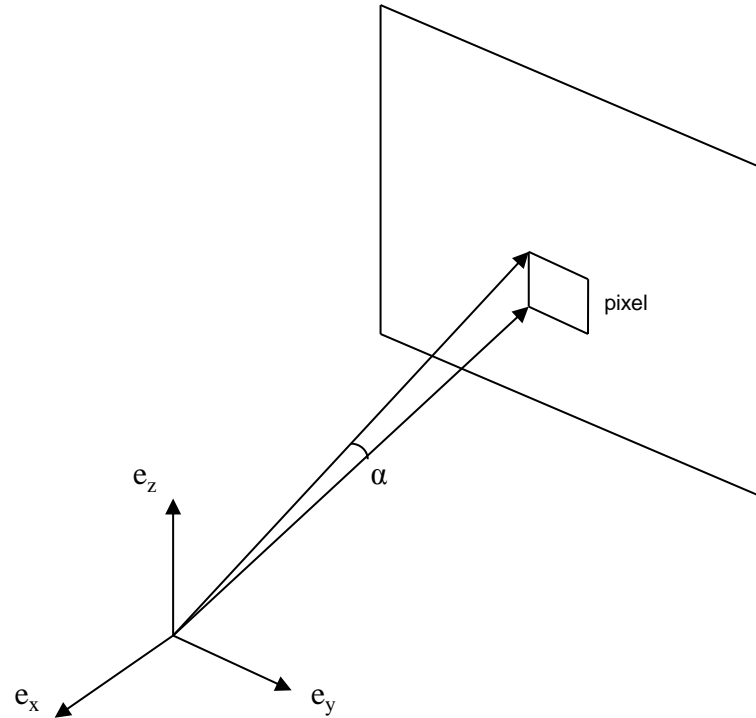


Rule of thumb:

“Use as close distance as possible”

Overlaps

- Pixel size = 0.1 deg
(Atlas, binning 2x2, dist=50mm)



Rule of thumb:

“First fine slice then go back with detector”

HW – Detector technology: Key metrics



Eos S2



Atlas S2



Titan S2

- Detectivity
- Dynamic range
- Speed
- Size
- Price

Detectivity: Detective Quantum Efficiency (DQE)

$$DQE = \frac{T_w \eta_{ph}}{1 + \frac{1}{g} + \frac{A(n_r^2 + i_d t)}{IT_w \eta_{ph} g^2}}$$

Diagram illustrating the components of the Detective Quantum Efficiency (DQE) equation:

- Window Scintillator**: Points to the term $T_w \eta_{ph}$ in the numerator.
- Gain**: Points to the term g in the denominator.
- Noise: Read and dark**: Points to the term $A(n_r^2 + i_d t)$ in the denominator.

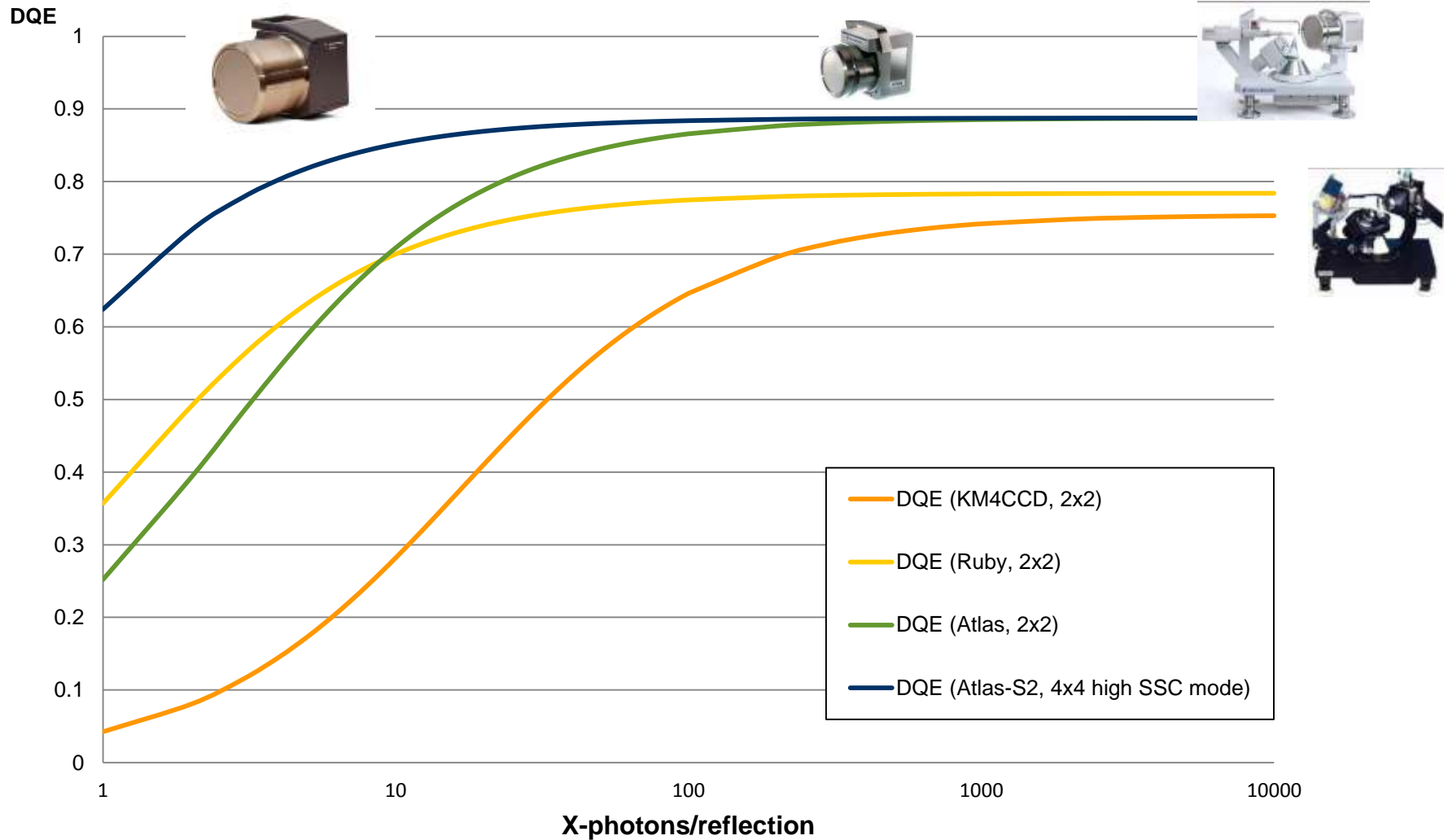
M. Stanton et al., J. Appl. Cryst. (1992). 25, 638-645



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Detective Quantum Efficiency (DQE) advances

DQE versus intensity

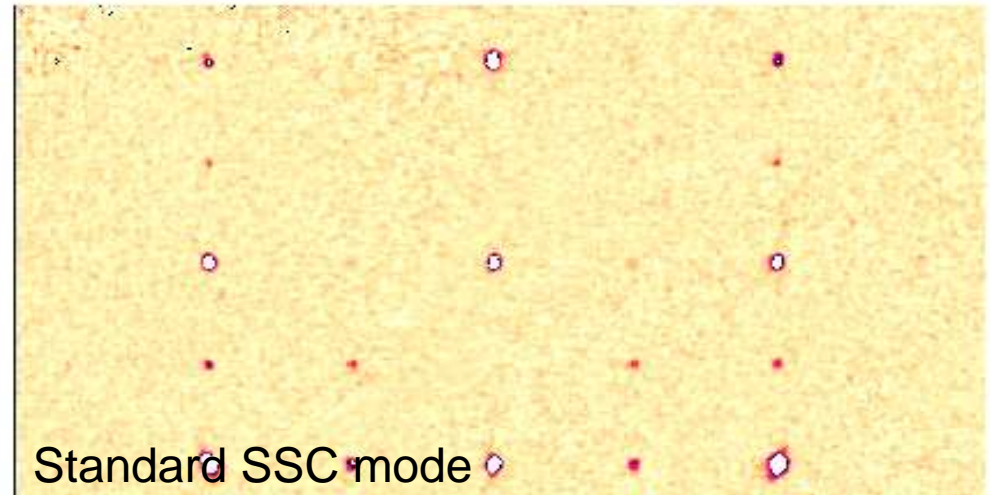


DQE for Mo radiation, peak dia 1mm at 60mm

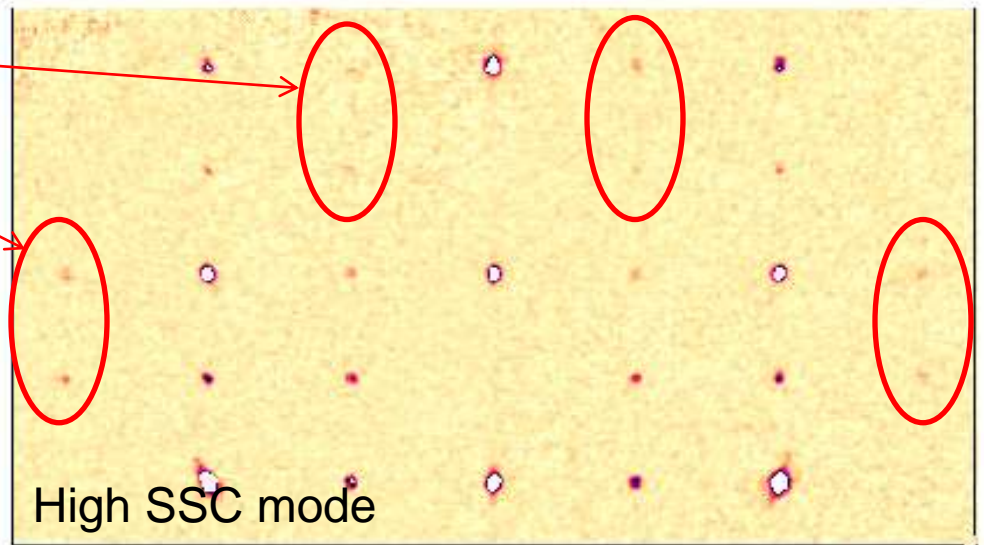
What is 'Smart sensitivity control' SSC

Precession image (Fe formate, 0kl plane)

- Smart Sensitivity Control allows for detecting very weak events!
- Like the ISO settings on digital cameras



These spots became visible only in high SSC mode!



State-of-the-art electronics provide:

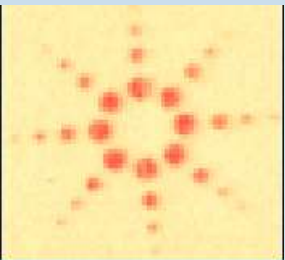
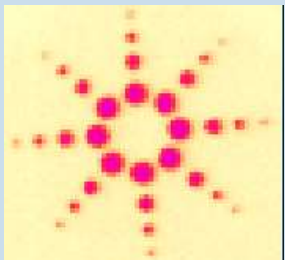
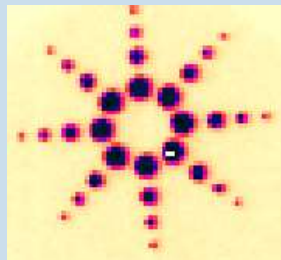
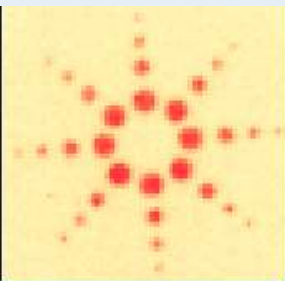
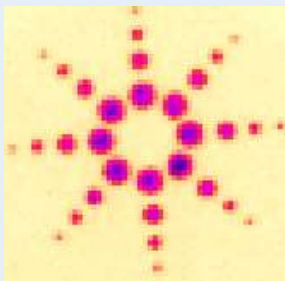
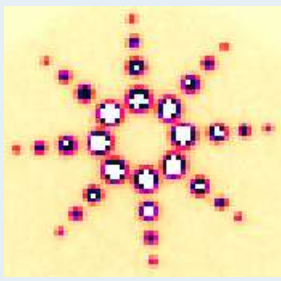
- Detectivity

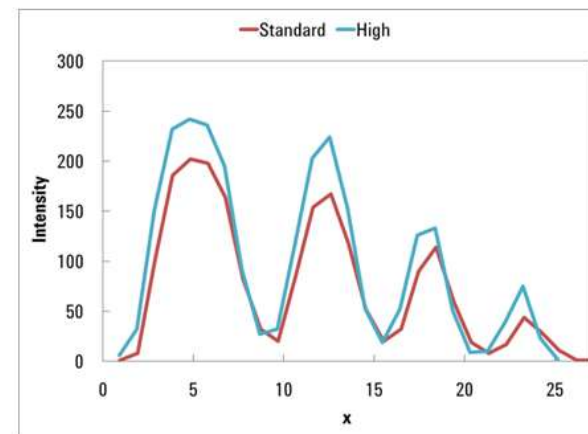
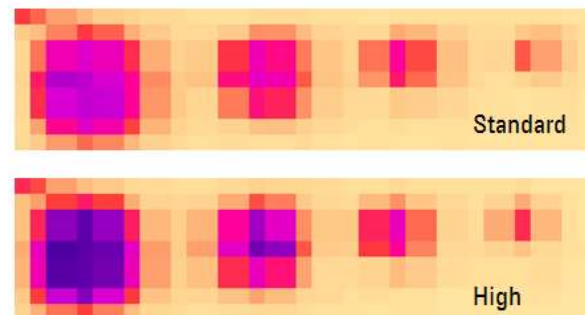


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Titan S2 camera SSC functionality


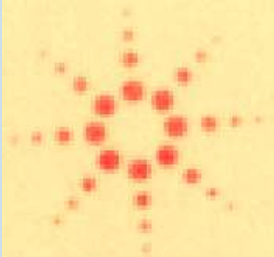
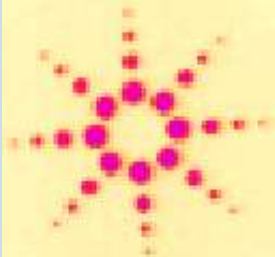
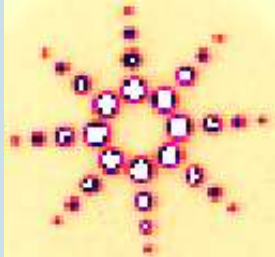
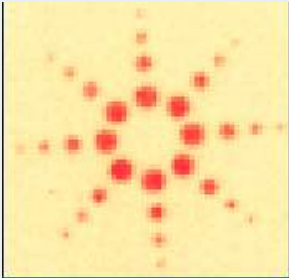
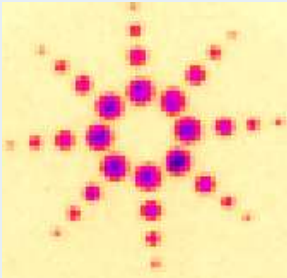
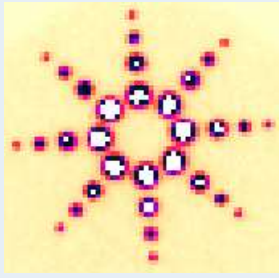
4x4 binning

Exposure time	0.5s	1s	2s
Standard SSC mode			
High SSC mode			



Titan S2 camera SSC functionality

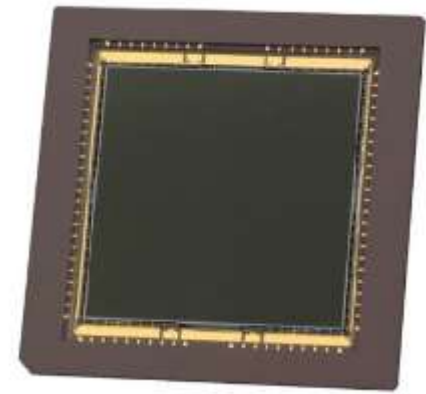
2x2 vs. 4x4 binning

Exposure time	0.5s	1s	2s	5s
2x2				
4x4				



HW – Detector tech: Full well/Dynamic

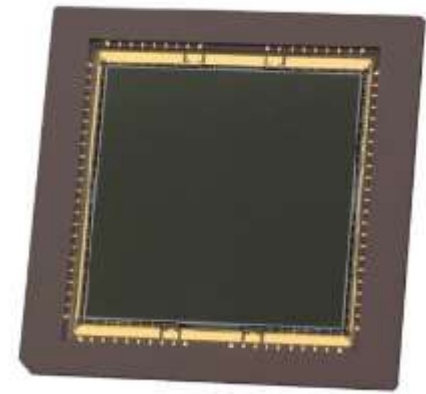
Detector generation	Full well with respect to base binning pix (e ⁻)	Relative
KM4CCD, Sapphire 2x2	256'000	1
Ruby 2x2	128'000	0.5
Atlas 2x2	550'000	2.1
Atlas – S2 4x4	8'800'000	34.3



- State-of-the-art electronics provide:
- Speed
 - Instant binning switch
 - Full well

HW – Detector tech: Read-out times

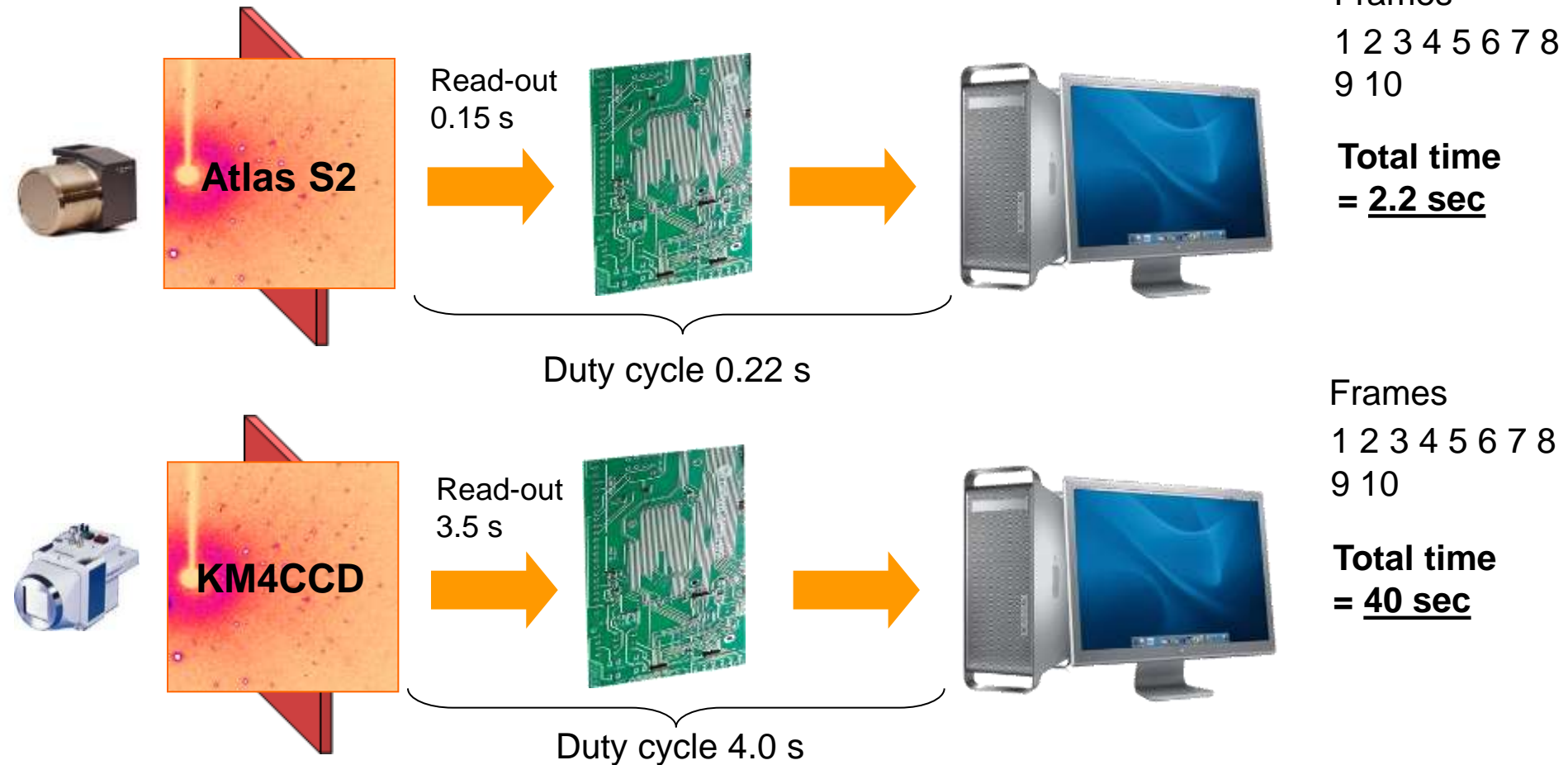
Detector generation	Readout frequency for 1x1 binned image	Relative
KM4CCD, Sapphire 1M	0.075Mhz	1
Ruby 4M	0.420Mhz	5.6
Atlas 4M	2.100Mhz	28
Atlas – S2 4M	4.400Mhz	56.7



State-of-the-art electronics provide:

- Speed

HW – Detector tech: Read-out times



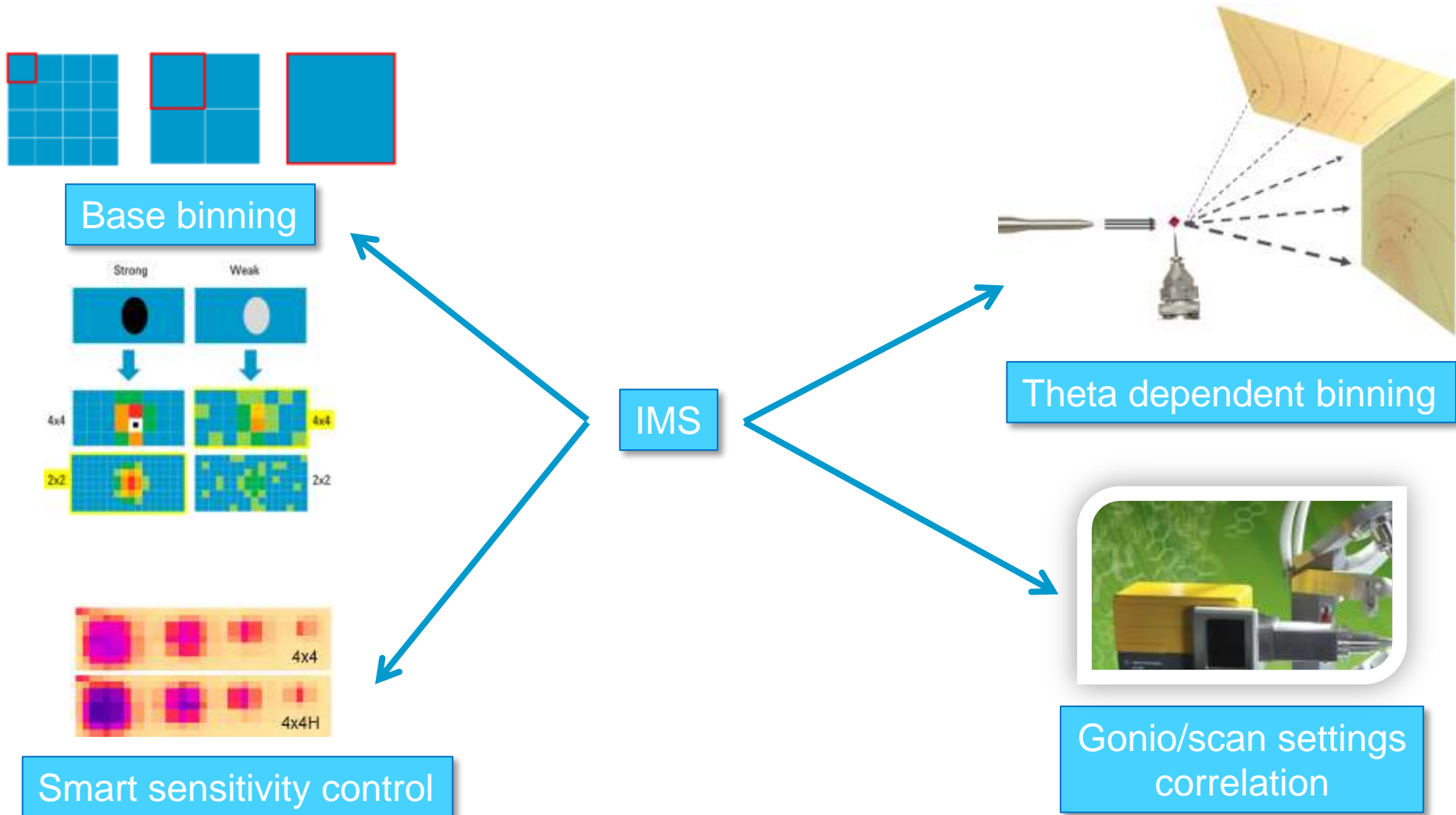
HW – Sources

Same sample 0.3mm, normal tubes (2kW, 0.5mm collimator), micro-focus (50W), GV1000 (1000W)



Source type	Integral intensity relative Enhance Mo
Enhance Mo	1
Enhance Cu	5
Ultra Cu	40
Nova Cu	120
Nova Cu 2 nd gen	240
GV1000	Up to 2400

Intelligent measurement system - IMS



How to increase data quality?

Extending exposure time

- Increase absolute detectivity
- Produce more overloads

Increasing redundancy

- Good for scaling
- Good for absorption correction

Rule of thumb:

“Extend exposure time for weak crystal”

„Increase redundancy for good scaling and absorption correction”



Twinning*: Challenges for the crystallographer

- Identify 'proper' unit cell(s); if possible at the screening/pre-experiment stage
- Reduce overlapping data
- De-convolute and correct data
- Solve the structure
- Refine in best possible way

*non-merohedric



Screening tool for quickly judging sample quality

SM Screening

Screening

Mount

Screening

>

PEAKS
UB fit with 70 obs out of 70 (100.0%)
UNIT CELL (CSD: 14 +437L)
PG: mm orthorhombic P
5.97(2) 9.05(2) 18.356(17)
90.02(13) 90.26(15) 90.2(2)
V = 992(5)

QUALITY
Resolution(A) N I/sig I/sig0
inf - 1.23 91 24.0 26.5
1.28- 1.23(last) 10 12.9 14.2

Well diffracting sample

Diff. limit: beyond 1.23 (theta res. limit) for I/sig=2.0
Mosaicity: e1=1.2, e2=1.2, e3=2.0 (deg), Iso=1.49 (deg)

Experiment - Complete data for publication

Name: exp_209

Detector=52.0mm, Res. = 0.837Ang, I/sig.=15.0, width=1.0deg, Movie, cryo off, Strategy: Complete data (default mode), Exposure: 1.0s 4.0s

Exposure time: 1.0 s

Start Pre-Exp. (5 min) Edit

Goniometer

Omega	Theta	Kappa	Phi	Distance
20.0	-35.0	0.0	0.0	52.0

Generator

kV	mA
50.0	0.80

SM Screening

Screening

Mount

Screening

>

PEAKS
UB fit with 98 obs out of 187 (52.4%)
UNIT CELL (CSD: 0 +0L)
PG: -1 anorthic/triclinic P
10.841(9) 19.320(8) 20.107(19)
73.92(5) 83.35(7) 80.15(5)
V = 3977(5)

QUALITY
Resolution(A) N I/sig I/sig0
inf - 0.91 749 0.7 10.8
1.05- 0.91(last) 83 0.5 7.1

Weakly diffracting sample

NOTE: Quality estimation may be unreliable - you can:
- Increase image binning to 4x4
- Increase exposure time
- Run pre-experiment with suggested exposure time
- Change sample

Experiment - Complete data for publication

Name: exp_101

User=maja user, Detector=43.0mm, Res. = 0.800Ang, I/sig.=15.0, width=1.0deg, Movie, cryo off, Strategy: Complete data (default mode), Exposure: 60.0s

Exposure time: 60 s

Start Pre-Exp. (35 min) Edit


Goniometer

Omega	Theta	Kappa	Phi	Distance
20.0	0.0	0.0	0.0	41.9

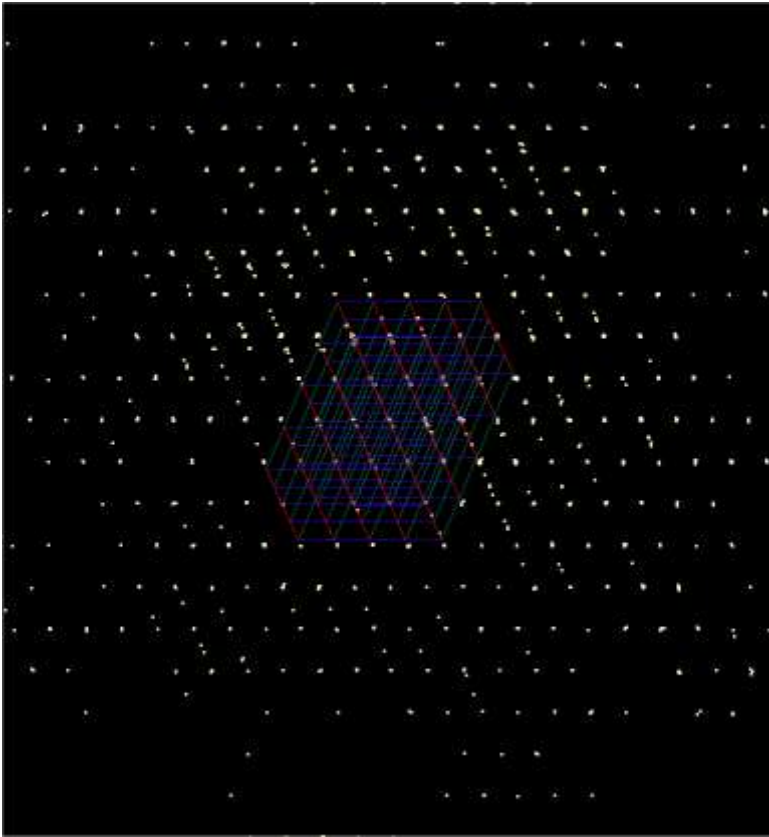
Generator

kV	mA
0.0	0.00

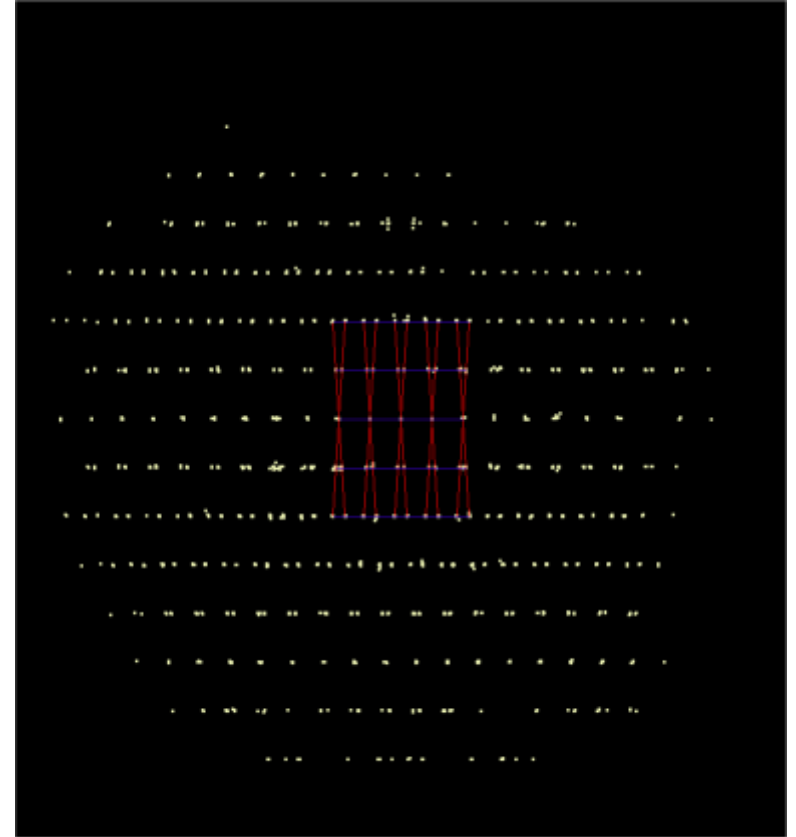
116

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Twin types



- Easy twin



- Super lattice “faker”

Easy twin

Lattice wizard (1.0.3.2)

Lattice

Current cell (CSD: 0 hits)
 7.052(3) 14.162(5) 16.673(5) 65.64(3) 85.12(3) 84.10(3) 1507.4(9)
 Lattice reduction
 selected cell
 7.0511 14.1637 16.6515 65.6629 85.1010 84.1126 aP 31
 reduced cell
 7.0511 14.1637 16.6515 65.6629 85.1010 84.1126 1505.5

PEAK TABLE
 Peak hunting table
 UB fit with 1336 obs out of 1777 (total:1777,skipped:0) (75.18%)

REFINEMENT MODEL
 Geometric
 beam: 0.04194 alpha: 50.04208 beta: -0.01668
 cell zero: -0.73025 th zero: -0.00479 ka zero: -0.00939
 Detector
 x-rot: 0.46124 y-rot: -0.20948
 z-rot: 521.80278 y-cent: 507.49276 distance: 60.30539
 Wavelength Mo (Ang): K1 0.70930 K2 0.71339 K3 0.83229

LATTICE
Current cell (CSD: 0 hits)
 7.052(3) 14.162(5) 16.673(5) 65.64(3) 85.12(3) 84.10(3) 1507.4(9)
 Lattice reduction
 selected cell
 7.0511 14.1637 16.6515 65.6629 85.1010 84.1126 aP 31
 reduced cell
 7.0511 14.1637 16.6515 65.6629 85.1010 84.1126 1505.5

PEAK TABLE
 Peak hunting table
 UB fit with 1336 obs out of 1777 (total:1777,skipped:0) (75.18%)

Mu-calculator (1.0.3): Absorption coefficient in mm-1

Cell and wavelength
 7.05249 14.16205 16.67339 65.642 85.120 84.103 1507.358
 Mo-radiation

Z: 1.00

Chemical formula: (e.g. C11 H10 S O2)
 Numbers follow elements; separate elements by space:

C80 H40 F24

Result
 3 element(s):
 H= 40.00(2.77); C= 80.00(65.93); F= 24.00(31.29)
 Formula wt: 1457.20 Mu(mm-1): 0.14
 Density: 1.605 Z: 1.00
 F(000): 840.00 At.vol: 10.47 Non-H at.vol: 14.49

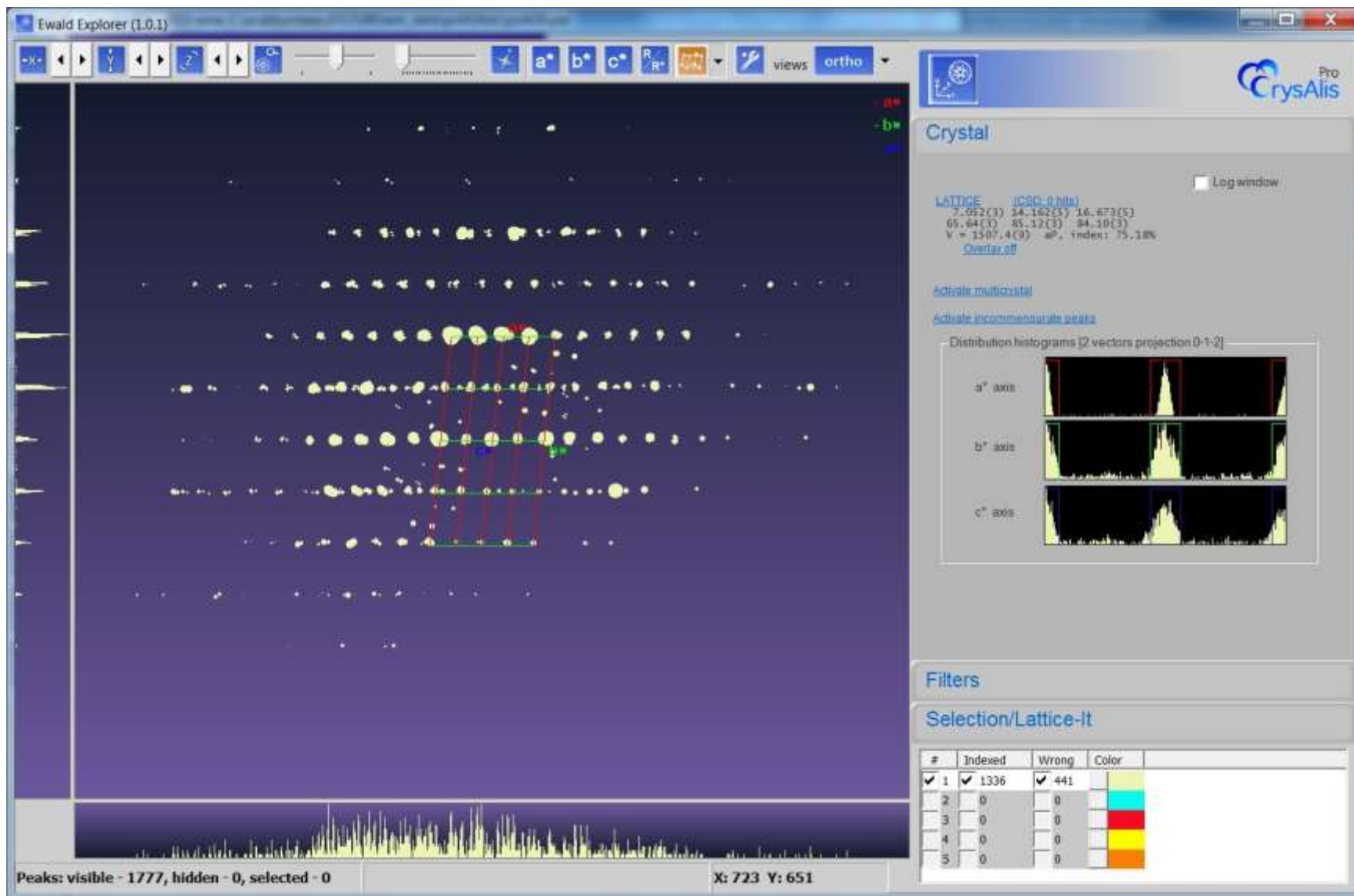
mu (mm-1) 0.14490 ☐ Edit mu

Note: If you change the chemical formula, you may need to reoptimize data reduction to make all output files (like .hkl, .cif, .p4 etc.) consistent for use with external programs (like Olex2, AutoChem, VESTA).

- Indexation <90%
- Chemical content consistent



Easy twin in new Ewald^{Pro}



Easy twin in new Ewald^{Pro}

The screenshot displays the Ewald Explorer (1.0.1) software interface. The main window shows a diffraction pattern with a grid of peaks. A context menu is open over the pattern, listing several options for twin finding and component identification. The right sidebar contains crystallographic data and distribution histograms. The bottom right corner features a table for peak selection and indexing.

