Méthodes de intégration: CrysAlis^{Pro}

Reciproque workshop - Ambleteuse

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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

↓

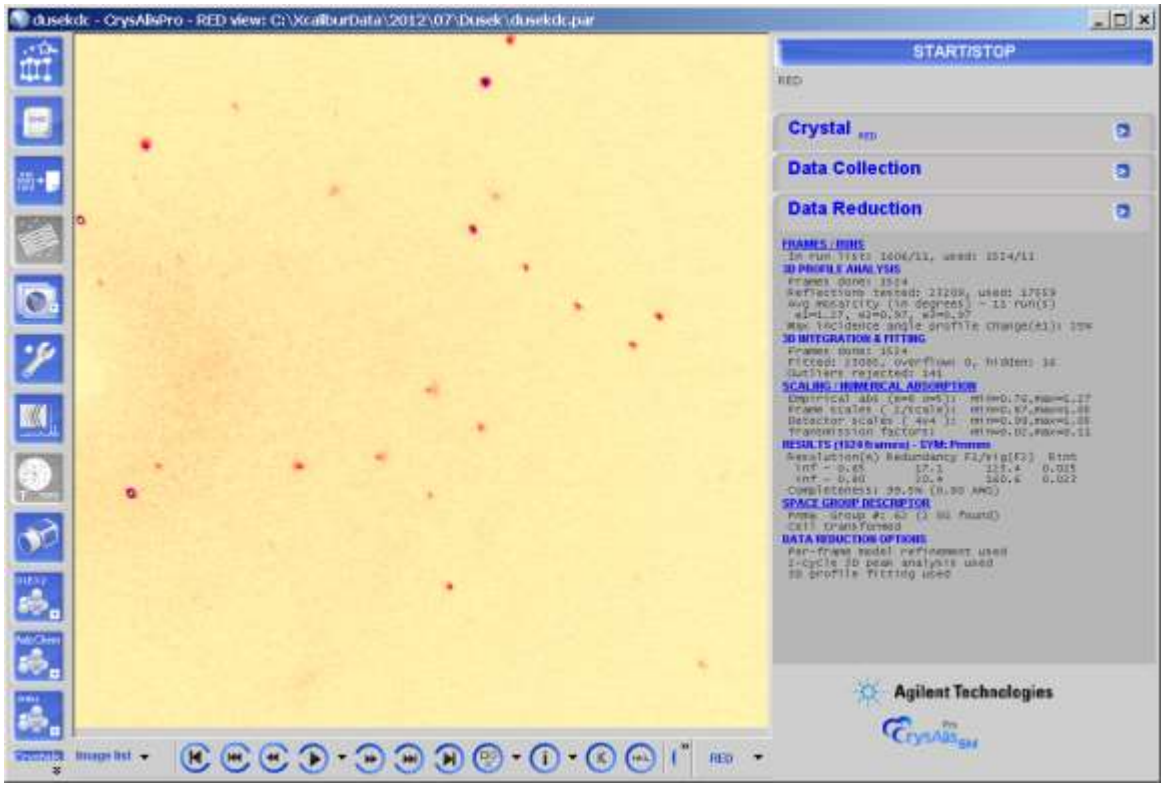
Profile learning

↓

Integration

↓

Post correction

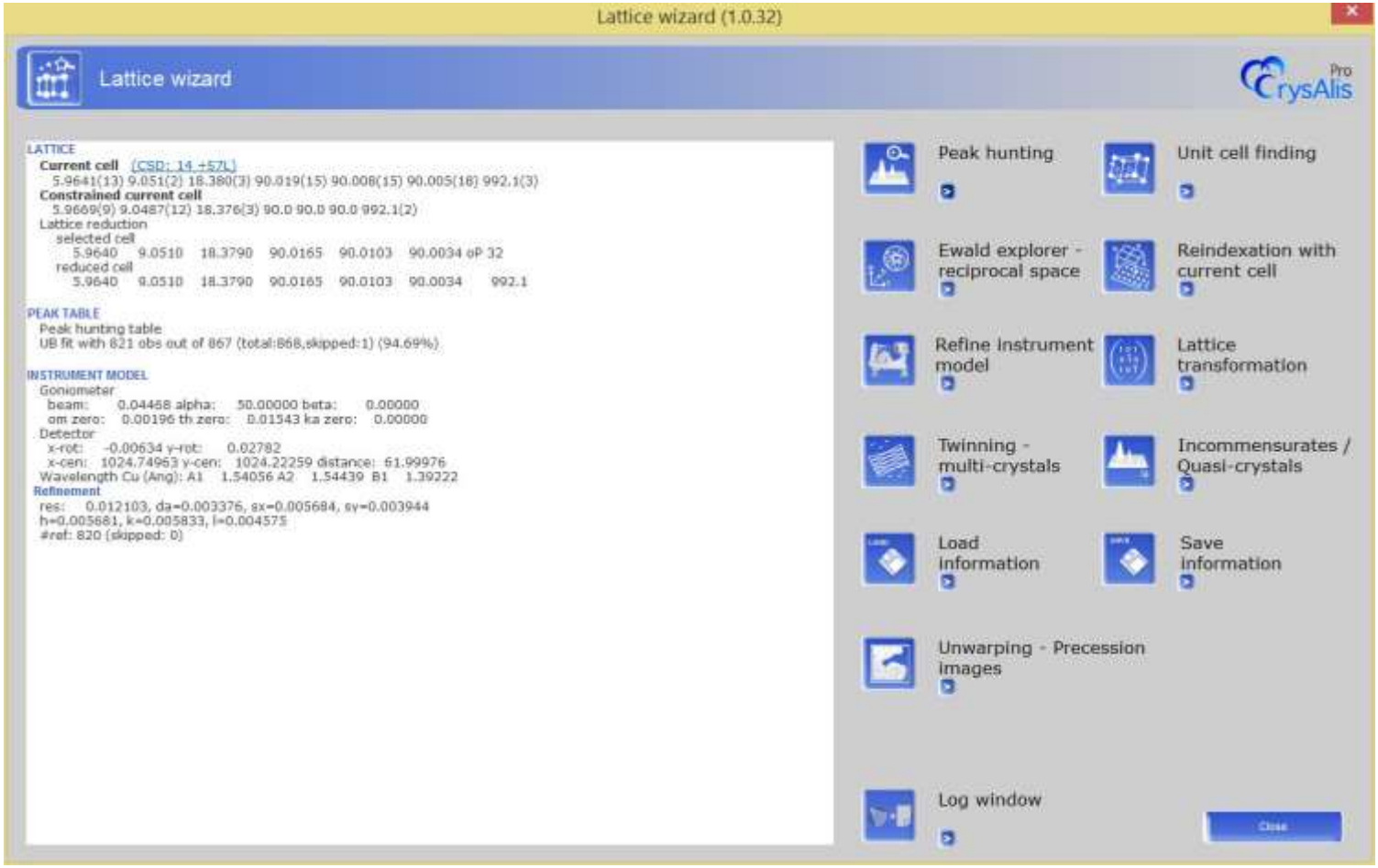


Unit cell finding

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