Crystal

LATICE [c2d0.0.rin](#)
7.092(3) 14.162(5) 16.673(5)
65.64(3) 85.12(3) 84.10(3)
V = 1507.4(6) aP, index: 75.18%
[Quintacof](#)

[Log window](#)

[Activate multiview](#)

[Activate incommensurate peaks](#)

Distribution histograms [2 vectors projection 0-T-2]

a* axis

b* axis

c* axis

Filters

Selection/Lattice-It

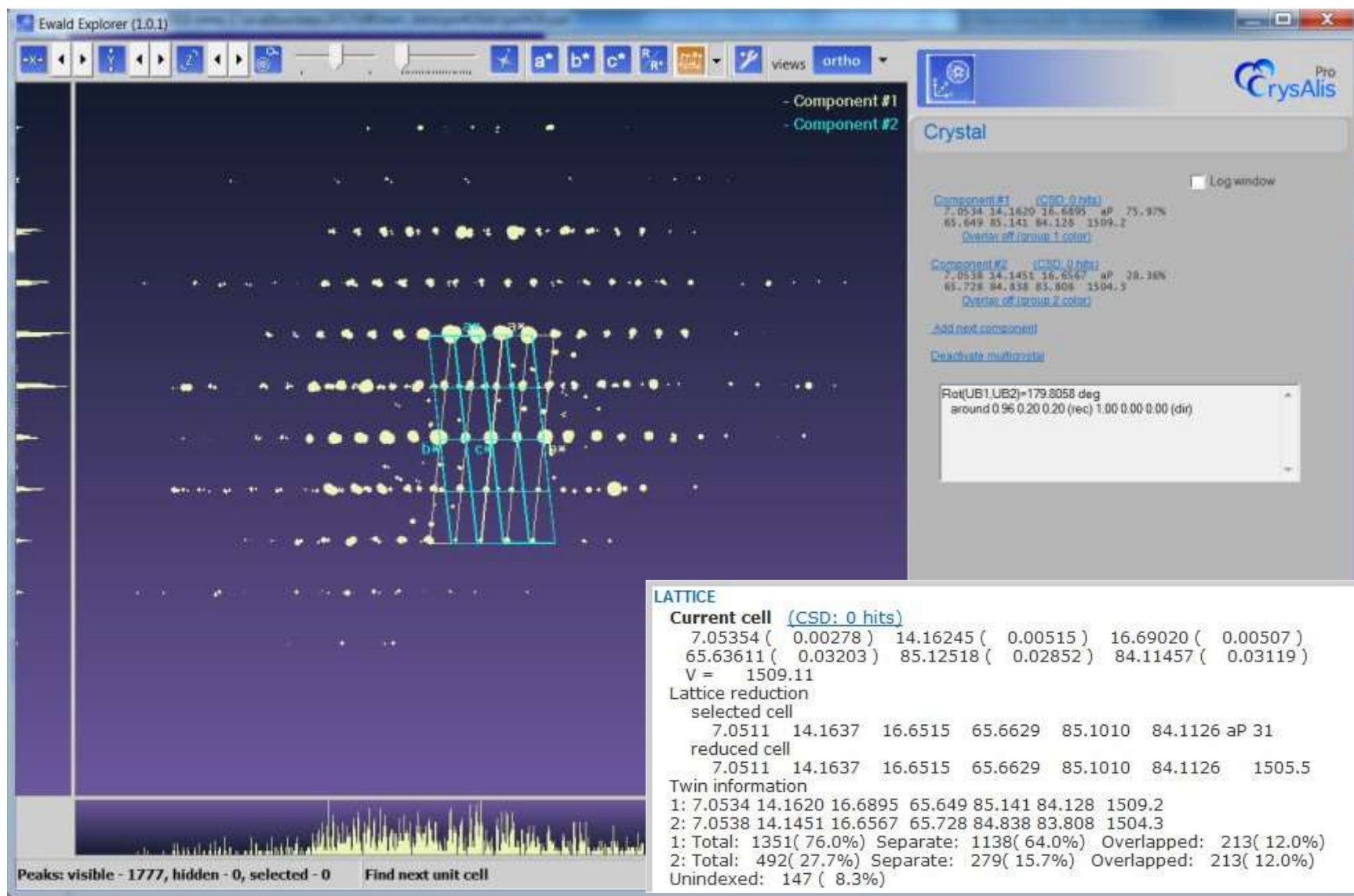
#	Indexed	Wrong	Color
✓ 1	✓ 1336	441	
2	0	0	
3	0	0	
4	0	0	
5	0	0	

Context Menu Options:

- Automatic twin finding
- Custom twin finding
- Find next component in wrong peaks
- Find next component in shown peaks
- Custom component in wrong peaks
- Custom component in shown peaks
- Go to UM TWIN: UB matrix setup for twins

Bottom Panel: X: 958 Y: 813

Easy twin in new Ewald^{Pro}



Super lattice faker

Lattice wizard (1.0.32)

Lattice wizard

LATTICE
Current cell (CSD: 0 hits)
38.427(5) 24.837(3) 45.727(5) 90.036(9) 108.228(11) 89.987(10) 41452(8)
Constrained current cell
38.425(5) 24.838(4) 45.733(6) 90.0 108.251(14) 90.0 41452(10)
Lattice reduction
selected cell
38.4282 24.8376 45.7274 90.0349 108.2283 89.9887 mI 27
reduced cell
24.8376 27.7682 36.3394 72.2609 70.0465 63.4572 20727.5

PEAK TABLE
Peak hunting table
UB fit with 966 obs out of 1077 (total:1077,skipped:0) (89.69%)

INSTRUMENT MODEL
Goniometer
beam: 0.06784 alpha: 50.04288 beta: -0.01668
om zero: -0.82838 th zero: 0.09567 ka zero: -0.90939
Detector
x-rot: 0.41740 y-rot: -0.07486
x-cent: 531.79221 y-cent: 505.32619 distance: 60.13336
Wavelength Mo (Ang): A1 0.70930 A2 0.71359 B1 0.63229

LATTICE
Current cell (CSD: 0 hits)
38.427(5) 24.837(3) 45.727(5) 90.036(9) 108.228(11) 89.987(10) 41452(8)
Constrained current cell
38.425(5) 24.838(4) 45.733(6) 90.0 108.251(14) 90.0 41452(10)
Lattice reduction
selected cell
38.4282 24.8376 45.7274 90.0349 108.2283 89.9887 mI 27
reduced cell
24.8376 27.7682 36.3394 72.2609 70.0465 63.4572 20727.5

PEAK TABLE
Peak hunting table
UB fit with 966 obs out of 1077 (total:1077,skipped:0) (89.69%)

Mu-calculator (1.0.3): Absorption coefficient in mm-1

Cell and wavelength
38.42671 24.83705 45.72677 90.036 108.228 89.987 41451.873
Mo-radiation
Z: 134

Chemical formula: (e.g. C11 H10 S O2)
Numbers follow elements; separate elements by space:
C14H12N3

Result
3 element(s):
H= 12.00(5.45); C= 14.00(75.64); N= 3.00(18.91);
Formula wt: 222.29 Mu(mm-1): 0.07
Density: 1.193 Z: 134.00
F(000): 17956.00 At.vol 10.67 Non-H at.vol 18.20

mu (mm-1) 0.07370 ☐ Edit mu

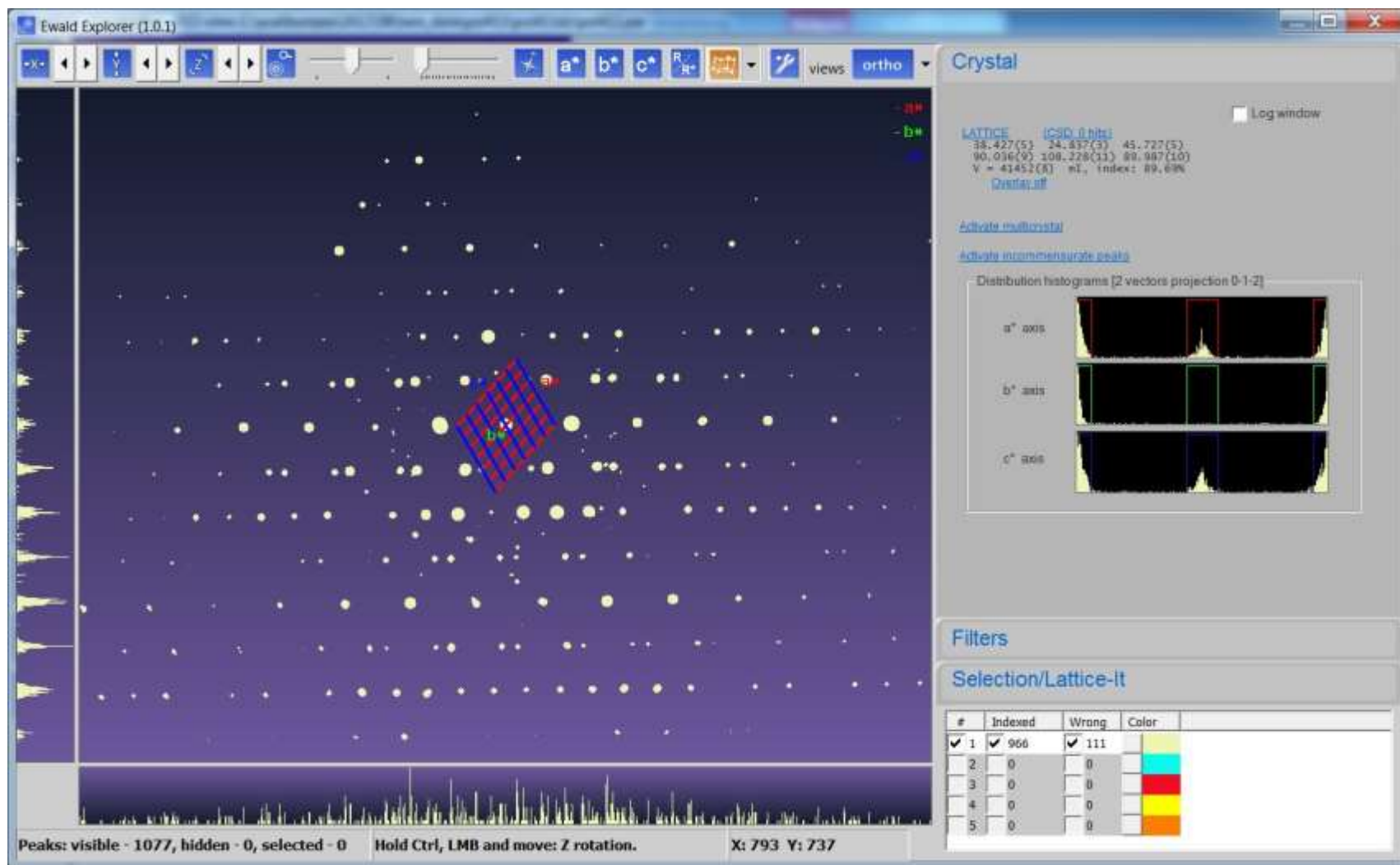
Note: If you change the chemical formula, you may need to reanalyze data reduction to make all output files (like .ins, .cif, .p4p etc.) consistent for use with external programs (like Olex2, AutoChem, WinGX)

Set mu and formula Cancel

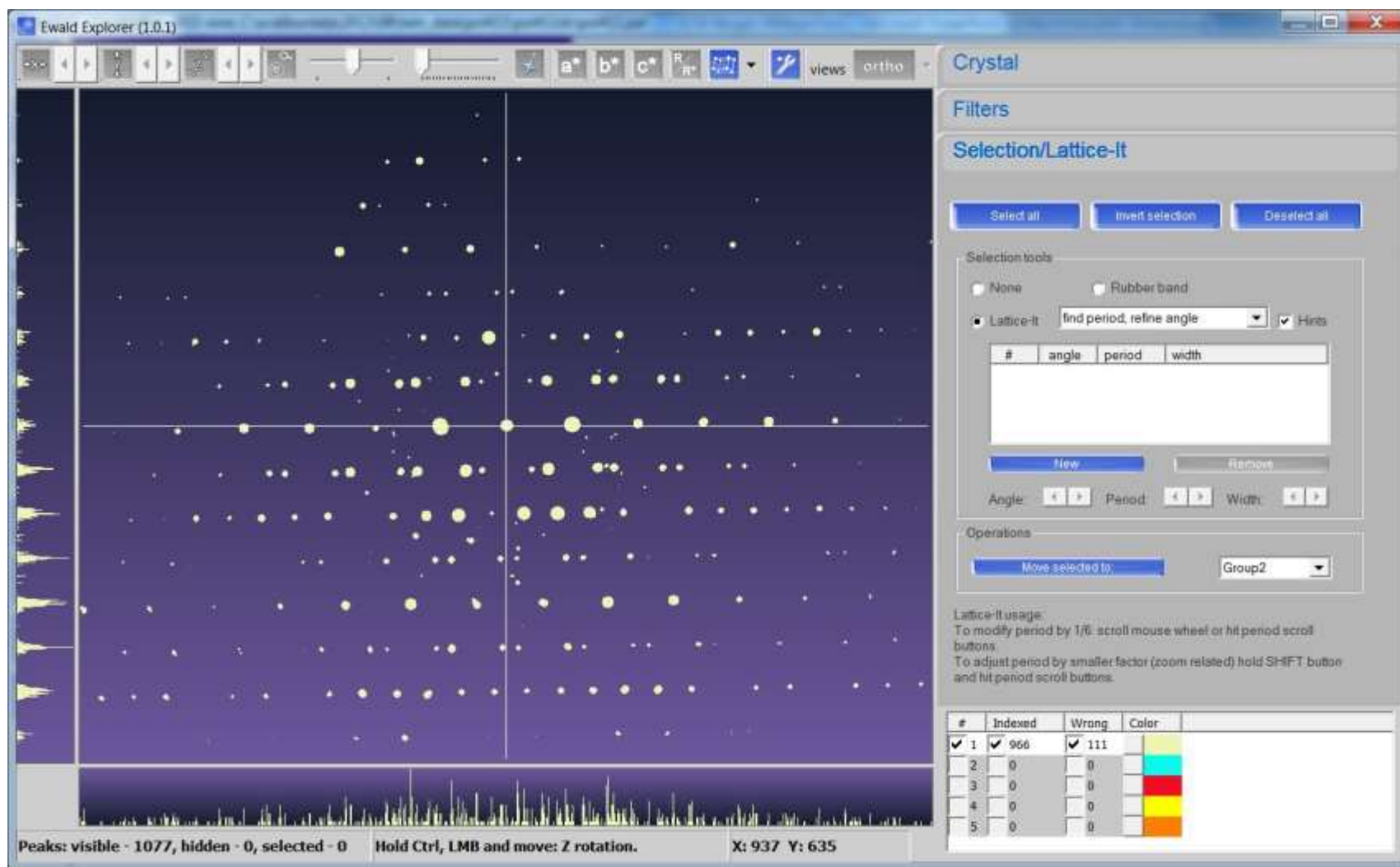
Log window
Close

- Indexation good/high
- Chemical content inconsistent (here Z=134)

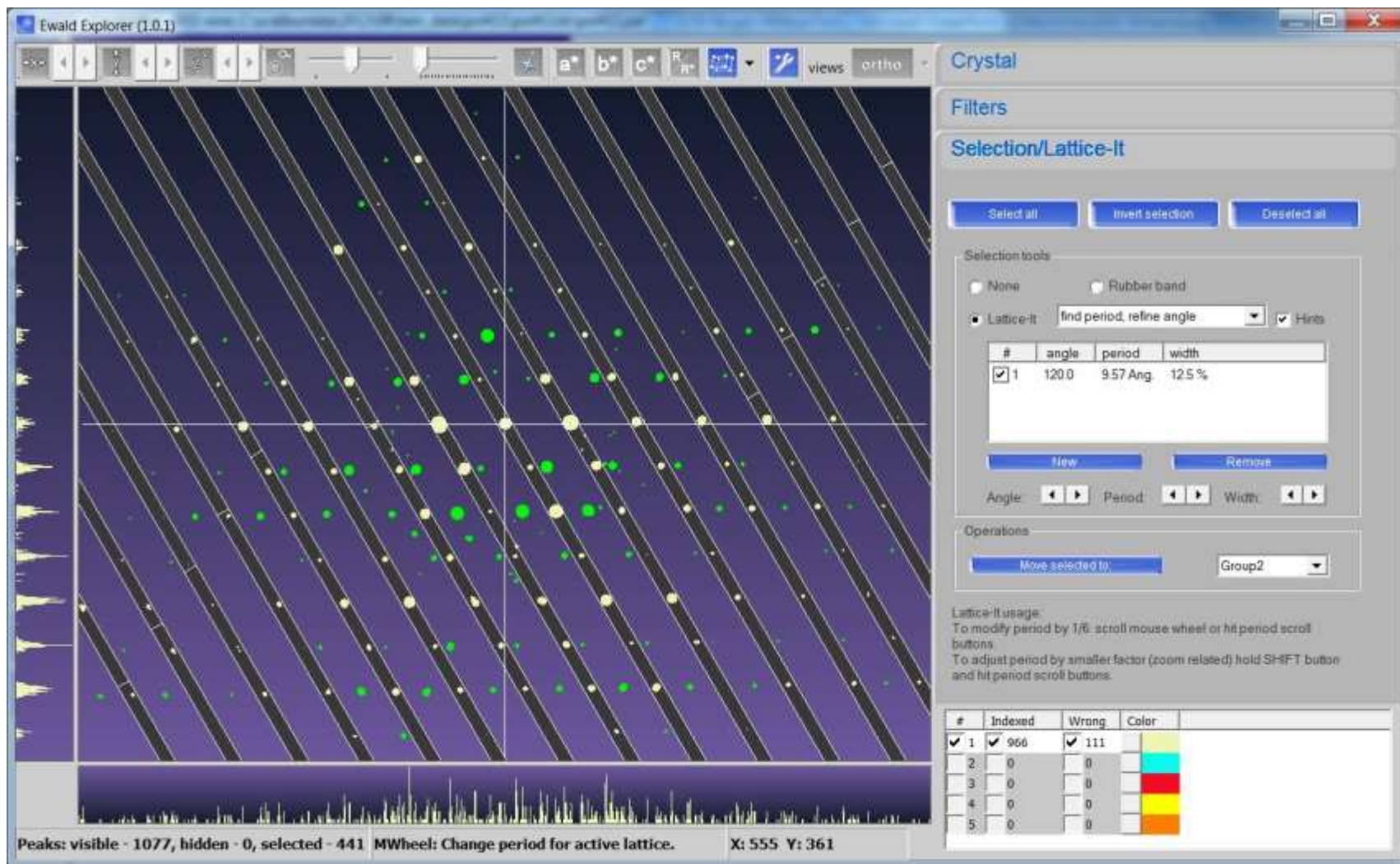
Super lattice faker in new Ewald^{Pro}



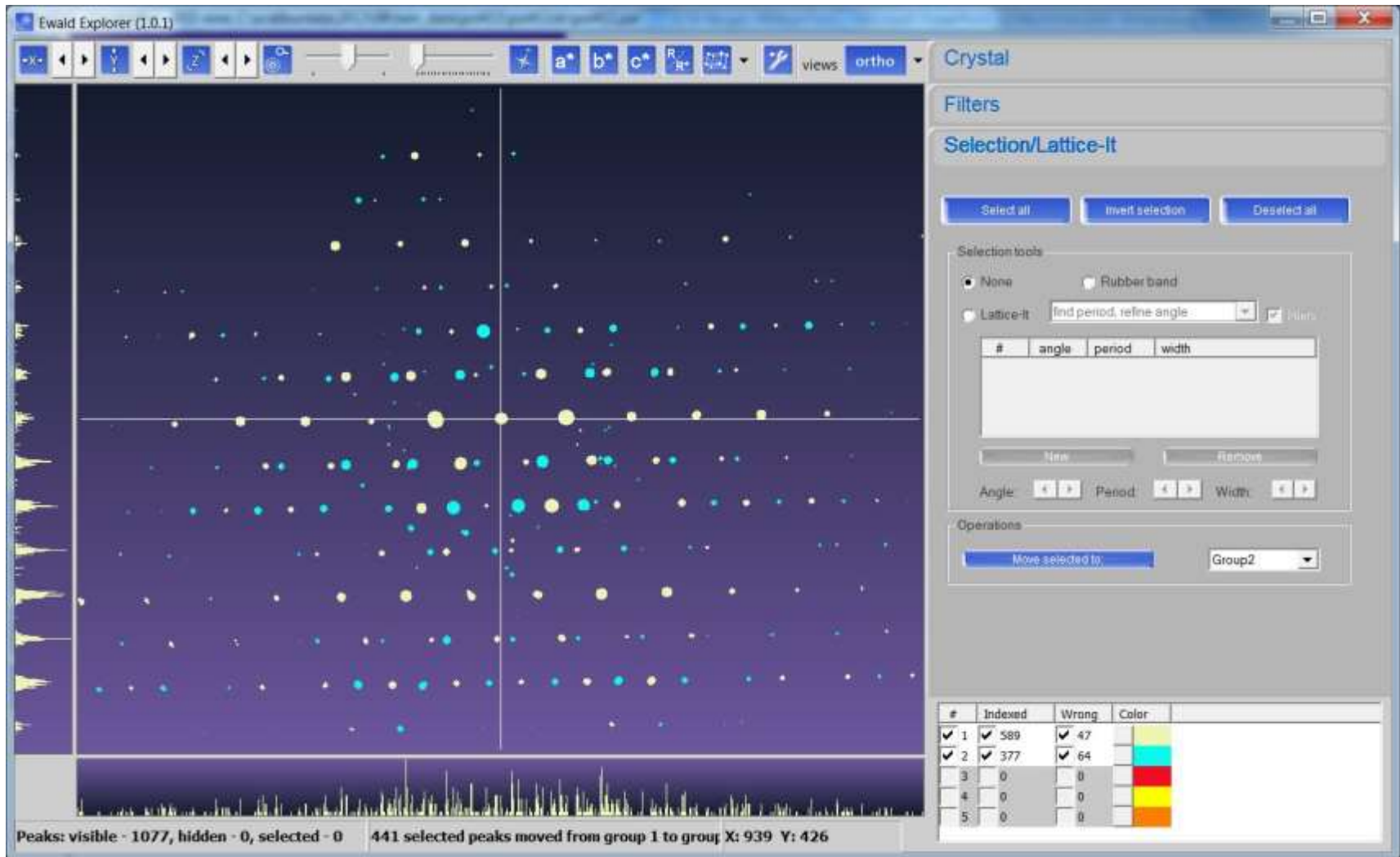
Super lattice faker



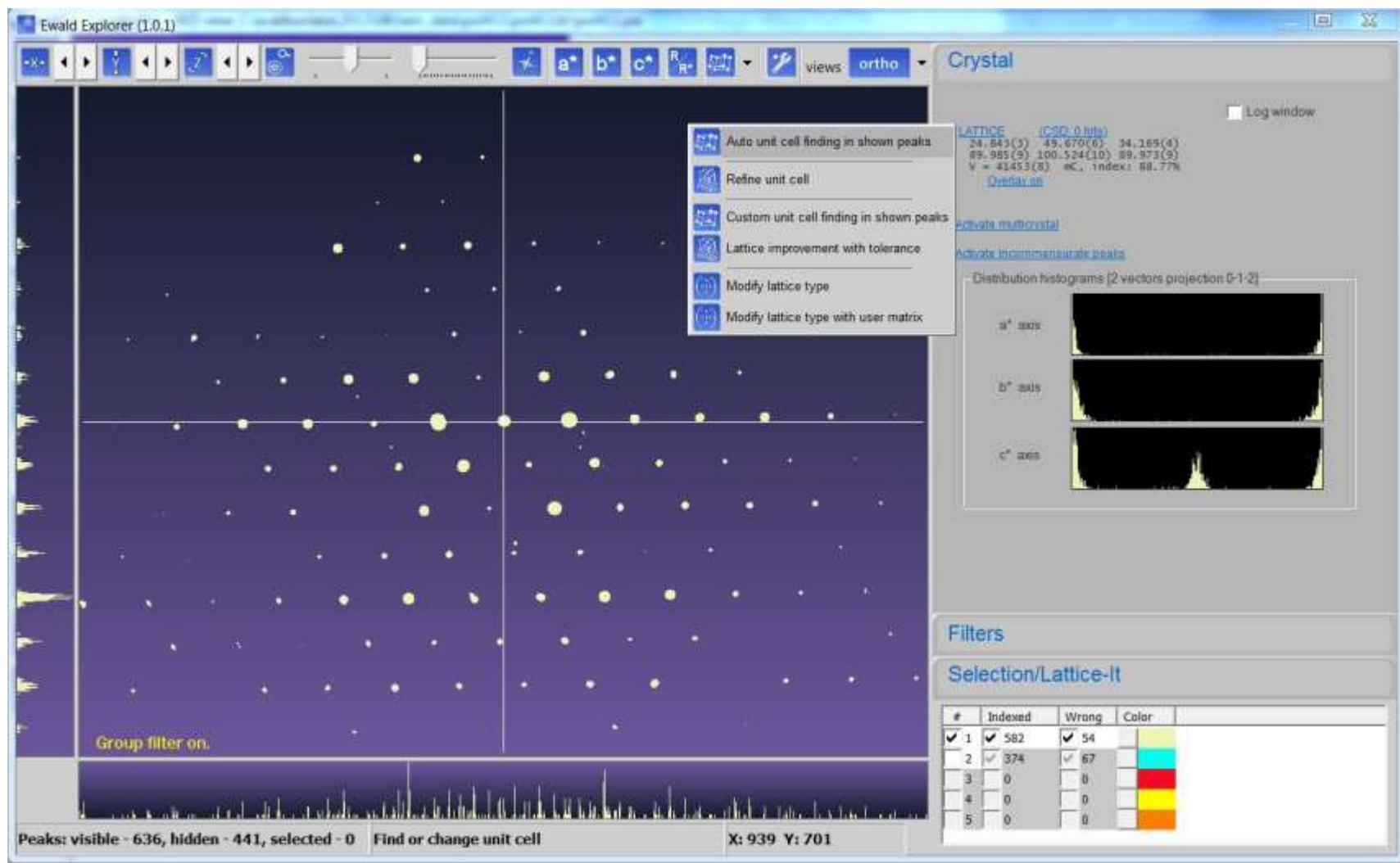
Super lattice faker



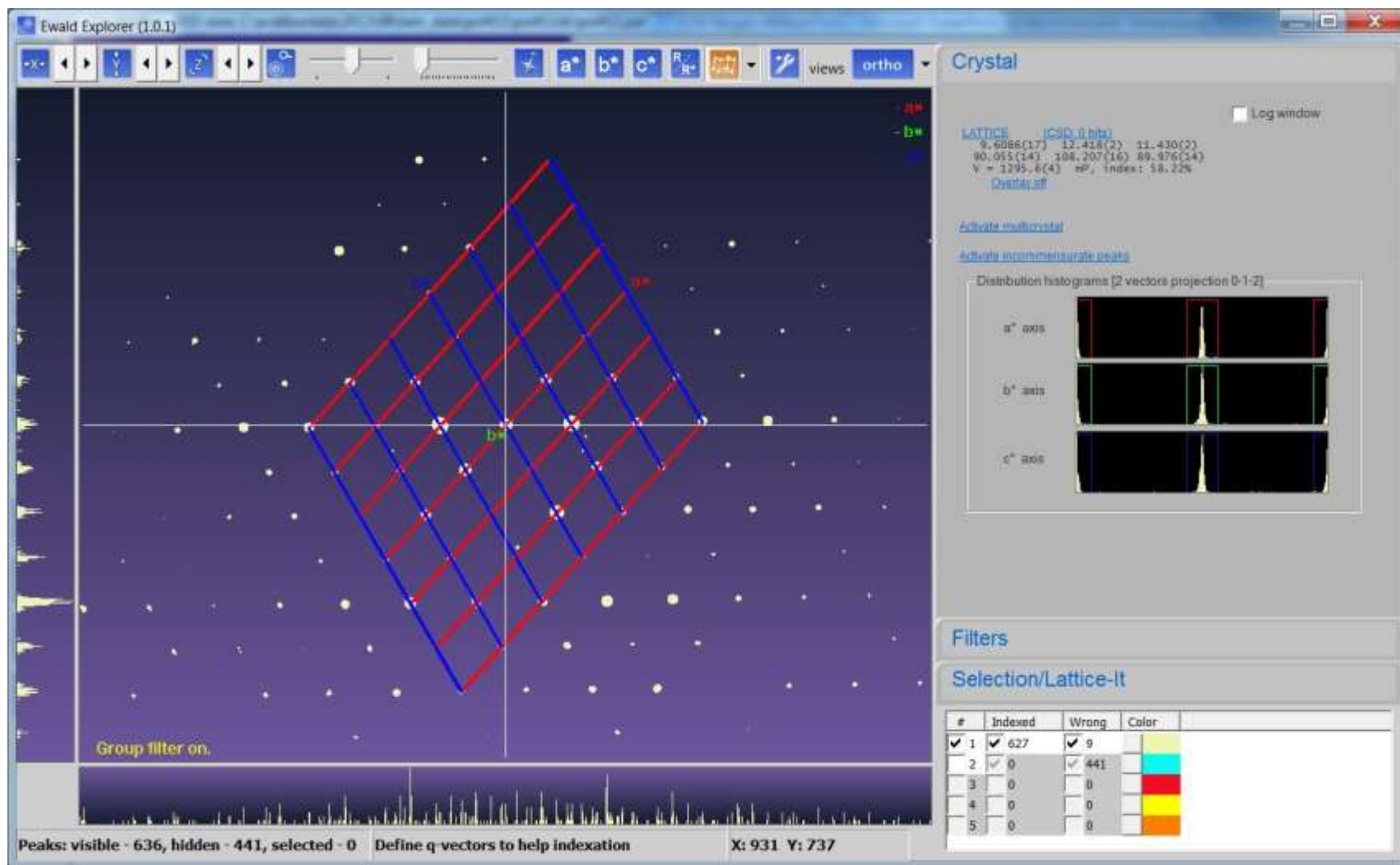
Super lattice faker



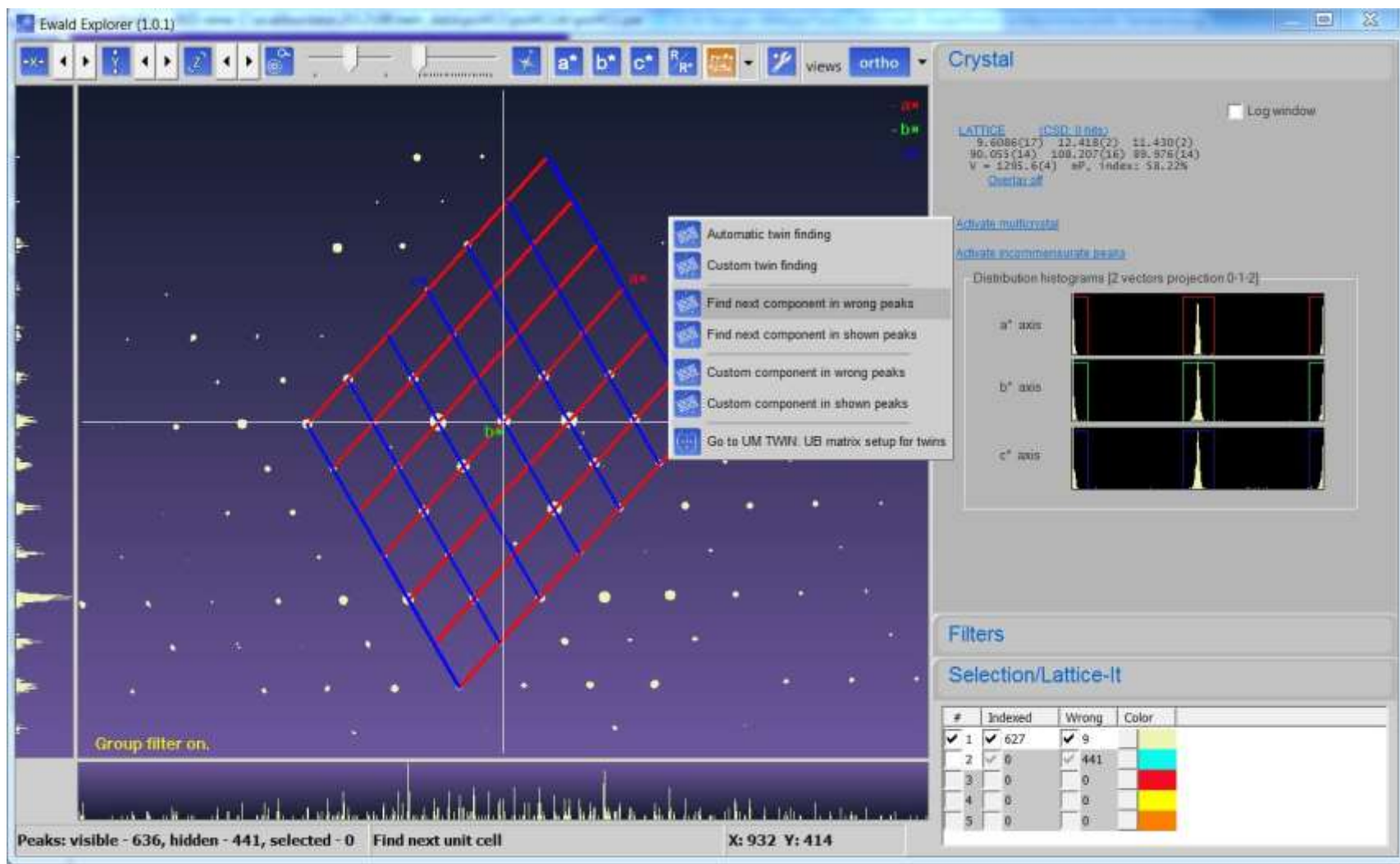
Super lattice faker



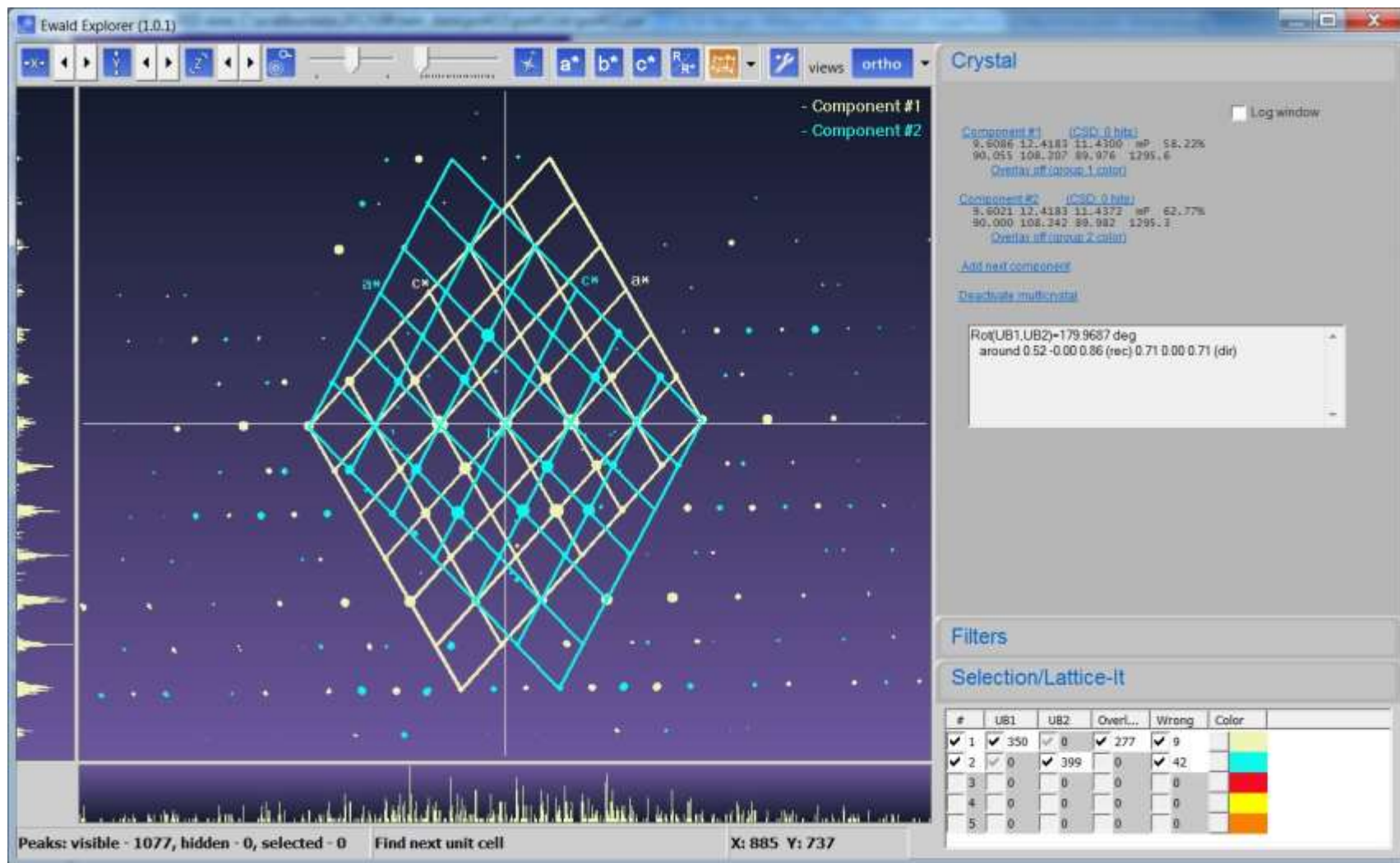
Super lattice faker



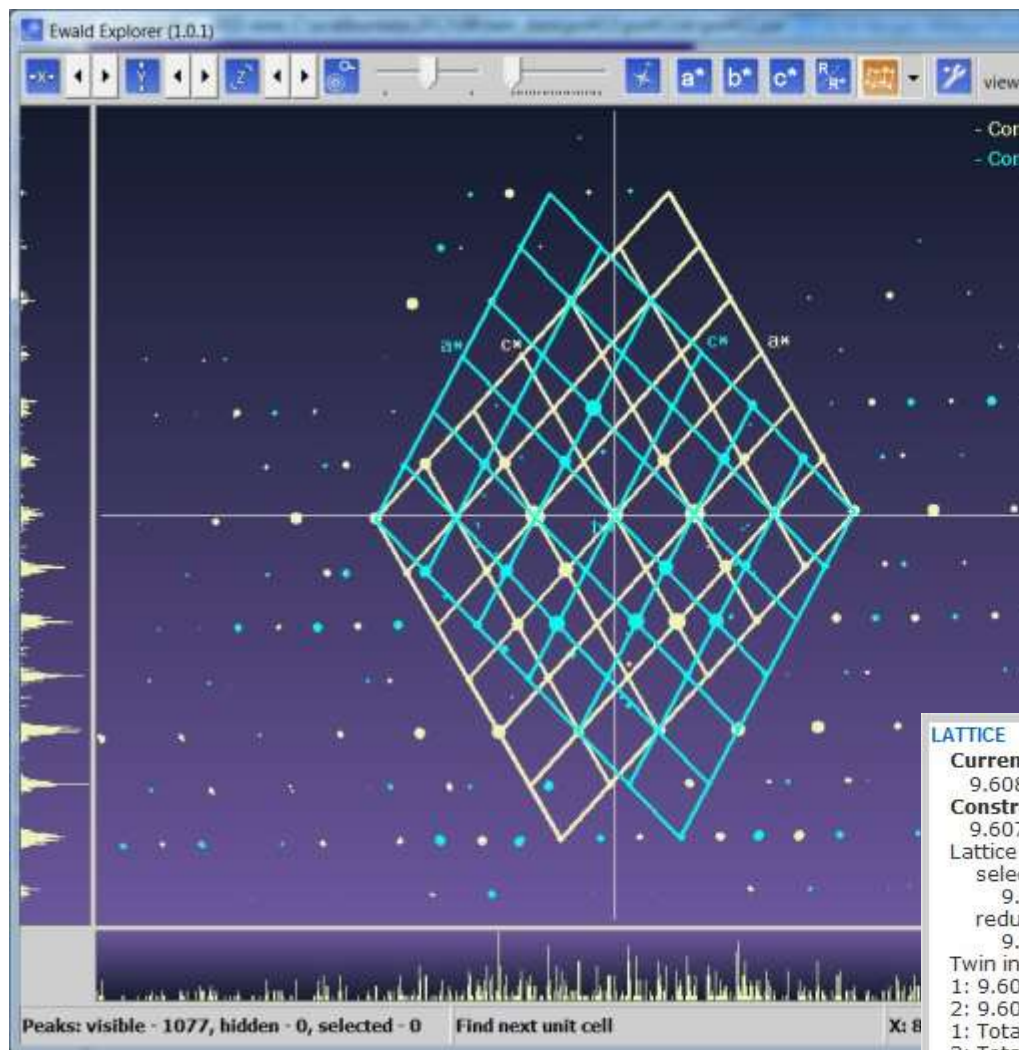
Super lattice faker



Super lattice faker



Super lattice faker



Mu-calculator (1.0.3): Absorption coefficient in mm⁻¹

Cell and wavelength
 9.60859 12.41834 11.43003 90.055 108.207 89.976 1295.581
 Mo-radiation

Z: 4.00

Chemical formula: (e.g. C₁₁H₁₀S₀₂)
 Numbers follow elements, separate elements by space:

C₁₄H₁₂N₃

Result
 3 element(s):
 H= 12.00(5.45) C= 14.00(75.64) N= 3.00(18.91)
 Formula wt 222.29 Mu(mm⁻¹) 0.07
 Density: 1.139 Z= 4.00
 F(000): 536.00 At.vol 11.17 Non-Het.vol 19.05

mu (mm⁻¹) 0.07039 ☐ Edit mu

Note: If you change the chemical formula, you may need to initialize data reduction to make all output files (like .act, .cif, .p4f etc.) consistent for use with external programs (like Olex2, AutoChem, WpCG)

LATTICE

Current cell ([CSD: 0 hits](#))

9.6086(17) 12.418(2) 11.430(2) 90.055(14) 108.207(16) 89.976(14) 1295.6(4)

Constrained current cell

9.607(3) 12.420(2) 11.4327(17) 90.0 108.24(2) 90.0 1295.6(5)

Lattice reduction

selected cell

9.6086 12.4183 11.4300 90.0545 108.2065 89.9764 mP 34

reduced cell

9.6086 11.4300 12.4183 89.9455 89.9764 71.7935 1295.6

Twin information

1: 9.6086 12.4183 11.4300 90.055 108.207 89.976 1295.6

2: 9.6021 12.4183 11.4372 90.000 108.242 89.982 1295.3

1: Total: 627(58.2%) Separate: 350(32.5%) Overlapped: 277(25.7%)

2: Total: 676(62.8%) Separate: 399(37.0%) Overlapped: 277(25.7%)

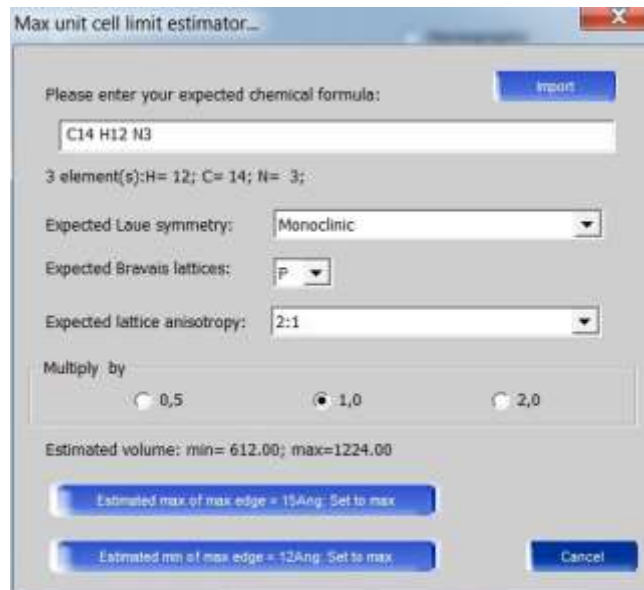
Unindexed: 51(4.7%)



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Other tools in Ewald^{Pro} to support twin handling

- Reflection grouping (up to 20 groups)
- Filters (intensity, d-value, runs [**mistake a moving sample as twin**])
- Custom unit cell finding



Max unit cell limit estimator...

Please enter your expected chemical formula:

3 element(s): H= 12; C= 14; N= 3;

Expected Laue symmetry:

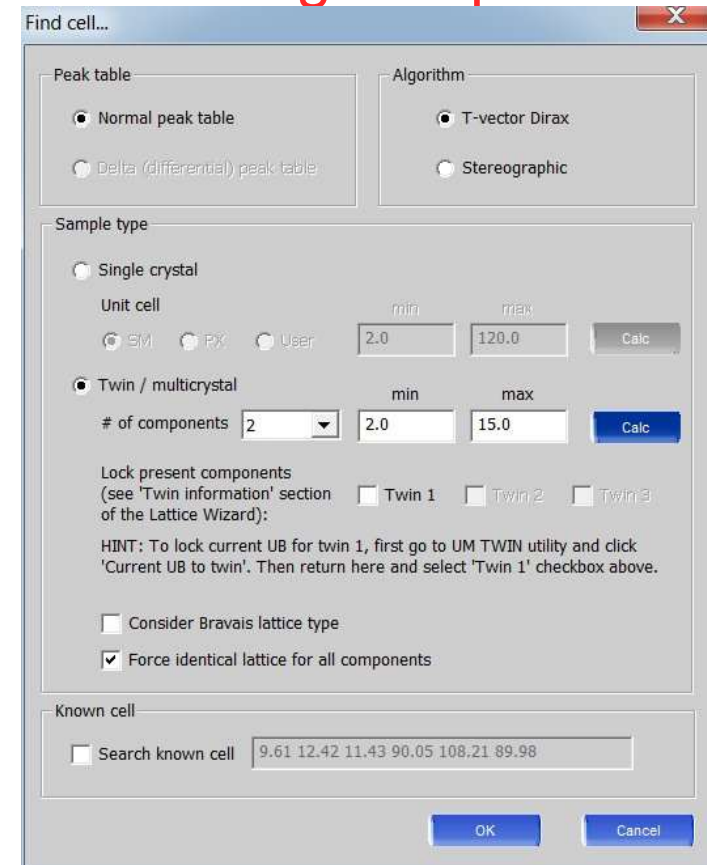
Expected Bravais lattices:

Expected lattice anisotropy:

Multiply by

☐ 0,5 ☒ 1,0 ☐ 2,0

Estimated volume: min= 612.00; max=1224.00



Find cell...

Peak table

☒ Normal peak table

☐ Delta (differential) peak table

Algorithm

☒ T-vector Dirax

☐ Stereographic

Sample type

☐ Single crystal

Unit cell

☒ SM ☐ PX ☐ User

min max

☒ Twin / multocrystal

of components

min max

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.

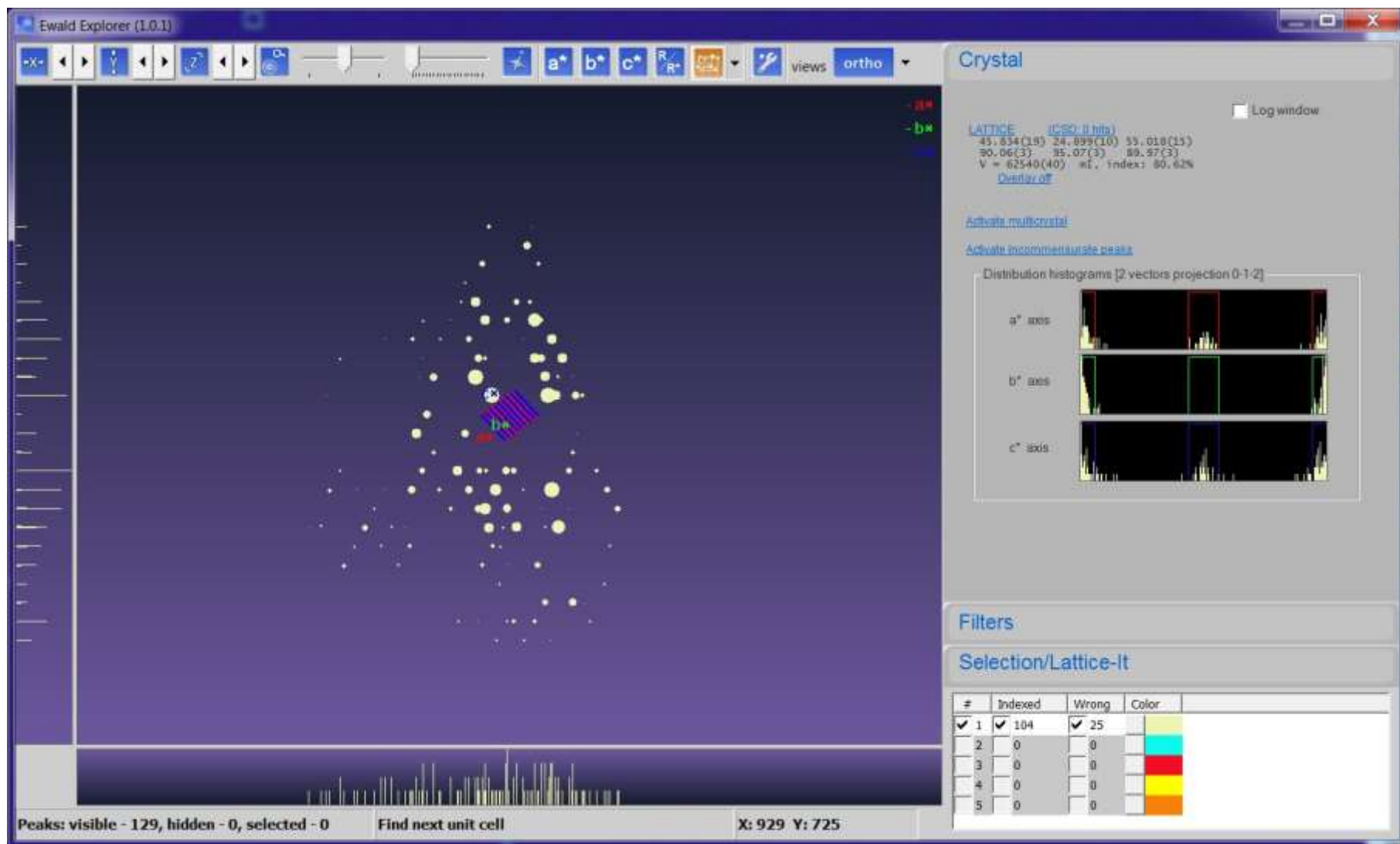
☐ Consider Bravais lattice type

☒ Force identical lattice for all components

Known cell

☐ Search known cell

Judging a twin from few reflections can be tricky...



Use the custom unit cell tool...

Find cell...

Peak table
☒ Normal peak table
☐ Delta (differential) peak table

Algorithm
☒ T-vector Dirax
☐ Stereographic

Sample type
☐ Single crystal
Unit cell
☒ SM ☐ PX ☐ User min max
2.0 120.0 Calc
☒ Twin / multocrystal
of components 2 min max
2.0 15.0 Calc
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 9.61 12.42 11.43 90.05 108.21 89.98

OK Cancel

Crystal

LATTICE ICSD: 011111
11.457(6) 12.434(6) 9.631(5)
90.02(4) 108.30(5) 89.96(4)
V = 1303(1) Å³ Z = 4 Index: 66.67%

Log window

Activate multibrain
Activate incommensurate peaks

Distribution histograms (2 vectors projection 0-1-2)

a* axis
b* axis
c* axis

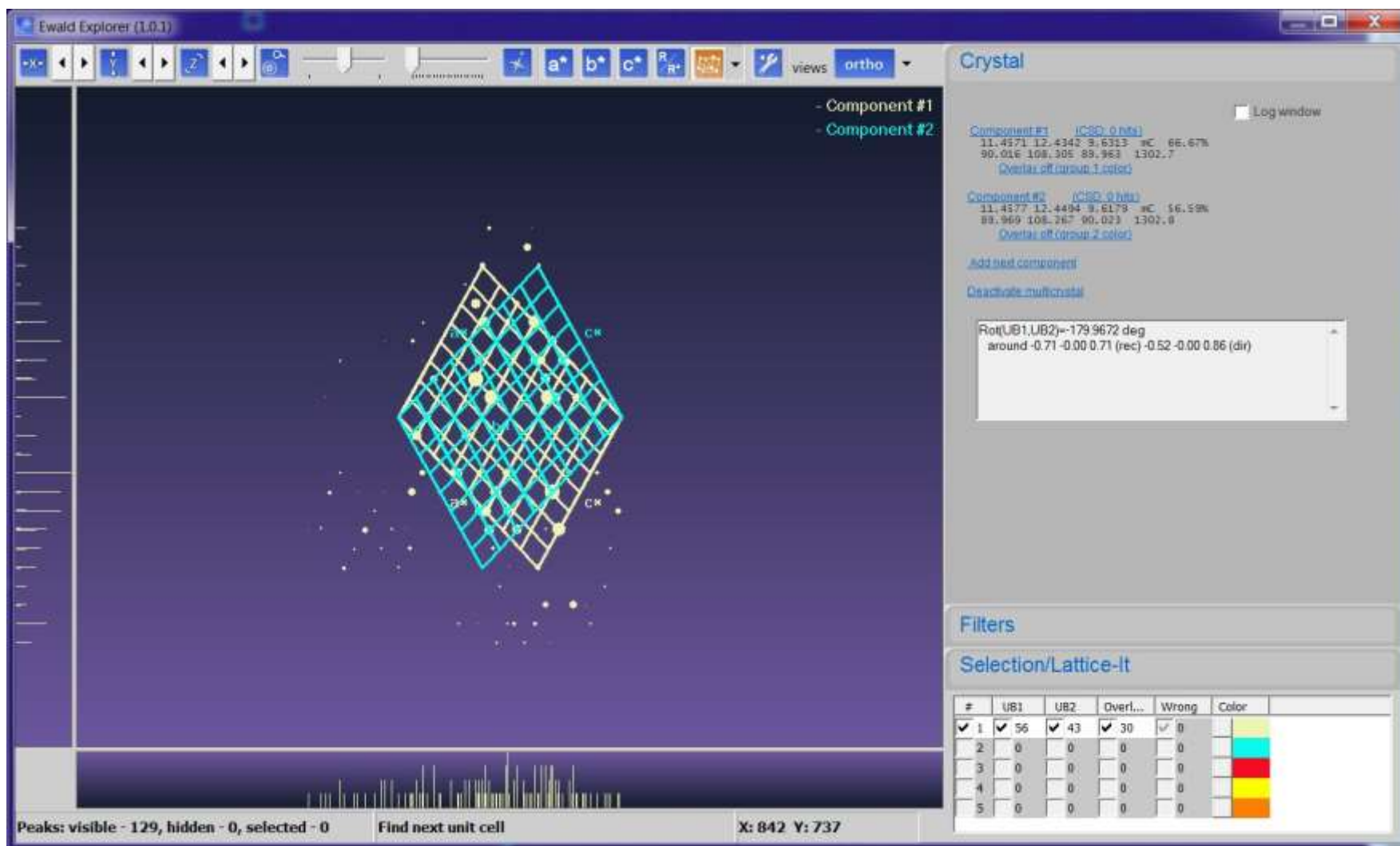
Filters

Selection/Lattice-It

#	Indexed	Wrong	Color
✓ 1	✓ 86	✓ 43	
2	0	0	
3	0	0	
4	0	0	
5	0	0	

Peaks: visible - 129, hidden - 0, selected - 0 Find or change unit cell X: 965 Y: 612

Find the twin from the wrong peaks...



Strategy with known twin law

Experiment Strategy (1.1.0)

Unit cell for Strategy Calculation ([CSD: 0.100](#))
Cell: 11.430(2) 12.418(2) 9.6086(17) 90.024(14) 108.207(16) 89.945(14) 1295.6(4) mC C-1attice 58.22% (627 of 1077 reflections) LatBox Wizard

Strategy parameters

- Resolution: ☐ Theta ☐ 2Theta 0.800
- Law group: ☐ Other 2/m (b-unique)
- ☒ Friedel mates are equivalent (uncheck for high quality absolute configuration data)
- Detector Distance 80.18

Strategy mode
Complete data for twins

Time prediction
default time: 10.00
☒ The same time for all theta positions
☐ Different time for each theta position
Scan width: 1.00

Settings/Options
Data collection/Start CCD processing Automate/Move/Cryo/Ref

Calculate New Strategy
Update Completeness

Mon Aug 06 07:21:55 2012
Completeness/Coverage tables
Completeness in 2/m (b-unique)

Strategy mode

Complete data for twins

limit 100.0 IUCr limit Max 99.74 %

Generates runs that reach completeness limit for all twin components

Completeness

Frame

Redundancy for completeness

Redundancy for coverage

Frame

Full sphere (F1)

The screenshot shows the CrysAlis Pro software interface for setting up an experiment strategy. The main window displays various parameters for a C-1attice unit cell. A dialog box titled 'Strategy mode' is open, showing the 'Complete data for twins' mode with a limit of 100.0 and a maximum completeness of 99.74%. Below the dialog, two graphs are displayed: 'Completeness' vs 'Frame' and 'Redundancy for completeness' vs 'Frame' for the 'Full sphere (F1)'.

Data reduction

CrysAlisPro: Data reduction (1.13)

Load new experiment

Full auto analysis (cell, red)

Data reduction with options



Proffit: CrysAlisPro data reduction assistant (1.0.26)

Simultaneous twin data reduction



Step 1: Orientation matrix for data reduction

UB - matrix:

```
-0.023887  0.014294  0.057126 ( 0.000009  0.000010  0.000011 )
-0.038447 -0.045998 -0.015443 ( 0.000010  0.000011  0.000011 )
 0.046928 -0.030486  0.050268 ( 0.000009  0.000010  0.000011 )
11.45724 ( 0.00186 ) 12.44279 ( 0.00203 ) 9.62081 ( 0.00158 )
89.94327 ( 0.01328 ) 108.28312 ( 0.01460 ) 89.96511 ( 0.01317 )
```

V = 1302.30

Selected cell (from UM rr/UM ttt/UM f):

14 11.4572 12.4428 9.6208 89.9433 108.2831 89.9651 mC

Auto analyse found P-lattice in peak hunting data!

Twin 1: 11.45724 12.44279 9.62081 89.9433 108.2831 89.9651 1302.30

Twin 2: 11.45006 12.44351 9.62704 90.0025 108.2504 90.1021 1302.65

Lattice extinctions (filter Bravais lattice extinctions)

☐ Don't use filter (P-lattice)

☒ Use filter for: C-lattice

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:

☐ Multi crystal

☒ Component 1

☒ Component 2

☐ Component 3

☐ Component 4

< Zurück

Weiter >

Fertig stellen

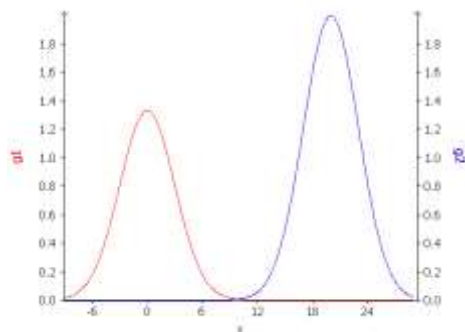
Abbrechen

Hilfe

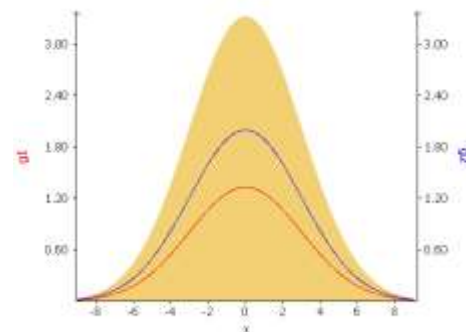


Agilent Technologies

Twin profile data

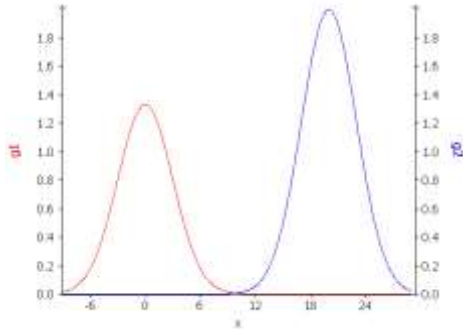


Fully separated

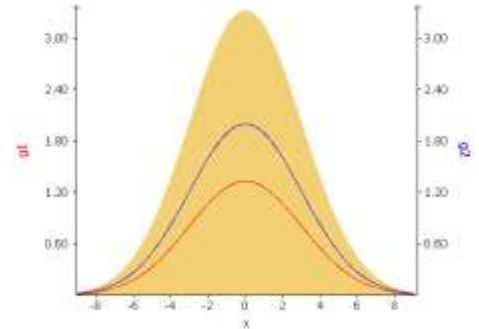


80% - 100%
Overlap data

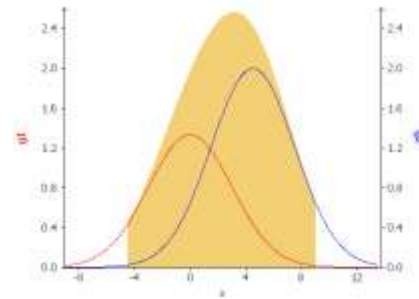
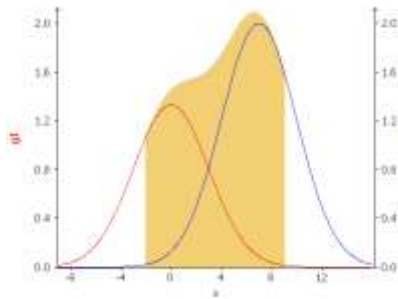
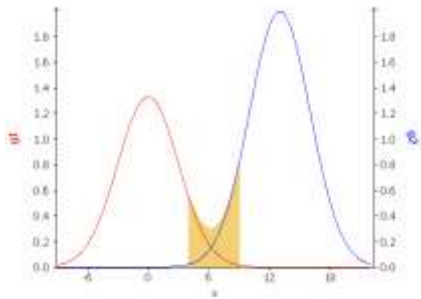
Twin profile data



Fully separated



80% - 100%
Overlap data



0%

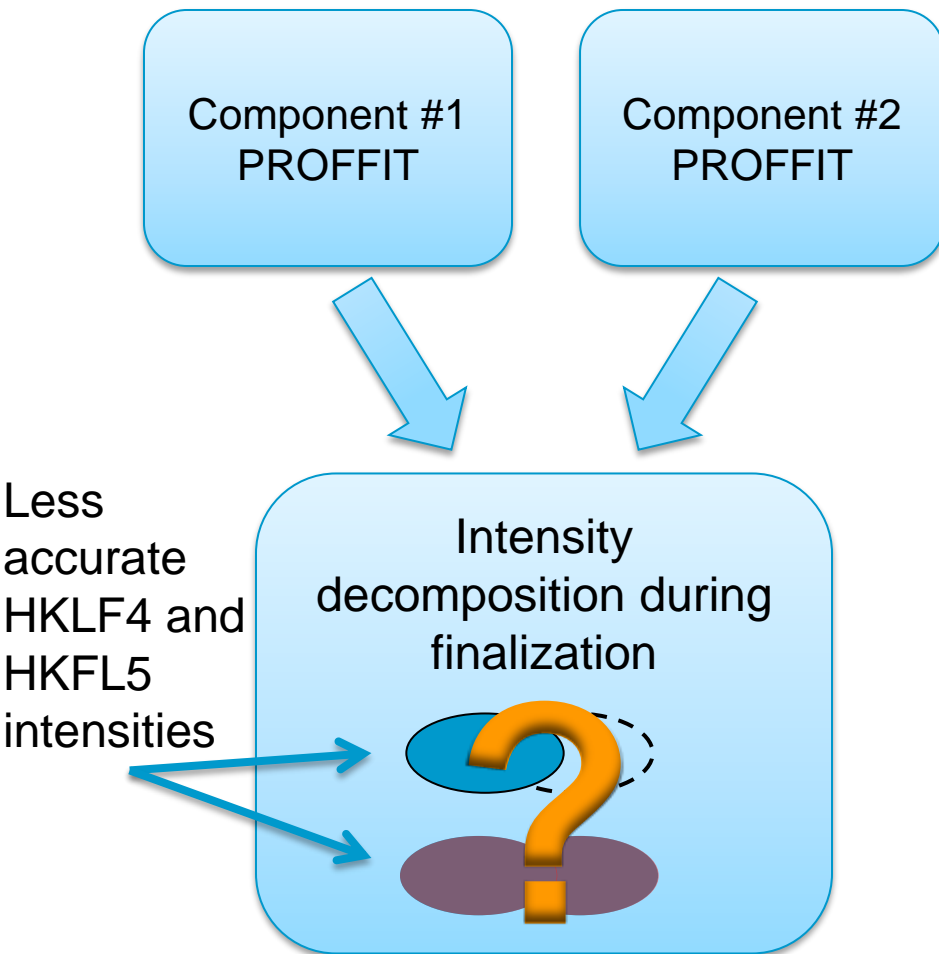
Deconvolution
possible

80%

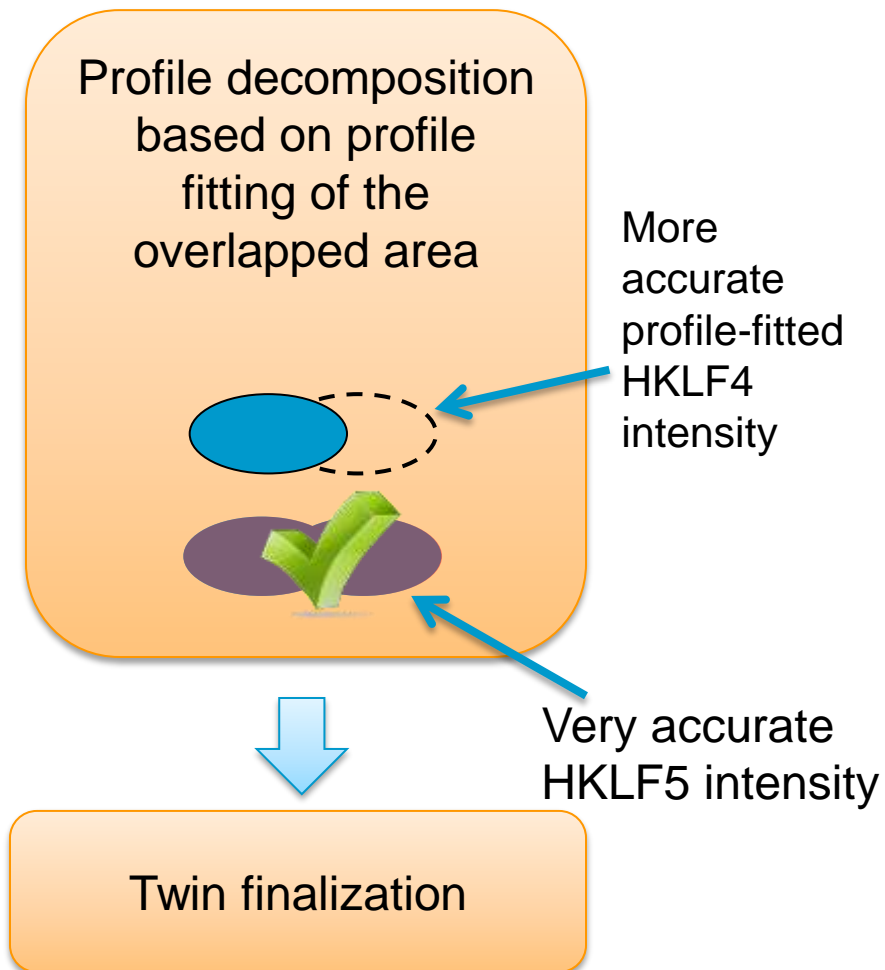
Current vs. new approach

New
37

Current twin integration

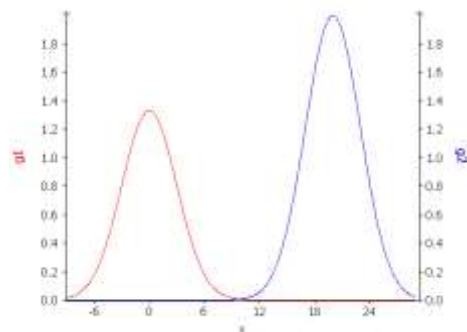


New simultaneous integration

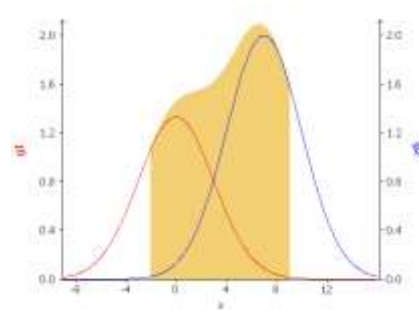


HKLF4 and HKLF5 play modes

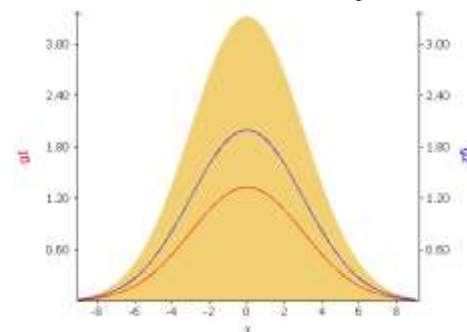
Fully separated



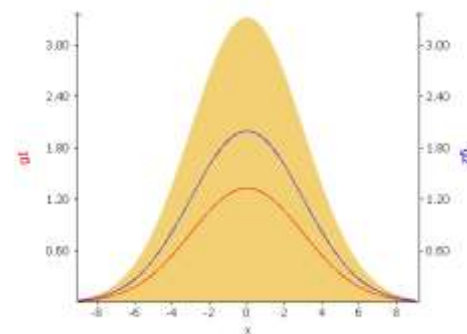
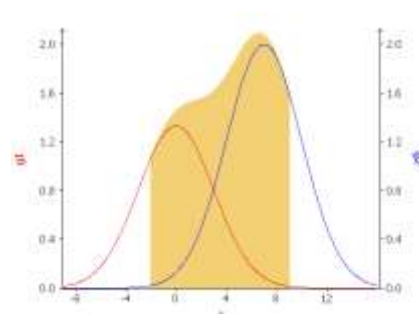
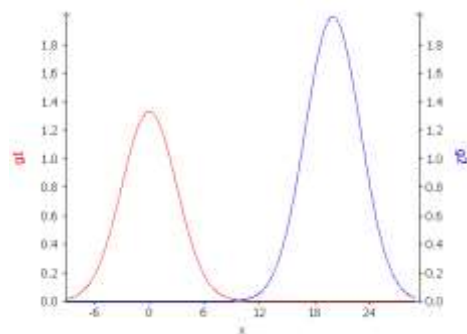
Partially overlapped



'Full' overlap



Component 1



Component 2

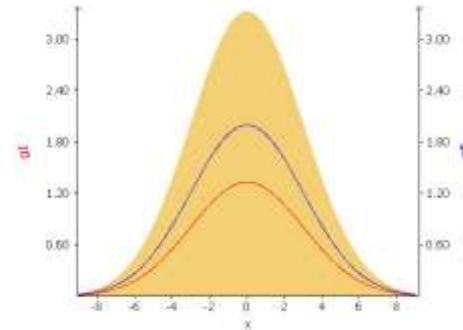
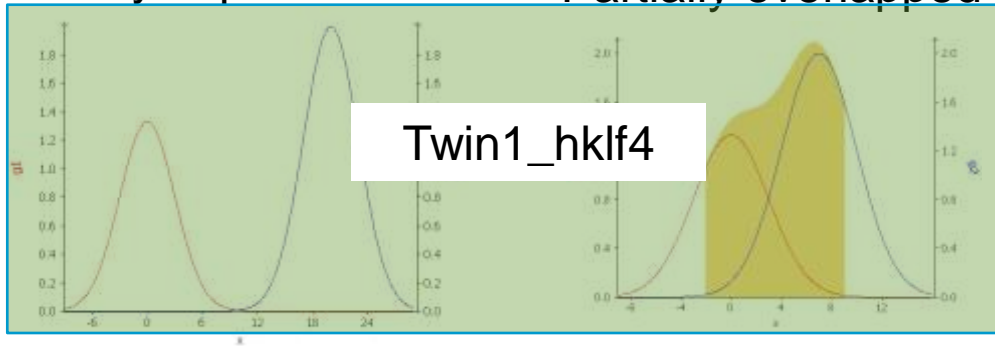
...
Component n

HKLF4 and HKLF5 play modes: 1st trial

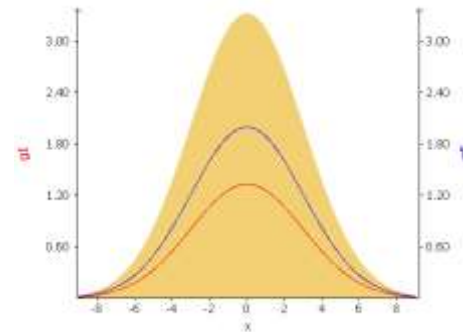
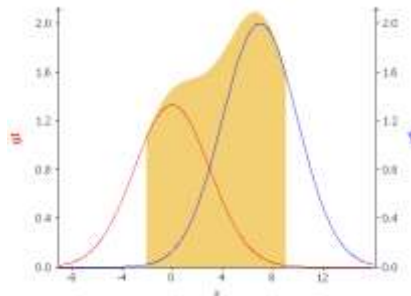
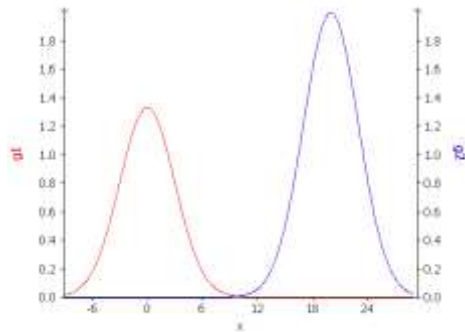
Fully separated

Partially overlapped

'Full' overlap



Component 1

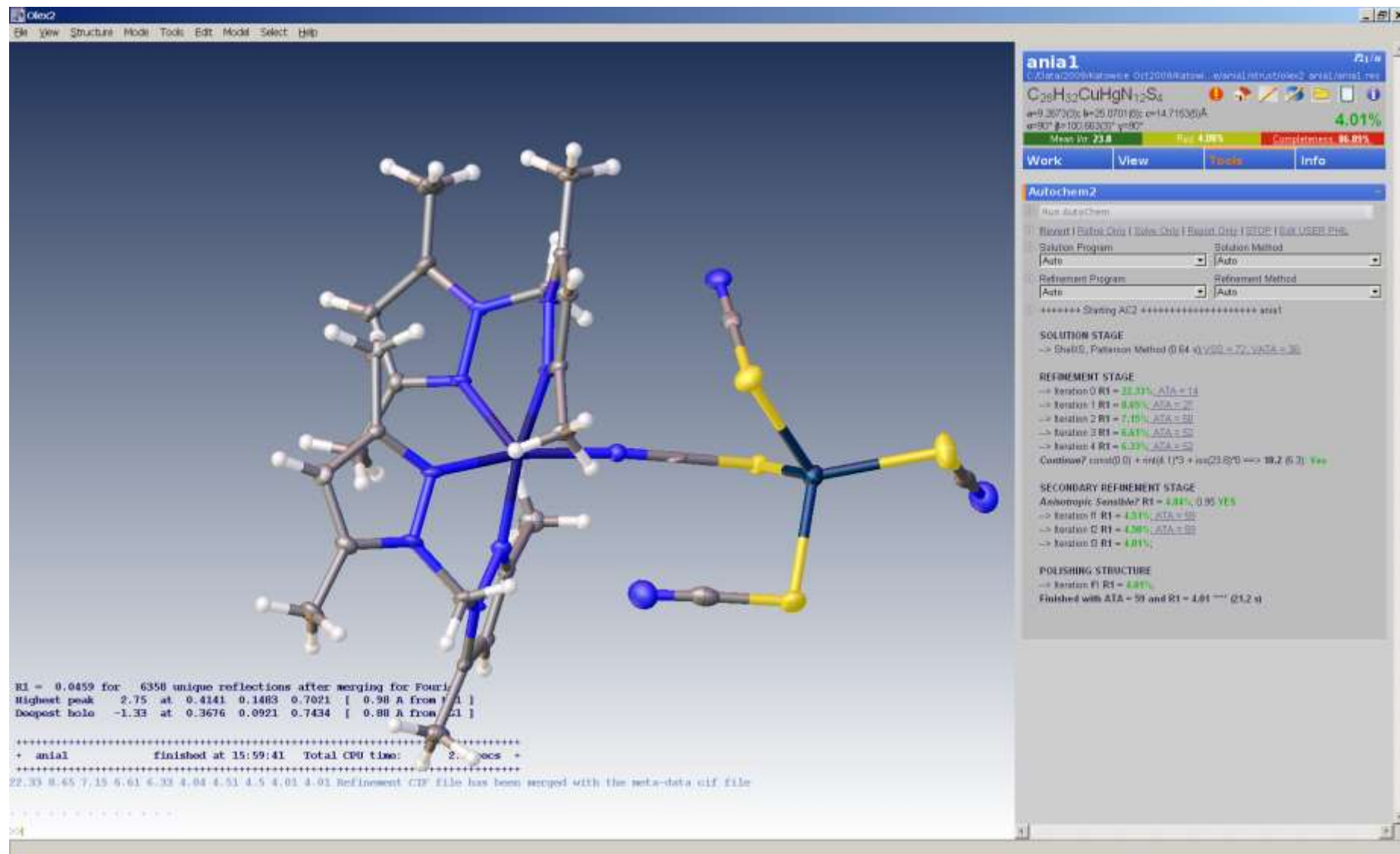


Component 2

...

Component n

Structure solve and refine using AutoChem^{2.0}

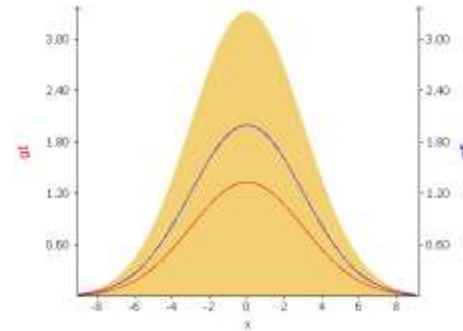
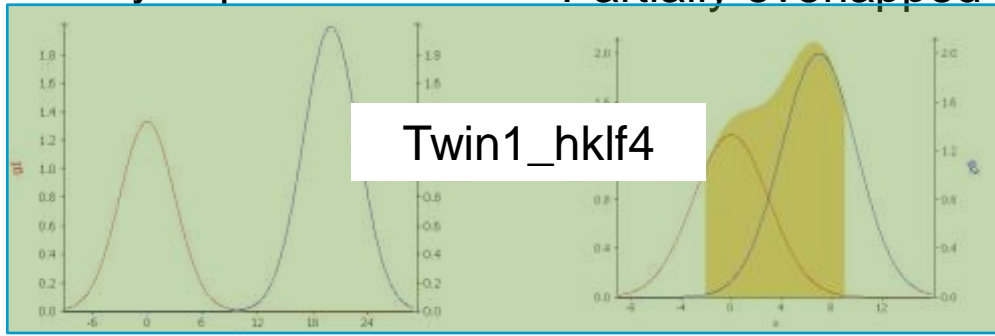


Get extra data in to solve the structure...

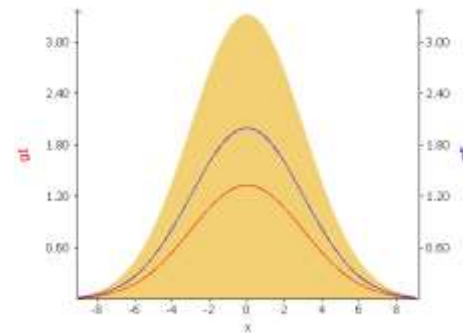
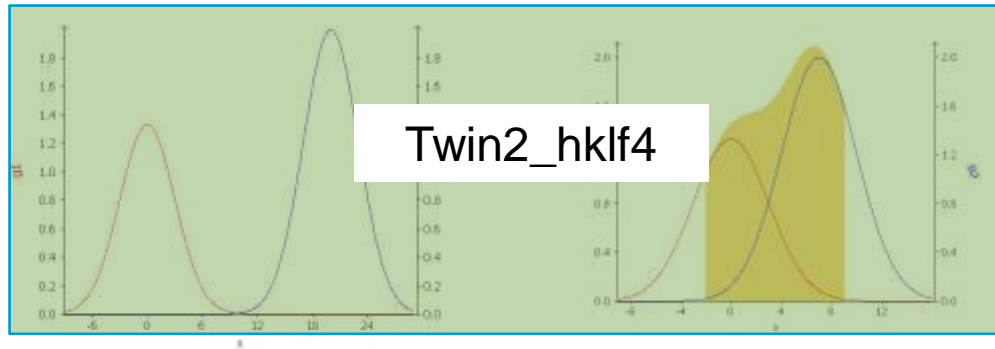
Fully separated

Partially overlapped

'Full' overlap



Component 1



Component 2

...

Component n

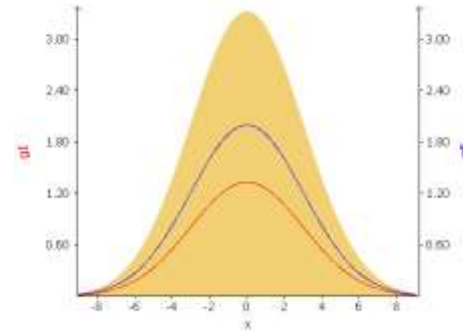
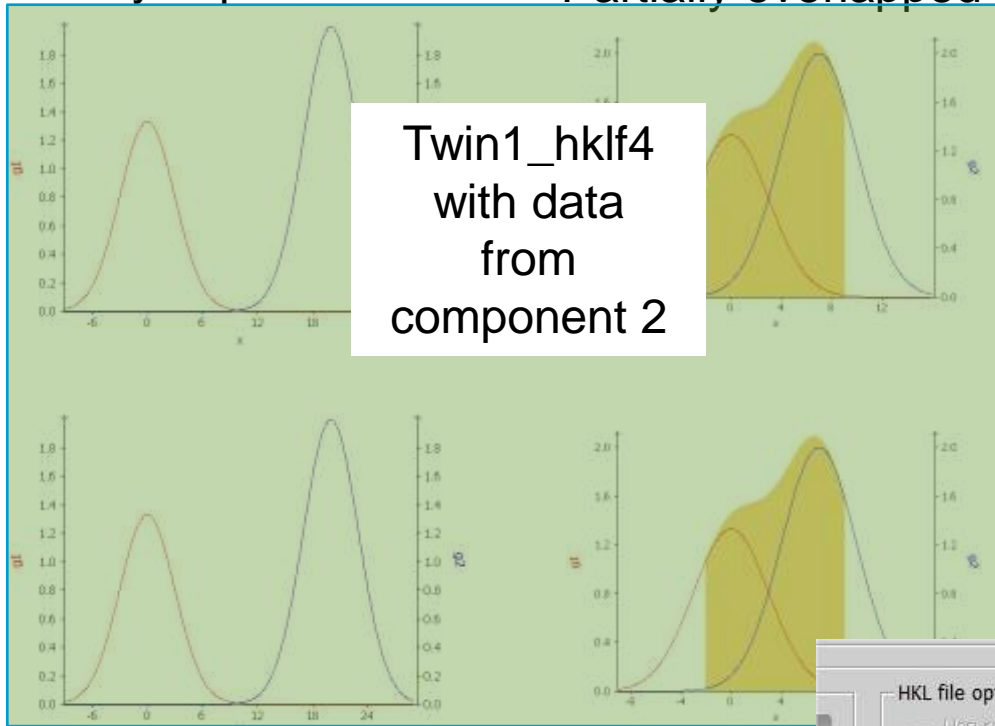


Get extra data in to solve the structure...

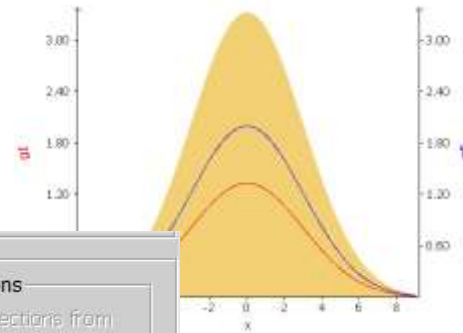
Fully separated

Partially overlapped

'Full' overlap



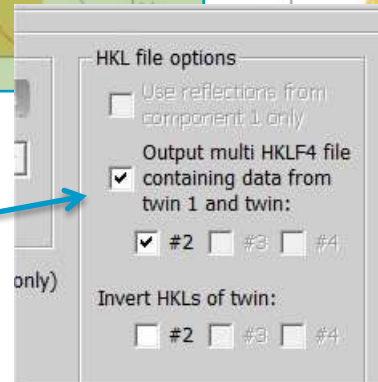
Component 1



Component 2

...

Component n

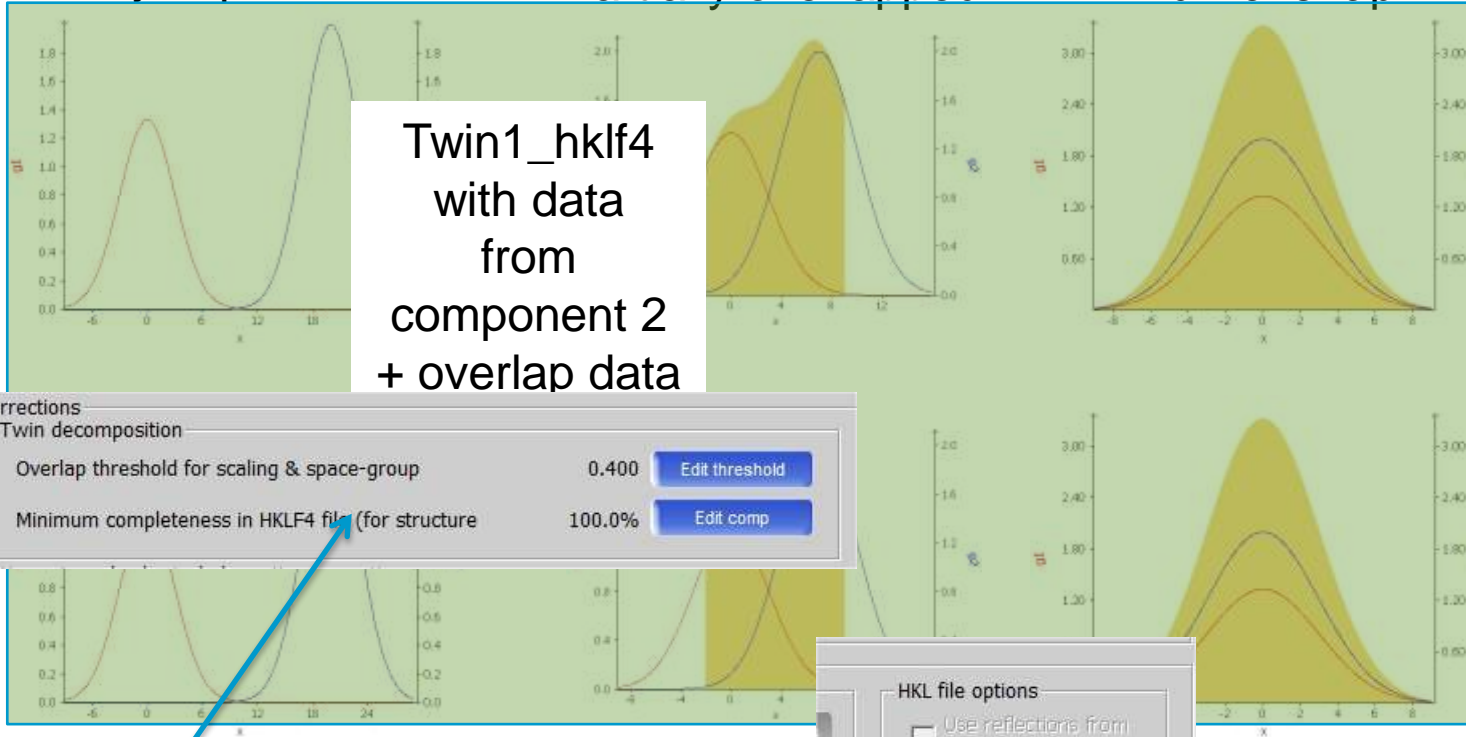


Not enough yet: Add full overlap data...

Fully separated

Partially overlapped

'Full' overlap



Component 1

Component 2

...
Component n

Corrections

Twin decomposition

Overlap threshold for scaling & space-group

0.400

Edit threshold

Minimum completeness in HKLF4 file (for structure)

100.0%

Edit comp



HKL file options

☐ Use reflections from component 1 only

☒ Output multi HKLF4 file containing data from twin 1 and twin:

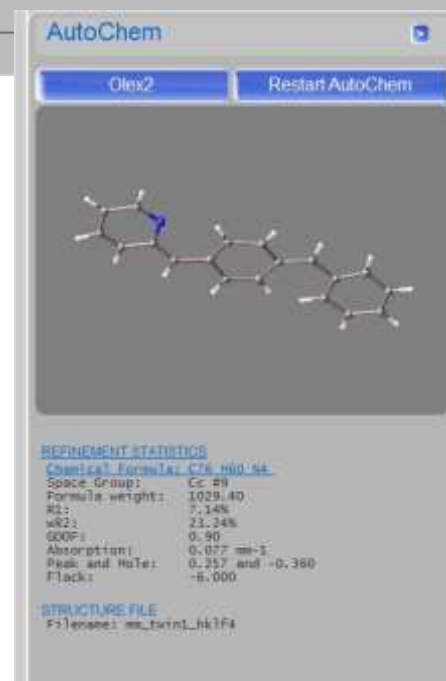
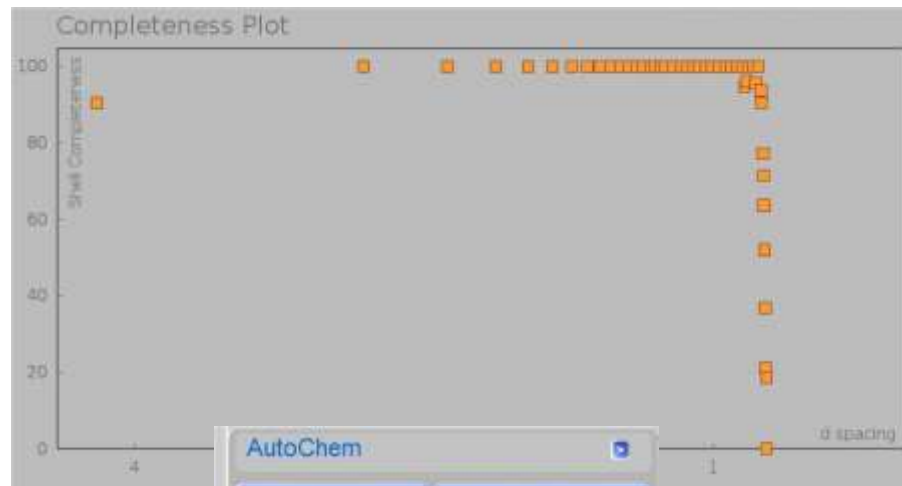
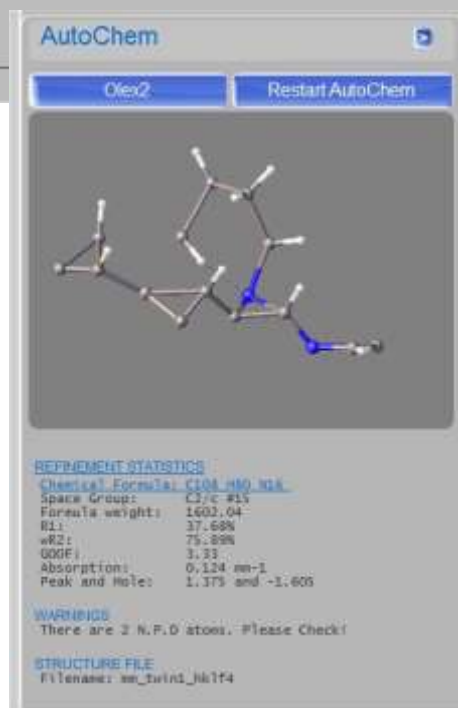
☒ #2 ☐ #3 ☐ #4

Invert HKLs of twin:

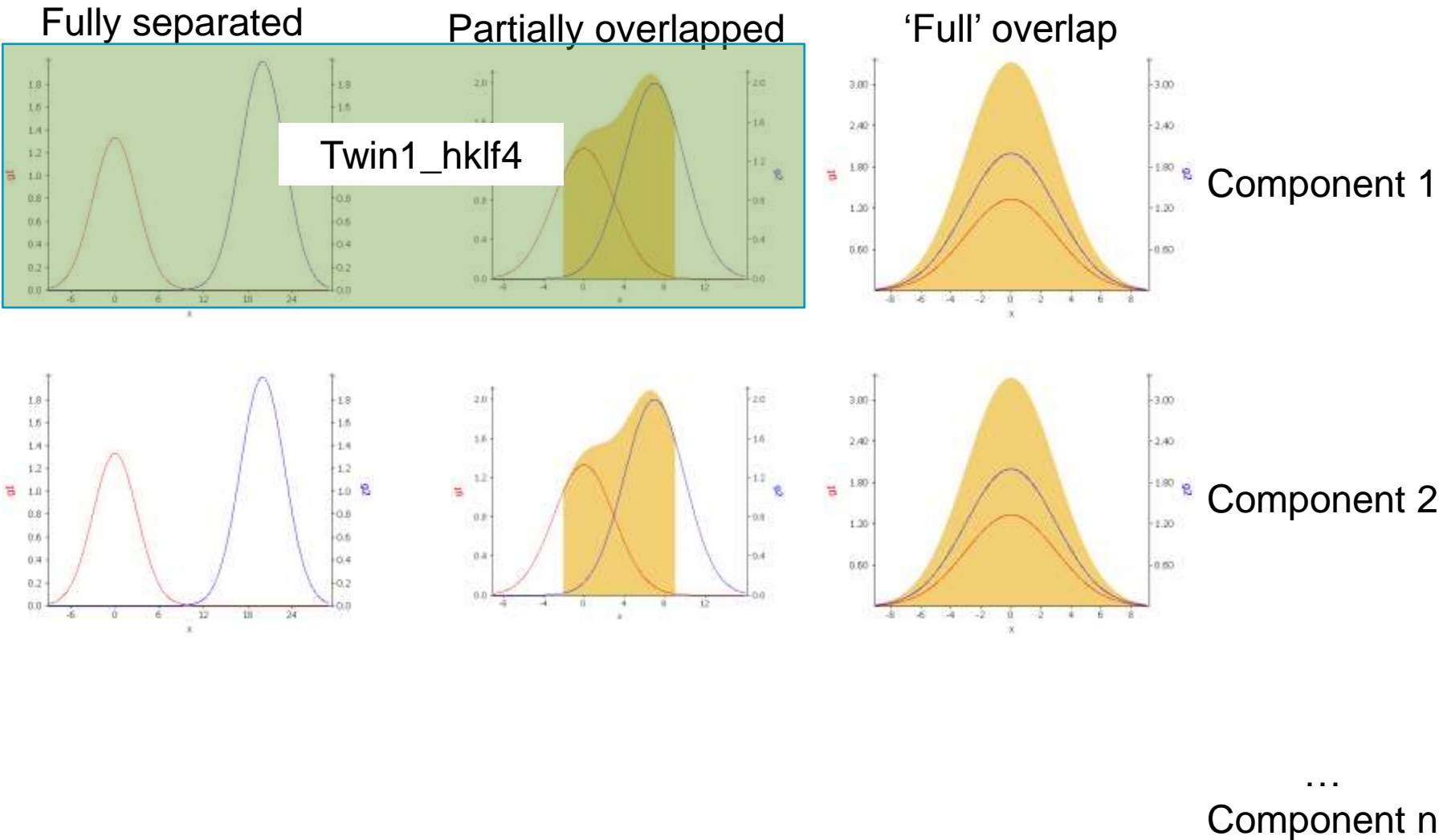
☐ #2 ☐ #3 ☐ #4



Why?