Unit cell finding

- Lattice wizard power tool



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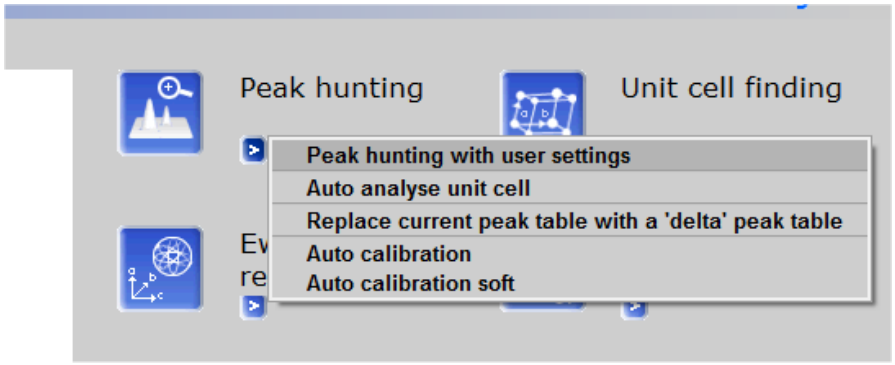
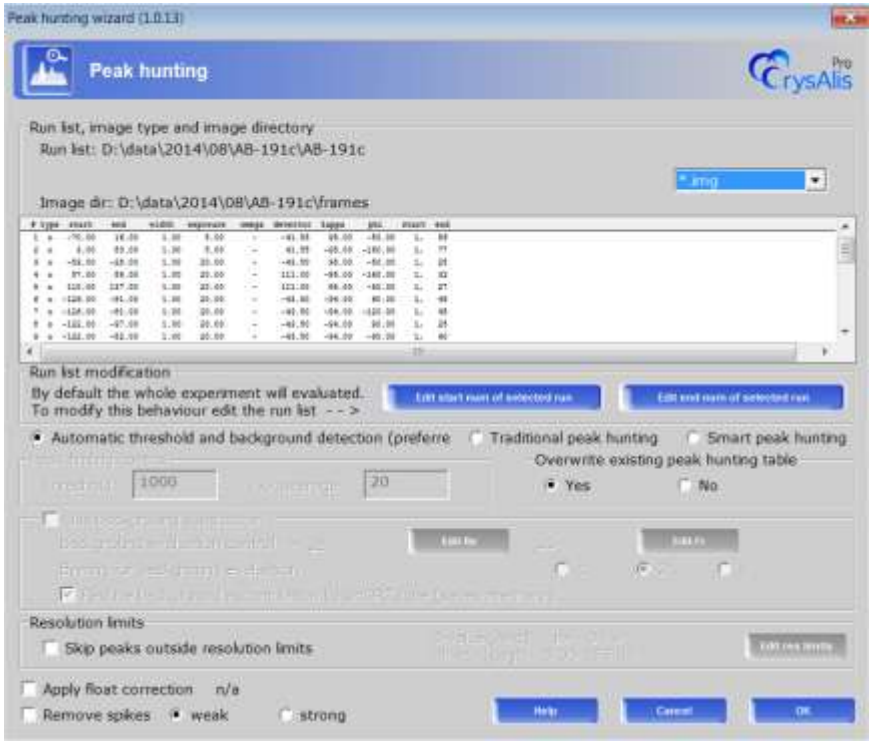
Unit cell finding – Peak hunting

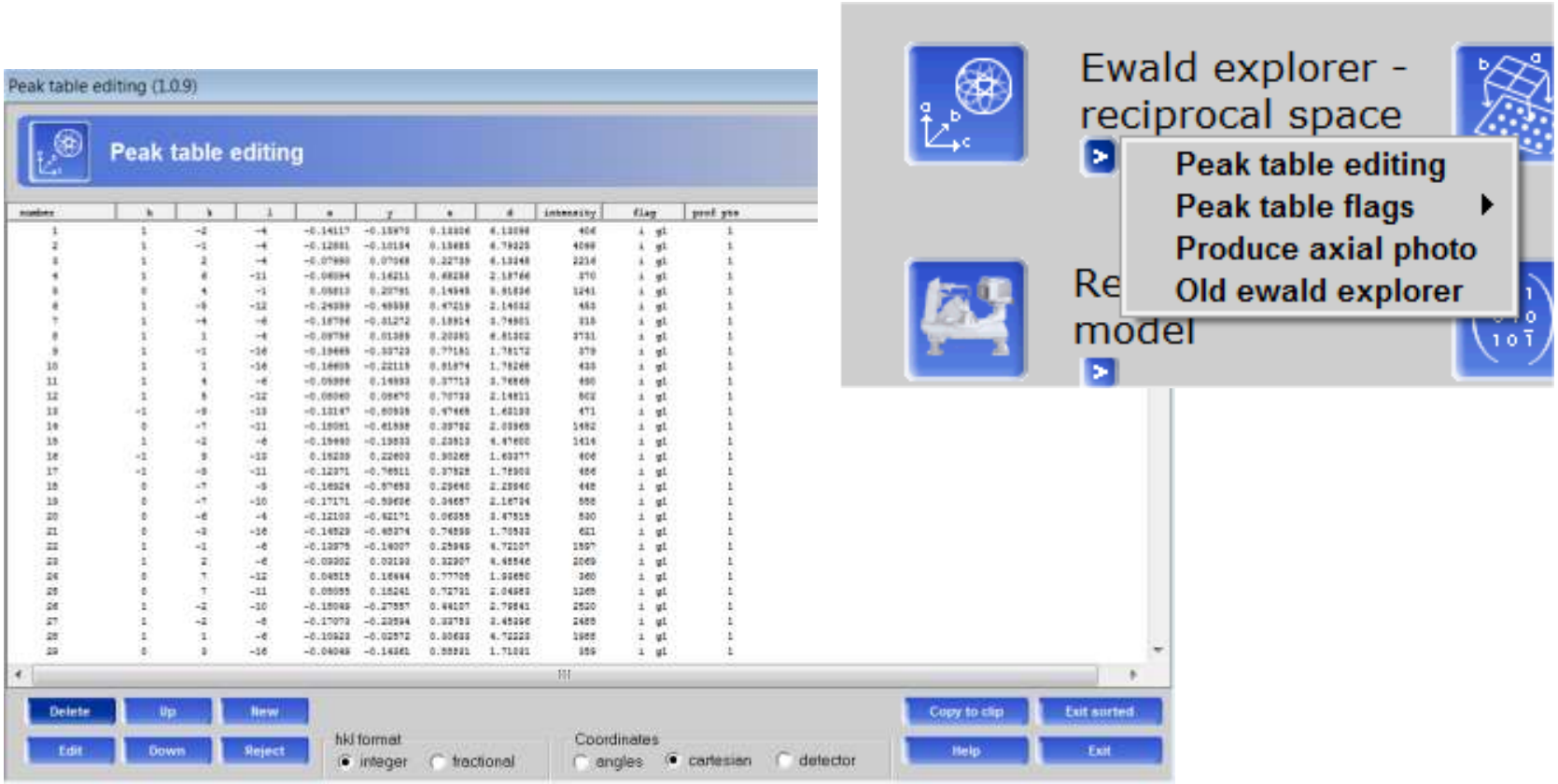
- Defaults work well for ‘normal’ crystals and twins
- Super-structures may require tweak