Solve done: Refine on good hklf4

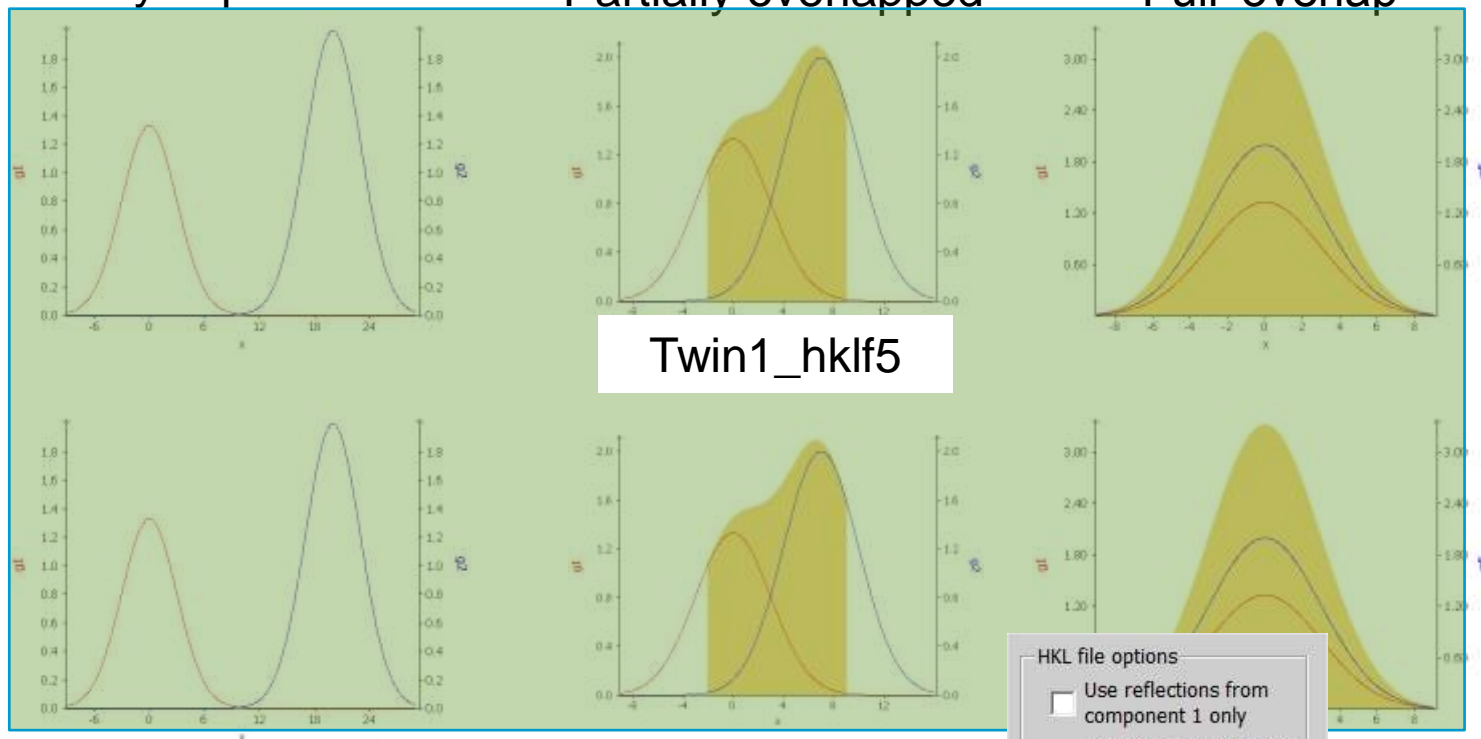


Solve done: Refine on good hklf5

Fully separated

Partially overlapped

'Full' overlap



Component 1

Component 2

...
Component n

Merging

☒ Merge data using: ☒ the same Laue class ☒ Use Friedel mates as equivalent

☐ Remove outliers ☐ different Laue class

1

Merging options

HKL file options

☐ Use reflections from component 1 only

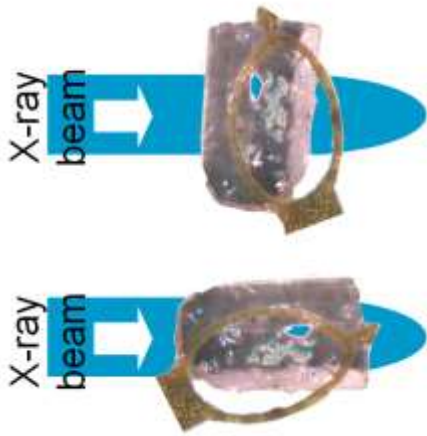
☐ Output multi HKLF4 file containing data from twin 1 and twin:

☒ #2 ☐ #3 ☐ #4

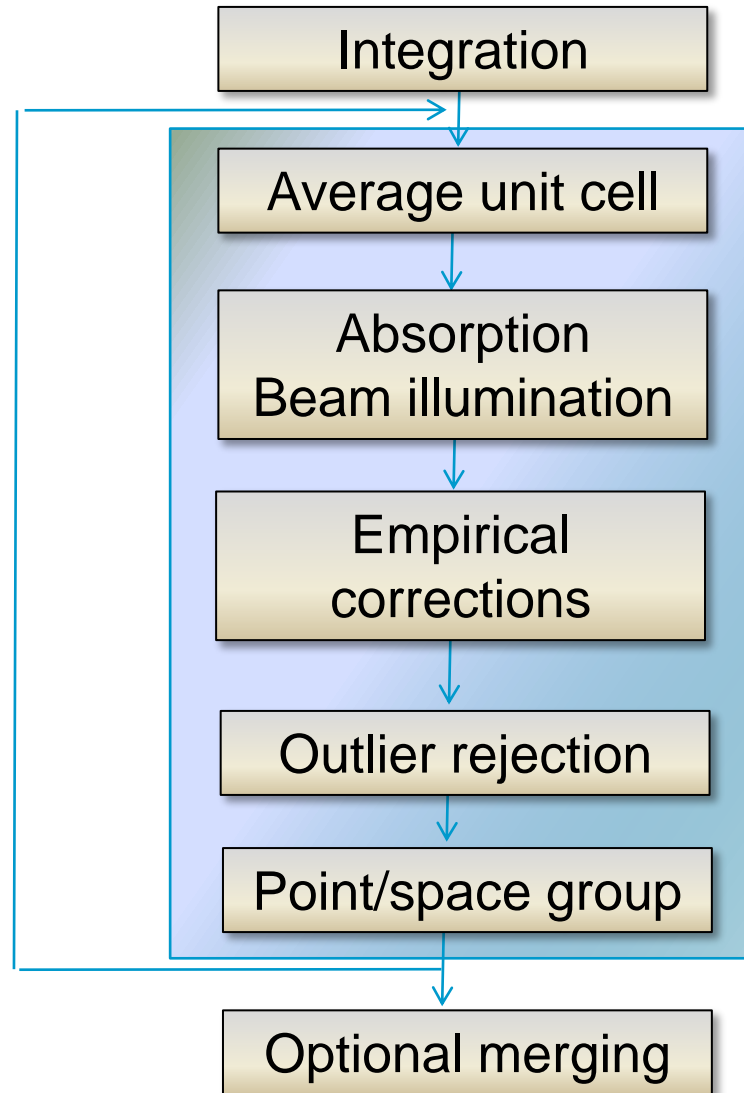
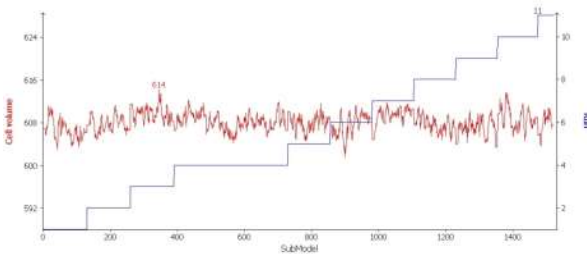
Invert HKLs of twin:

☐ #2 ☐ #3 ☐ #4

Good data quality through full post corrections



PG changed?

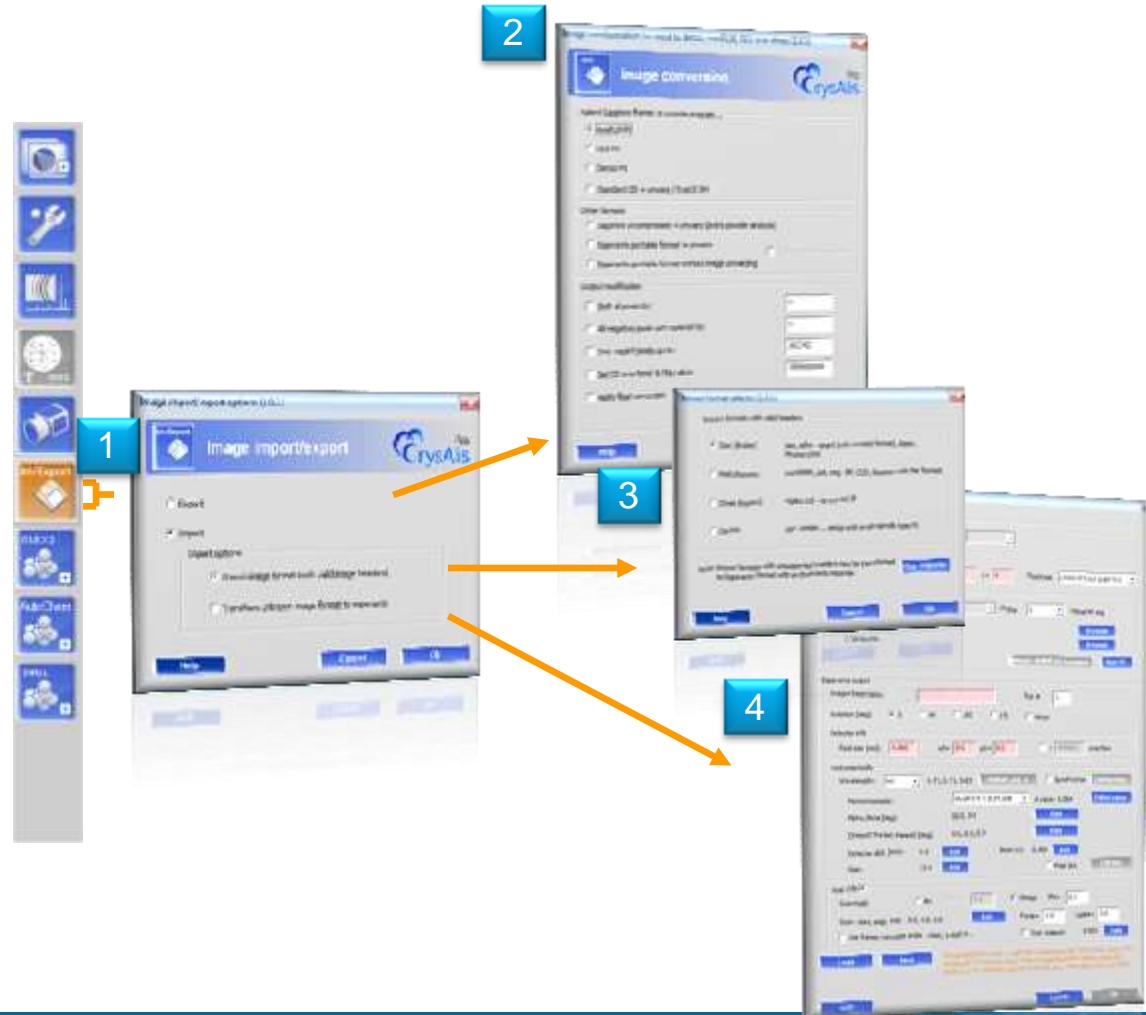


Summary - twin

- Twins can be recognized early on during screening/pre-experiment
- With the graphic and computational tools in Ewald^{Pro} twin assignment is easy.
- De-convolution of overlap data gives good HKLF4 files.
- Sometimes solution boot-strapping requires different play modes
- HKLF4 and 5 files can be easily conditioned for top data quality with absorption, beam illumination and empirical corrections.

Import/export from/to external formats

1. Easier access to import/export options (on power toolbar).
2. Organized export options.
3. Importing external images on one clique (instead of typing commands).
4. Esperanto importer for non standard image types.



SAXI CREATERUNLIST

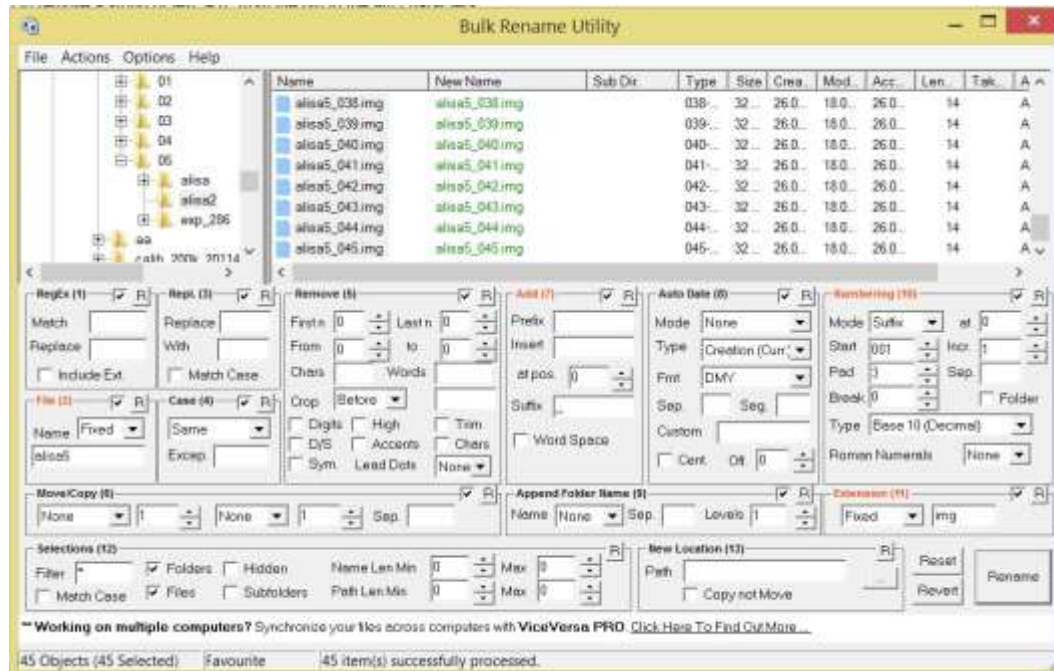
- ## MAR EXPERIMENTSETUP



DECTRIS CREATERUNLIS

Import/export

Dc rit/mar experimentsetup/dtrek createrunlist etc



- For some import tools image renaming might be required.
- There 50+ tools available on the internet. Here we would like to present a very powerful one: 'bulk rename'
- It has the possibility to pad names with zero to get names like aa_0001.xx.
- You can also inject run numbers for multi-run imports...



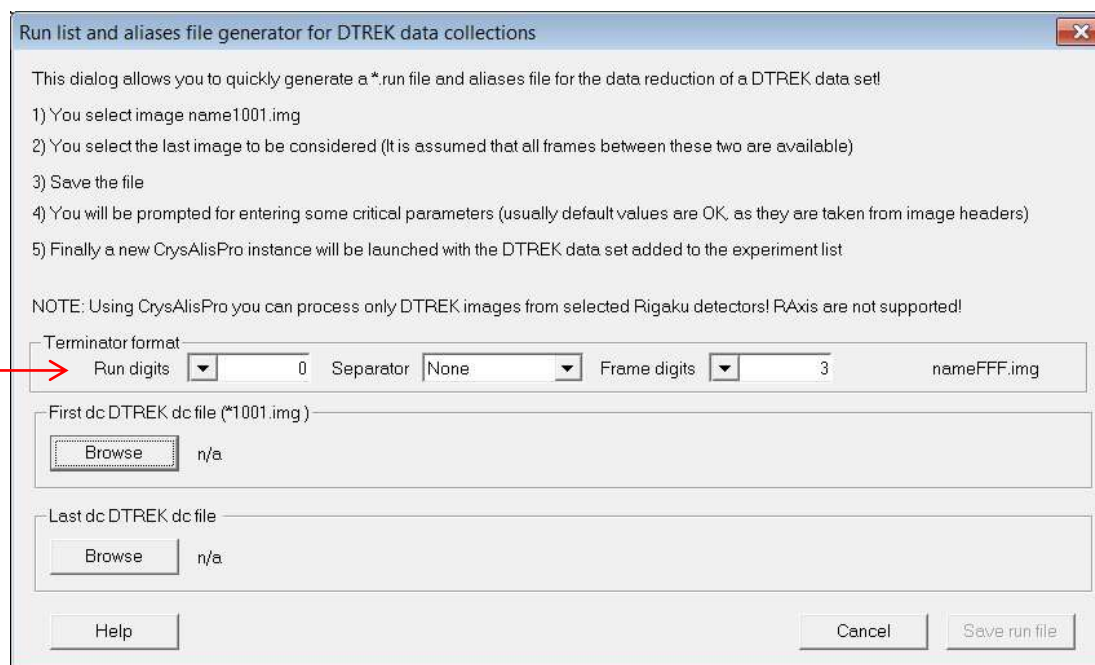
New bruker formats supported (CMOS)

- Data from photon100 detectors can be processed.
- The following observations can be made:
 - The Mo gain of cameras varies from 260 to 450 in spite of specification
 - The image gain is typically of the order 260 to 450 and the images are not divide as the Apex images in spite the of the header value e-/ADU 40.
 - The 3 gaps of the Radeye chips are clearly visible and should be rejected with dc rejectrect for better data. The most offending one is the middle one.
 - The offsets of the detector jump around significantly, so that smart background is the only method of choice.
 - In spite of spec. and marketing material the chip shows blooming effects. The noise level behind beam stop is 150-200e- for exp <5sec, meaning >10 higher noise than ApexII.
 - At same detector opening angle the Photon100 requires 2*longer exposure to reach the same R1 as ApexII for strong diffractors. For medium 3-4 and for weak >7 times. The detectivity seems very low as compared to ApexII.
 - Frame overhead is 4-8sec from frame time stamps in shuttered mode.
 - Cmos frames are large: 2Mb



New rigaku formats supported (CCD and Pilatus)

- New header format with CCD and Pilatus key words supported.
- The following observations can be made:
 - The Pilatus Rigaku frames are large: 1.2Mb, the cbf has 0.3Mb.
 - The dtrek createrunlist now handle awkward naming schemes
 - Pilatus use all Pixel detector corrections (parallax, Si efficiency etc.) and handles gaps.



Esperanto with bit field compression and pixel detector support

- For our Esperanto importer we now can use Agilent bitfield compression for Esperanto files. The format is not documented yet.
- There was also a pixel detector flag added to support the implemented pixel detector corrections (`[dsithicknessmmmforpixeldetector]` - thickness of Si for pixel detectors; the presence of this number signals a pixel detector. Pixel detector gap zones are marked with -1).
- Publication describes the Esperanto format

Single-crystal diffraction at the Extreme Conditions beamline P02.2: procedure for collecting and analyzing high-pressure single-crystal data

André Rothkirch, G. Diego Gatta, Mathias Meyer, Sébastien Merkel,
Marco Merlini and Hanns-Peter Liermann

J. Synchrotron Rad. (2013). **20**, 711–720

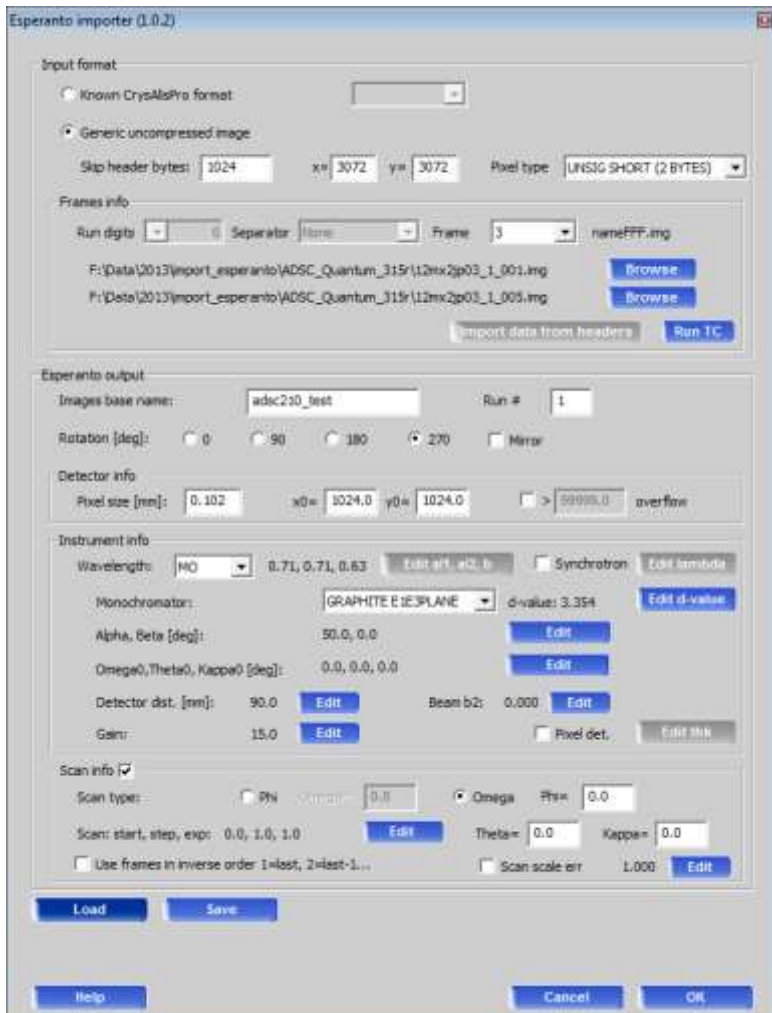


Generic image format 'Esperanto' generator

- Use of Esperanto format for unknown image formats with no compression or known formats with strange instrument configs
- Esperanto fully supports 4 circle instruments.
- Command 'dc rit' rit = raw image transform.
- But it also supports the known formats to handle 'unusual', obstinate images.
- Pixel detectors have an automatic dead zone detection based on the special value -1.
- The Esperanto generator uses a proprietary Agilent bit field format. To get back the uncompressed Esperanto version, please use the export function.



Dc rit: ADSC 315



Command dc rit

Header bytes 1024, x 3072 y 3072 and other info from text header (f.ex with total commander)

Then esperanto createrunlist

Slight play in EwaldPro to get the center right.

Dc rit: ADSC 210

Esperanto importer (1.0.2)

Input format:

- ☐ Known CrysAlisPro format
- ☒ Generic uncompressed image

Skip header bytes: 1024 x: 2048 y: 2048 Pixel type: UNSIG SHORT (2 BYTES)

Frames info:

Run digits: 0 Separator: None Frame: 3 name: PPF.png

F:\Data\2013\import_esperanto\ADSC_Quantum_210r\12mx1p05_1_001.png [Browse](#)

F:\Data\2013\import_esperanto\ADSC_Quantum_210r\12mx1p05_1_005.png [Browse](#)

[Import data from headers](#) [Run TC](#)

Esperanto output:

Images base name: adsc210_test Run #: 1

Rotation [deg]: ☐ 0 ☐ 90 ☐ 180 ☒ 270 ☐ Mirror

Detector info:

Pixel size [nm]: 0.102 x0: 1024.0 y0: 1024.0 ☐ > 99999.0 overflow

Instrument info:

Wavelength: MO 0.71, 0.71, 0.63 [Edit all](#) [a12, b](#) ☐ Synchrotron [Edit lambda](#)

Monochromator: GRAPHITE EIE PLANE d-value: 3.354 [Edit d-value](#)

Alpha, Beta [deg]: 50.0, 0.0 [Edit](#)

Omega0, Theta0, Kappa0 [deg]: 0.0, 0.0, 0.0 [Edit](#)

Detector dist. [mm]: 72.0 [Edit](#) Beam b2: 0.000 [Edit](#)

Gain: 15.0 [Edit](#) ☐ Pixel det. [Edit this](#)

Scan info ☒

Scan type: ☐ Phy ☒ Omega ☐ Phi ☐ Omega ☐ Phi ☐ Omega ☐ Phi

Scan start, step, exp: 0.0, 1.0, 1.0 [Edit](#) Theta: 0.0 Kappa: 0.0

☐ Use frames in inverse order 1=last, 2=last-1... ☐ Scan scale err: 1.000 [Edit](#)

[Load](#) [Save](#)

[Help](#) [Cancel](#) [OK](#)

- Command dc rit
- Header bytes 1024, x 2048 y 20482 and other info from text header (f.ex with total commander)
- Then esperanto createrunlist
- Slight play in EwaldPro to get the center right.

Dc rit: MAR165ccd

- Command dc rit
- Known format MAR (mccd)
- Then esperanto createrunlist
- Slight play in EwaldPro to get the center right.

Esperanto importer (1.0.2)

Input format

☒ Known CrysAlisPro format MAR marNNNN, pck, img

☐ Generic uncompressed image

Skip header bytes: x= y= Pixel type: UNSIG SHORT (2 BYTES)

Frames info

Run digits: 0 Separator: None Frame: 3 nameFFFF.img

F:\Data\2013\import_esperanto\MarCCD165\data_01_001.mccd Browse

F:\Data\2013\import_esperanto\MarCCD165\data_01_010.mccd Browse

Import data from headers Run TC

Esperanto output

Images base name: Run # 1

Rotation [deg]: ☐ 0 ☐ 90 ☒ 180 ☐ 270 ☐ Mirror

Detector info

Pixel size [mm]: x0= y0= ☐ > 99999.0 overflow

Instrument info

Wavelength: CU 1.54, 1.54, 1.39 Edit a1, a2, b ☐ Synchrotron Edit lambda

Monochromator: GRAPHITE E1E3PLANE d-value: 3.354 Edit d-value

Alpha, Beta [deg]: 50.0, 0.0 Edit

Omega0, Theta0, Kappa0 [deg]: 0.0, 0.0, 0.0 Edit

Detector dist. [mm]: 90.0 Edit Beam b2: 0.000 Edit

Gain: 1.0 Edit ☐ Pixel det. Edit thk

Scan info ☐

Scan type: ☒ Phi ☐ Omega 57.0 ☐ Omega ☐ Phi 75.0

Scan: start, step, exp: 75.0, 1.0, 20.0 Edit Theta= Kappa=

☐ Use frames in inverse order 1=last, 2=last-1,...

☐ Scan scale err 1.000 Edit

Load Save

Help Cancel OK

Dc rit: A200 detector

- Command dc rit
- Header bytes 3584, x 2048 y 20482 and other info from text header (f.ex with total commander)
- Then esperanto createrunlist
- Slight play in EwaldPro to get the center right.

Dc rit: Diamond ID 19 Dectris turned

Esperanto importer (1.0.1)

Input format

☒ Known CrysAlisPro format. DECTRIS

☐ Generic uncompressed image

Frames info

Run digits: 1 Separator: Underscore Frame: 5 nameR_FFFF.png

Browse Browse

Import data from headers Run TC

Esperanto output

Images base name: PAHAD_01deg_ Run #: 1

Rotation [deg]: ☐ 0 ☐ 90 ☐ 180 ☒ 270 ☐ Mirror

Detector info

Pixel size [mm]: 0.172 x0: 300.0 y0: 323.0 > 99999.0 overflow

Instrument info

Wavelength: 1.068 1.068 0.000 0.000 Edit list... ☒ Synchrotron Edit kinematics

Monochromator: MIRROR/SYNCHROTRON Polfact: 0.990 Edit polfact

Alpha, Beta [deg]: 50.0, 0.0 Edit

Omega0, Theta0, Kappa0 [deg]: 90.0, 0.0, 0.0 Edit

Detector dist. [mm]: 100.0 Edit Beam: 0.000 Edit

Gain: 1.0 Edit ☒ Pixel det. Edit pix

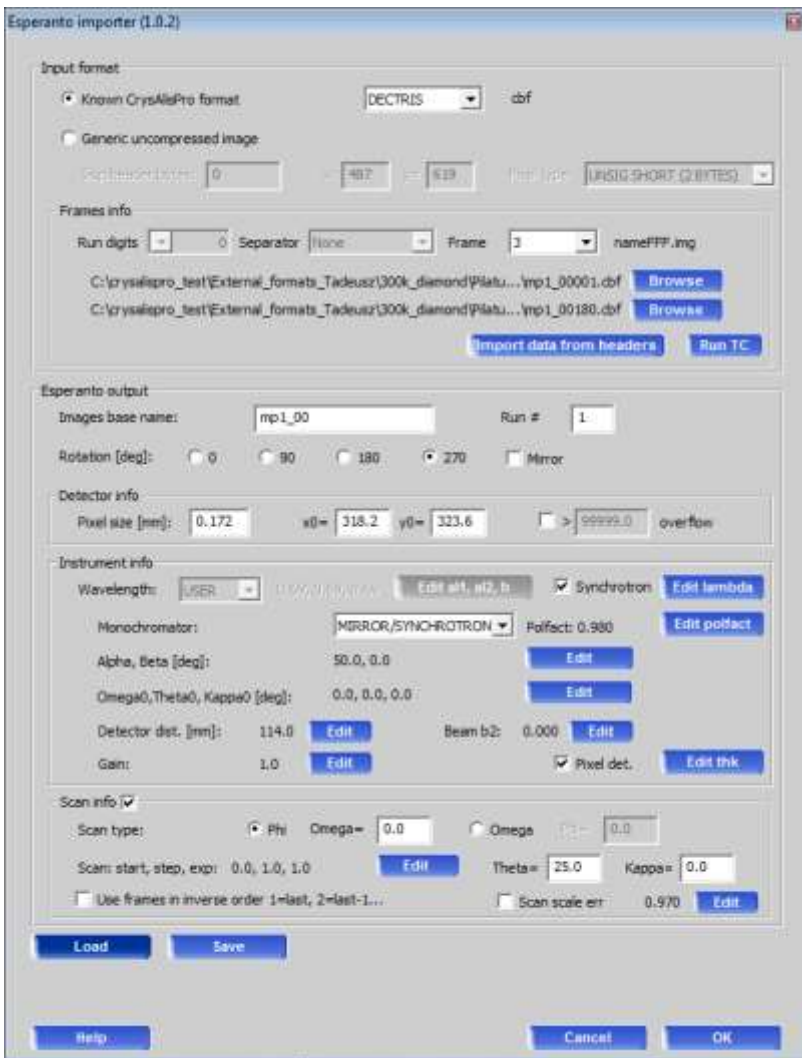
Scan info

Scan scale err 0.970 Edit

Load Save Help Cancel OK

- Command dc rit
- Use of known format dectris. Header values are read.
- Camera turned 270deg. Non-square detector is padded by zeros.
- The header scan values are wrong by 3% (Scan scale err 0.97)
- Then esperanto createrunlist
- Slight play in EwaldPro to get the center right.
- The several cycles to refine instrument model.

Dc rit: Diamond ID 19 Dectris turned II



- Command dc rit
- Use of known format dectris. Header values are read.
- Camera turned 270deg. Non-square detector is padded by zeros.
- This data had NO scan scale error!
- Then esperanto createrunlist
- Slight play in EwaldPro to get the center right.
- The several cycles to refine instrument model.

Dc rit: IPDS

- Command dc rit/Import button on power toolbar
- Read detector information from the sum file of IPDS (0.15mm pix, cen x=600, y=600)
- Stoe char as pixel type; .xi files (this is OD compression...)
- Make sure to use resolution limit due to round IP image (Mo typical 0.809Ang)



Dc rit: dtrek frame from Japanese synchrotron



- Command dc rit/Import button on power toolbar
- The issue was here that the dtrek image was turn 90deg relative to the inhouse image. The dtrek createrunlist would not work on this.
- Thus the 'dc rit' command can be used to handy obstinate known images...

Dc rit: xpad detector



- Command dc rit/Import button on power toolbar
- The xpad detector is developed in France
- One of it's raw format can be channelled through the Esperanto importer
- As the header info is unknown, it has to be given in the scan info section.
- Provide the raw data file contains the -1 marker for pixel detectors the esperanto createrunlist command will automatically create a ccd file with dead zones.
- Such formats have to be transformed run by run as there is only one field for scan info.

Overview

- Incommensurate structures
- Theoretical background
- Practical handling in CrysAlis^{Pro}



Incommensurate structures – Overview

- History
- Recognition of incommensurate structures in CrysAlis
- Symmetry limitation due to q-vector
- Data reduction
- Interface to Jana2006



History

- Satellite reflections are known since a long time -> super structures
- IUCR meeting Tokyo: de Wolff and Janner present a paper on 'Sodium carbonate' with satellites which are incommensurate to the main lattice

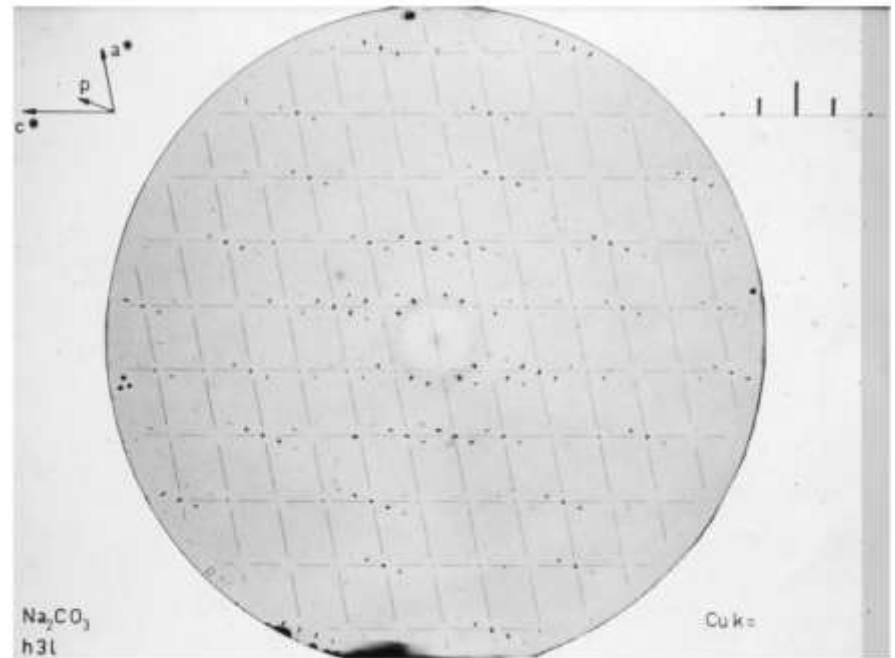
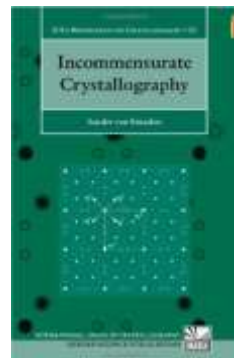


FIG. 1.11. Diffraction pattern of Na₂CO₃ (retigram; similar to a precession photograph). The measured X-ray diffraction has been overlaid with thin lines, highlighting the reciprocal lattice of the basic structure. p indicates the modulation wave vector. Reprinted from Tuinstra and Fraase Storm (1972) by courtesy of F. Tuinstra (Delft, The Netherlands).



From: Incommensurate Crystallography (IUCr Monographs on Crystallography), Sander Van Smaalen von Oxford University Press (7. Juni 2007)

History

- Aperiodic distortions

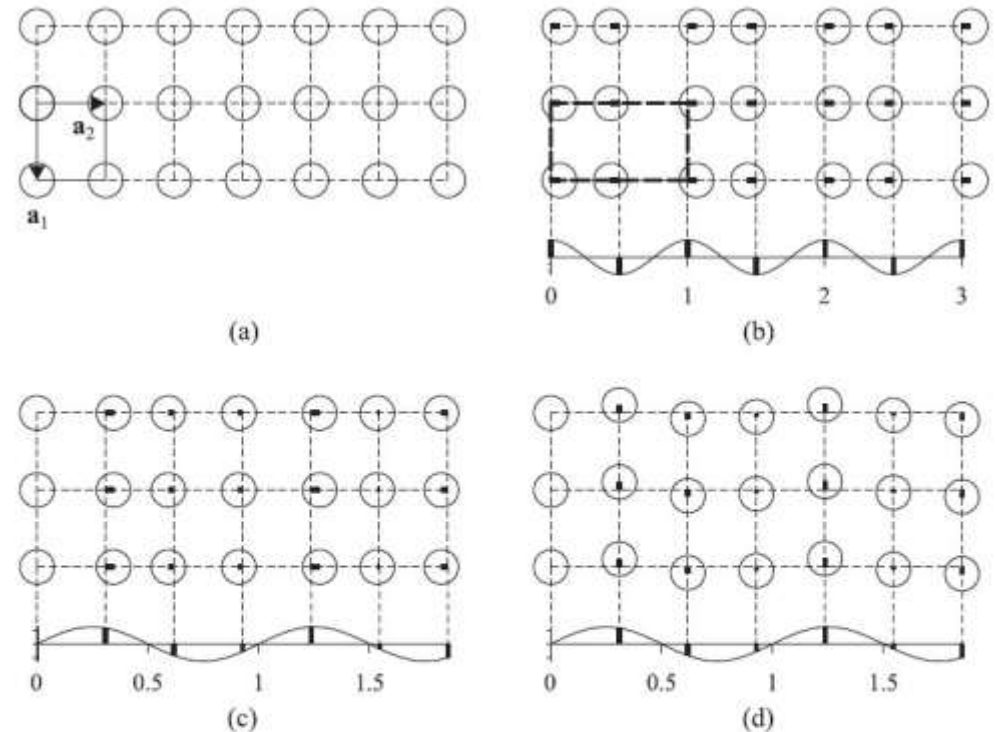


FIG. 1.2. Crystal structures with displacement modulations. (a) Basic structure. (b) Twofold superstructure with the supercell indicated by heavy dashed lines. (c) Incommensurate longitudinal modulation. (d) Incommensurate transversal modulation. Lattices of the periodic basic structures are indicated by dashed grids. Circles denote atoms that are shifted out of lattice periodic positions by varying amounts given by the heavy bars. Numbers count periods of the modulation waves $u(\bar{x}_4)$ [eqn (1.7)].

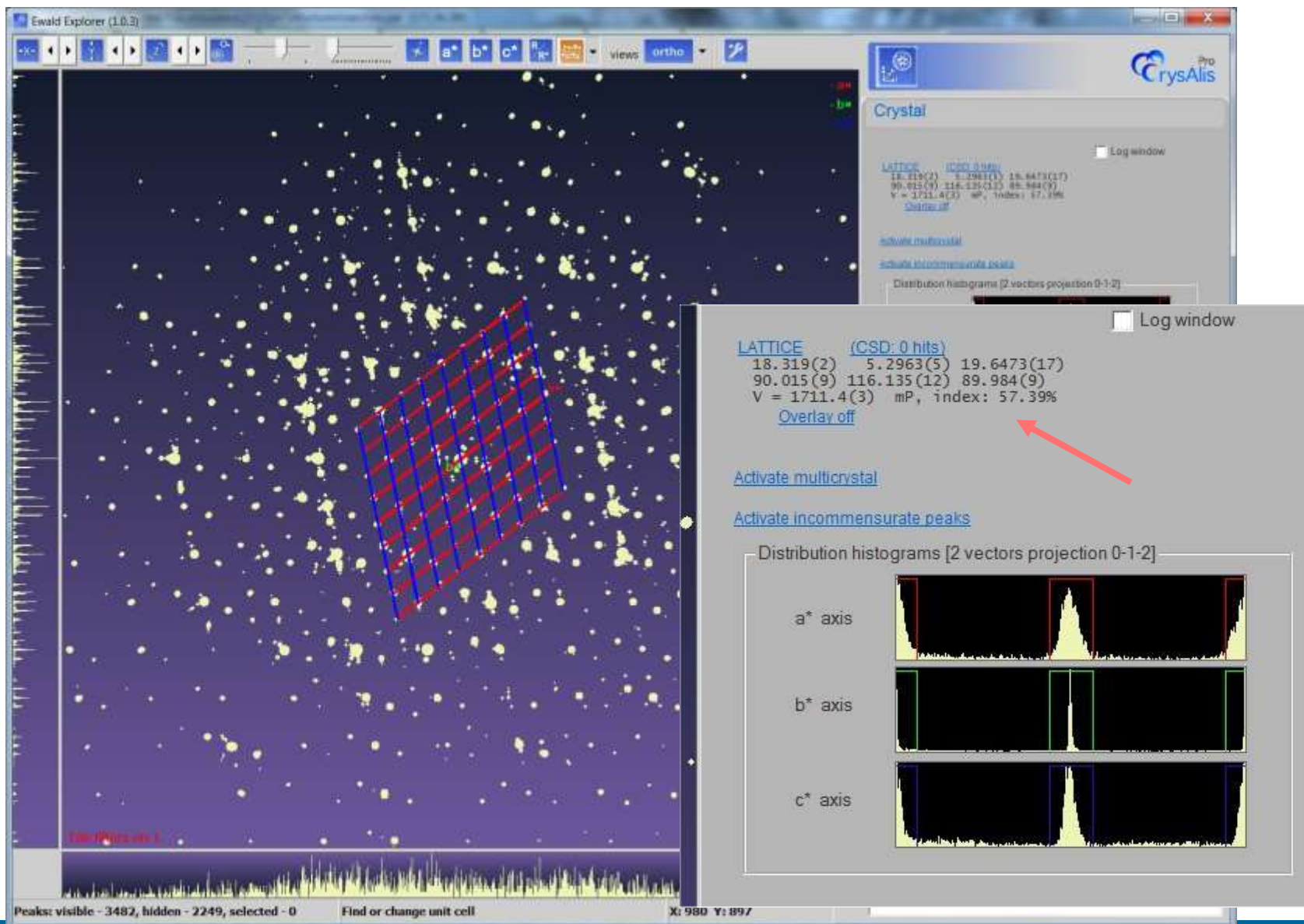
From: Incommensurate Crystallography (IUCr Monographs on Crystallography), Sander Van Smaalen von Oxford University Press (7. Juni 2007)

Recognition of incommensurate structures in CrysAlis

- Case Na_2CO_3
- Normal unit cell finding fails: Super cell found
- **Health warning**: samples of Naco are water sensitive and this is the baest sample ever after IUCr Tokyo: a multi-crystal/twin with 3 components: but component 1 is 80%



Naco: Super cell found



Naco: look for basic cell – play with custom cell

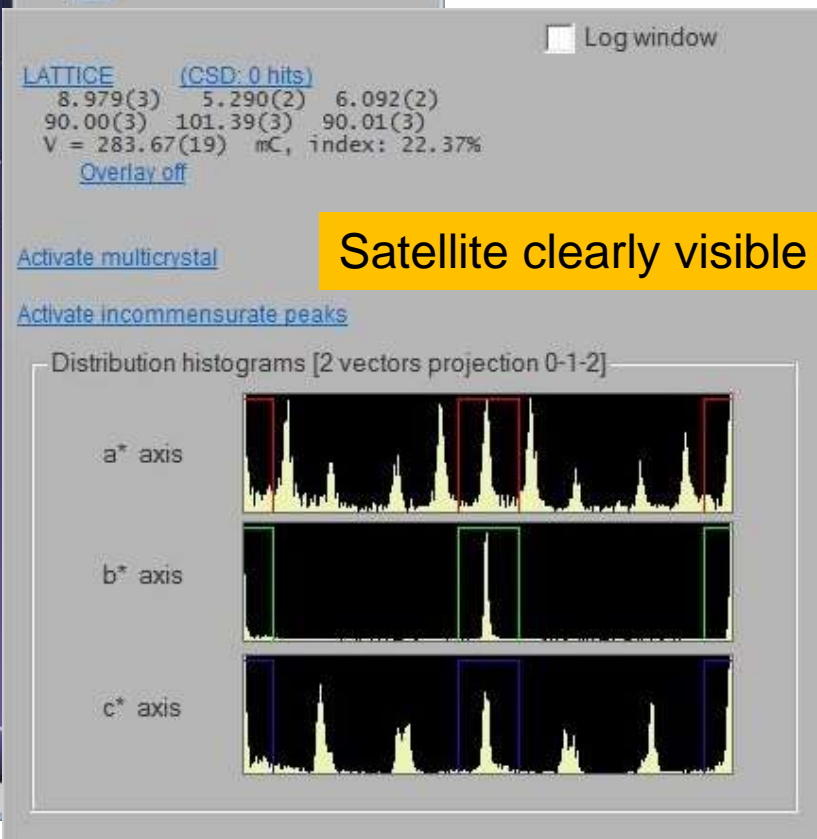
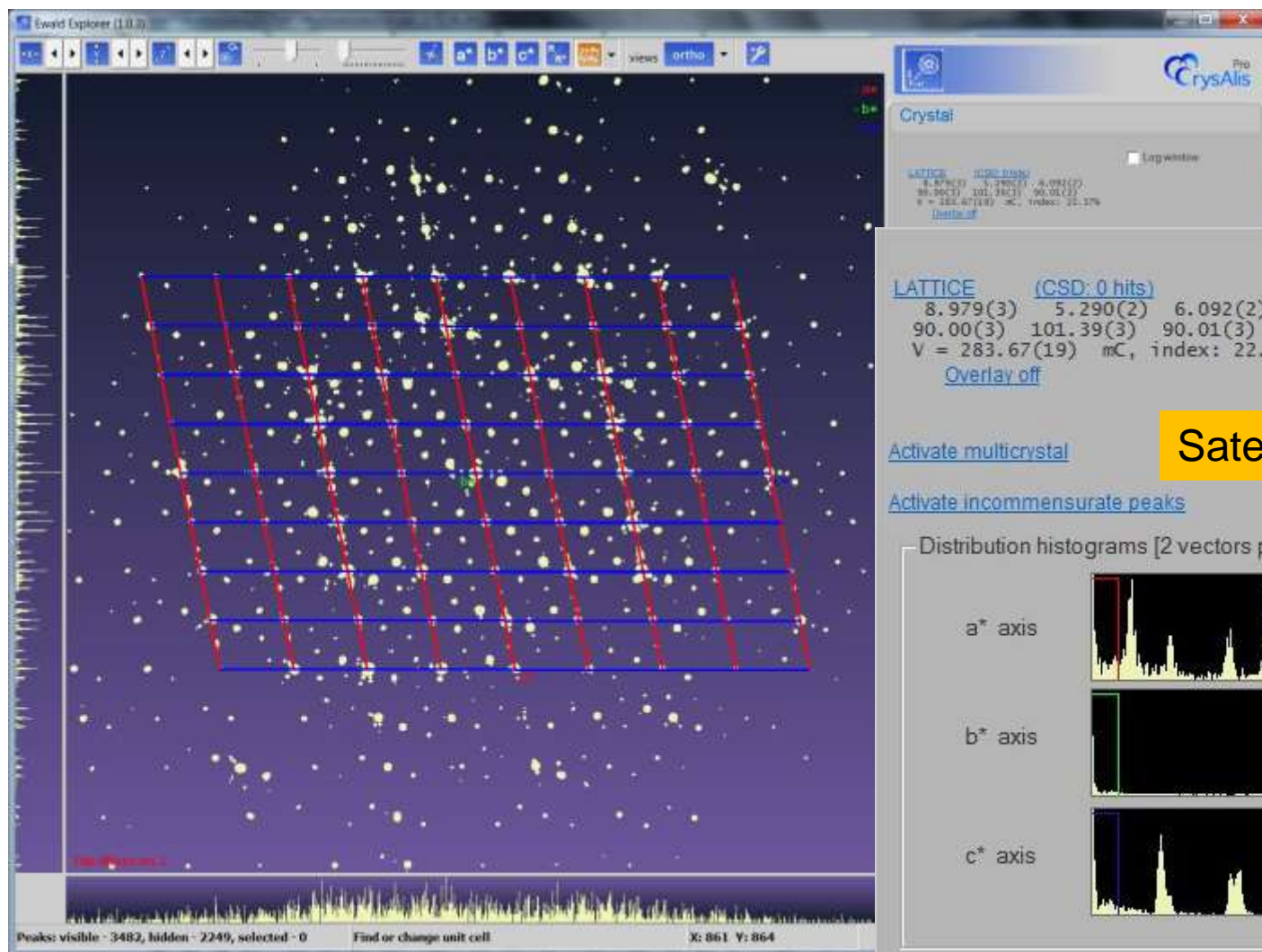
The screenshot displays the Ewald Explorer (1.0.3) software interface. The main window shows a diffraction pattern with a unit cell overlay. A red arrow points to the 'Find cell...' dialog box, which is open. The dialog box has several sections:

- Peak table:** Radio buttons for 'Normal peak table' (selected) and 'Delta (differential) peak table'.
- Algorithm:** Radio buttons for 'T-vector Dirax' (selected) and 'Stereographic'.
- Sample type:** Radio buttons for 'Single crystal' (selected) and 'Twin / multycrystal'.
- Unit cell:** Radio buttons for 'SM', 'PX', and 'User' (selected). Below are 'min' and 'max' input fields for 'a' (2.0 and 18.0) and 'b' (2.0 and 120.0). A 'Calc' button is present.
- Lock present components:** Checkboxes for 'Twin 1', 'Twin 2', and 'Twin 3'.
- HINT:** Text explaining how to lock current UB for twin 1.
- Consider Bravais lattice type:** A checkbox.
- Force identical lattice for all components:** A checked checkbox.
- Known cell:** A checkbox for 'Search known cell' and a text field containing '18.32 5.30 19.65 90.02 116.13 89.98'.

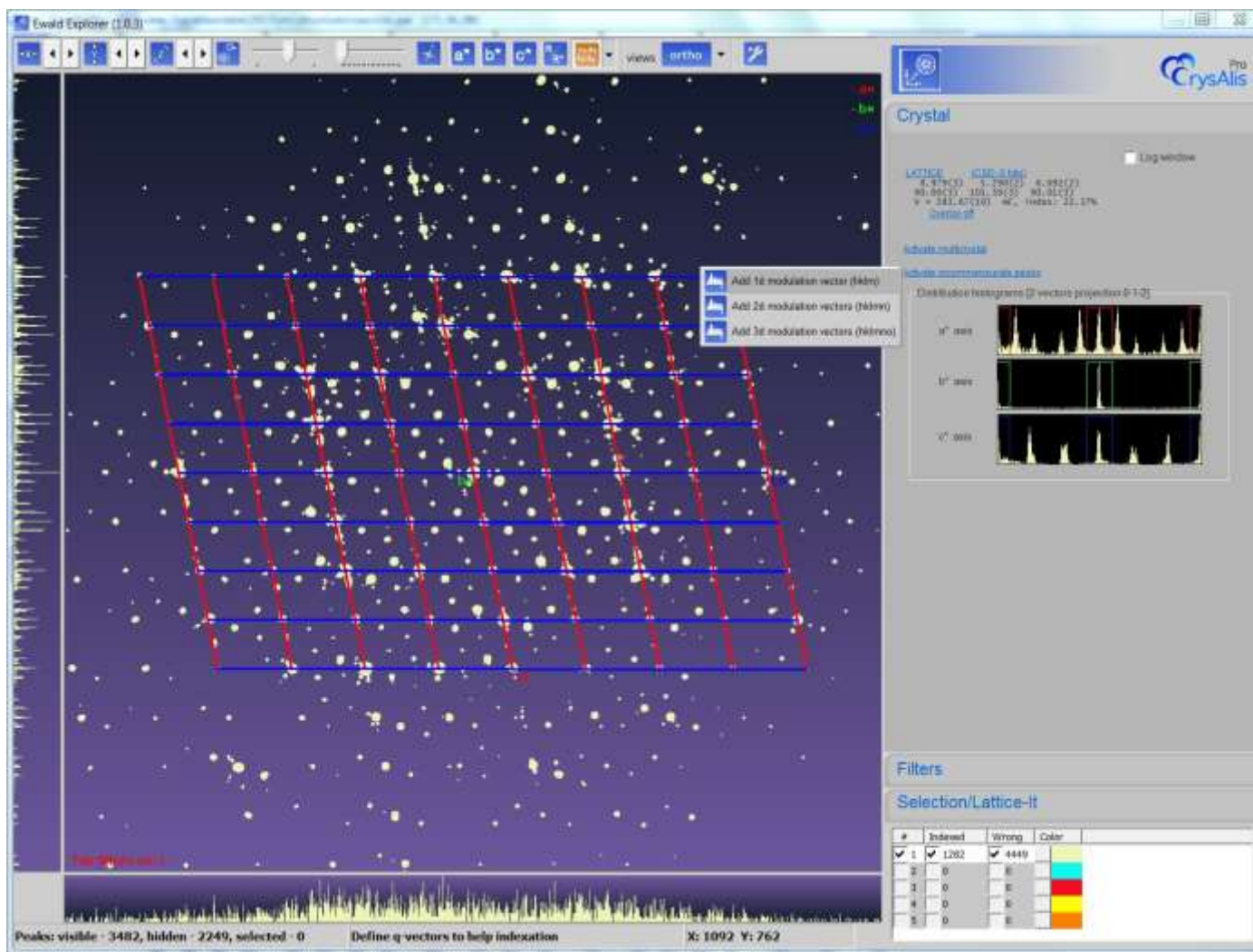
The main window also shows a 'Crystal' panel on the right with lattice parameters and a 'Filters' panel at the bottom right with a 'Selection/Lattice-It' table.

#	Indexed	Wrong	Color
1	✓ 3289	✓ 2642	Yellow
2	0	0	Cyan
3	0	0	Red
4	0	0	Yellow
5	0	0	Orange

Naco: basic cell – indexation poor but simple q-vector

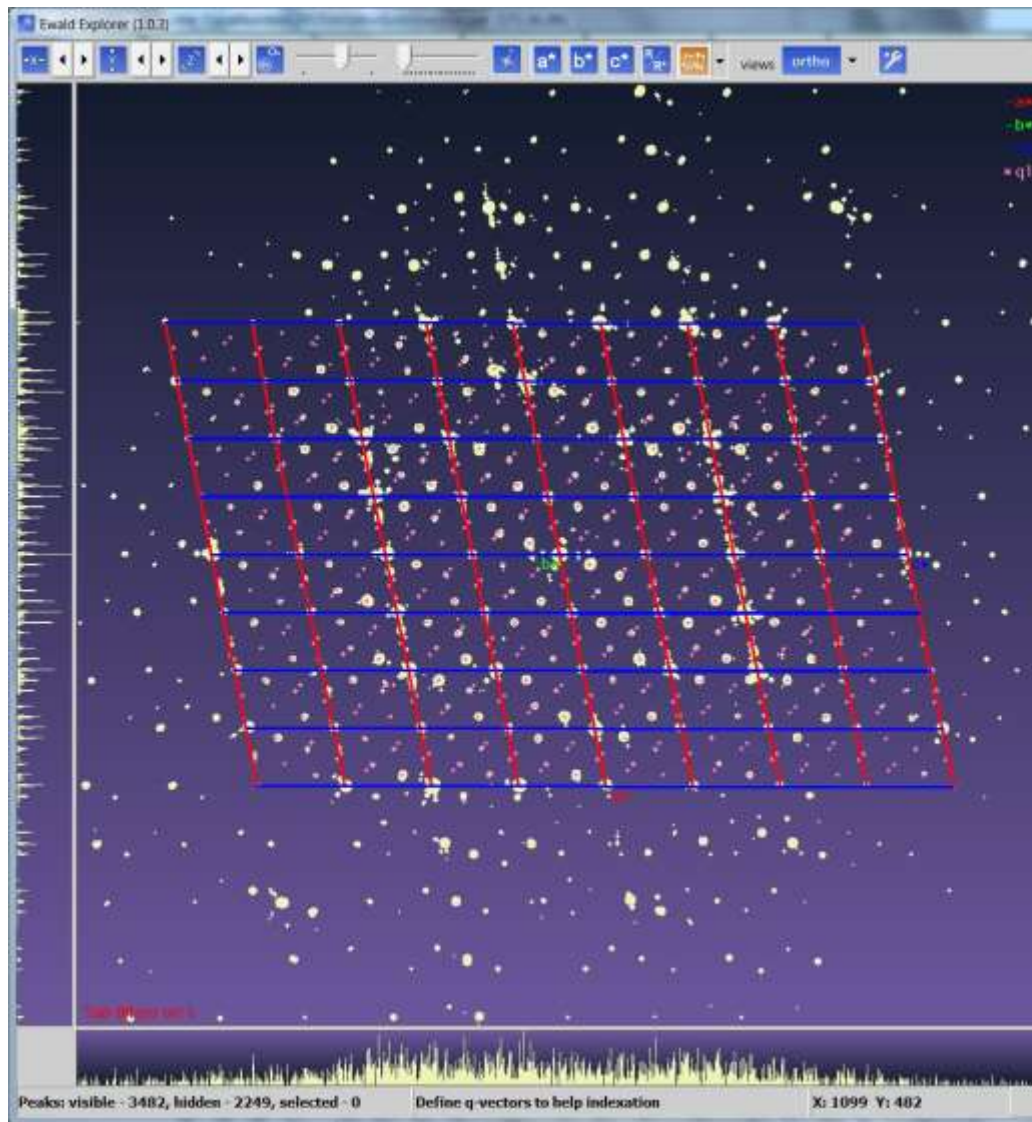


Naco: add q-vector



Naco: add q-vector

Click modify to change the values: rough guess is enough



Note: q-vectors have to confront lattice symmetry!

Naco: refine q-vector inspect results

Do q-vector refinement

Please enter mm max crith k1 q1 q2 q3 [q1 q2 q3 [q1 q2 q3]] (5,8,11 values)

4 0.05000 0.18359 0.00002 0.32119

OK Cancel

Additional command information

Indexed total: 3340 => 58.25%

Unindexed: 2391

Skipped: 0

Refinement Cycle: 4

Main reflections: 828

Satellites: (1) 1428 (2) 766 (3) 197 (4) 23

Indexed total: 3340 => 58.25%

Unindexed: 2391

Skipped: 0

--> New OM:

0.00350444 (0.00000573) 0.13033941 (0.00000487) -0.02110593 (0.00000858)

-0.04650485 (0.00000394) 0.03027405 (0.00000711) 0.07747373 (0.00000889)

0.06504390 (0.00000677) 0.00459330 (0.00000608) 0.08713369 (0.00000753)

--> Lattice Parameters:

5.297715 (0.002816) 6.104783 (0.003104)

89.890905 (0.003237) 101.410381 (0.044723) 90.003047 (0.028237)

245.378 (0.471)

--> Modulation Vector 1:

0.183589 (0.000512) -0.000020 (0.000213) 0.321186 (0.000365)

Modulation Vector 1:

OM SETVECTOR 0.18359 -0.00002 0.32119

NADA QVECTOR 4 0.05000 0.18359 -0.00002 0.32119

Filters

Selection/Lattice-It

#	Indexed	Inc	Wrong	Color
✓ 1	✓ 926	✓ 2414	✓ 2391	
2	0	0	0	
3	0	0	0	
4	0	0	0	

Naco: clean the picture...

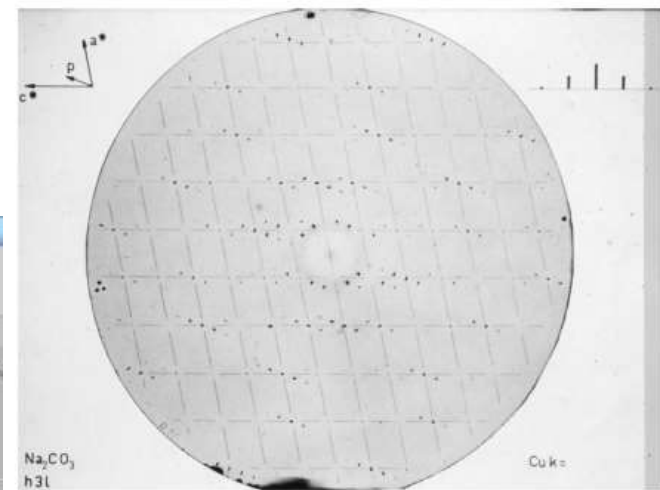
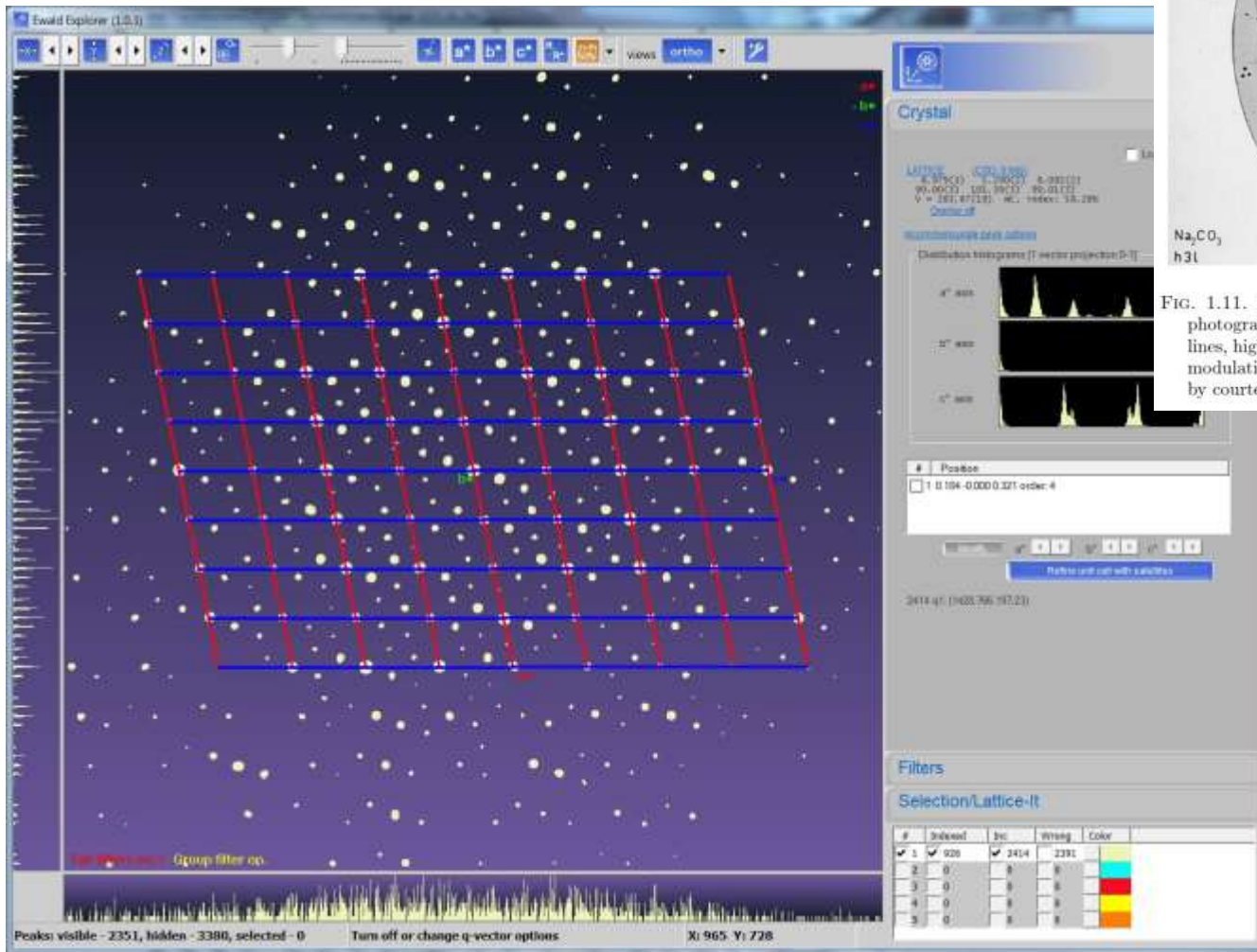


FIG. 1.11. Diffraction pattern of Na_2CO_3 (retigram; similar to a precession photograph). The measured X-ray diffraction has been overlaid with thin lines, highlighting the reciprocal lattice of the basic structure. p indicates the modulation wave vector. Reprinted from Tuinstra and Fraase Storm (1972) by courtesy of F. Tuinstra (Delft, The Netherlands).



- Group checkmarks
- Hide wrong

Naco: Lattice wizard

Lattice wizard (1.0.32)

Lattice wizard

LATTICE

Current cell (CSD: 0 hits)
8.979(3) 5.290(2) 6.092(2) 90.00(3) 101.39(3) 90.01(3) 283.67(19)

Constrained current cell
8.978(4) 5.2922(15) 6.091(3) 90.0 101.40(5) 90.0 283.7(2)

Lattice reduction

selected cell	8.9683	5.2852	6.0802	90.0149	101.4613	90.0097 mC	10
reduced cell	5.2045	5.2053	6.0802	80.1513	80.1345	61.0231	141.2

Incommensurate/quasi-crystal information
q(1): 0.1836(5) 0(2e-004) 0.3212(4)

PEAK TABLE

Peak hunting table
UB fit with 2351 obs out of 2351 (total:5731,skipped:3380) (100.00%)
926 main refl.; 2414 q1 satellites
By order
2414 q1: (1428,766,197,23)

INSTRUMENT MODEL

Goniometer
beam: -0.06378 alpha: 50.05931 beta: -0.04694
om zero: -0.11655 th zero: 0.51155 ka zero: -0.00763

Detector
x-rot: 0.15208 y-rot: 0.41264
x-cen: 533.86586 y-cen: 482.45755 distance: 72.23148
Wavelength Mo (Ang): A1 0.70930 A2 0.71359 B1 0.63229

Pro CrysAlis

Peak hunting

Unit cell finding

Ewald explorer - reciprocal space

Reindexation with current cell

Refine instrument model

Lattice transformation

Twinning - multi-crystals

Incommensurates / Quasi-crystals

Load information

Save information

Unwarping - Precession images

Log window

- Sorting
- Cleaning
- q-vector refinement



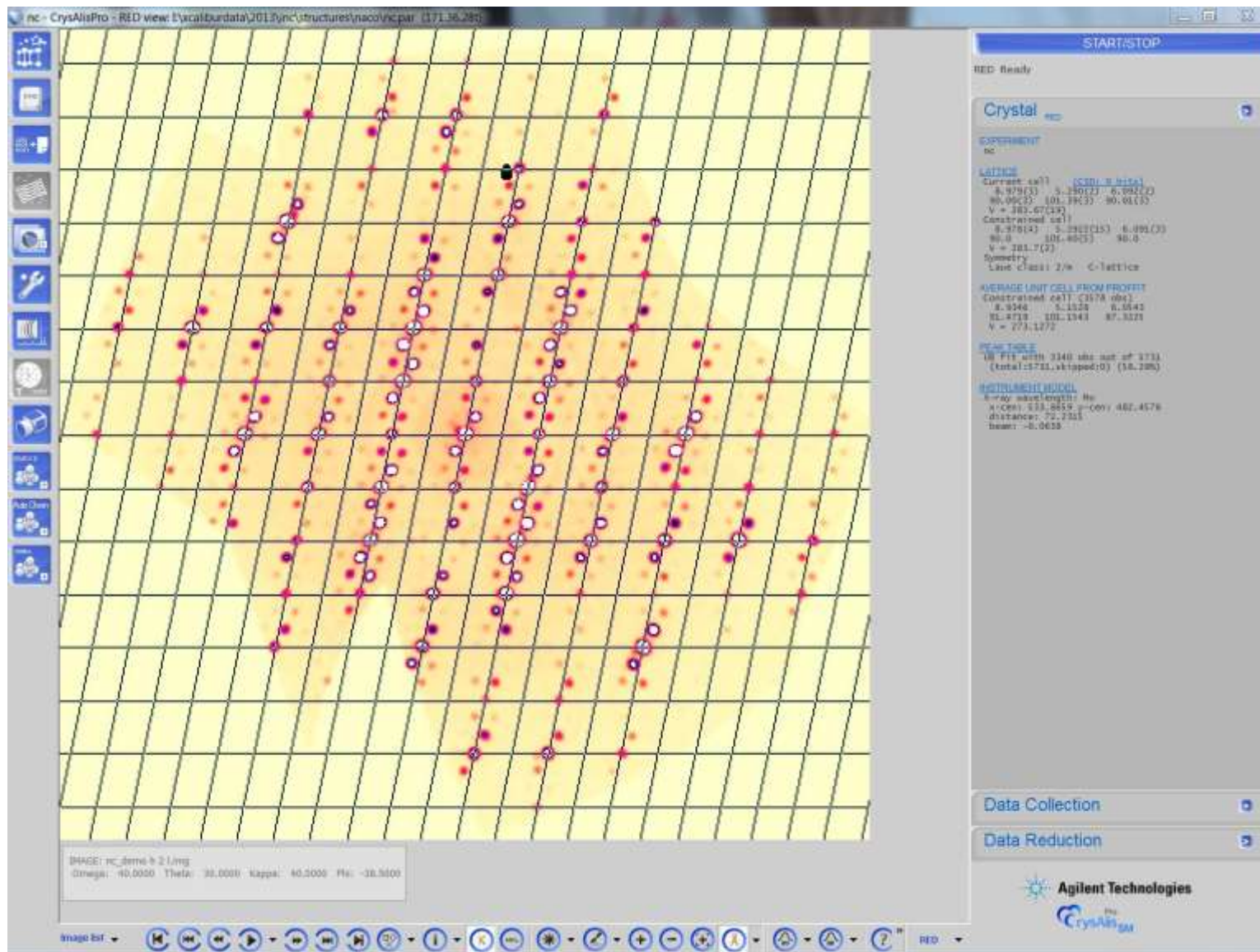
Naco: Run unwarp to gain better understanding

The screenshot shows the CrysAlis Pro software interface. The main window displays a 2D diffraction pattern with contour lines and labels 3.96, 2.77, and 2.12. A dialog box titled 'UNWARP: Add a reconstruction layer' is open in the center. The dialog box contains the following fields and options:

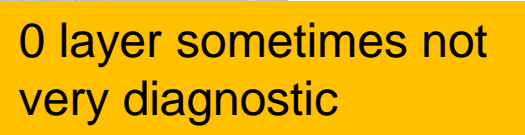
- Vectors:**
 - ☒ One pixel layer
 - ☐ Integrative layer
 - O:** 0 0 0
 - L1:** 1 0 0
 - L2:** 0 1 0
 - Resolution (Ang):** 0.00000
 - ☐ Use L1, L2, O/Os/Os as confusion (prevold)
- Optional 2d Laue symmetry averaging:**
 - no 2D Laue averaging
 - Tip: Use 2d Laue averaging to fill your images due to missing coverage! But it takes time!
 - ☐ Use mirror plane
 - ☐ Use inversion center
- Output name:** demo h 0 1
- Tip: Choose a meaningful name like 'h k l' with '0's'. This helps to find your images back.

The right sidebar shows the 'Crystal' and 'EXPERIMENT' data. The 'Crystal' section includes the lattice parameters: $a = 10.00(3)$, $b = 10.00(3)$, $c = 10.00(3)$, $\alpha = 90.0^\circ$, $\beta = 90.0^\circ$, $\gamma = 90.0^\circ$. The 'EXPERIMENT' section includes the peak statistics: 10 files with 3340 obs out of 3373 (Total 15731, skipped 69) (54.29%).

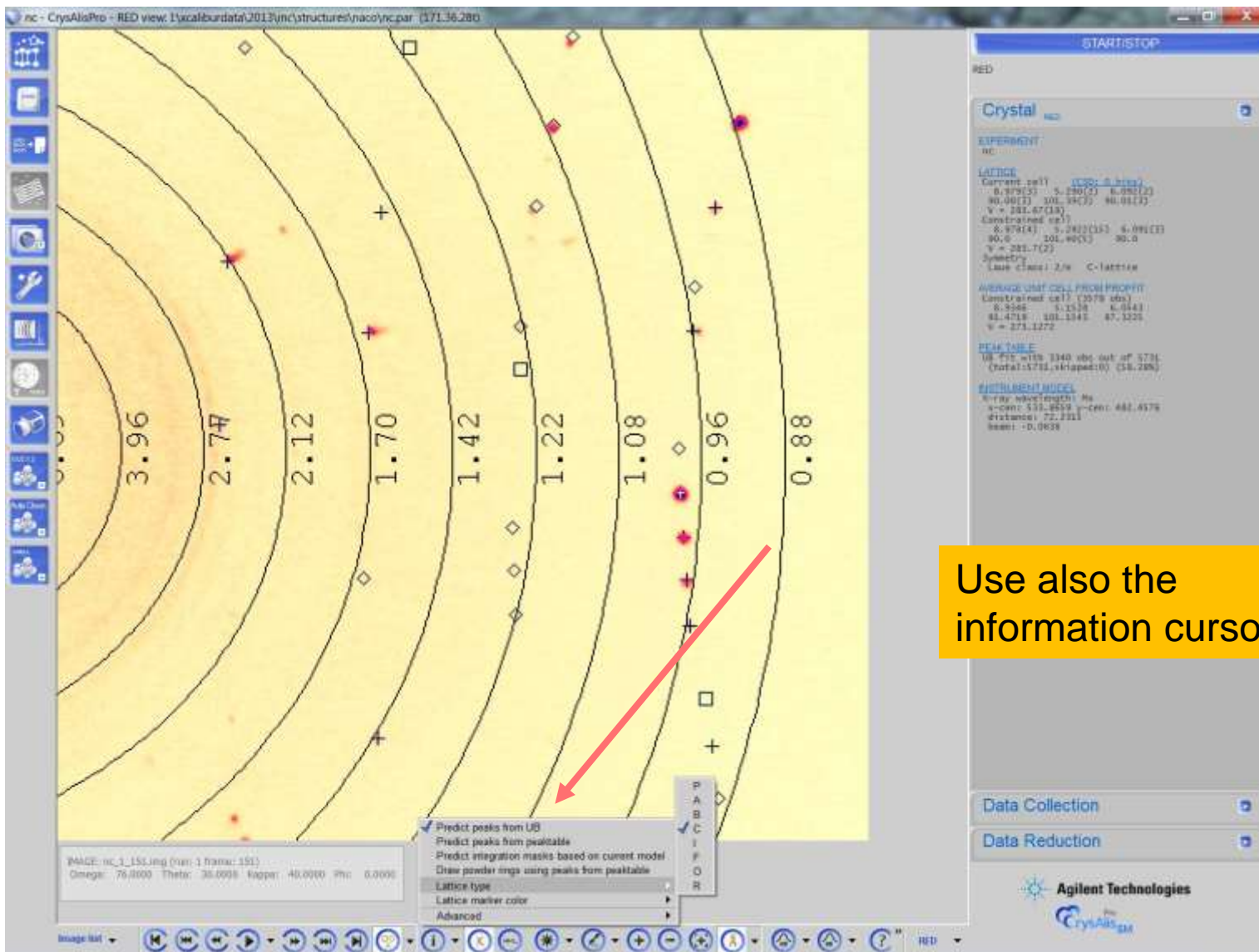
Naco: Run unwarp to gain better understanding: h 2 I



Naco: Run unwarp to gain better understanding: h 0 l

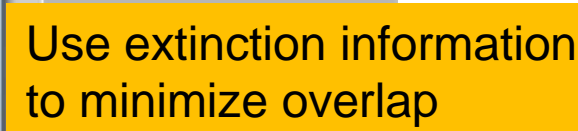


Naco: Check reflection predictions



Use also the
information cursor

Naco: Data reduction



Naco: Alternative way to formulate extinctions

The screenshot displays the CrysAlisPro software interface. On the left, a 'Command shell' window shows the following text:

```
DC options: EXTINGT
DC EXTINGT
  0 Extinction conditions:
DC EXTINGT ?
```

In the center, a file explorer window lists various command files, with 'dc extinct' selected.

On the right, the 'dc extinct' command documentation is displayed. It includes the following text:

dc extinct

This command allows the user to add custom extinction rules to the data reduction process. These rules have following general form:

$$mh * h + mk * k + ml * l [+ mm * m [+ mn * n [+ no * o]]] = mp * p + co$$

With the eight command line numbers you are able to build all crystallographic conditions (and also non-crystallographic ones), for example to analyse super structures. For example see below.

DC EXTINGT hkclass mh mk ml [mm [mn [mo]]] mp co

Parameters

hkclass: reflection class (hkl,hkm,hklmn,hklmno,hk0,h0l,0kl,h00,0k0,00l)

mh mk ml mm mn mo mp: h, k, l, [m, [n, [o]]] p multipliers.

co: integer constant

Examples

dc extinct hkl 1 1 1 2 1 = l- lattice $1^*h + 1^*k + 1^*l = 2^*p + 1$

dc extinct hkl 1 1 0 2 1 = C- lattice $1^*h + 1^*k + 0^*l = 2^*p + 1$

dc extinct hk0 1 0 0 2 1 = a-glide perp. c $1^*h + 0^*k + 0^*l = 2^*p + 1$

dc extinct hklm 1 1 0 1 2 1 = C- lattice plus hyperspace centering $1^*h + 1^*k + 0^*l + 1^*m = 2^*p + 1$

Note: you can also combine conditions:

-d-

dc extinct 0kl 0 1 1 4 1 = d-glide perp. a $0^*h + 1^*k + 1^*l = 4^*p + 1$ condition $k+l=4n+1$

dc extinct 0kl 0 0 1 2 1 = d-glide perp. a $0^*h + 0^*k + 1^*l = 2^*p + 1$ l odd

dc extinct 0kl 0 1 0 2 1 = d-glide perp. a $0^*h + 1^*k + 0^*l = 2^*p + 1$ k odd



Naco: Check results

nc - CrysAlisPro - RED view: I:\xcaliburdata\2013\inc\structures\naco\nc.par (171.36.28)

Shell command window (Esc - interrupts)

Command shell

RED

Force auto scroll

Transparent

	intensity measured	kept	unique	redundancy	F2	F2/sig(F2)	Rint	Rsigma
1599685-	18923	1306	1272	254	5.0	137167.50	133.40	0.009
18918-	4444	1213	1208	254	4.8	9459.02	34.68	0.022
4443-	1583	1204	1193	254	4.7	2775.95	18.96	0.032
1569-	611	1242	1232	254	4.9	1008.56	10.95	0.053
611-	204	1156	1142	254	4.5	421.59	6.98	0.090
281-	129	1144	1136	254	4.5	198.75	4.23	0.174
128-	53	1120	1104	254	4.4	86.39	2.24	0.324
53-	21	1097	1084	254	4.3	35.30	1.03	0.618
20-	-1	1060	1058	254	4.2	9.00	0.32	0.923
-1-	-84	955	947	255	3.7	-14.46	-0.33	0.851
1599685-	-84	11497	11378	2541	4.5	16813.69	23.21	0.011

Statistics vs resolution - point group symmetry: C2/m (b-unique)

resolution(A)	# measured	# kept	# unique	average redundancy	mean F2	mean F2/sig(F2)	Rint	Rsigma	Rsign
inf-2.21	1505	1478	259	5.7	30699.19	41.23	0.010	0.010	0.0
2.20-1.68	1420	1411	256	5.5	30033.65	36.50	0.011	0.011	0.0
1.67-1.43	1247	1236	258	4.8	23065.95	28.18	0.009	0.010	0.0
1.43-1.27	1330	1326	257	5.2	17219.50	22.85	0.009	0.011	0.0
1.27-1.15	1152	1144	260	4.4	12176.42	19.67	0.012	0.014	0.0
1.15-1.07	1034	1022	258	4.0	14988.04	18.22	0.009	0.011	0.0
1.06-1.00	1189	1184	255	4.6	6597.70	12.91	0.016	0.018	0.0
1.00-0.95	934	921	258	3.6	5953.23	11.35	0.015	0.016	0.0
0.95-0.88	918	910	254	3.6	6909.91	12.26	0.018	0.021	0.0
0.88-0.78	760	746	226	3.3	4534.19	11.35	0.023	0.023	0.0
inf-0.78	11497	11378	2541	4.5	16813.69	23.21	0.011	0.012	0.0
inf-0.80	11488	11369	2532	4.5	16820.10	23.21	0.011	0.012	0.0

Redundant output table is not printed for incommensurate reflections!

Reading tabbin file: "I:\xcaliburdata\2013\inc\structures\naco\nc_proffitpeak"

Incommensurate/quasi-crystal information

UM SET/VECTOR 0.18375 -0.00005 0.32117 4

NADA QVECTOR 4 0.05000 0.18375 -0.00005 0.32117

Data reduction ended at Mon Feb 04 21:47:50 2013

Options RED

Close

START/STOP

RED Ready

Crystal

Data Collection

Data Reduction

PROCESS/STATUS

In run list: 1127/11, used: 1180/1

3D PROFILE ANALYSIS

Frames done: 1580

Reflections tested: 11479, used: 1148

Avg intensity (1/s degree): 1.5 mm(a)

Q2=0.94, Q3=0.81, Q4=0.51

Max incidence angle profile change(a1): 140

3D INTEGRATION & FITTING

Frames done: 1540

Fitted: 11358, overflow: 0, hidden: 0

Outliers rejected: 119

EVALUATION INPUT FILE

Filename: ms171623

EVALUATION OUTPUT FILE

Filename: ms171629

SCALING/STATISTICAL INFORMATION

Empirical abs (w=0.001): w=0.97, max=1.08

Frame scaled C2/scale): w=0.96, max=1.10

Detector scale (444.31) w=0.90, max=1.09

RESULTS (1540 frames): 3D C2/m (b-unique)

Resolution(A) Redundancy F2/sig(F2) Rint

inf - 0.78 4.5 23.2 0.011

inf - 0.80 4.5 23.2 0.011

Completeness: 94.1% (0.60 ANG)

SPECIAL

Incommensurate structures handling ON

DATA REDUCTION OPTIONS

3D profile fitting used

Agilent Technologies

CrysAlisPro



Naco: Strength of satellites

Main reflections

resolu- tion(Å)	# measured	# kept	# unique	average redundancy	mean F2	mean F2/sig(F2)	Rint	Rsigma	RsigmaA
inf-0.78	1274	1230	290	4.2	101051.65	88.11	0.008	0.009	0.005

Satellite reflections

inf-0.79	10223	10148	2251	4.5	6603.53	15.34	0.016	0.019	0.019
----------	-------	-------	------	-----	---------	-------	-------	-------	-------

1. order satellite reflections

inf-0.79	2592	2547	577	4.4	22747.40	40.22	0.012	0.015	0.012
----------	------	------	-----	-----	----------	-------	-------	-------	-------

2. order satellite reflections

inf-0.82	2564	2553	565	4.5	2976.23	13.65	0.024	0.030	0.039
----------	------	------	-----	-----	---------	-------	-------	-------	-------

3. order satellite reflections

inf-0.80	2561	2550	562	4.5	449.45	4.88	0.075	0.099	0.127
----------	------	------	-----	-----	--------	------	-------	-------	-------

4. order satellite reflections

inf-0.81	2506	2498	547	4.6	132.35	2.39	0.189	0.244	0.300
----------	------	------	-----	-----	--------	------	-------	-------	-------

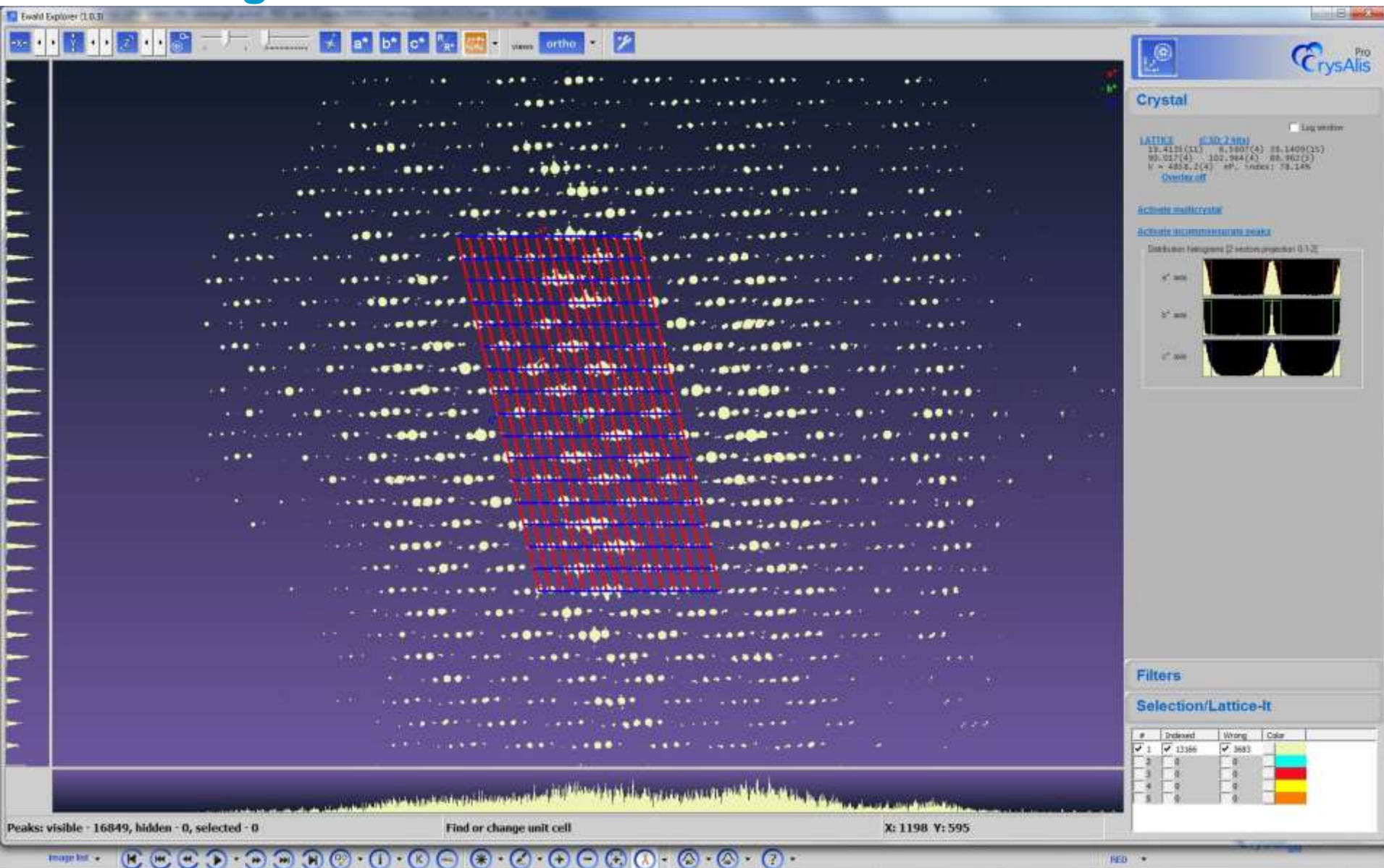


Recognition of incommensurate structures in CrysAlis

- Case Oldenburg
- Sample with high order satellites
- Normal unit cell finding fails: Super cell found



Oldenburg:



Oldenburg:

Ewald Explorer (1.0.3)

Find cell...