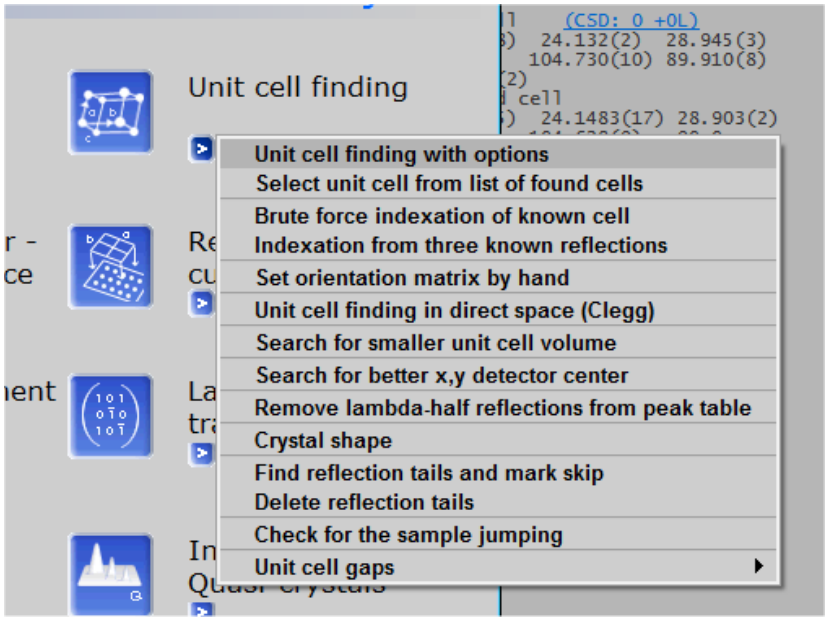


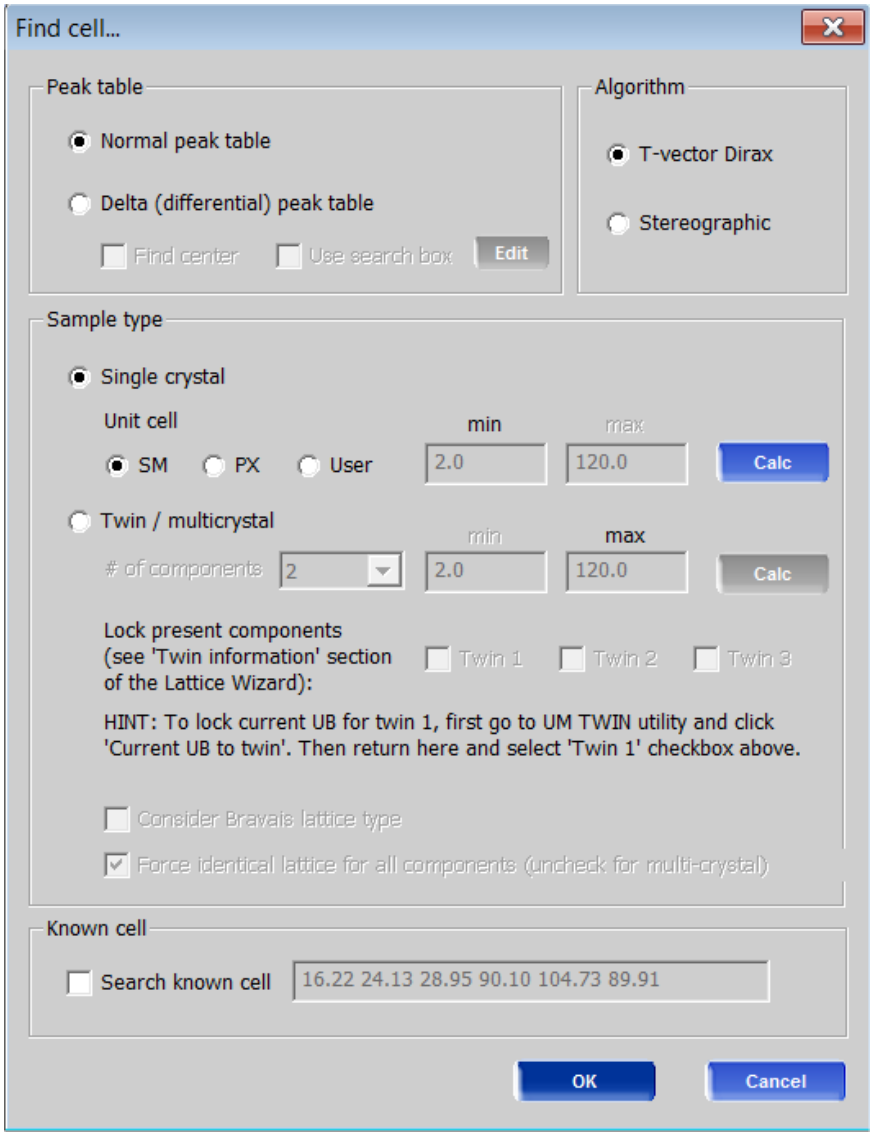
Unit cell finding – Peak hunting

- Data filtering via peak table

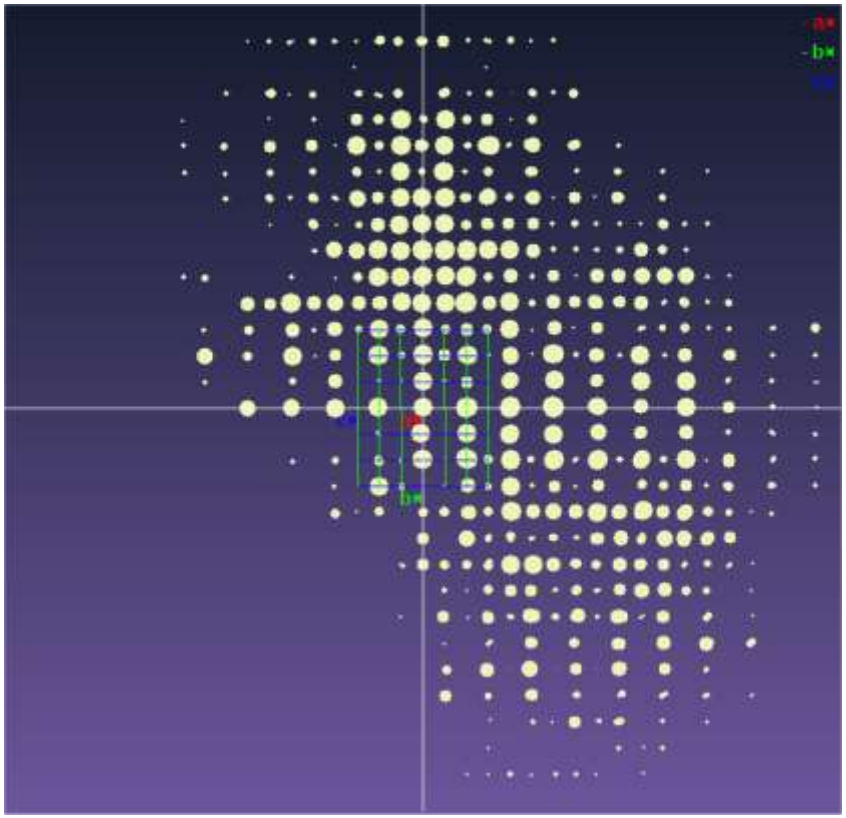


Unit cell finding – Automatic unit cell finding

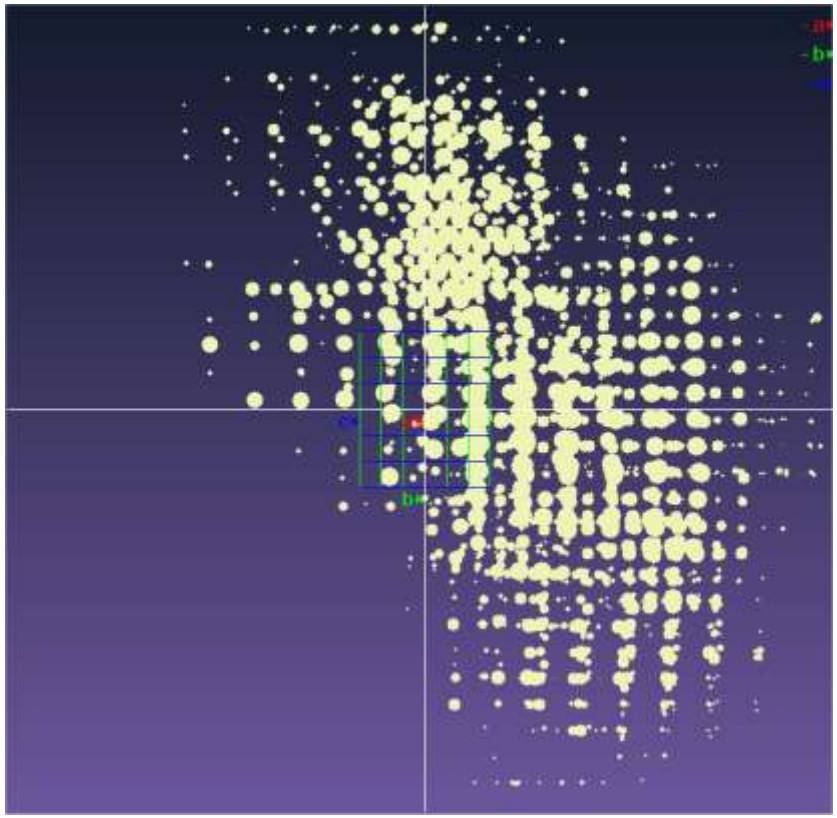




Unit cell finding – Influence of bad instrument model



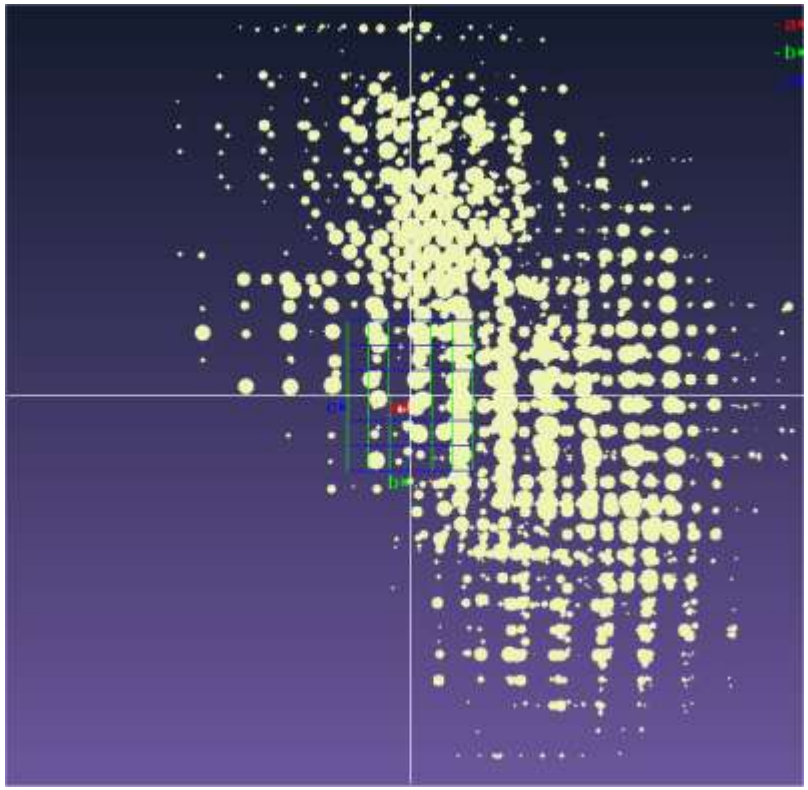