Peak table

- ☒ Normal peak table
- ☐ Delta (differential) peak table

Algorithm

- ☒ T-vector Dirax
- ☐ Stereographic

Sample type

- ☒ Single crystal
 - Unit cell limits
 - ☐ SM ☐ PX ☒ User
 - min: 2.0 max: 22
 - Calc**
- ☐ Twin / multycrystal
 - # of components: 2
 - min: 2.0 max: 120.0
 - Calc**

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 (uncheck for multi-crystal)

Known cell

☐ Search known cell 19.41 6.56 39.14 90.02 102.96 89.96

OK **Cancel**

Crystal

LATICE **hkl: 1 0 0**

10.1690(10) 8.5560(10) 13.1191(13)
90.029(11) 112.284(9) 89.881(10)
V = 209.17(16) Å^3 mp, index: 24, 42%

[Qunitex off](#)

[Activate multycrystal](#)

[Activate incommensurate peaks](#)

Distribution histograms (2 sections projection 0.1/2)

Filters

Selection/Lattice-It

#	Indexed	Wrong	Color
✓ 1	✓ +114	✓ 12735	
2	0	0	
3	0	0	
4	0	0	
5	0	0	

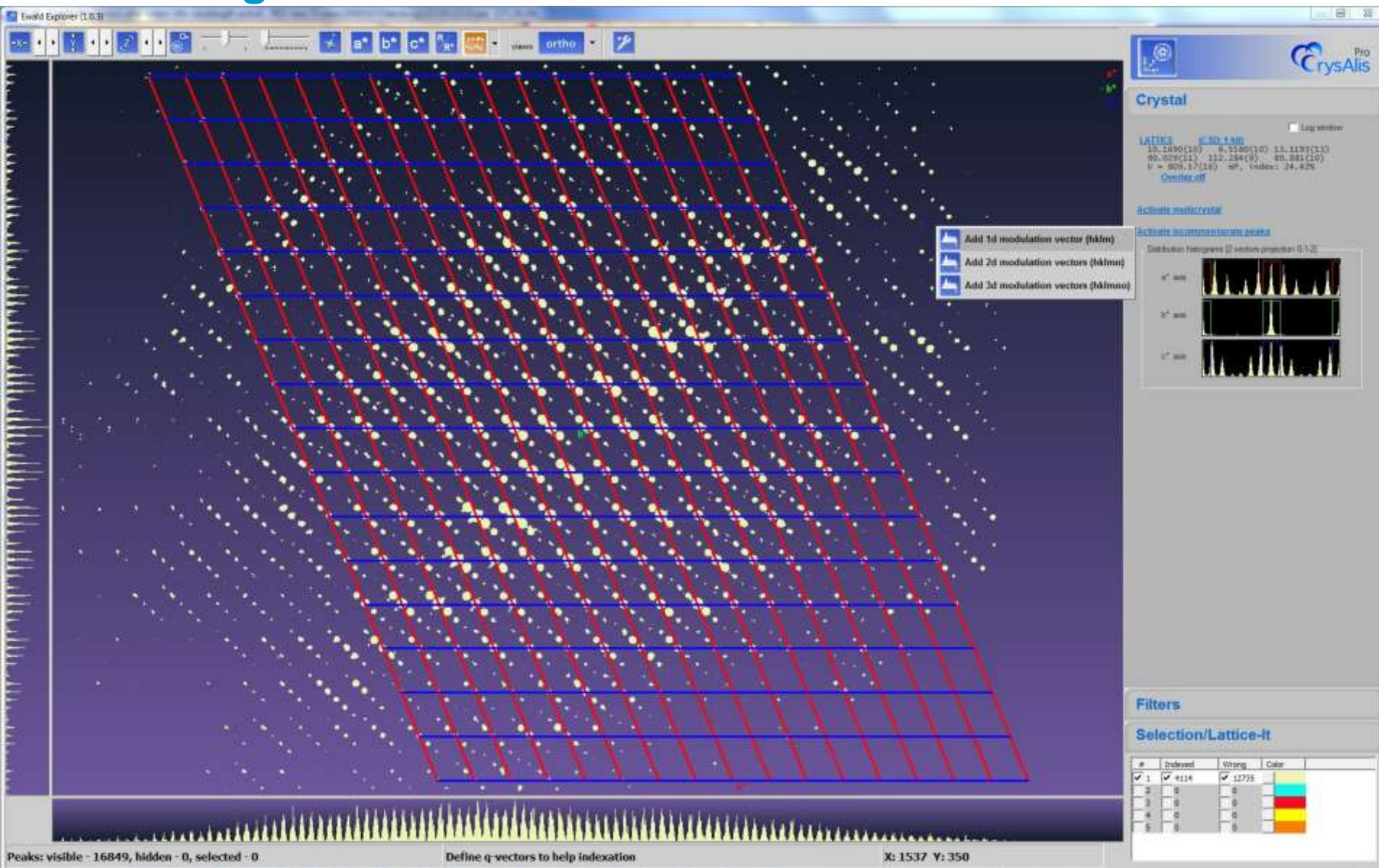
Peaks: visible - 16849, hidden - 0, selected - 0

Open Check CSD tool

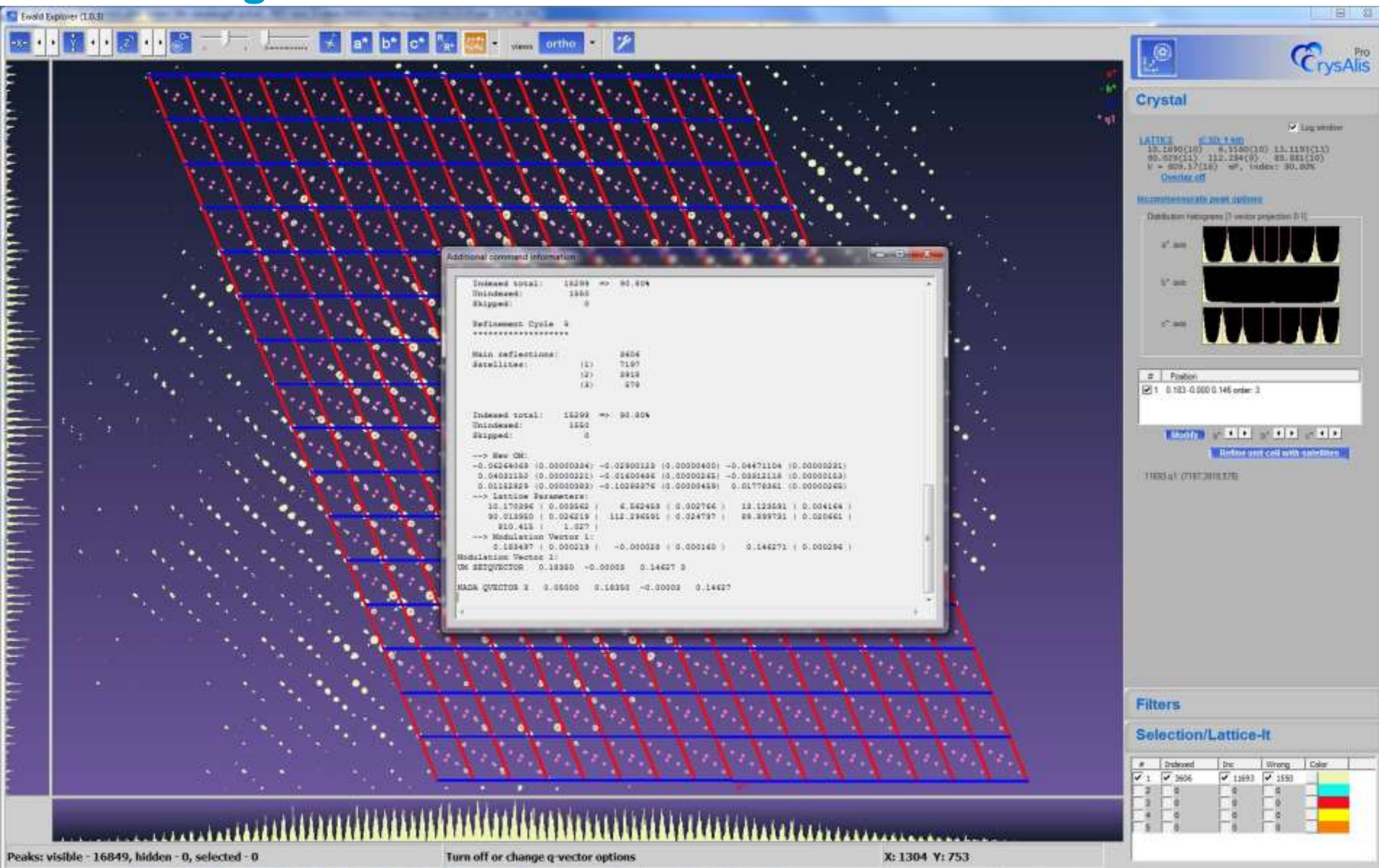
X: 1038 Y: 816





Oldenburg:



Oldenburg:



Lattice wizard (1.0.32)


Lattice wizard


LATTICE

Current cell (CSD: 1 hit)
 10.1690(10) 6.5580(10) 13.1193(13) 90.029(11) 112.284(9) 89.881(10) 809.57(16)

Constrained current cell
 10.1676(15) 6.5561(5) 13.1328(18) 90.0 112.368(16) 90.0 809.57(17)

Lattice reduction
 selected cell
 10.1687 6.5585 13.1183 90.0295 112.2816 89.8858 mP 35
 reduced cell
 6.5585 10.1687 13.1183 67.7184 89.9705 89.8858 809.6

Incommensurate/quasi-crystal information
 q(1): 0.1835(2) 0(1.6e-004) 0.1463(3)

PEAK TABLE













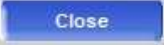
Peak hunting table
 UB fit with 15303 obs out of 16849 (total:16849,skipped:0) (90.82%)
 3606 main refl.; 11697 q1 satellites
 By order
 11697 q1: (7197,3918,578,4)

INSTRUMENT MODEL

Goniometer
 beam: -0.11081 alpha: 49.81236 beta: -0.01532
 om zero: -0.42381 th zero: -0.07605 ka zero: 0.01549

Detector
 x-rot: -0.02924 y-rot: -0.07001
 x-cen: 1022.04058 y-cen: 1036.48085 distance: 60.05314

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
 Twinning - multi-crystals
 Incommensurates / Quasi-crystals
 Load information
 Save information
 Unwarping - Precession images
 Log window
 Close

Profit: CrysAlisPro data reduction assistant (1.0.25)

Profile fitting data reduction

Step 1: Orientation matrix for data reduction

```
UB - matrix:
-0.062641  -0.029001  -0.044711  ( 0.000008  0.000012  0.000006 )
 0.040312  -0.016005  -0.033121  ( 0.000006  0.000009  0.000004 )
 0.011528  -0.102884  0.017784  ( 0.000011  0.000017  0.000008 )
10.17029 ( 0.00097 )  6.56246 ( 0.00096 ) 13.12359 ( 0.00133 )
90.01393 ( 0.01053 ) 112.29657 ( 0.00921 ) 89.89980 ( 0.01027 )

V =      810.41
Selected cell (from UM xx/UM ttt/UM f):
35  10.1704  6.5625 13.1236 90.0139 112.2966 89.8998 mP
q(1) vector: 0.1835 -0.0000 0.1463
```

Lattice extinctions (filter Bravais lattice extinctions):

☒ Don't use filter (P-lattice)

☐ Use filter for:

Incommensurate structures:

☐ Normal data reduction (HKL)

☒ Single q-vector

☐ Other (reduction list)

Twinning/Multi crystal (activated by UM TWIN entries):

☐ Use automatic twin/multi crystal data reduction with the following components: ☐ Multi crystal

☐ Component 1 ☐ Component 2 ☐ Component 3 ☐ Component 4



Oldenburg: Strength of satellites

Main reflections

resolu- tion(A)	# measured	# kept	# unique	average redundancy	mean F2	mean F2/sig(F2)	Rint	Rsigma	RsigmaA
inf-0.80	6588	6543	1813	3.6	22536.12	18.98	0.019	0.022	0.022

Satellite reflections

inf-0.80	55590	54668	16211	3.4	2911.76	6.00	0.049	0.061	0.082
----------	-------	-------	-------	-----	---------	------	-------	-------	-------

1. order satellite reflections

inf-0.80	13890	13827	4055	3.4	8502.10	12.87	0.025	0.031	0.048
----------	-------	-------	------	-----	---------	-------	-------	-------	-------

2. order satellite reflections

inf-0.80	13897	13827	4052	3.4	2024.81	6.17	0.056	0.067	0.116
----------	-------	-------	------	-----	---------	------	-------	-------	-------

3. order satellite reflections

inf-0.80	13905	13743	4065	3.4	540.78	2.84	0.168	0.188	0.272
----------	-------	-------	------	-----	--------	------	-------	-------	-------

4. order satellite reflections

inf-0.80	13898	13271	4039	3.3	466.63	1.96	0.328	0.318	0.328
----------	-------	-------	------	-----	--------	------	-------	-------	-------

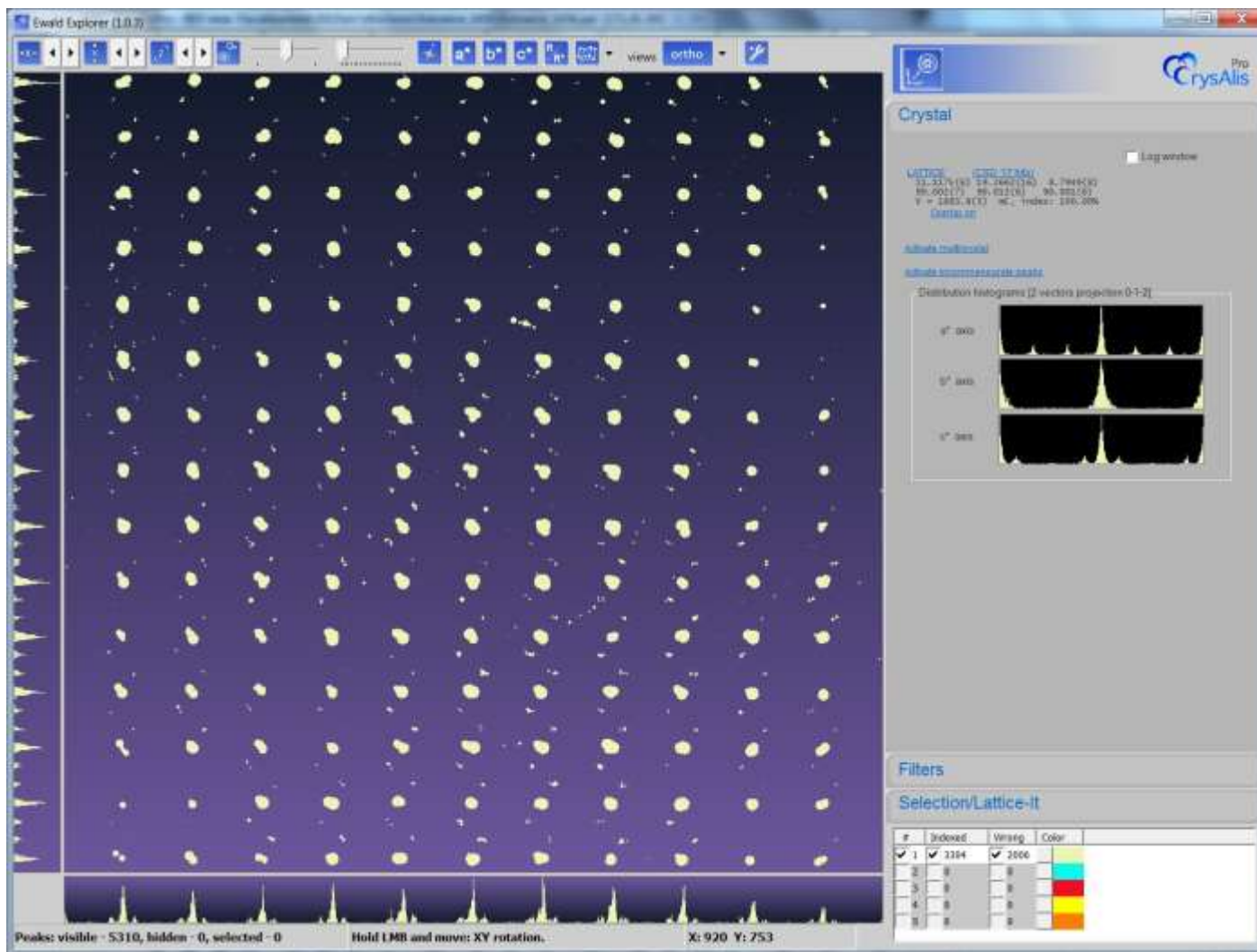


Recognition of incommensurate structures in CrysAlis

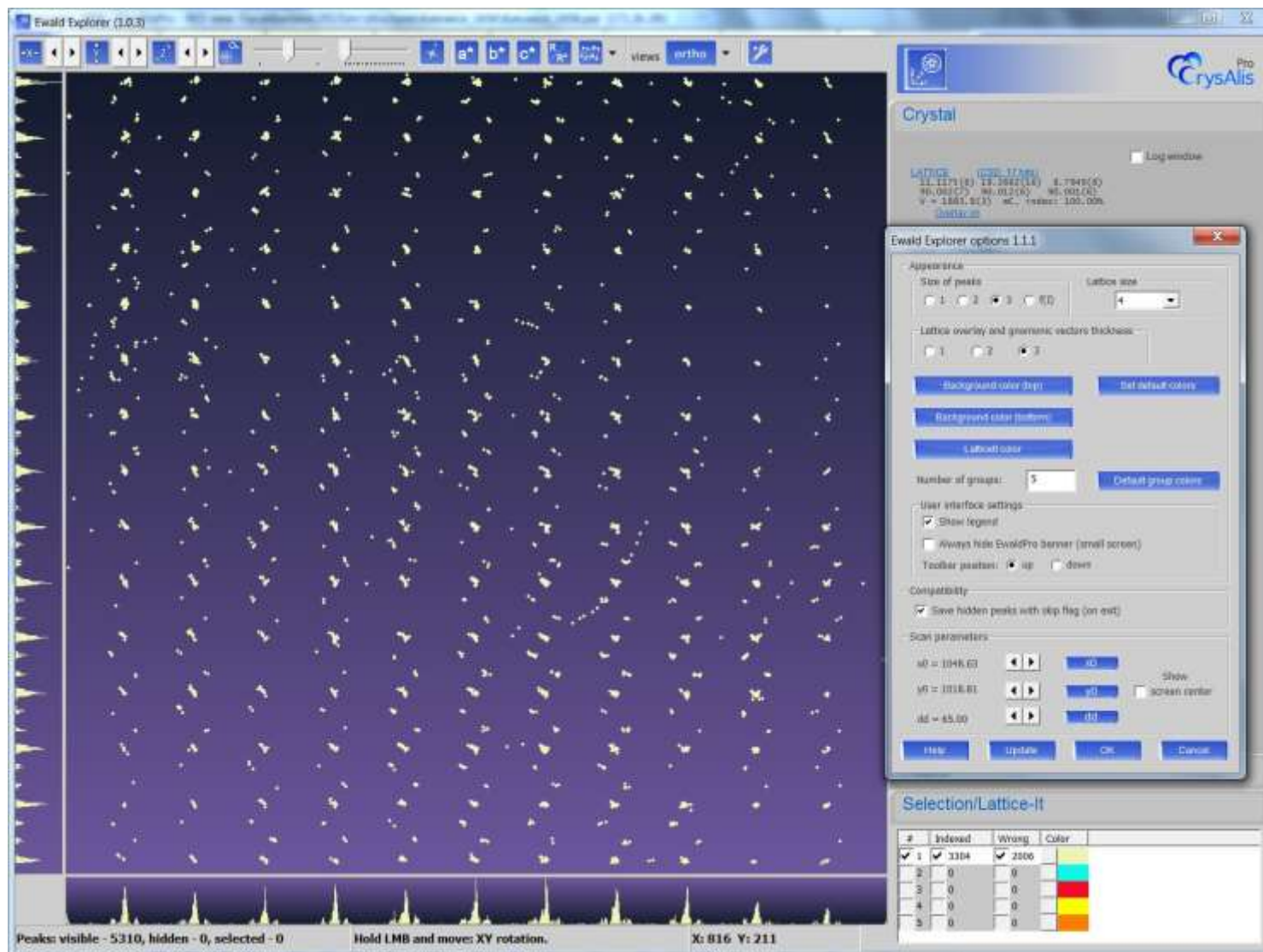
- Case katowice_165K
- Mistaking commensurate super-lattice as incommensurate!
- Normal unit cell finding fails: Super cell found



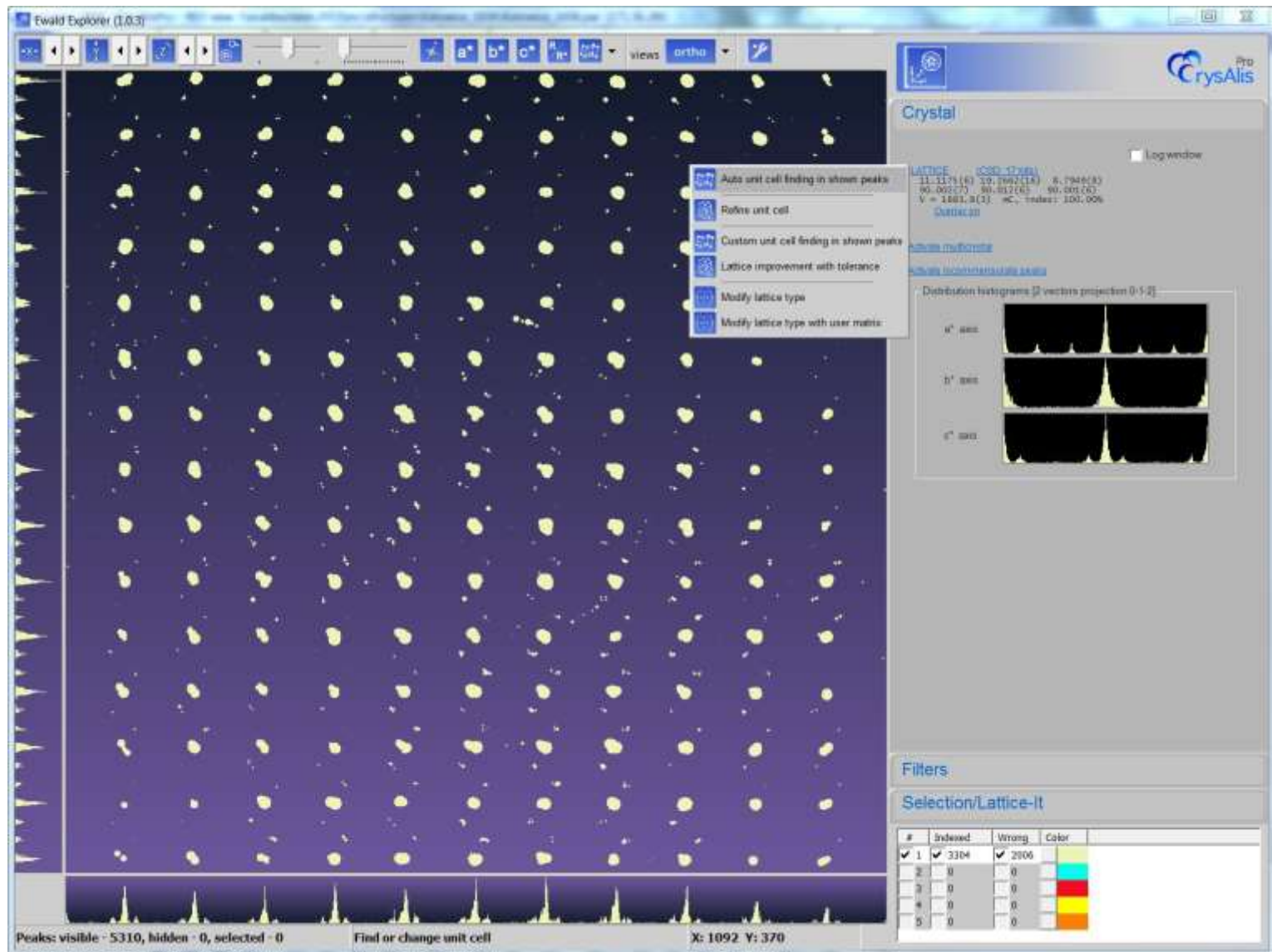
Katowice_165K:



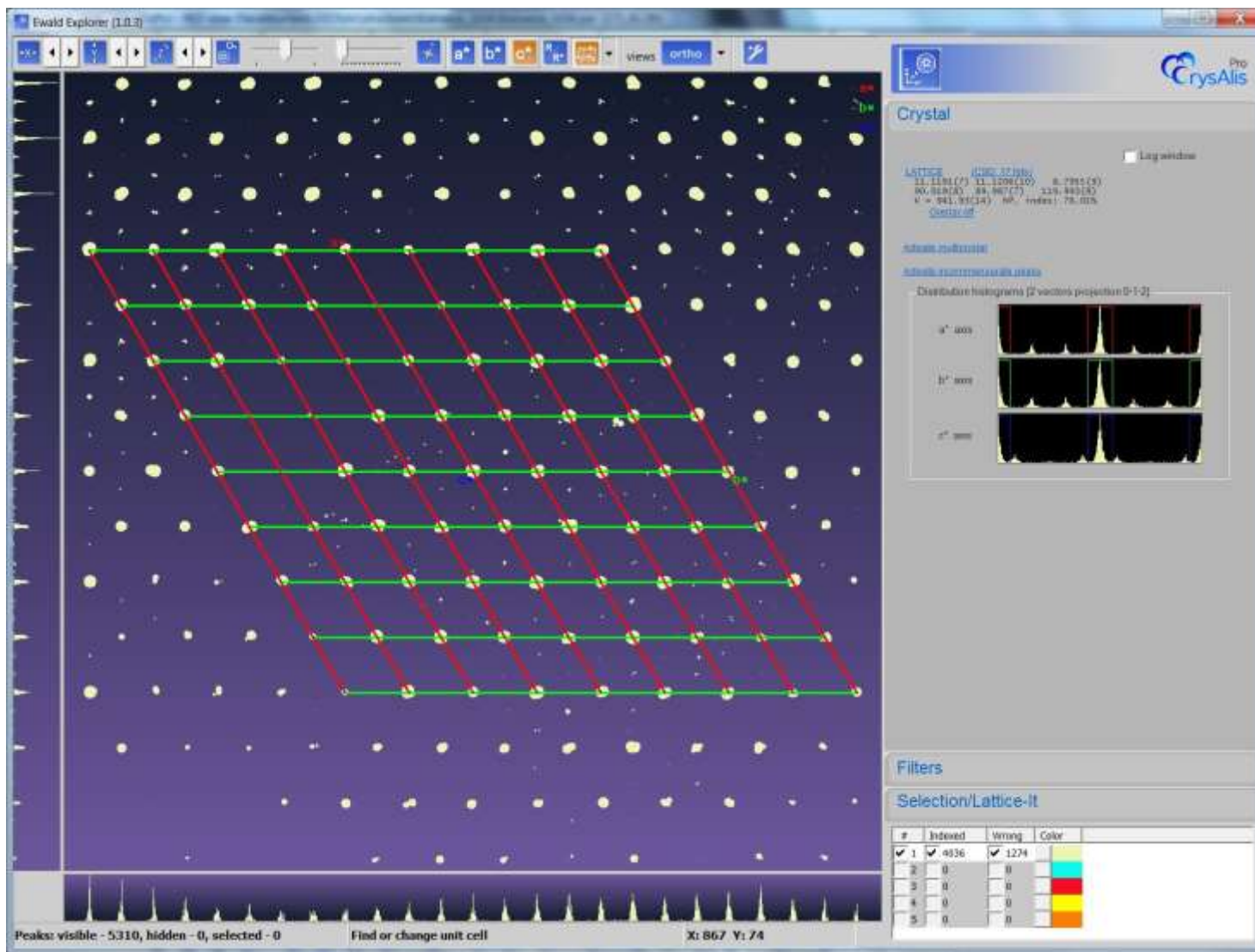
Katowice_165K:



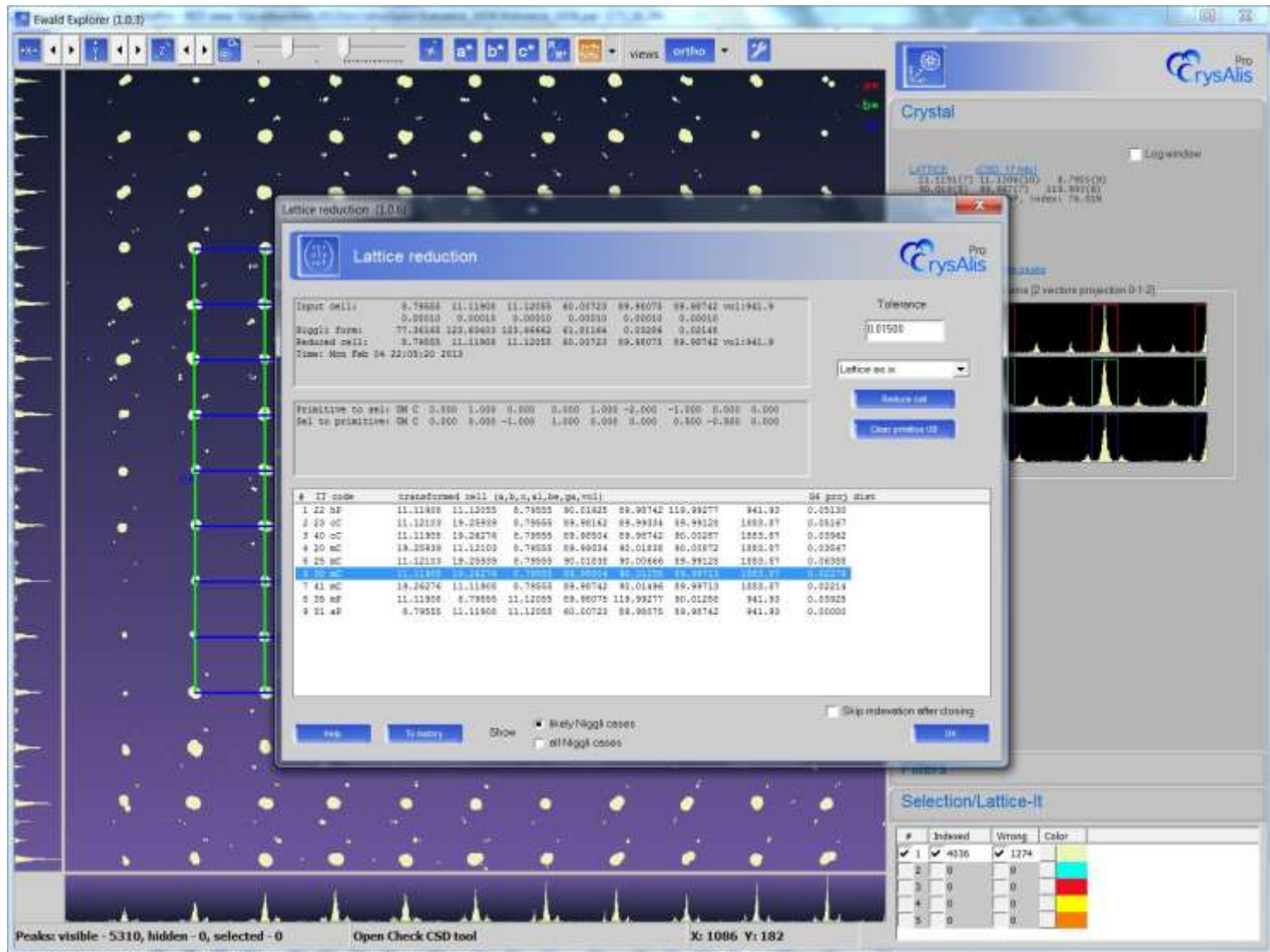
Katowice_165K:



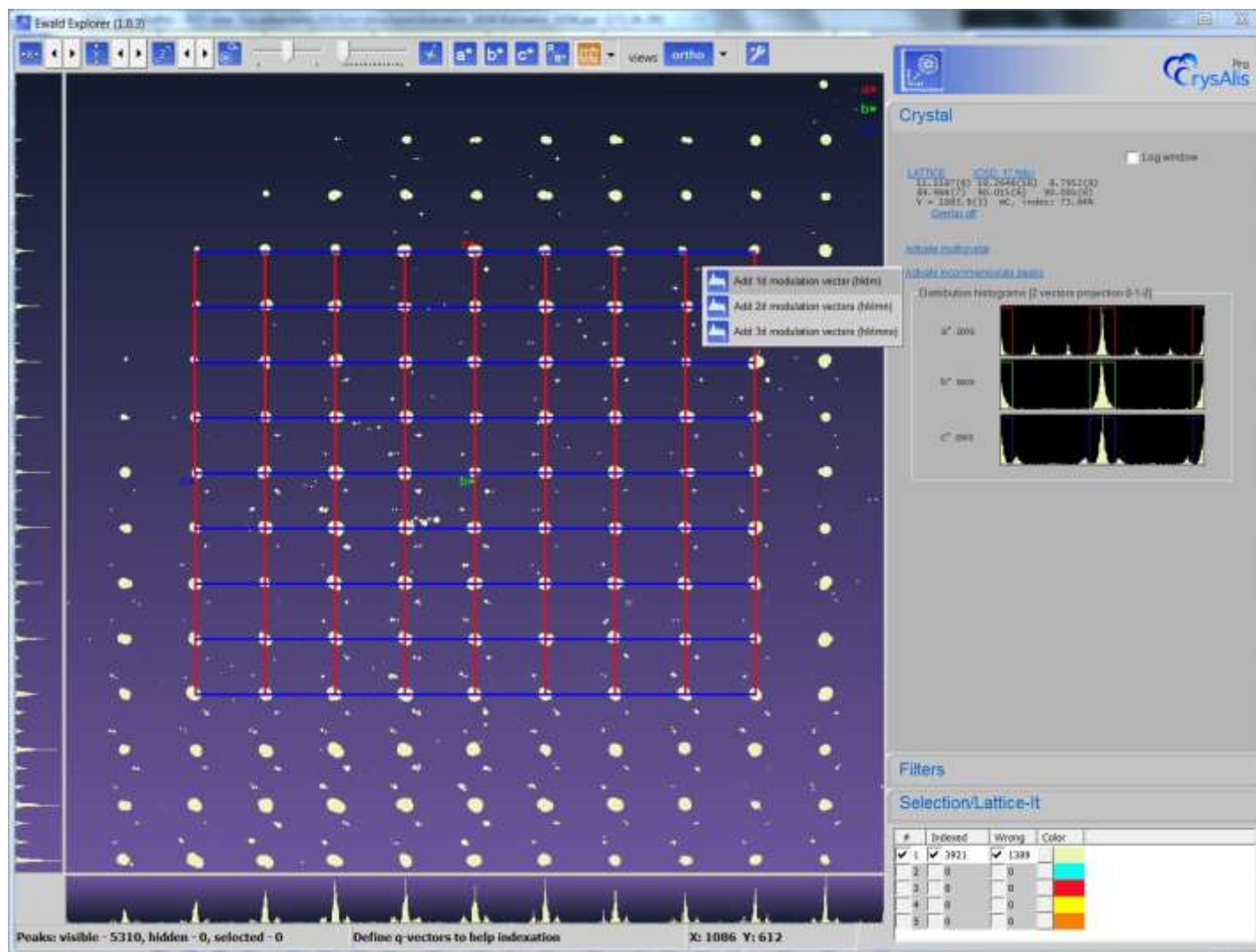
Katowice_165K:



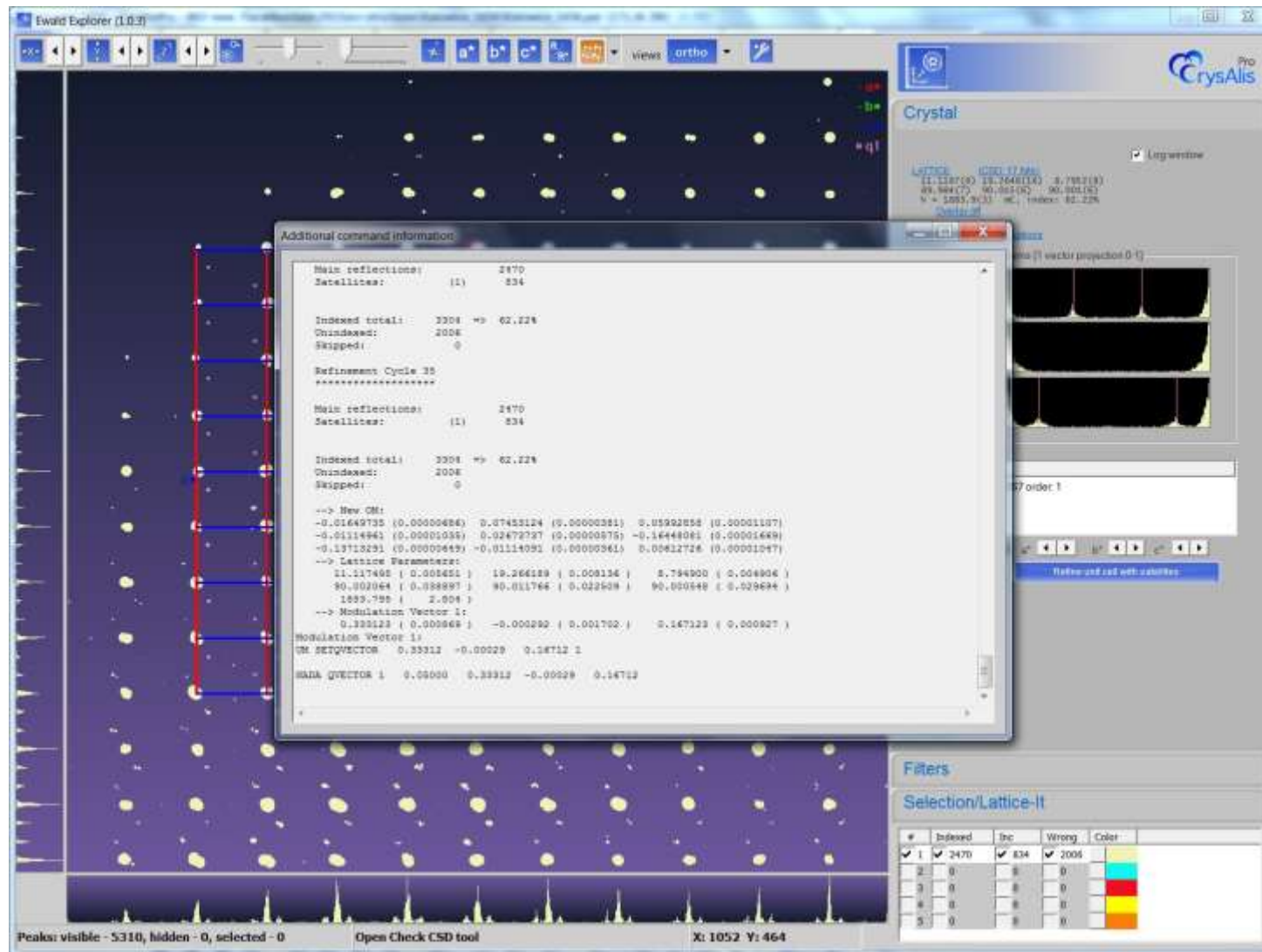
Katowice_165K:



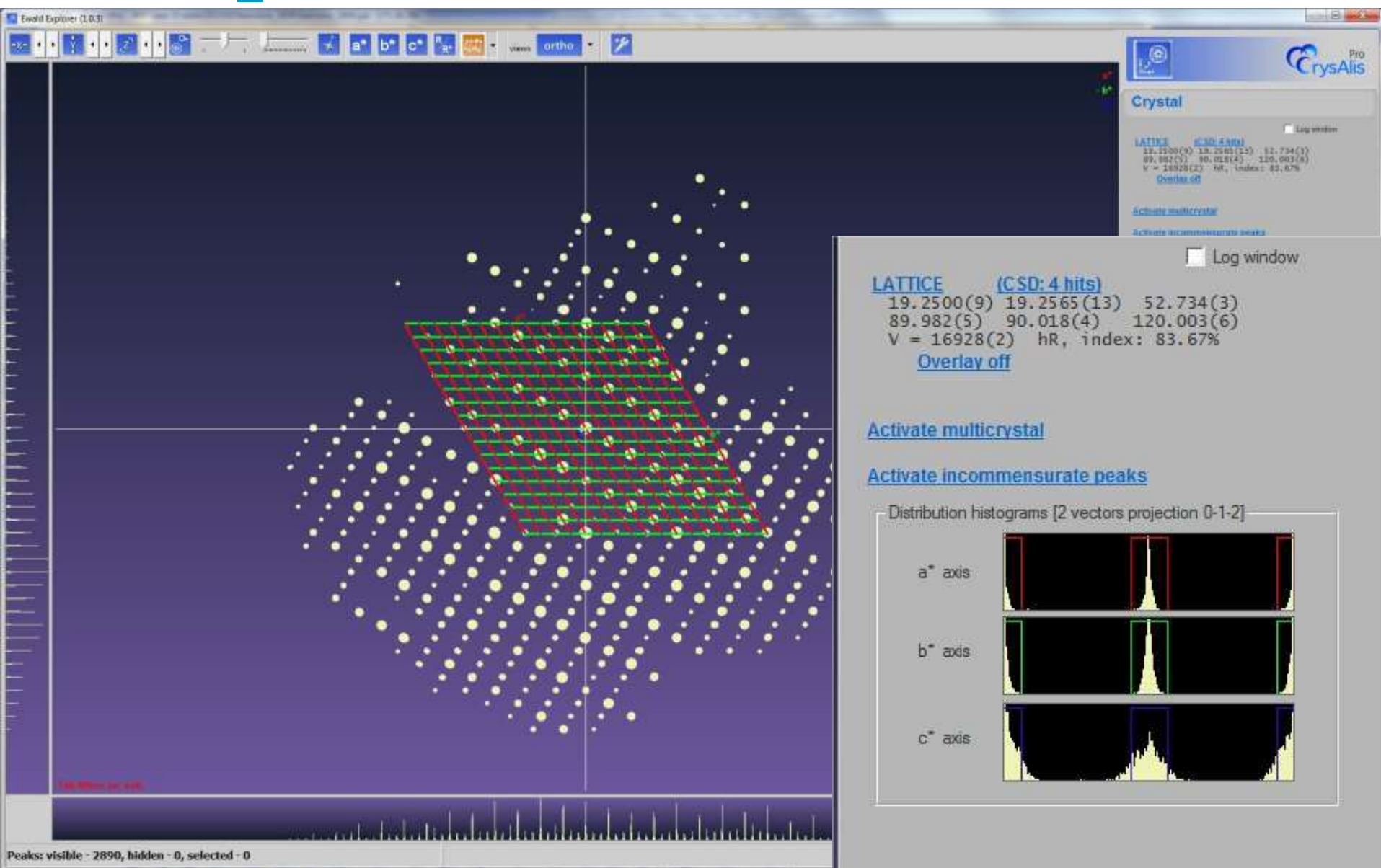
Katowice_165K:



Katowice_165K:



Katowice_165K:

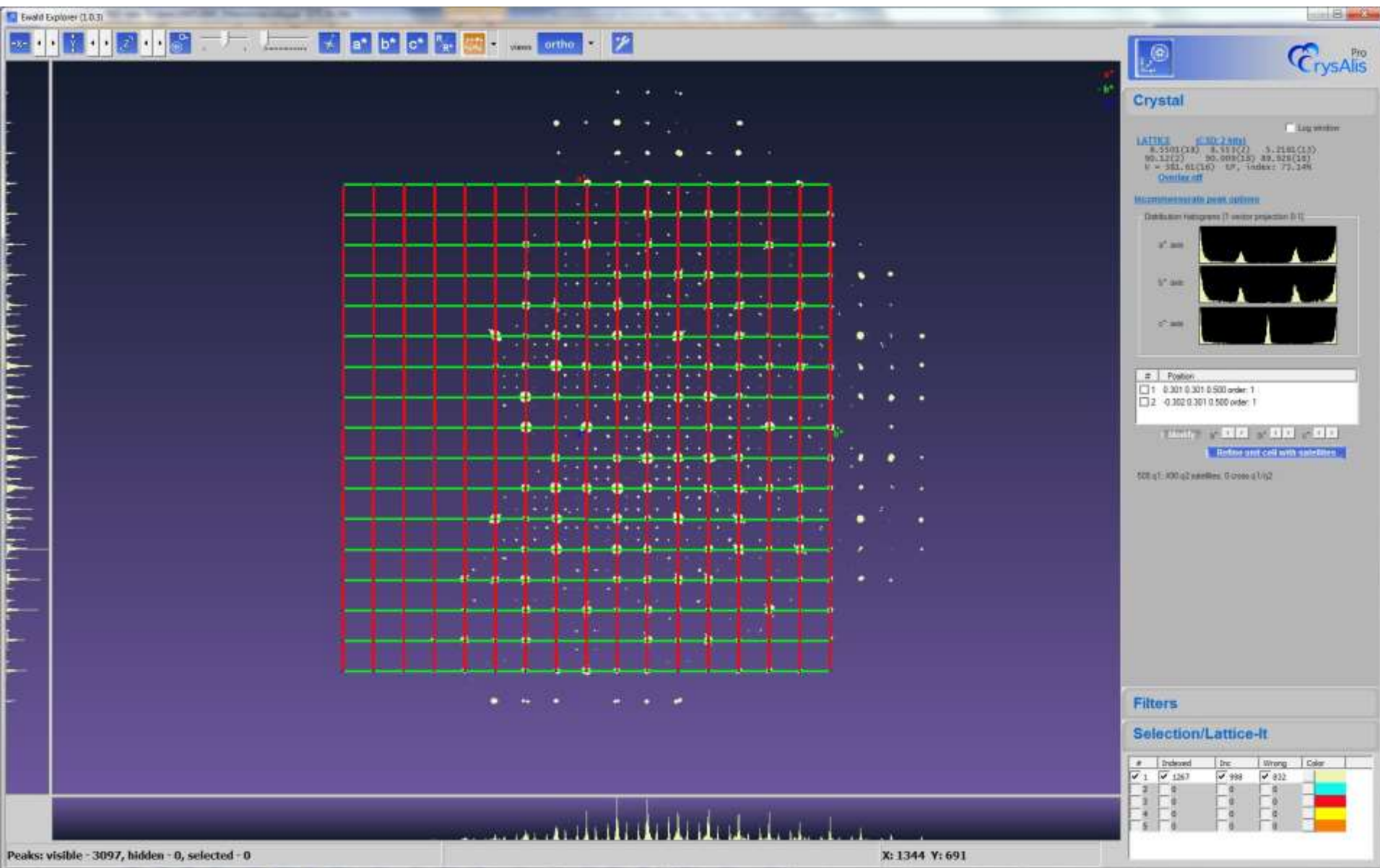


Recognition of incommensurate structures in CrysAlis

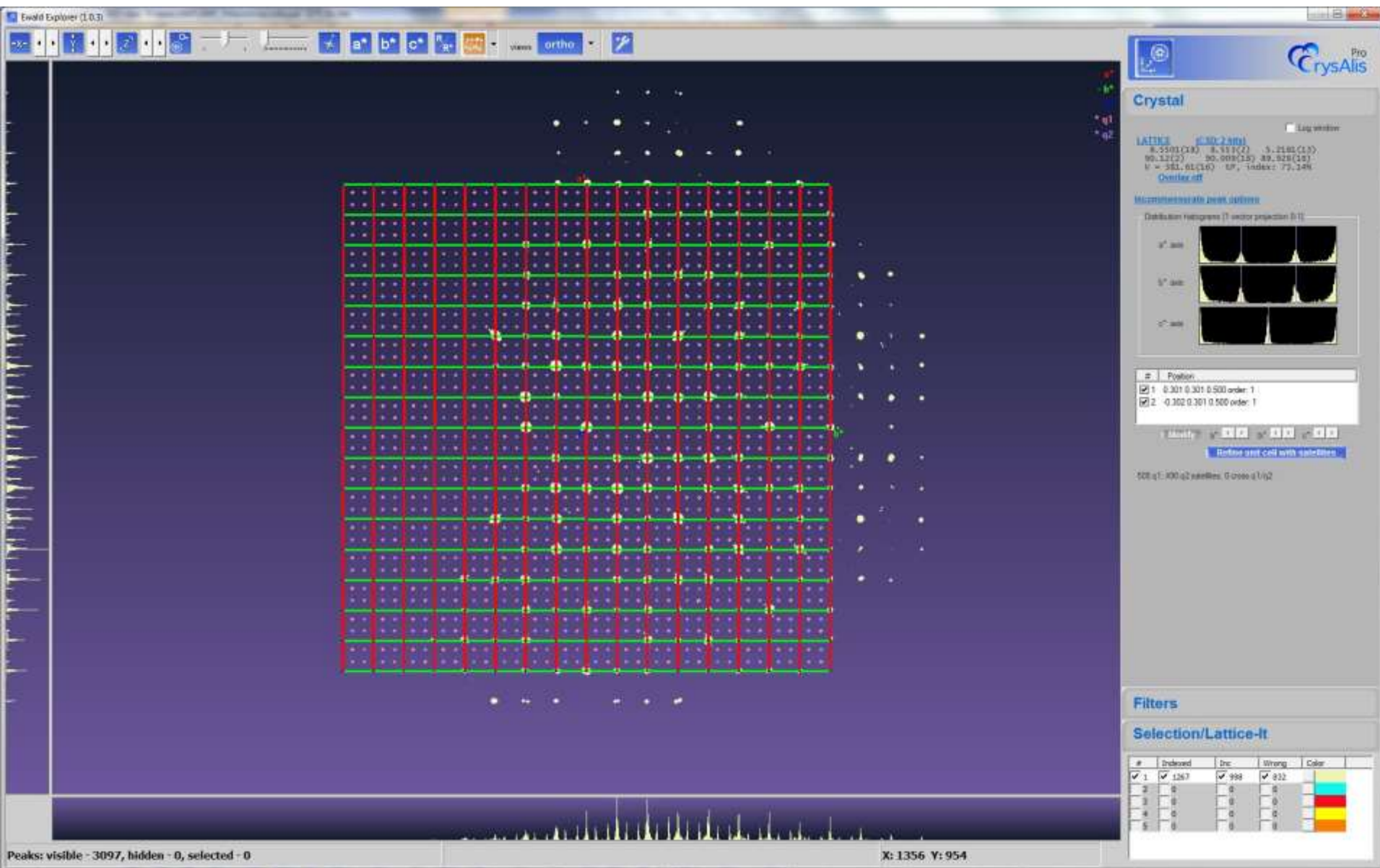
- Case fresno/raccolta
- Tetragonal sample with 2(!) q-vectors!
- Symmetry dictates the orientations



Fresno:



Fresno:



Lattice wizard (1.0.32)

Lattice wizard

LATTICE

Current cell [\(CSD: 2 hits\)](#)
 8.5501(18) 8.553(2) 5.2181(13) 90.12(2) 90.009(18) 89.928(18) 381.61(16)

Constrained current cell
 8.5411(13) 8.5411(13) 5.2311(18) 90.0 90.0 90.0 381.61(16)

Lattice reduction
 selected cell
 8.5418 8.5480 5.2137 89.8598 89.9784 89.9610 tP 21
 reduced cell
 5.2137 8.5418 8.5480 89.9610 89.8598 89.9784 380.7

Incommensurate/quasi-crystal information
 q(1): 0.3012(15) 0.3014(18) 0.5000(12) q(2): -0.3015(19) 0.301(2) 0.5002(13)

PEAK TABLE

Peak hunting table
 UB fit with 2265 obs out of 2265 (total:3097,skipped:832) (100.00%)
 1267 main refl.; 508 q1 ; 490 q2 satellites; 0 cross q1/q2

INSTRUMENT MODEL

Goniometer
 beam: 0.07153 alpha: 49.94538 beta: 0.00990
 om zero: 0.15902 th zero: -0.50465 ka zero: 0.24275

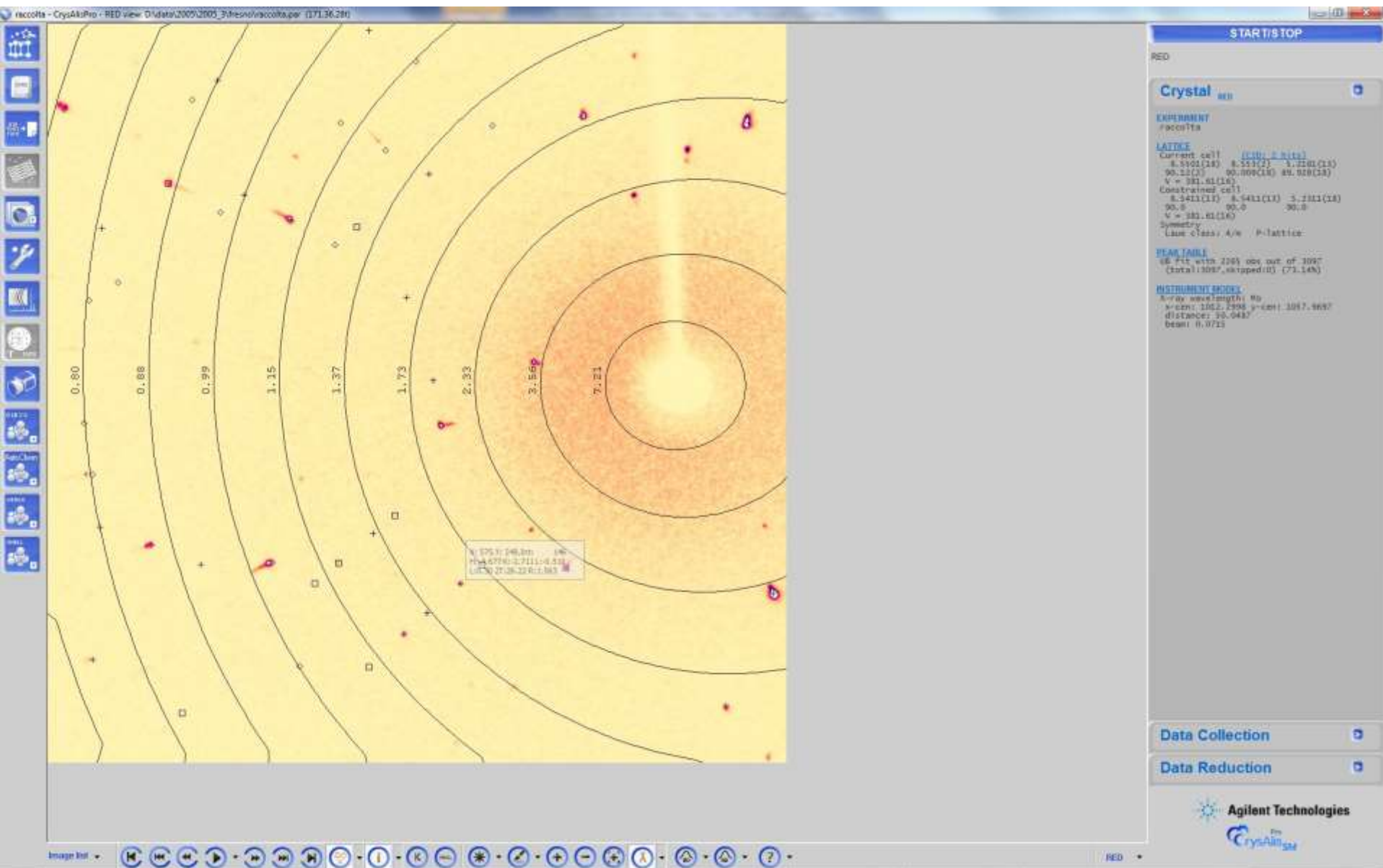
Detector
 x-rot: -0.56156 y-rot: 0.11022
 x-cen: 1012.29978 y-cen: 1057.96971 distance: 50.04869

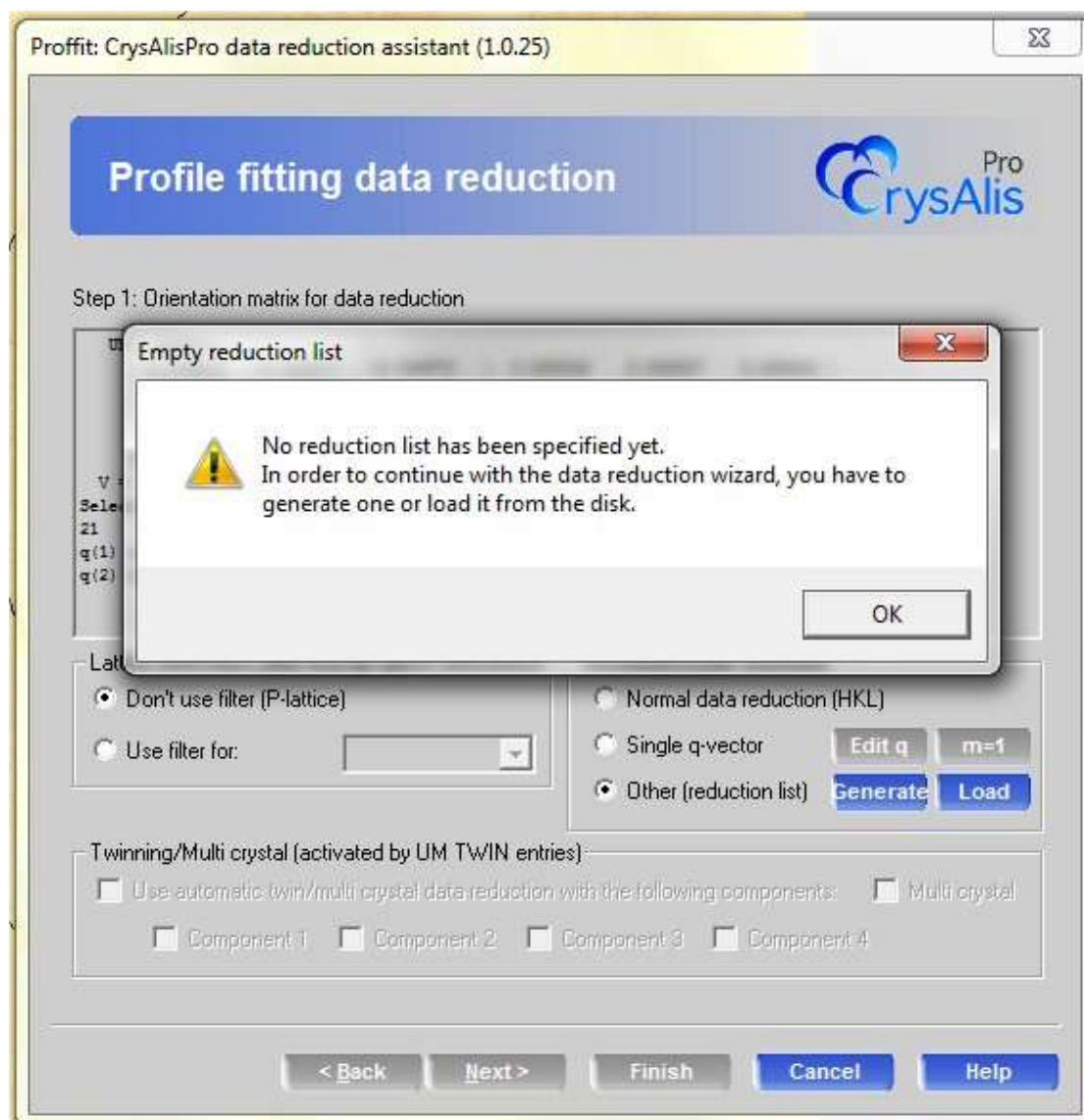
Wavelength Mo (Ang): A1 0.70930 A2 0.71359 B1 0.63229

Peak hunting
 Unit cell finding
 Ewald explorer - reciprocal space
 Reindexation with current cell
 Refine instrument model
 Lattice transformation
 Twinning - multi-crystals
 Incommensurates / Quasi-crystals
 Load information
 Save information
 Unwarping - Precession images
 Log window



Close

Fresno:





Proffit reduction list generation (1.0.3)

 **Generate hkl list (hkl,hklm,hklmn etc)** 

CrysAlis hkl reduction lists (*.dcred*)

Min/Max

d-value (Ang) hkl max (from resolution limit): 13 13 8

hkl max: 13 13 8

q-vectors

q(1): 0.3012 0.3014 0.5000 mmax: 1

q(2): -0.3015 0.3010 0.5002 nmax: 1

q(3): omax: 0

☒ Quasi-crystal ☐ Incommensurate

q limit: 0.0000

Options

☐ Use cross q-vector reflections max index for cross reflections: 0

Extinction rules

No extinction rules specified

HINT: You can use DC EXTINGT to add extinction rules and DC CLEAREXTINGT to remove selected or all rules from the list

Fresno: Strength of satellites

DC RED/RRP INFO: Incommensurate/quasi-crystal statistics

Main reflections

Statistics vs resolution - point group symmetry: P4/m

resolu- tion(A)	# measured	# kept	# unique	average redundancy	mean F2	mean F2/sig(F2)	Rint	Rsigma	RsigmaA
inf-0.80	1602	1591	421	3.8	128244.12	18.87	0.023	0.024	0.037

Satellite reflections

inf-0.80	6695	6521	4033	1.6	1392.20	1.88	0.358	0.324	0.426
----------	------	------	------	-----	---------	------	-------	-------	-------



Export to Jana

- Via hkl or CIF

```
_diffrn_reflns_theta_min 4.5503
_diffrn_reflns_theta_max 29.4106
_diffrn_reflns_theta_full 26.3154
_diffrn_measured_fraction_theta_full 0.9568
_diffrn_orient_matrix_UB_11 0.0761045000
_diffrn_orient_matrix_UB_12 -0.0229269000
_diffrn_orient_matrix_UB_13 -0.0397729000
_diffrn_orient_matrix_UB_21 0.0041613000
_diffrn_orient_matrix_UB_22 0.0664606000
_diffrn_orient_matrix_UB_23 -0.0811103000
_diffrn_orient_matrix_UB_31 0.0330885000
_diffrn_orient_matrix_UB_32 0.0442330000
_diffrn_orient_matrix_UB_33 0.1019252000
_diffrn_measurement_details
;
#_ type_ start_ end_ width_ exp.time_
1 omega -99.58 50.42 1.0000 30.0000
omega_ theta_ kappa_ phi_ frames
- -23.0000 -10.0000 30.0000 150
;
#_ type_ start_ end_ width_ exp.time_
2 omega -86.80 14.20 1.0000 30.0000
omega_ theta_ kappa_ phi_ frames
- -15.0000 -70.0000 0.0000 101
;
_diffrn_measurement_method '\w scans'
_reflns_odcompleteness_completeness 95.68
_reflns_odcompleteness_theta 26.32
_reflns_odcompleteness_iscentric 1
;
loop_
_hkl_oxdiff_h
_hkl_oxdiff_k
_hkl_oxdiff_l
_hkl_oxdiff_m
_hkl_oxdiff_n
_hkl_oxdiff_f2
_hkl_oxdiff_sig
_hkl_oxdiff_ba
_hkl_oxdiff_frac_h
_hkl_oxdiff_frac_k
_hkl_oxdiff_frac_l
2 1 0 0 0 144968. 3413.03 2 2.00000 1.00000 0.00000
1 -2 0 0 0 140331. 2963.35 1 1.00000 -2.00000 0.00000
-2 -1 0 0 0 138886. 3098.56 1 -2.00000 -1.00000 0.00000
-2 -1 0 0 0 137255. 3336.35 2 -2.00000 -1.00000 0.00000
2 0 0 0 0 84763.1 2668.13 2 2.00000 0.00000 0.00000
-2 0 0 0 0 85509.2 2688.20 2 -2.00000 0.00000 0.00000
0 -2 0 0 0 83962.7 2045.80 1 0.00000 -2.00000 0.00000
2 -1 0 0 0 140081. 3503.16 2 2.00000 -1.00000 0.00000
-1 -2 0 0 0 141876. 2698.34 1 -1.00000 -2.00000 0.00000
2 -1 0 0 0 140580. 2998.18 1 2.00000 -1.00000 0.00000
-2 1 0 0 0 138181. 3547.67 2 -2.00000 1.00000 0.00000
2 -2 0 0 0 208573. 4578.08 2 2.00000 -2.00000 0.00000
```

Data reduction finalizing - rrp file to hkl file (1.6.6)

Chemical formula
Not available [Edit formula](#)

Absorption correction
☐ Apply face based absorption correction
Analytical absorption correction
No face information available!

[Show face list](#)

☐ Apply

4/m (tetra-c) ☐ Don't use outlier rejection
5.21004 90.19213 90.01299 89.96898
equivalent

Overlap+twin reject
[Error model options](#)
0.60000 6.00000 [Edit rej. par](#)
100.0000 [Edit overlap rej](#)
100.0000 [Edit twin rej](#)

Limits, filters and lattice extinction filters
☒ Neg intensity sigma limit -3.0 [Edit sig limit](#)
☐ Resolution d-value (Ang) inf: 0.80 [Edit res limits](#)
2theta (deg) 0.00-52.63
☒ No filter ☐ use filter for: P-lattice
[Filters](#) 0 active filters

Use in external programs
n (for absorption correction)/special formats:
[Edit batch](#)

User modifications
☐ Export .eqv file
☐ Export Stoe *.crs
☐ Export sadabs *_1.raw;...
☒ Export CIF file
☐ Export MTZ file
☒ Scaling and empirical absorption
☐ Space group determination (GRAL)
☒ Completeness (0.80 Ang)
☐ Structure solution (AutoChem)

[MTZ merging options](#)
[Edit ABSPACK](#)
[Space group options](#)
[Edit resolution](#)
[AutoChem options](#)

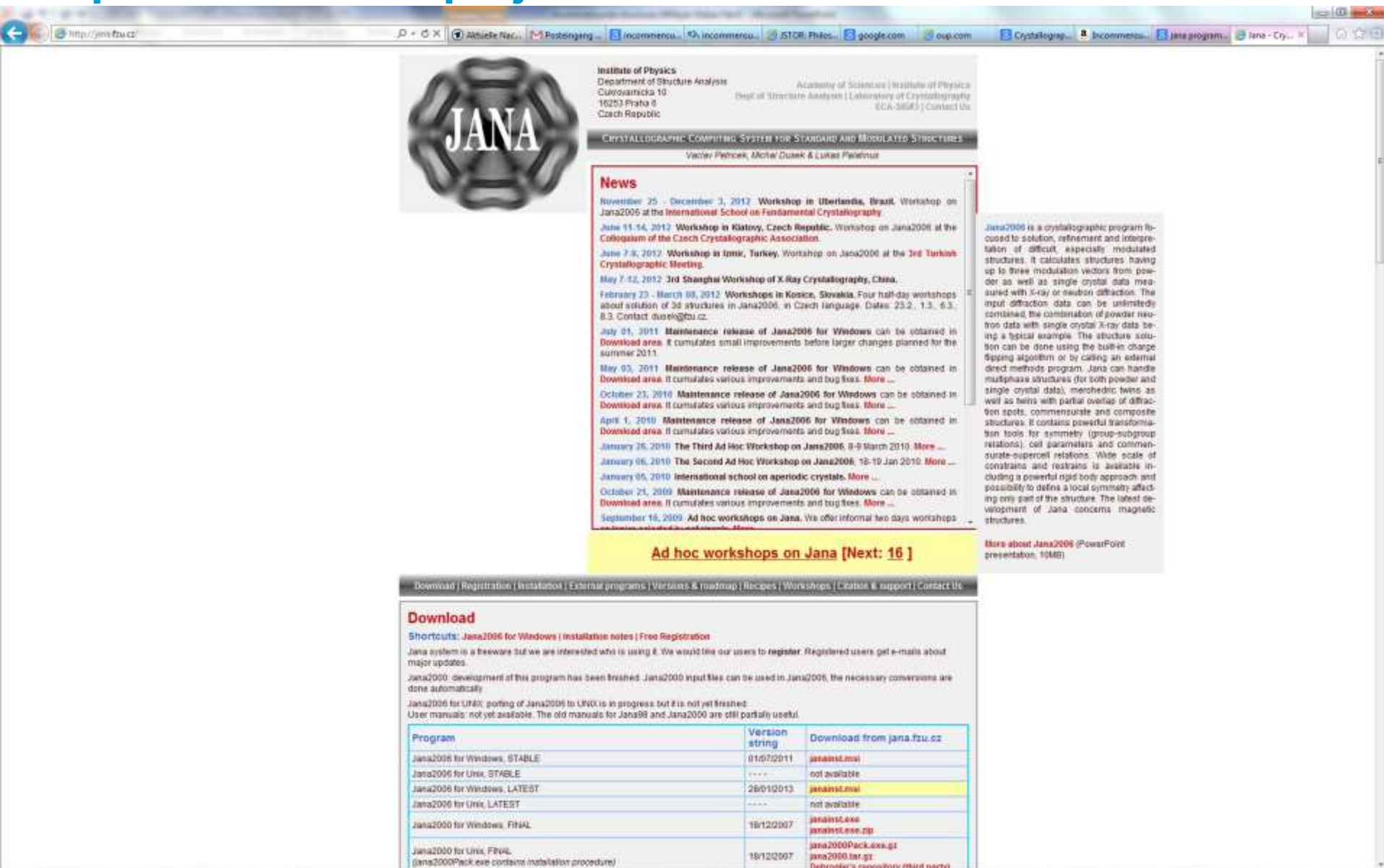
[OK](#) [Cancel](#)

Add hkl info



Agilent Technologies

Export to Jana: <http://jana.fzu.cz/>



The screenshot shows the official website for Jana2006, a crystallographic software package. The page features a header with the Jana logo and contact information for the Institute of Physics at the University of Science and Technology, Prague. A central 'News' section lists various workshops and updates from 2009 to 2012. A sidebar on the right provides a brief overview of the software's capabilities. The main content area includes a 'Download' section with a table of available versions and links to registration and installation notes.

Institute of Physics
Department of Structure Analysis
Cukrovarnická 10
16253 Praha 8
Czech Republic

Academy of Sciences | Institute of Physics
Dept of Structure Analysis | Laboratory of Crystallography
ECA-5603 | Contact Us

CRYSTALLOGRAPHIC COMPUTING SYSTEM FOR STANDARD AND MODULATED STRUCTURES
Václav Petrášek, Michal Dusek & Lukáš Palatinus

News

- November 25 - December 3, 2012 Workshop in Uberlandia, Brazil. Workshop on Jana2006 at the International School on Fundamental Crystallography.
- June 11-14, 2012 Workshop in Klatovy, Czech Republic. Workshop on Jana2006 at the Colloquium of the Czech Crystallographic Association.
- June 7-9, 2012 Workshop in Izmir, Turkey. Workshop on Jana2006 at the 3rd Turkish Crystallographic Meeting.
- May 7-12, 2012 3rd Shanghai Workshop of X-Ray Crystallography, China.
- February 23 - March 08, 2012 Workshops in Kosice, Slovakia. Four half-day workshops about solution of 3d structures in Jana2006, in Czech language. Dates: 23.2., 1.3., 6.3., 8.3. Contact: dusek@fzu.cz.
- July 01, 2011 Maintenance release of Jana2006 for Windows can be obtained in [Download area](#). It cumulates small improvements before larger changes planned for the summer 2011.
- May 03, 2011 Maintenance release of Jana2006 for Windows can be obtained in [Download area](#). It cumulates various improvements and bug fixes. [More ...](#)
- October 23, 2010 Maintenance release of Jana2006 for Windows can be obtained in [Download area](#). It cumulates various improvements and bug fixes. [More ...](#)
- April 1, 2010 Maintenance release of Jana2006 for Windows can be obtained in [Download area](#). It cumulates various improvements and bug fixes. [More ...](#)
- January 26, 2010 The Third Ad Hoc Workshop on Jana2006, 8-9 March 2010. [More ...](#)
- January 06, 2010 The Second Ad Hoc Workshop on Jana2006, 18-19 Jan 2010. [More ...](#)
- January 05, 2010 International school on aperiodic crystals. [More ...](#)
- October 21, 2009 Maintenance release of Jana2006 for Windows can be obtained in [Download area](#). It cumulates various improvements and bug fixes. [More ...](#)
- September 16, 2009 Ad hoc workshops on Jana. We offer informal two days workshops.

Ad hoc workshops on Jana [Next: 16]

[Download](#) | [Registration](#) | [Installation](#) | [External programs](#) | [Versions & roadmap](#) | [Recipes](#) | [Workshops](#) | [Citation & support](#) | [Contact Us](#)

Download

Shortcuts: [Jana2006 for Windows](#) | [Installation notes](#) | [Free Registration](#)

Jana system is a freeware but we are interested who is using it. We would like our users to **register**. Registered users get e-mails about major updates.

Jana2000: development of this program has been finished. Jana2000 input files can be used in Jana2006, the necessary conversions are done automatically.

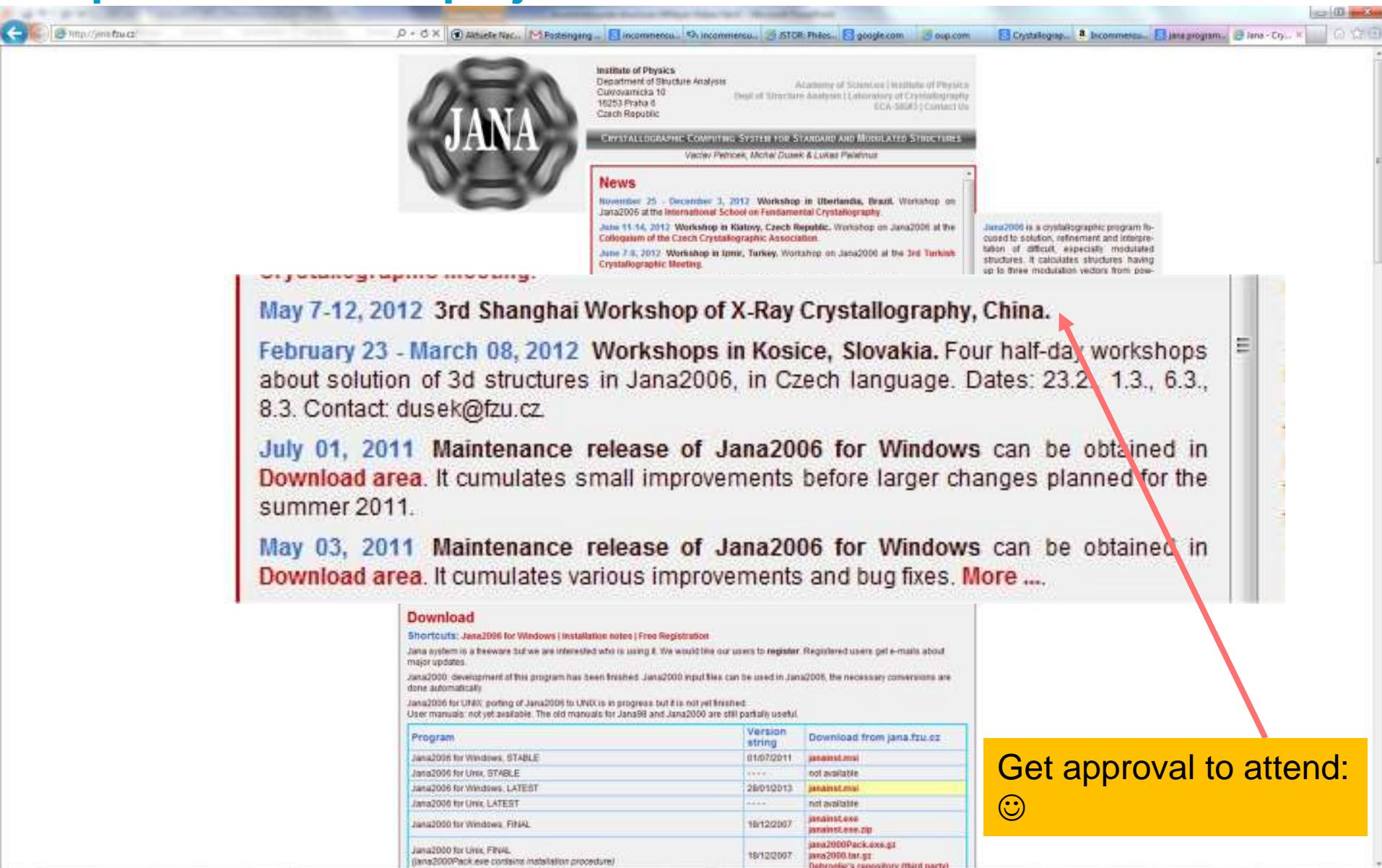
Jana2006 for UNIX: porting of Jana2006 to UNIX is in progress but it is not yet finished.
User manuals: not yet available. The old manuals for Jana98 and Jana2000 are still partially useful.

Program	Version string	Download from jana.fzu.cz
Jana2006 for Windows, STABLE	01/07/2011	janainst.msi
Jana2006 for Unix, STABLE	----	not available
Jana2006 for Windows, LATEST	28/01/2013	janainst.msi
Jana2006 for Unix, LATEST	----	not available
Jana2000 for Windows, FINAL	18/12/2007	janainst.exe janainst.exe.zip
Jana2000 for Unix, FINAL (Jana2000Pack.exe contains installation procedure)	18/12/2007	jana2000pack.exe.gz jana2000.tar.gz Debian's repository (third party)

More about Jana2006 (PowerPoint presentation, 10MB)



Export to Jana: <http://jana.fzu.cz/>



The screenshot shows the Jana2006 website. At the top, there's a logo and contact information for the Institute of Physics, Department of Structure Analysis, Curie Institute 10, 16253 Praha 8, Czech Republic. Below this, there's a 'News' section with several entries. A red arrow points from a yellow box at the bottom right to the 'May 03, 2011' news entry.

May 03, 2011 Maintenance release of Jana2006 for Windows can be obtained in **Download area**. It cumulates various improvements and bug fixes. **More ...**

Download

Shortcuts: [Jana2006 for Windows](#) | [Installation notes](#) | [Free Registration](#)

Jana system is a freeware but we are interested who is using it. We would like our users to **register**. Registered users get e-mails about major updates.

Jana2000: development of this program has been finished. Jana2000 input files can be used in Jana2006, the necessary conversions are done automatically.

Jana2006 for UNIX: porting of Jana2006 to UNIX is in progress but it is not yet finished.