Good instrument model



Bad instrument model:

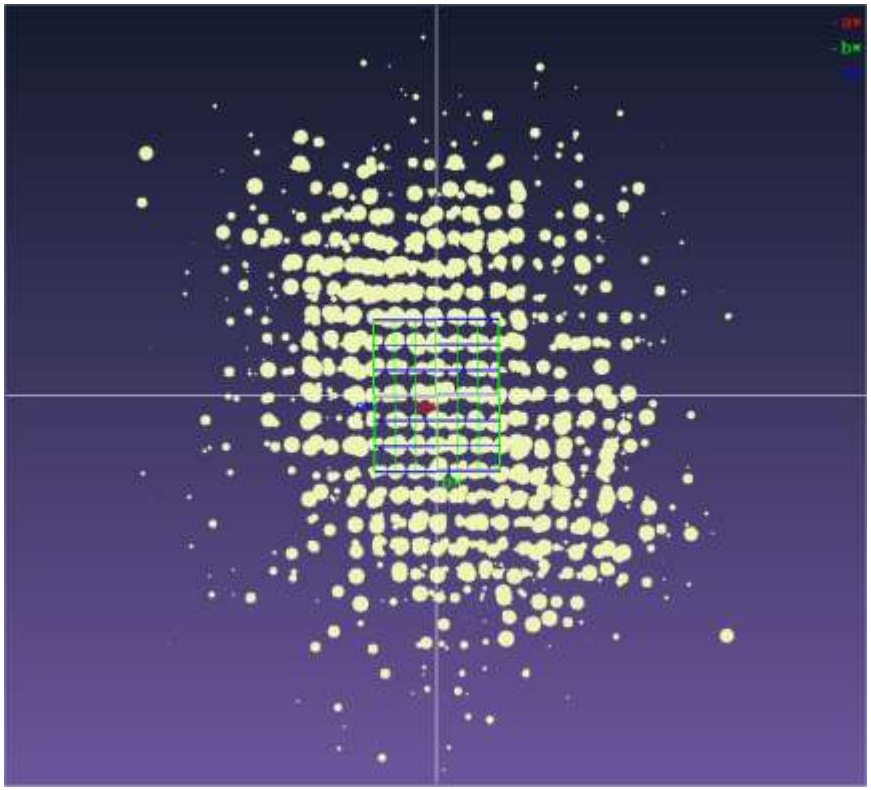
Beam center deviation 40 pix

Unit cell finding – Automatic unit cell finding



Bad instrument model:

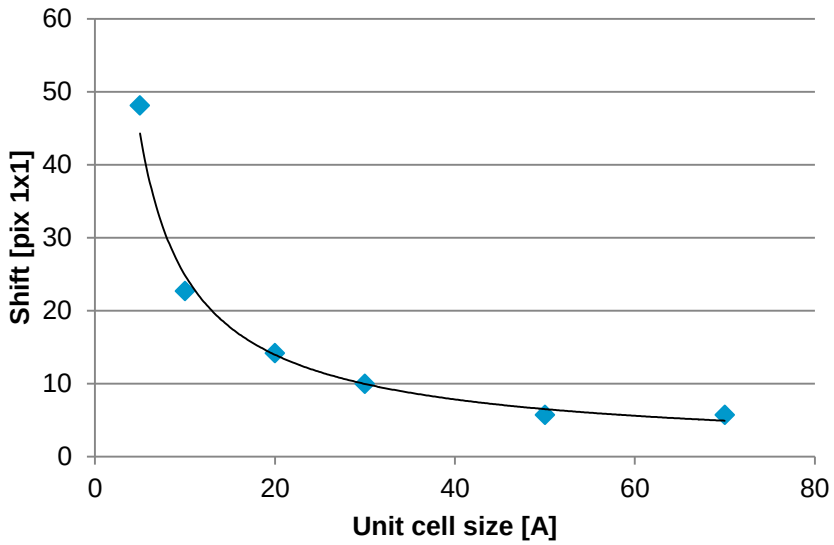
Beam center deviation 40 pix

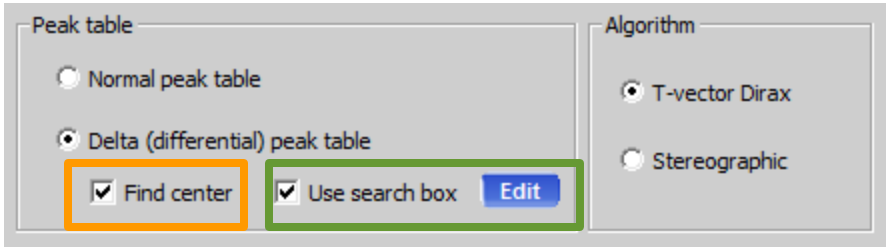


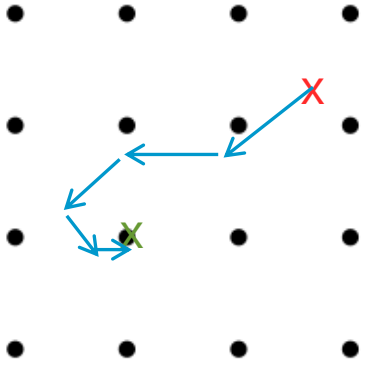
Bad instrument model:

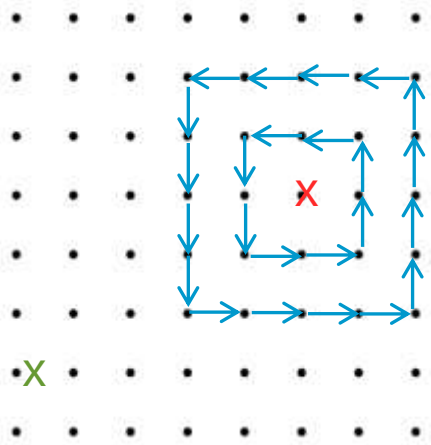
Delta-peak table

Unit cell finding – Automatic unit cell finding





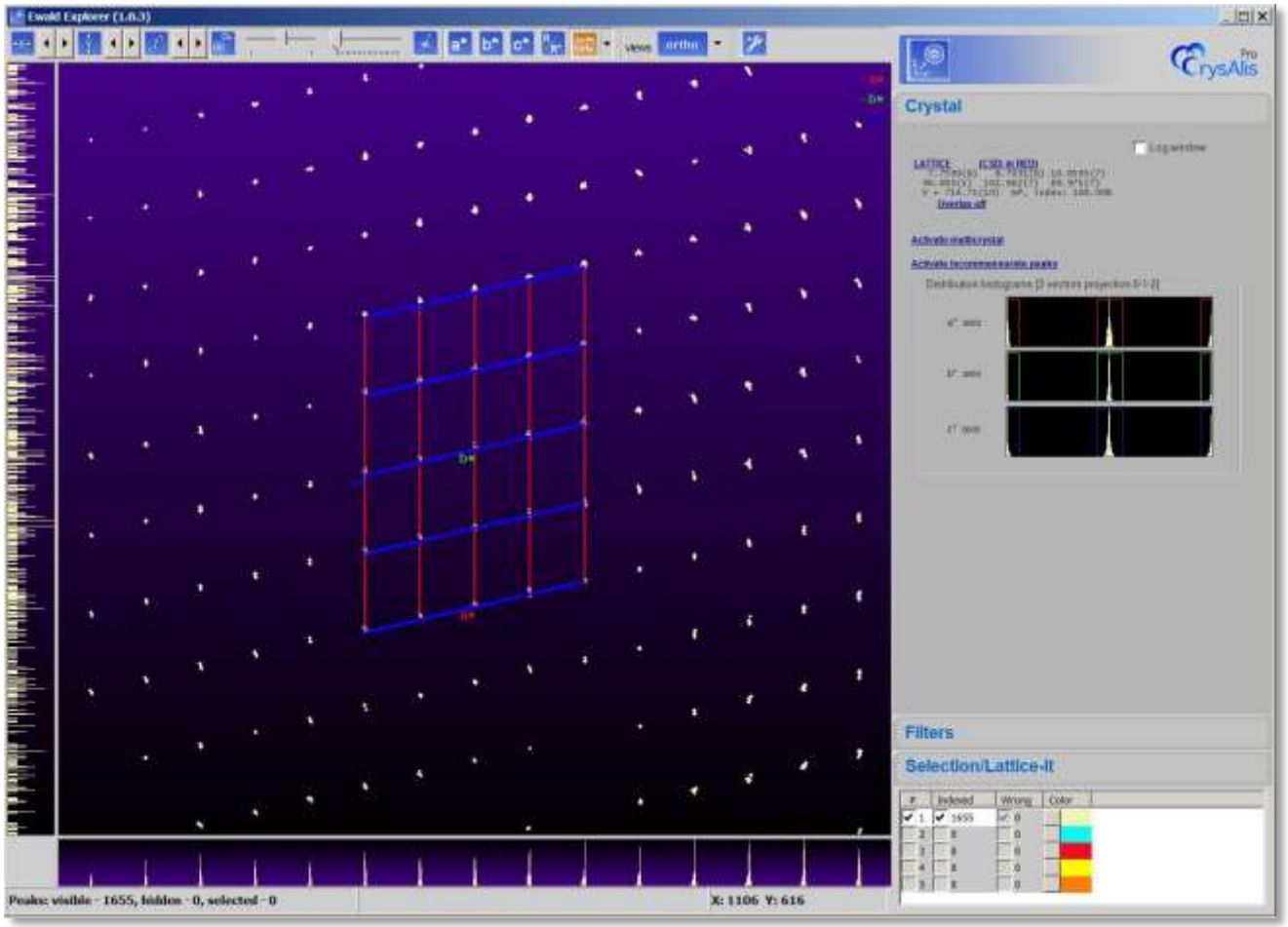




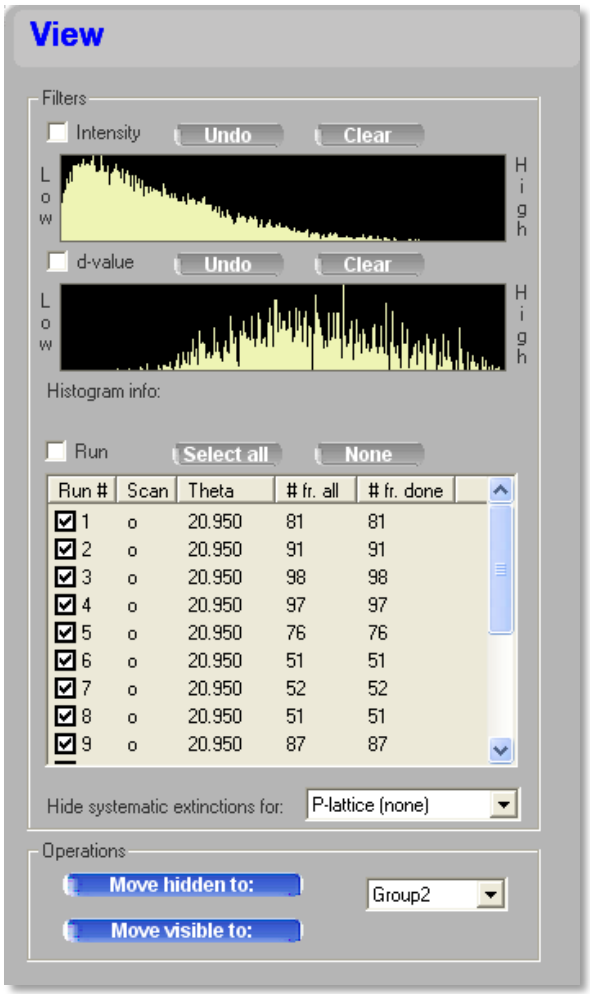
Power Tools

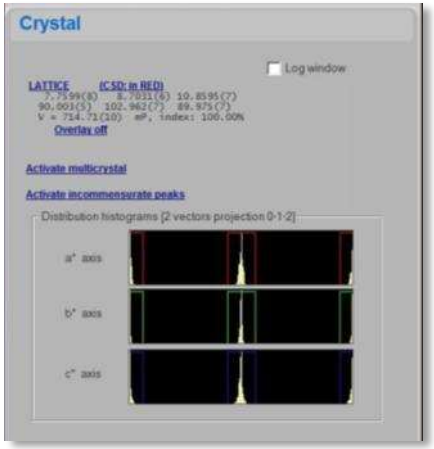
Ewald^{Pro}

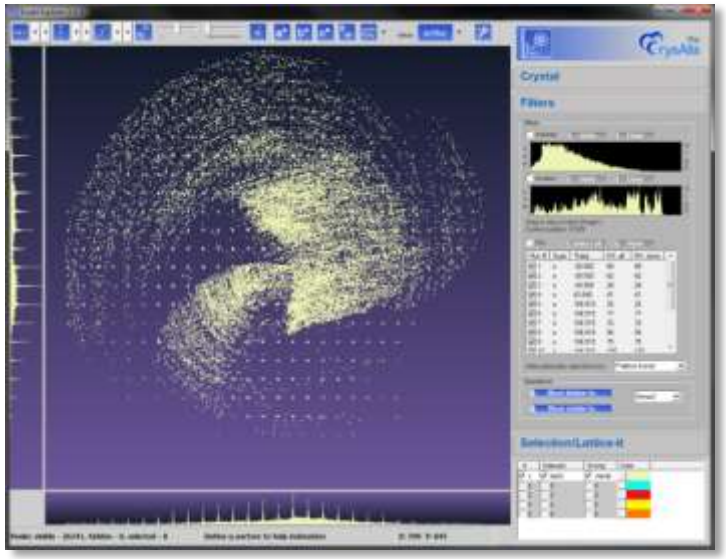
- User-friendly, powerful reciprocal lattice viewer
- Twin/Multi-lattice visualisation
- Drag filter tools – simple & intuitive
- Lattice^{IT} tool for defining periodicity
- Reflection grouping
- Incommensurate overlay tools

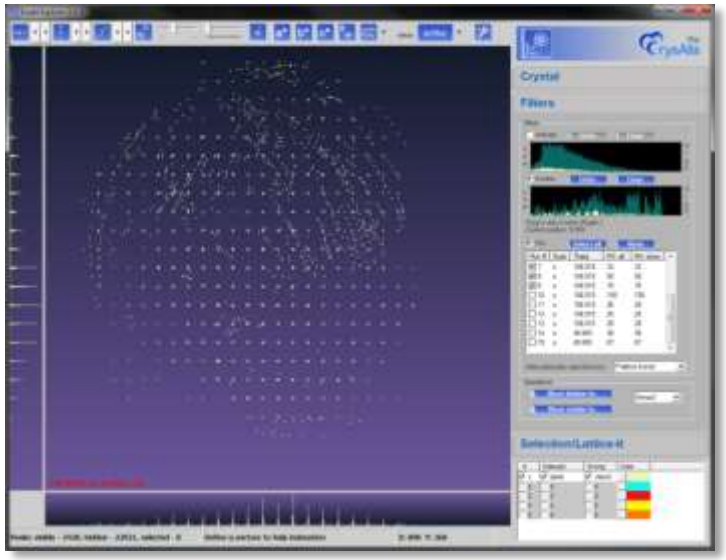


Ewald^{Pro} – Functional Tabs









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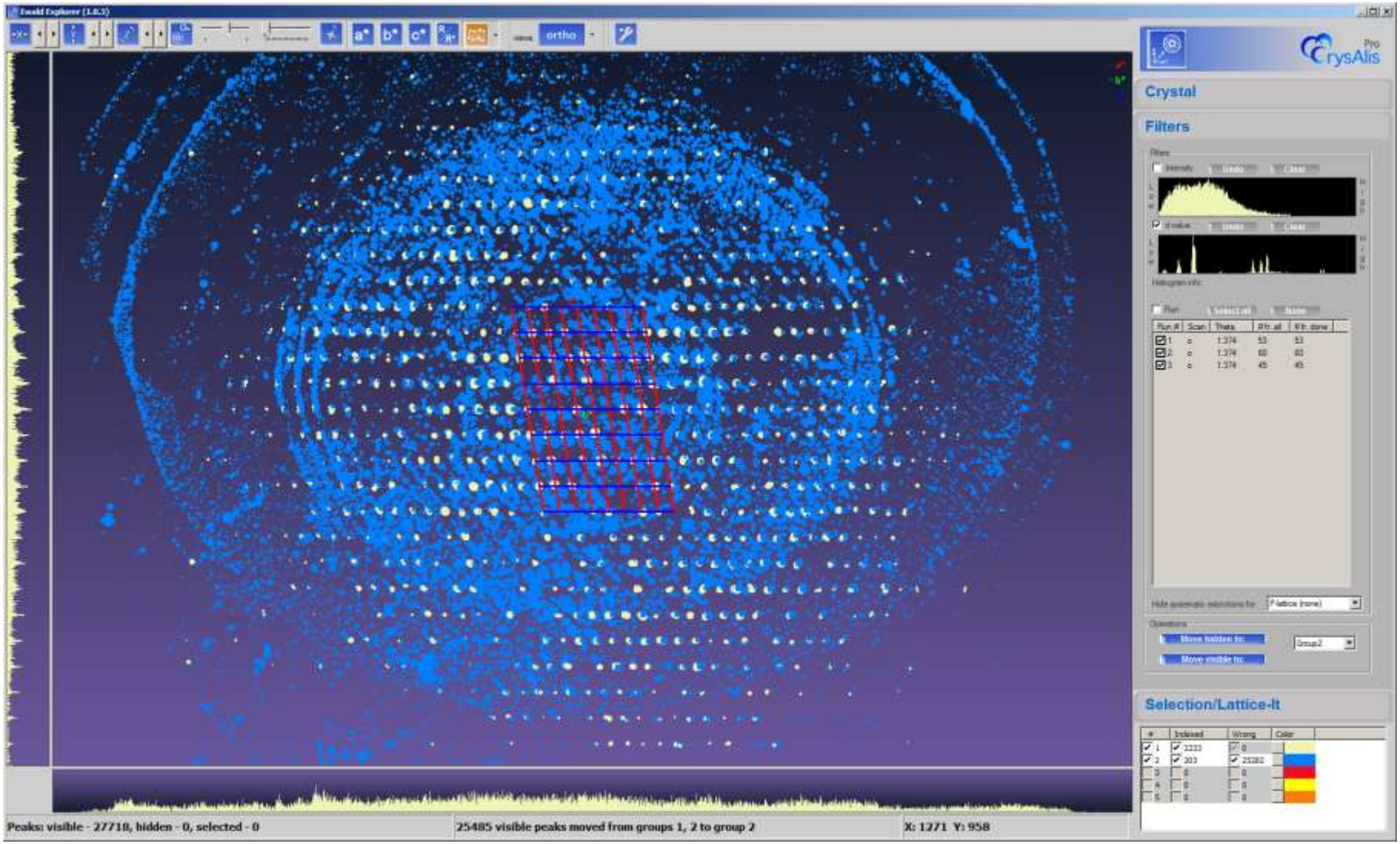
Ewald^{Pro} – Ice Ring Example



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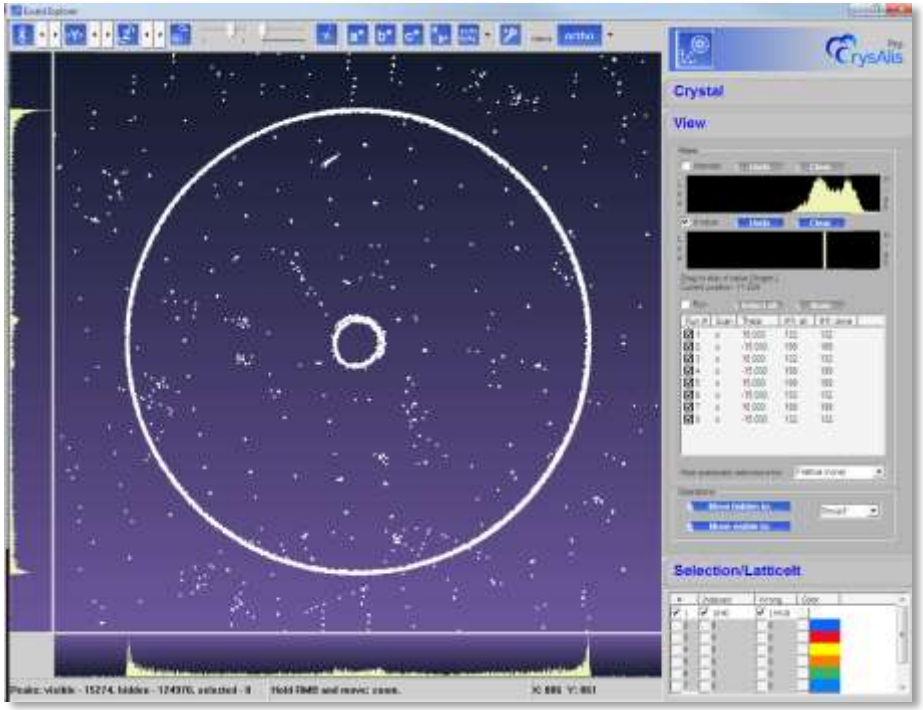
Ewald^{Pro} – Groups