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Program	Version string	Download from jana.fzu.cz
Jana2006 for Windows, STABLE	01/07/2011	janainst.msi
Jana2006 for Unix, STABLE	----	not available
Jana2006 for Windows, LATEST	28/01/2013	janainst.msi
Jana2006 for Unix, LATEST	----	not available
Jana2000 for Windows, FINAL	18/12/2007	janainst.exe janainst.exe.zip
Jana2000 for Unix, FINAL (Jana2000Pack.exe contains installation procedure)	18/12/2007	jana2000Pack.exe.gz jana2000.tar.gz Debian's repository (third party)

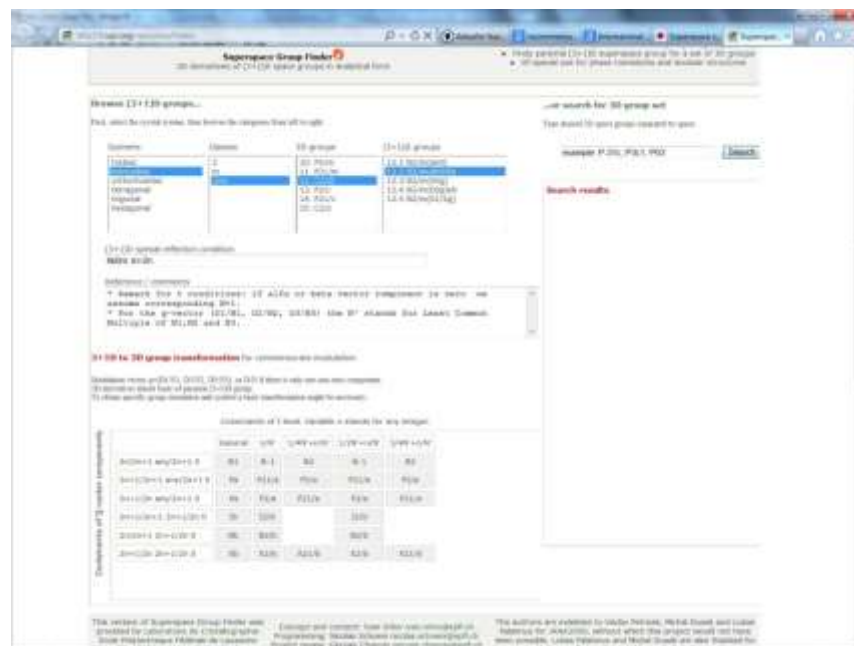
Get approval to attend:

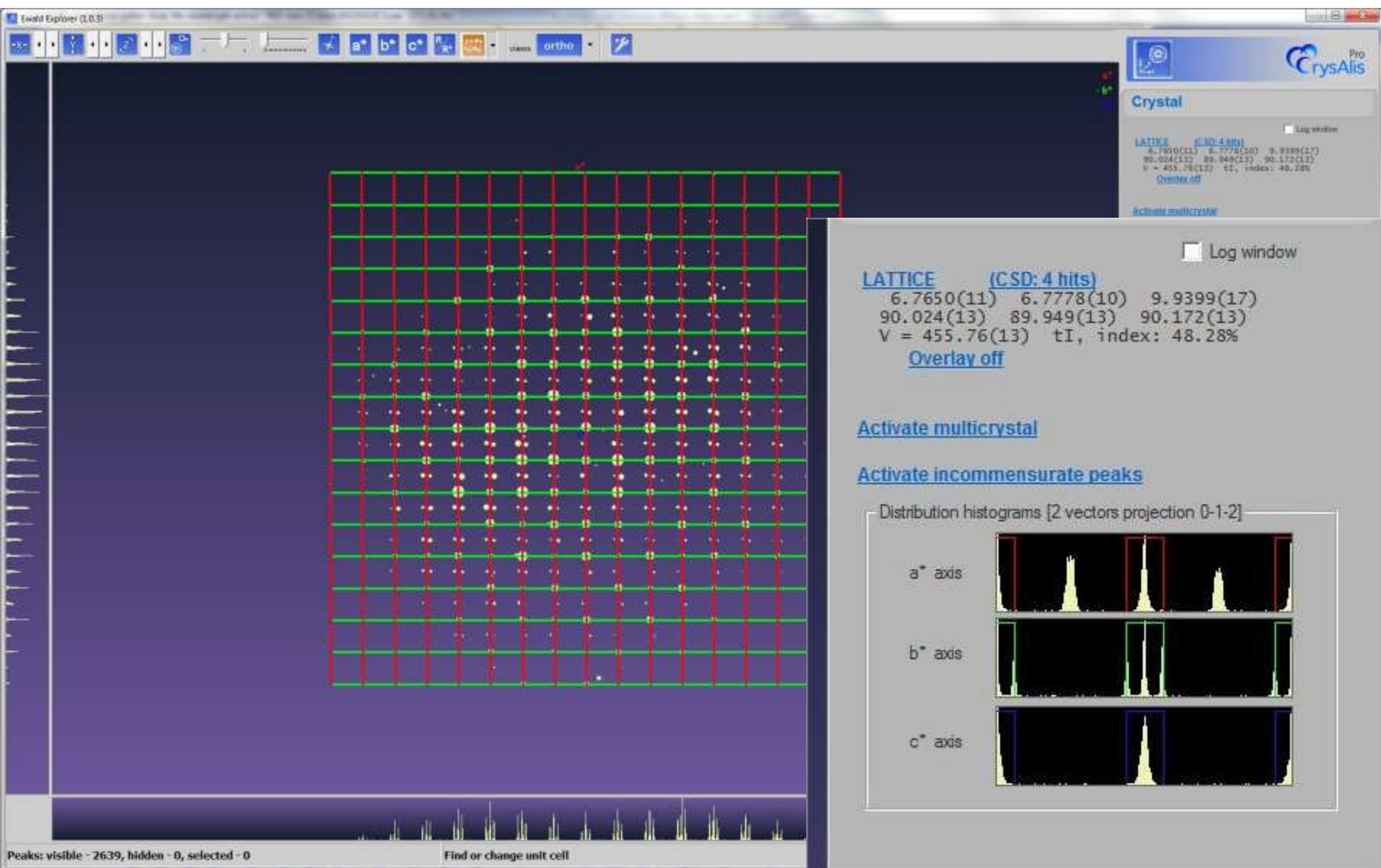


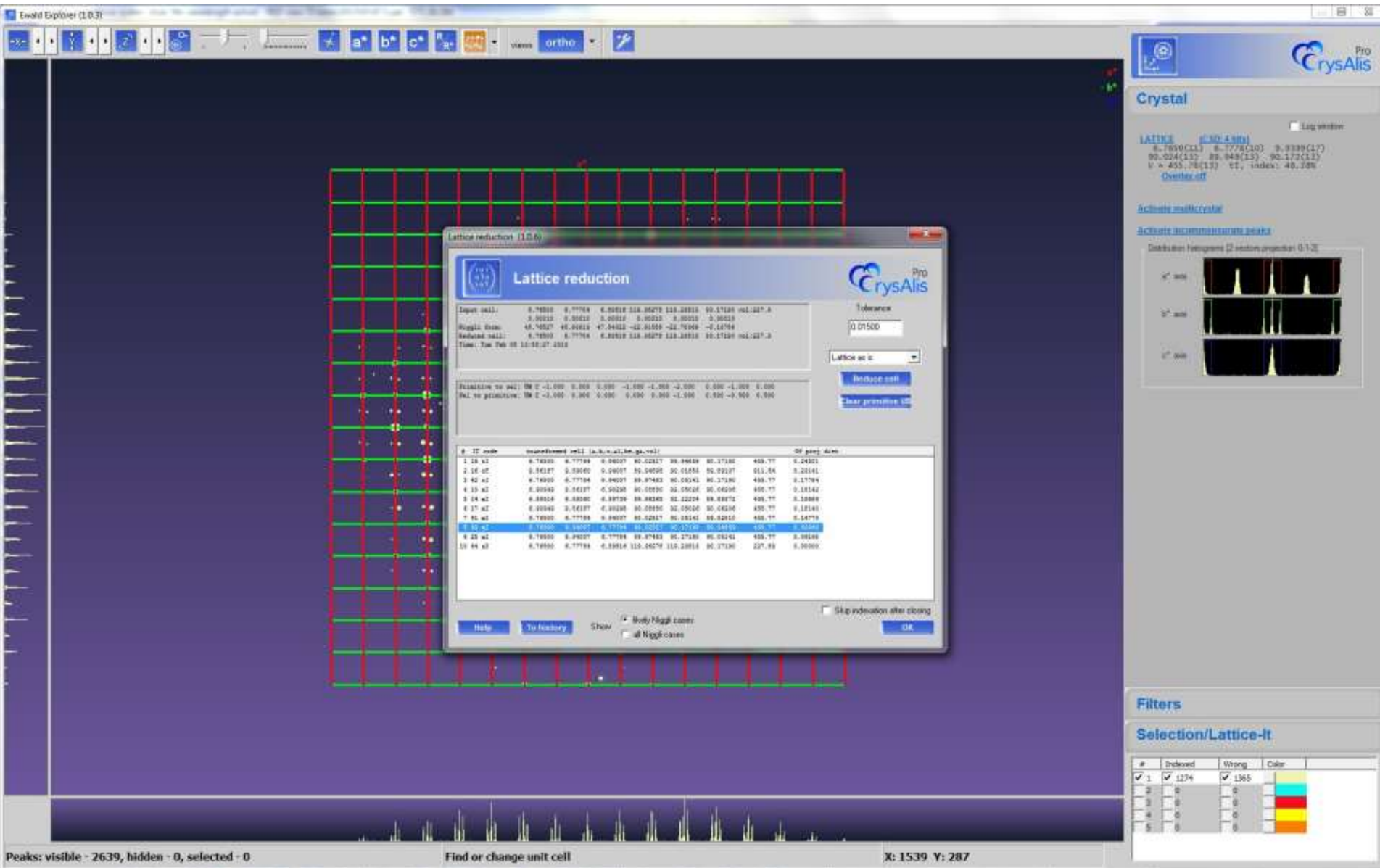
Agilent Technologies

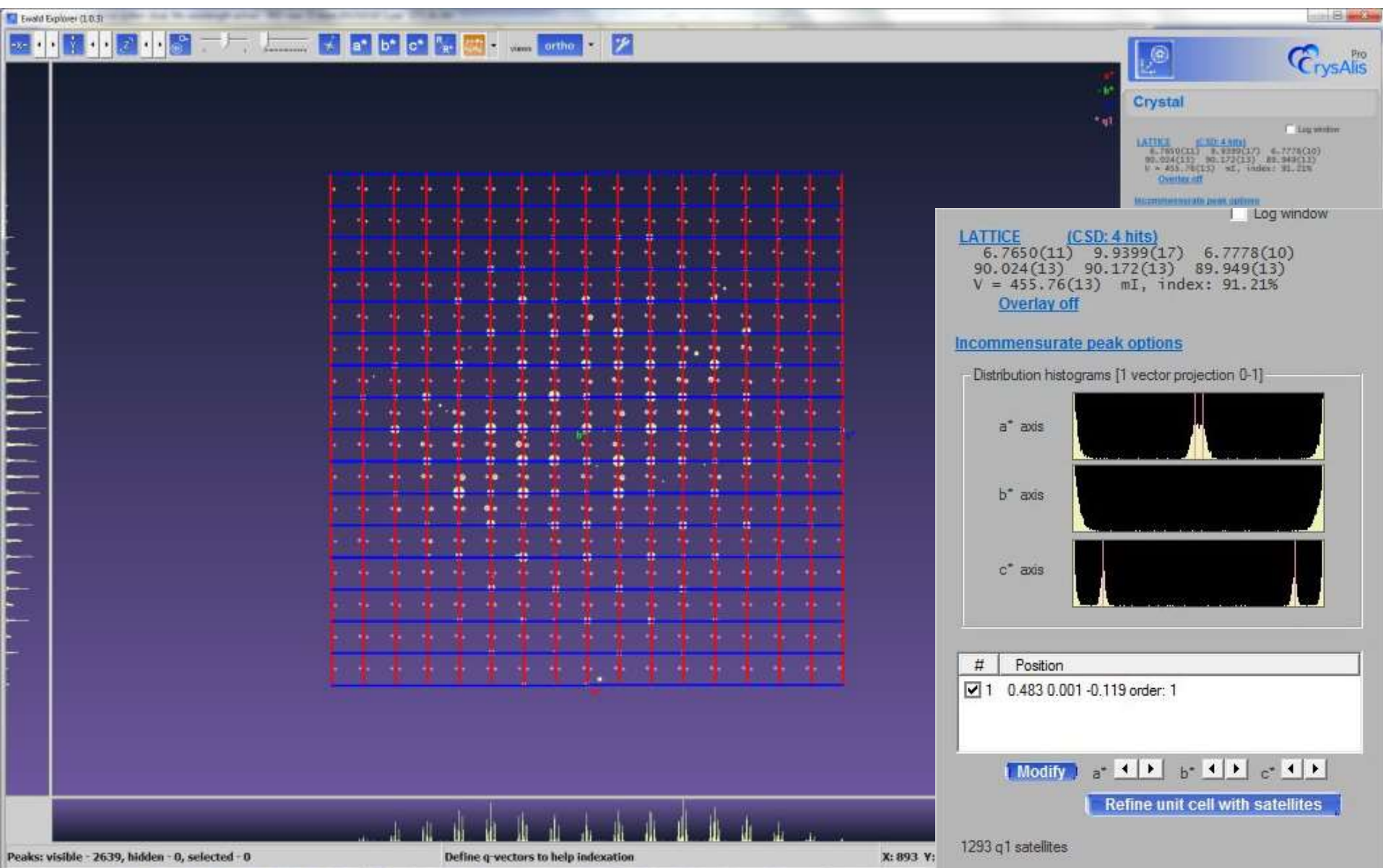
Recognition of incommensurate structures in CrysAlis

- Case IC/1
- Pseudo tetragonal sample. Satellite show the symmetry reduction.
- Satellites with two components dictates monoclinic (<http://superspace.epfl.ch/>; <http://it.iucr.org/resources/finder/>)



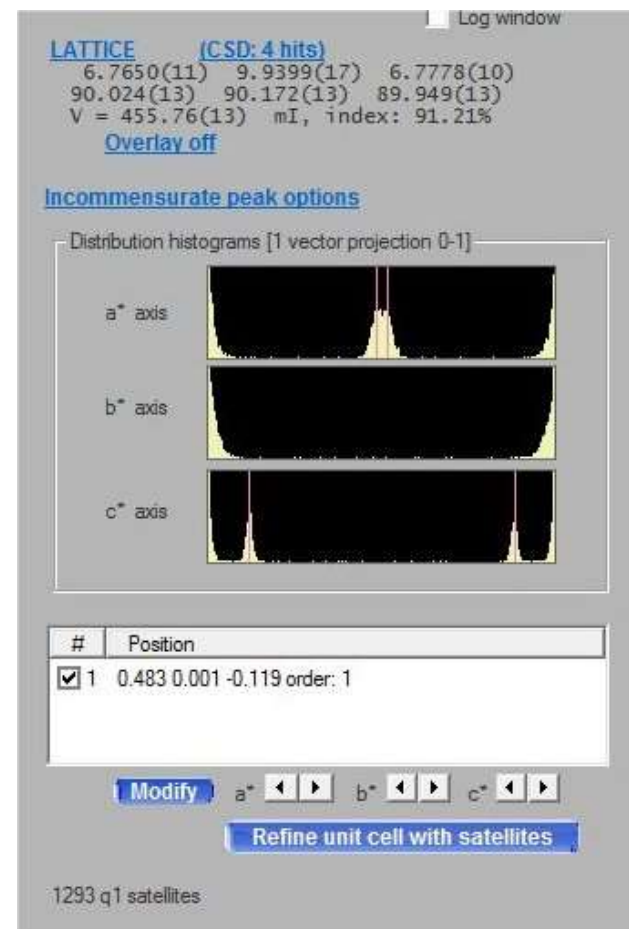




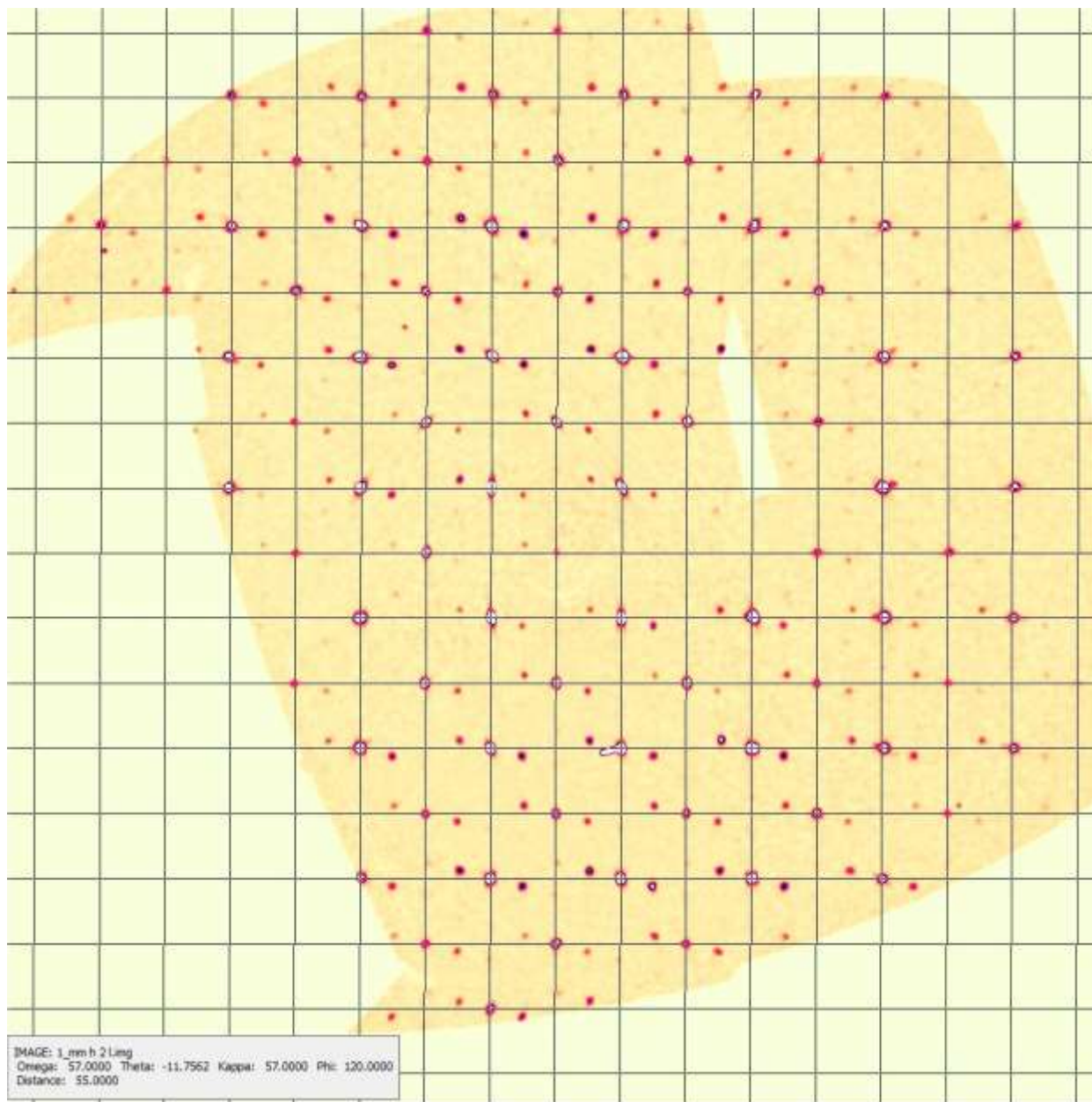


Watch out: Ewald are projections

- In pseudo-orthorhombic: 0.48 0 0.119 roughly the same as 0.48 0 -0.119
- Cross check predictions



IC:



IC: Strength of satellites

DC RED/RRP INFO: Incommensurate/quasi-crystal statistics

Main reflections

Statistics vs resolution - point group symmetry: P2/m (b-unique)

resolu- tion(A)	# measured	# kept	# unique	average redundancy	mean F2	mean F2/sig(F2)	Rint	Rsigma	RsigmaA
inf-0.80	1400	1358	494	2.7	115665.31	24.92	0.025	0.026	0.032

Satellite reflections

inf-0.80	5918	5866	2233	2.6	6711.75	5.10	0.069	0.077	0.136
----------	------	------	------	-----	---------	------	-------	-------	-------

1. order satellite reflections

inf-0.80	2968	2954	1121	2.6	12464.12	8.40	0.056	0.063	0.115
----------	------	------	------	-----	----------	------	-------	-------	-------

2. order satellite reflections

inf-0.80	2950	2912	1112	2.6	876.41	1.76	0.248	0.274	0.431
----------	------	------	------	-----	--------	------	-------	-------	-------

Powder diffraction with CrysAlisPro

- Agilent XRD instruments are single crystal diffractometers. They use almost parallel X-ray beams for sample illumination. The geometry is open beam, hence no Soller slits reduce background.
- The instrument is calibrated for the single crystal application. The calibration is only exact at the 'near' calibration point. Experiments at distances other than the near point rely on the model interpolation in conjunction with 'refine model'. This means that the instrument model might not be as accurate as required for powder diffraction, but reasonable good for single crystal application.
- Powder diffraction, like single crystal diffraction is a volume effect. For good results a reasonable amount of sample has to be brought into the beam preferably with little dull diffraction material like capillary, glass stick, excessive oil/grease etc. We recommend loop or Mitigen mounts as they have low background diffraction.



Sample mounting

- There are many successful mounting techniques. For good results a reasonable amount of sample has to be brought into the beam preferably with little dull diffraction material like capillary, glass stick, excessive oil/grease etc. We recommend loop or Mitigen mounts as they have low background diffraction.
- Make sure to have a good ground powder, as for a powder diffractometer experiment.
- Try to increase the amount of material to a similar volume as single crystal. Powder diffraction is weak. The more sample volume the better. The more sample volume to dull volume the better.



Wavelength

- While the Gemini let's you the choice of Mo or Cu, powder diffraction will generally be done with Cu for it's diffraction power. Exceptions are heavy absorbers or HP setups.



Resolution, distance, binning

- The resolution of a powder diffraction experiment depends on several factors: scintillator, distance, source, binning, sample divergence.
- The resolution is limited due to the scintillator thickness (40-80microns). The scintillator resolution is $2 \times \text{scintillator thickness}$. Eos has a front pixel size of: $1 \times 1 = 33\text{microns}$; $2 \times 2 = 66\text{microns}$; $4 \times 4 = 132\text{microns}$. Atlas has a front pixel size of: $1 \times 1 = 50\text{microns}$; $2 \times 2 = 100\text{microns}$; $4 \times 4 = 200\text{microns}$. So 2×2 and 4×4 binning are reasonable with respect to resolution. Your camera's 1×1 pixel size you find on the 'instrument model I' tab.
- The source divergence is of the order of ($5\text{-}10\text{mRad} = 0.3\text{-}0.6\text{deg}$). For a divergent beam doubling the distance will also double the size. So generally the close distance is fine. The long distance may only lower the background ($1/r^2$).



Instrument calibration

- As was said: The instrument is calibrated for the single crystal application. The calibration is only exact at the 'near' calibration point. Experiments at distances other than the near point rely on the model interpolation in conjunction with 'refine model'. This means that the instrument model might not be as accurate as required for powder diffraction, but reasonable good for single crystal application.
- How to find the near calibration point? On the tab 'Distance calibration' there is a section 'Close distance calibration'. For this distance the powder extraction is the most accurate. We will treat the case of an arbitrary distance later. It also requires that the calibration is valid for the current setup.
- The instrument calibration is only required if the machine setup is uncertain, or someone changed X-ray optics settings.



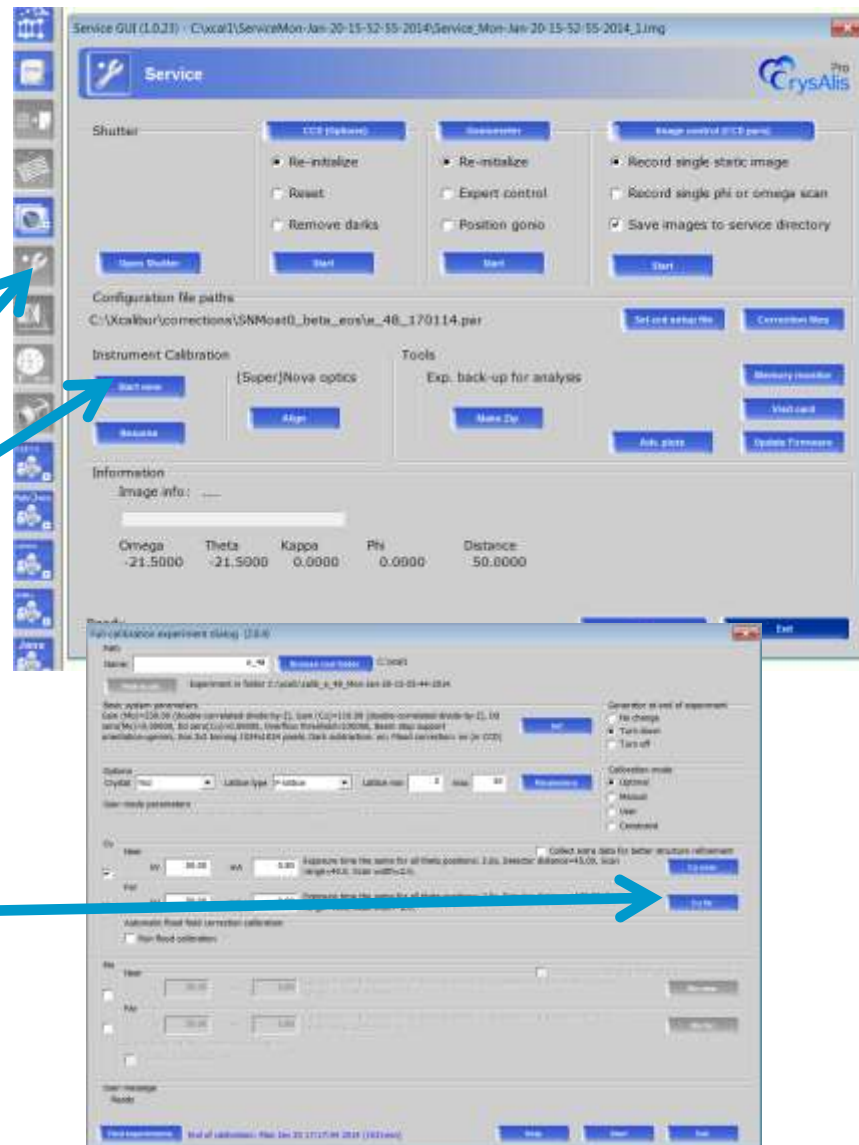
Instrument calibration 2

- As said on the previous slide. The 'near point distance' is fine for powder diffraction experiments. It is recommended to use this setup for powder diffraction. Then no calibration is required.
- There is no special calibration for powder experiments. The instrument is calibrated with the single crystal test sample.
- For powder experiments at arbitrary distances refer to later slides. Here a special procedure might be required.



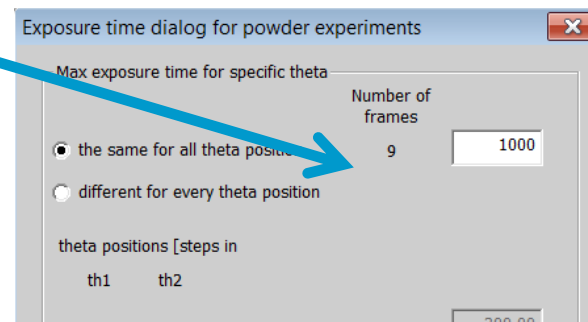
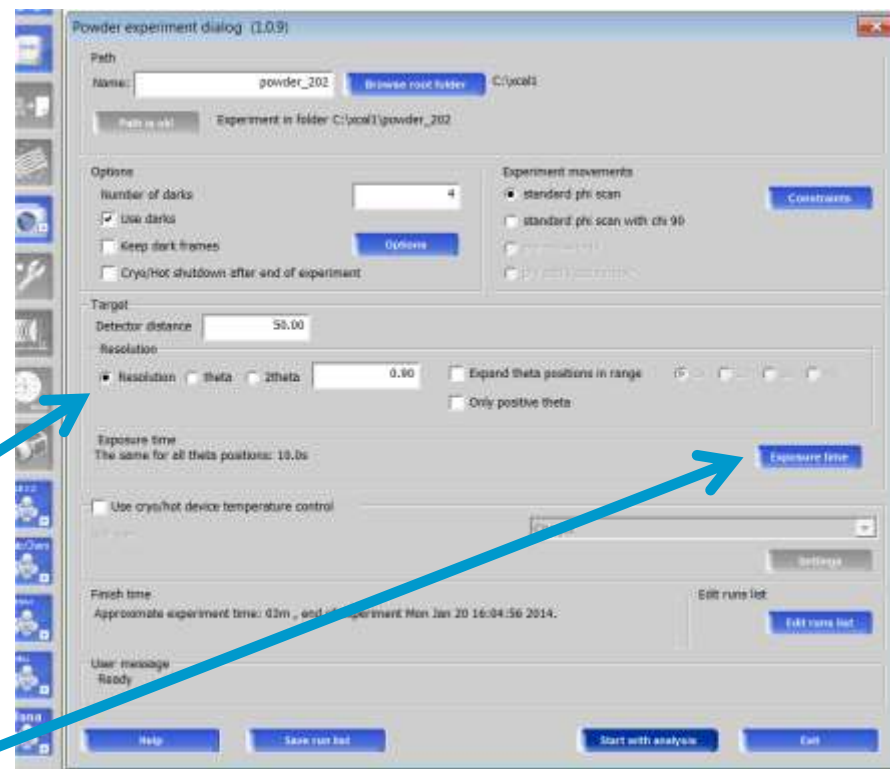
Do an instrument calibration (only if required!!!)

- The instrument calibration can be easily archived:
- Mount the standard sample ylid. Make sure the sample very well centered and checked in 8 positions.
- Open the service wrench power tool.
- There click on 'Instrument calibration' -> start new.
- Select the required wavelength for calibration (here Cu) and click start. The default settings generally are fine. If you have a weak source or small ylid you might want to increase the default exposure by clicking on 'Cu near'/'Cu far' and adjusting the exposure time there...



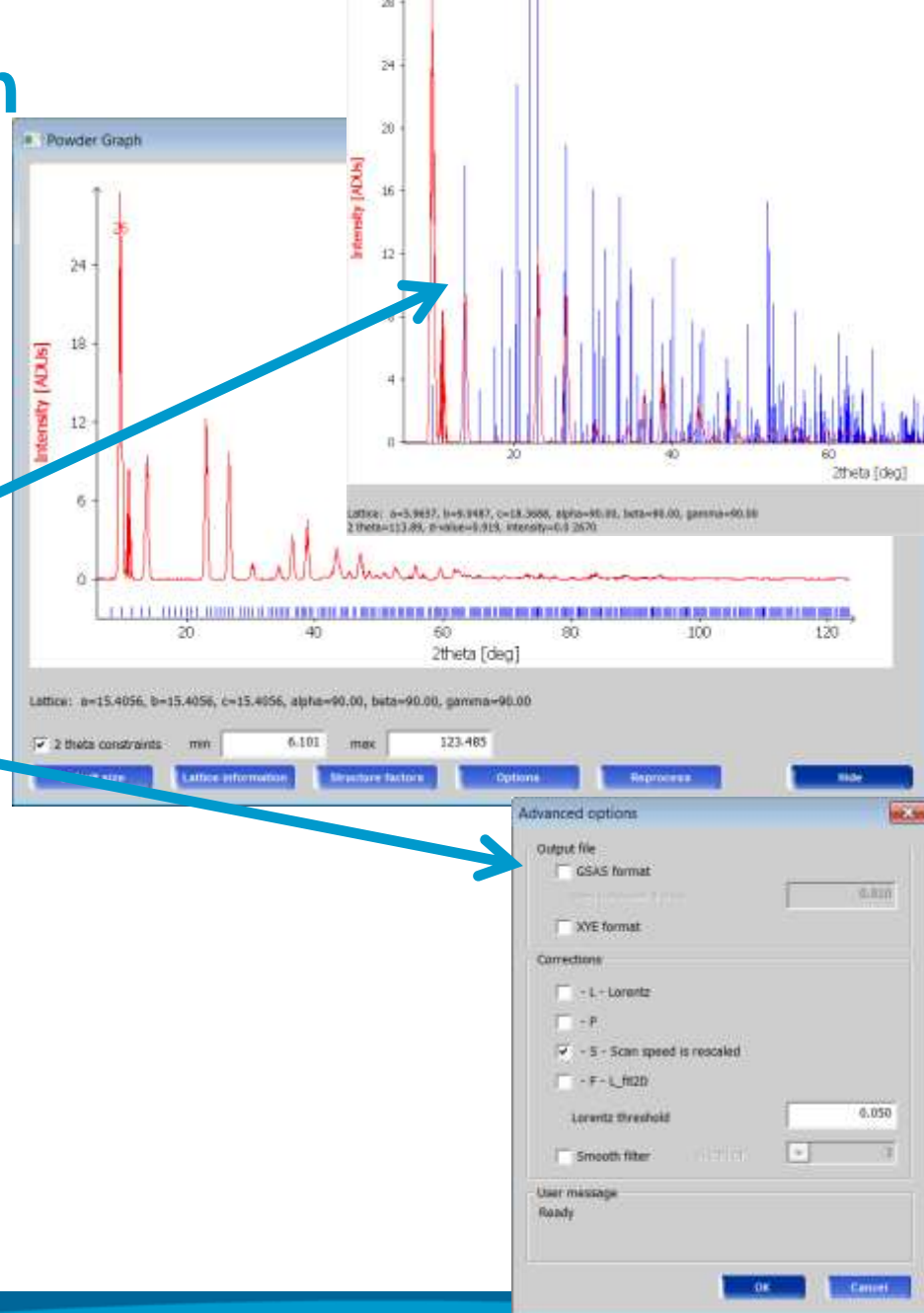
Powder experiments

- After a successful calibration you can use the instrument for powder experiments at the near calibration point.
- Mount your sample with F12 and center it well
- Click on the Powder power tool to plan the experiment.
- Put the distance roughly to the near point (55.0234 can be 55mm). Select the target resolution. Expand theta will put extra settings to the experiment.
- Set the exposure time: Note that powders may require very long times. The instrument will chop very long exposures to several images and add them
- Click on 'Start with analysis' to do the experiment



Powder pattern extraction

- During the experiment the pattern will be automatically extracted.
- 'Lattice information' allows to put lattice line marker on the plot. If you have a structure res file you can even put 'Structure factors'
- 'Options' changes the extraction options. After changing the option you might need to click on reprocess. (Look at the history window output for details...)
- You can zoom the pattern



Example of history window output

```
POWDER DATA ANALYSIS INFO: Started at Mon Jan 20 16:12:59 2014
POWDER CORRECTIONS: Corrections flags: S
POWDER CORRECTIONS: lorentz threshold: 0.050
POWDER CORRECTIONS: smooth filter not applied!
                    L-correction is not applied
                    P-correction is not applied
                    Scan speed is rescaled to 20.00 s
                    F-L_fit2D is not applied

POWDER INFO: Resetting 2896 append bins with thetamin= 0.00050 thetamax= 61.74229
POWDER RADIAL INFO: 1523 of 2896 valid bins with 2theta: 53.44657 to 118.36608
Powder pattern copied into file (C:\xcall\powder_203\powder_203_powder)!
POWDER INFO: image theta -86.000, Exposure time=1.00
POWDER INFO: Statistics: valid min pixel=-35, valid max pixel=10889, theta min=26.72, theta max=59.18
POWDER INFO: valid pixels=997424, valid bins=1523, masked pixels=51152, skipped pixels=0, bad pixels=0
POWDER INFO: Append data to 2896 append bins with thetamin= 0.00050 thetamax= 61.74229
POWDER RADIAL INFO: 2624 of 2896 valid bins with 2theta: 6.48442 to 118.36608
Powder pattern copied into file (C:\xcall\powder_203\powder_203_powder)!
POWDER INFO: image theta -31.529, Exposure time=1.00
POWDER INFO: Statistics: valid min pixel=-35, valid max pixel=377431, theta min=3.24, theta max=59.18
POWDER INFO: valid pixels=893150, valid bins=2624, masked pixels=155426, skipped pixels=0, bad pixels=0
POWDER INFO: Append data to 2896 append bins with thetamin= 0.00050 thetamax= 61.74229
POWDER RADIAL INFO: 2633 of 2896 valid bins with 2theta: 6.10053 to 118.36608
Powder pattern copied into file (C:\xcall\powder_203\powder_203_powder)!
POWDER INFO: image theta 31.529, Exposure time=1.00
POWDER INFO: Statistics: valid min pixel=-36, valid max pixel=258718, theta min=3.05, theta max=59.18
POWDER INFO: valid pixels=976192, valid bins=2633, masked pixels=72384, skipped pixels=0, bad pixels=0
POWDER INFO: Append data to 2896 append bins with thetamin= 0.00050 thetamax= 61.74229
POWDER RADIAL INFO: 2753 of 2896 valid bins with 2theta: 6.10053 to 123.48457
Powder pattern copied into file (C:\xcall\powder_203\powder_203_powder)!
POWDER INFO: image theta 90.933, Exposure time=1.00
POWDER INFO: Statistics: valid min pixel=-36, valid max pixel=11279, theta min=3.05, theta max=61.74
POWDER INFO: valid pixels=997314, valid bins=2753, masked pixels=51223, skipped pixels=39, bad pixels=0
POWDER DATA ANALYSIS INFO: Finished at Mon Jan 20 16:13:08 2014
Warning: F2 of ( 0,-2,-2) was modified from 1403567.3 to 999999.0
Warning: F2 of (-1,-1,-3) was modified from 1040141.6 to 999999.0
Warning: F2 of (-1,-1, 3) was modified from 1038842.9 to 999999.0
Reading 1565 observations from C:\xcall\calib_e_48_Mon-Jan-13-19-00-06-2014\Mo\Near45mm\Mo_Near45mm.hkl
```

- Note that the extracted pattern will land in a file.



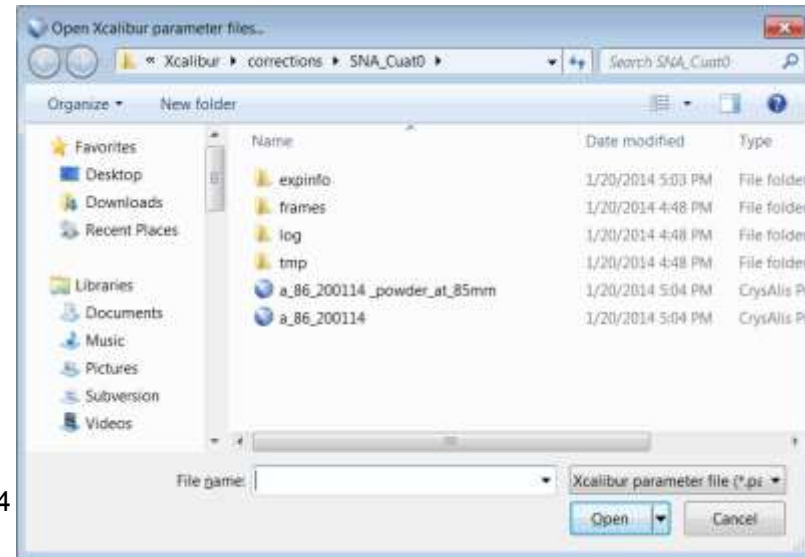
Powder experiments at arbitrary distances (PEAAD)

- The previous slides showed how to exploit the 'near calibration point' for the occasional powder experiment.
- 'Real professionals' might want to choose their experiment distance.
- The following slide will show the procedure on making a custom (x)par file for powder experiments at arbitrary distance.
- As an example we will use 85mm on Cu wavelength on an Atlas SN



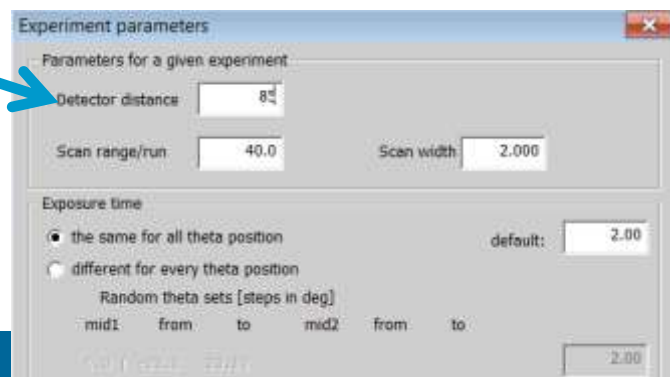
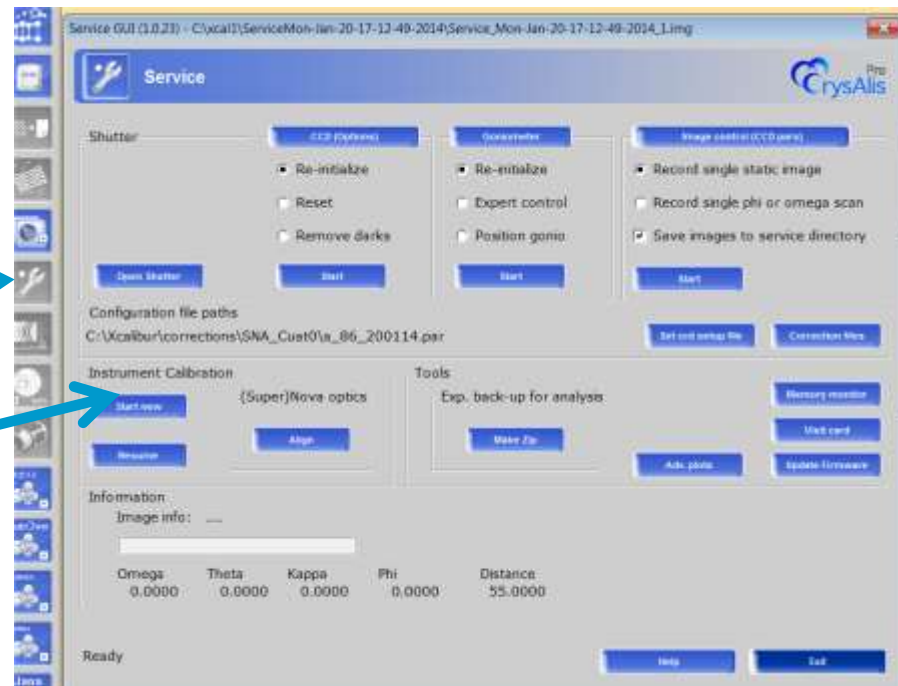
PEAAD 1

- Make a copy of your current par file: Mine is 'a_86_200114.par'. The powder one I will call 'a_86_200114_powder_at_85mm.par'.
- The new par is selected as the new ccd configuration file (under 'Configuration file paths' -> 'Set ccd setup file').
- The software prompts to restart the software, which I accept.
- I may be prompted to select to proper binning. This can be avoided by also making a copy of 'a_86_200114_1_1.img' in the frames folder to 'a_86_200114_powder_at_85mm_1_1.img' and in the root folder 'a_86_200114.run' to 'a_86_200114_powder_at_85mm.run'
- I will now proceed the with ylid calibration at a single distance: here 85mm.



PEAAD 2 – single distance calibration

- Mount the standard sample ylid. Make sure the sample very well centered and checked in 8 positions.
- Open the service wrench power tool.
- There click on 'Instrument calibration' -> start new.
- I select the required wavelength for calibration (here Cu). Only the 'Near' model, edit the 'Cu near' the distance to 85 mm, close all sub dialogs and click on 'Find experiments' and the 'Start'



PEAAD 3

- After the successful calibration I can proceed with powder experiments at that distance
- The 'a_86_200114_powder_at_85mm.par' calibration is now explicitly for 85mm. **Do not** use this setup for single crystal work!
- To go back to the standard single crystal calibration: Open the service power tool (wrench) and select under 'Configuration file paths' -> 'Set ccd setup file' the file 'a_86_200114.par' and proceed with system restart.

Powder reference samples

- There are a number of NIST powder standard samples available. E.g. Si, CsI, LaB_6 and others.
- A powder standard can also be used for instrument model calibration. But it is not an automated procedure. The procedure is explained in 'ITS 29 - Comments on powder model refinement'. This procedure is difficult, but possible. The single crystal method is strictly preferred.



Powder: common errors

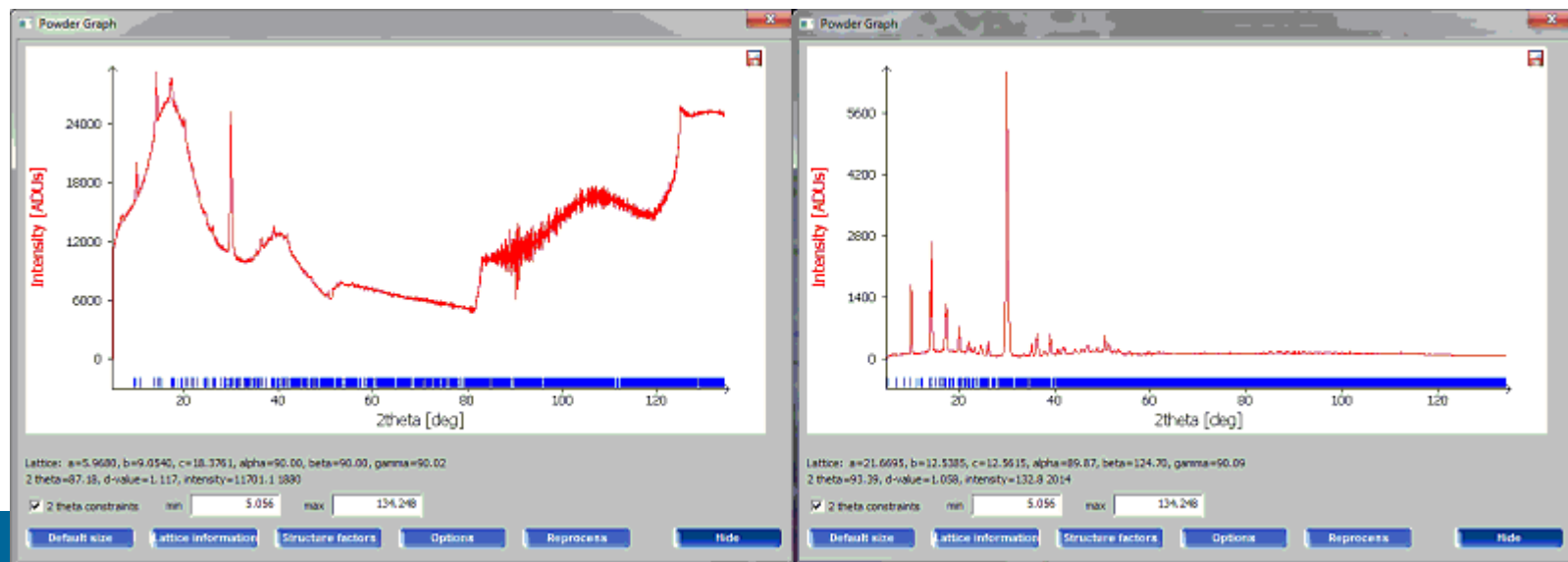
- As is clear from the previous slides, the precise powder extraction relies on the instrument calibration at the used distance. As the detector drive does not maintain the center as it drives back using the instrument model away from the near point is the most frequent user error.
- Also an invalid instrument calibration may cause extraction errors. Here the user has 'changed' the system willing or unwillingly (e.g. SN optics move by hand/arm collision with the user). A quick ylid calibration will repair this flaw.
- The instrument calibration at the near point is head on. The use of the valid distance calibration and using the powder extraction at the far point might incur a scale error of up to 2.0 promille (will depend on orientation of the detector drive). For fingerprinting this might be still fine.



xx gandolfi:

powder patterns from single crystal experiments

- Gandolfi method images a single crystal on film as powder diagram
- You can do the same using a single crystal data set and doing a powder pattern extraction.
- The difference to a normal powder extraction is: background subtraction before image accumulation. The results are striking
- Now you can deposit new mineral structures with experimental powder pattern extracted from your single crystal data! A super sensitive Gandolfi camera.
- See also issue 81: **ITS 81 XX GANDOLFI - Background computations for powder analysis**



Powder: technical

- To accumulate a powder pattern we generate a theta-bin vector with N, where N is automatically selected between 512 and 32000 based on θ_{\min} and θ_{\max} (only the GSAS extraction has a fixed pitch theta step).
- Concurrent pattern extraction runs frame by frame. Offline processing accumulates frames of the same detector setting. In case of 'Scan time rescaling' the exposure times are accumulated and normalized to 20s. In the 'Gandolfi case' exposure time is normalized by the 'background computation sequence'.
- Each pixel has a defined θ value and will accumulate it in the closest bin $X_{\text{raw},i}$ as X-ray counts (ADUs/system gain). In parallel we accumulate the number of hits C_i and the corrected counts $X_{\text{corr},i}$ (optional L, P and scan time rescale).
- The final pattern is outputted in the following way: The $\sigma(X_{\text{raw},i}) = \sqrt{X_{\text{raw},i}}$ and $\sigma(X_{\text{corr},i}) = \sigma(X_{\text{raw},i}) X_{\text{corr},i} / X_{\text{raw},i}$. We output as $I_i \rightarrow X_{\text{corr},i} / C_i$ and $\sigma(I_i) \rightarrow \sigma(X_{\text{corr},i}) / C_i$. For proper plotting an additional rescale is applied on the $\max(I_i; (i \text{ 1 to } N))$ is <10 .



Thank you for listening!

Find out more at

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