Groups:

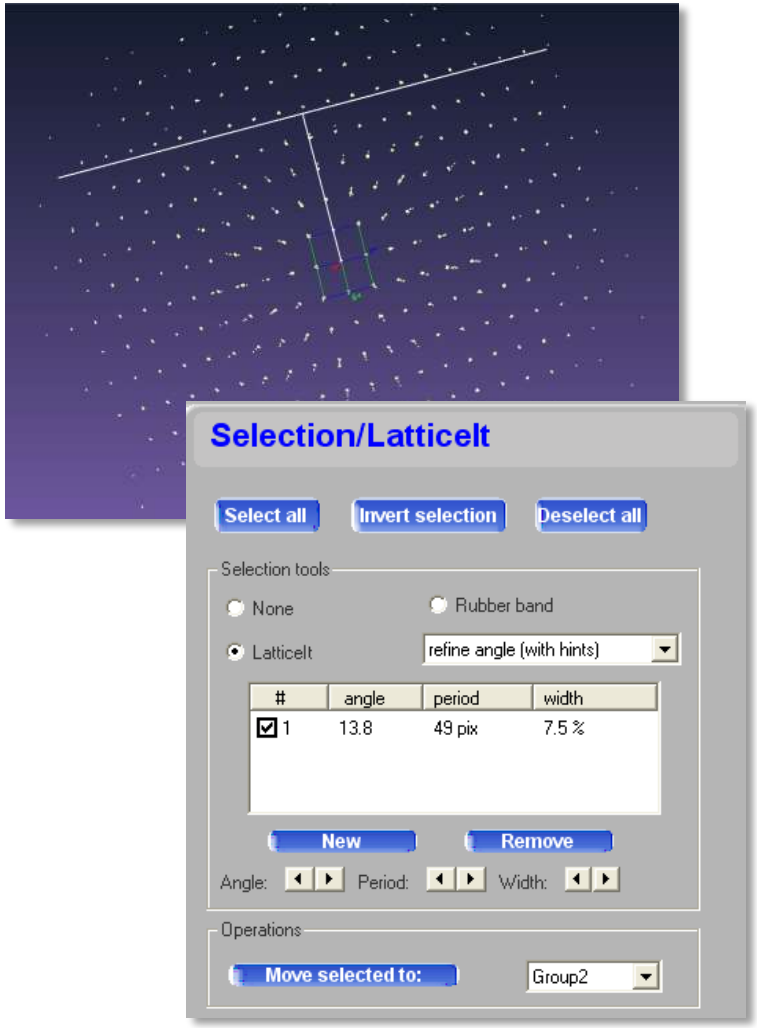
- Indexation quality
- Twin components
- Resolution range
- Incommensurates
- Specific features (weak/strong peaks, powder rings, etc.)

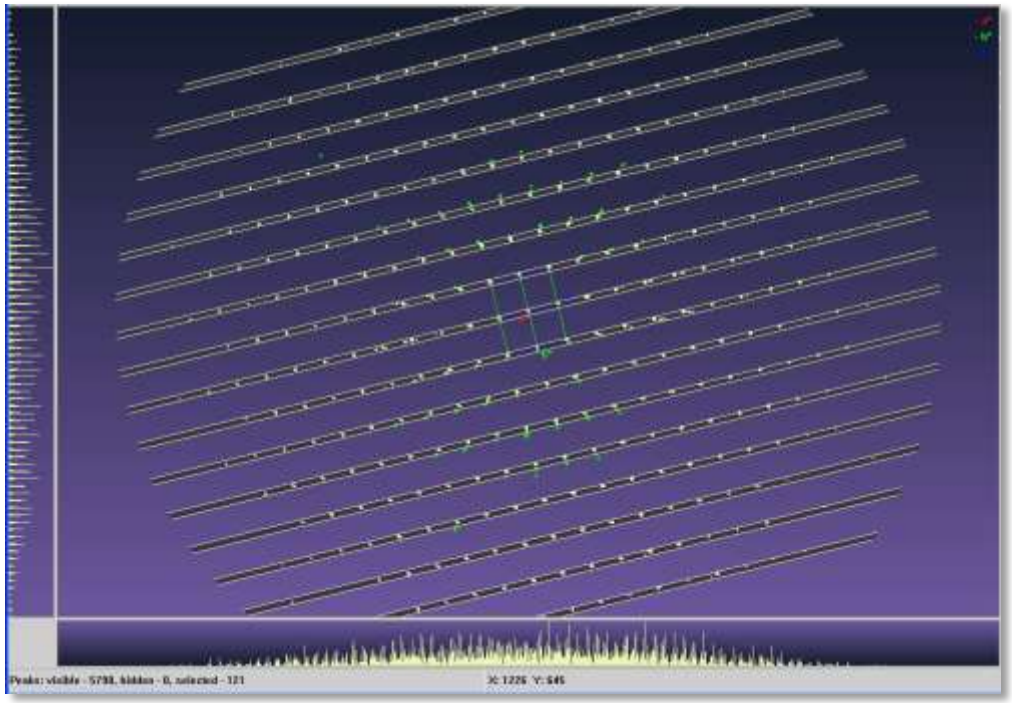
#	Indexed	Wrong	Color
1	1715	36	
2	0	0	
3	0	0	
4	0	0	
5	0	0	

Group1

#	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}





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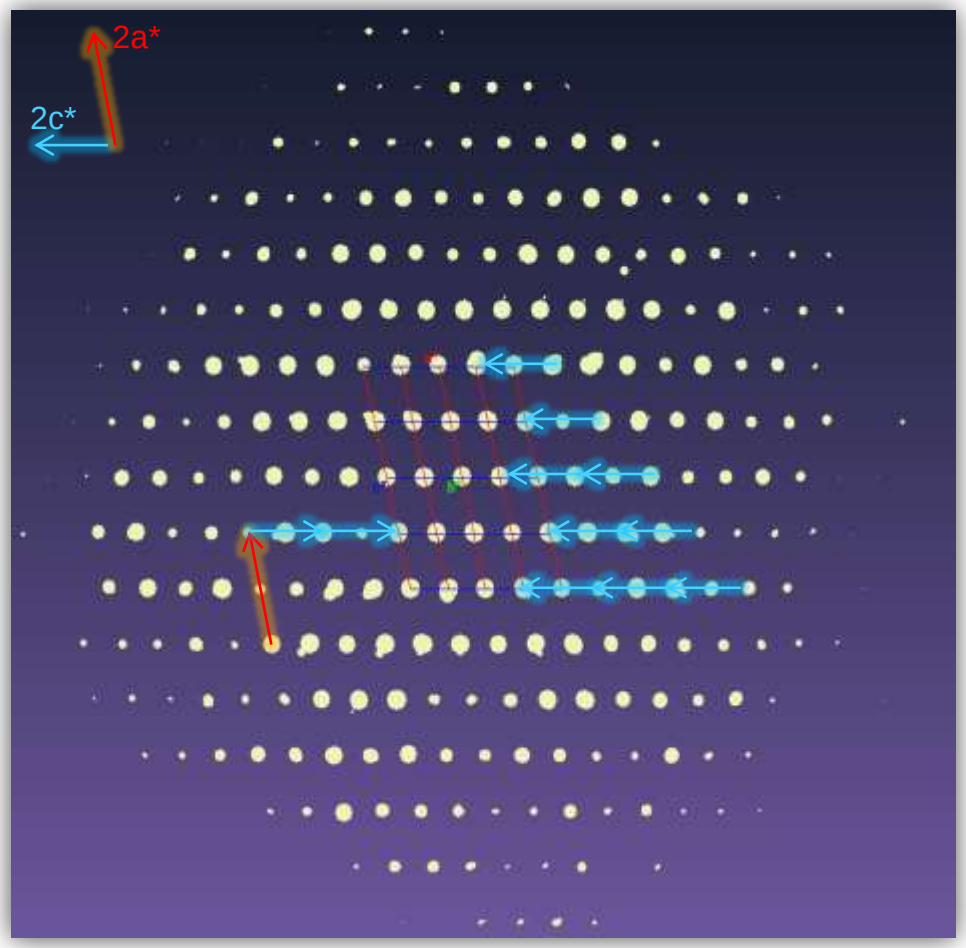
EwaldPro – new view mode: collapse peaks

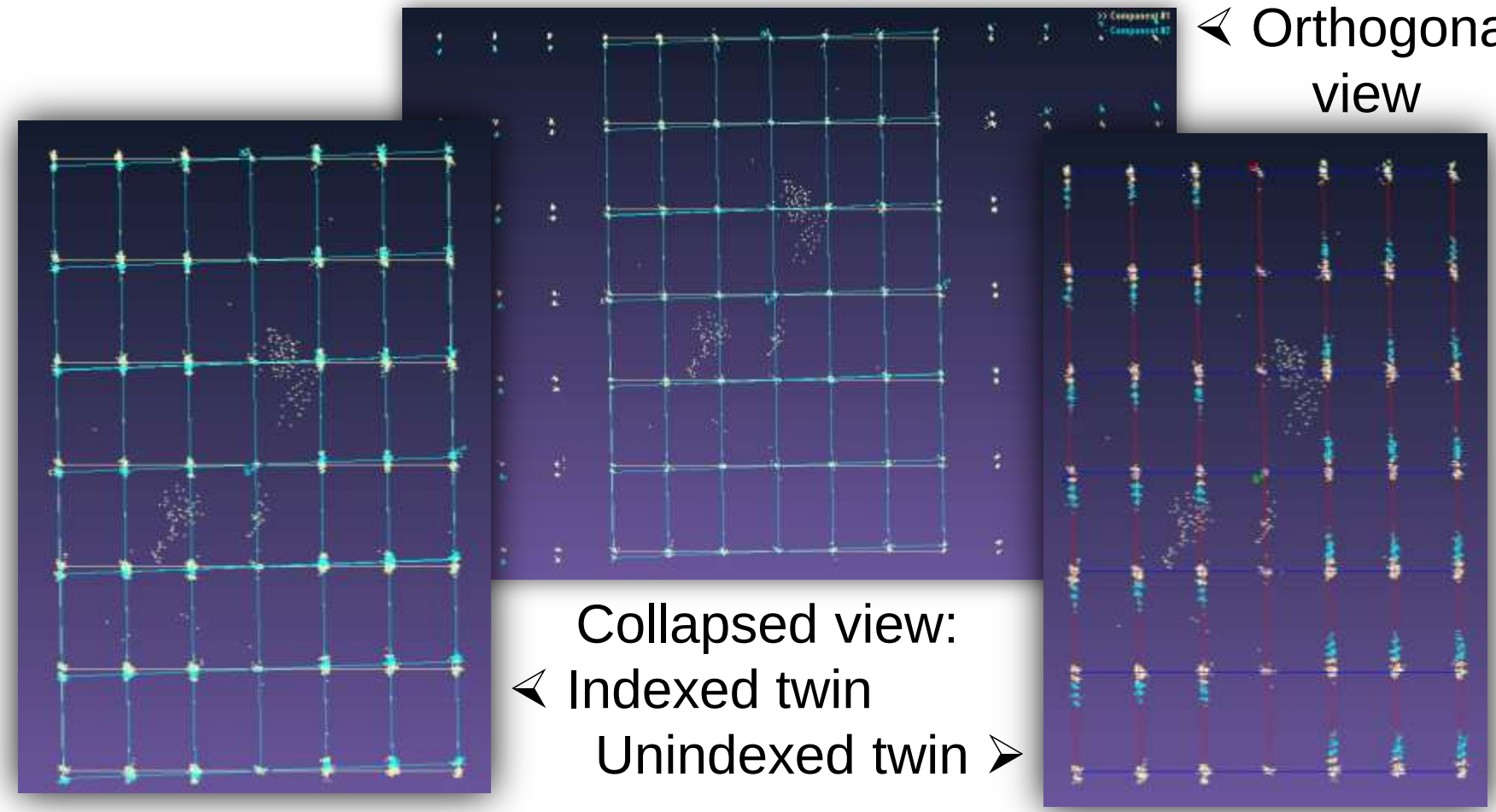
Collapse peak view – construction



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



➢ Orthogonal view

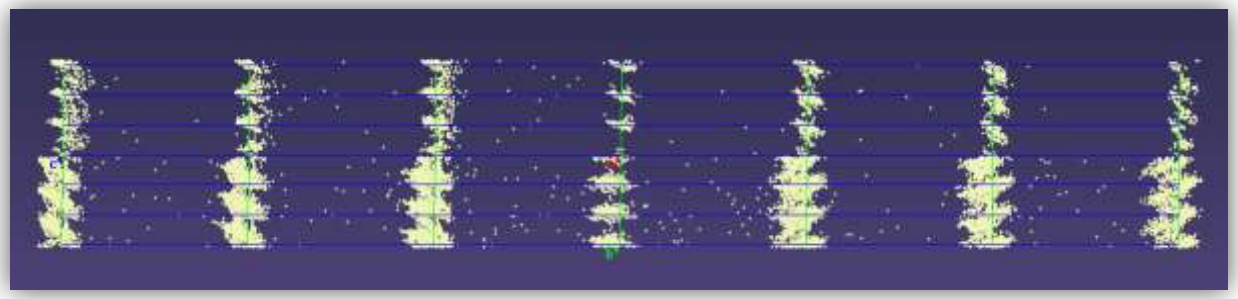
Collapsed view:

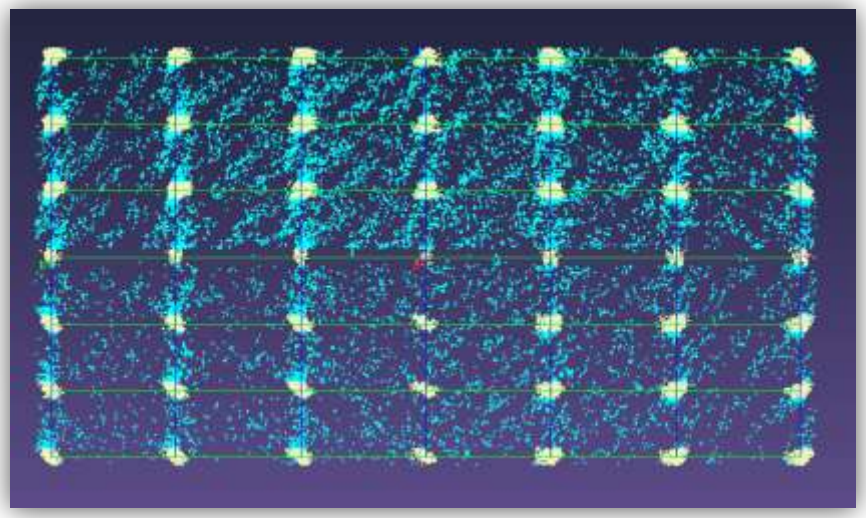
➢ Indexed twin

Unindexed twin ➢

EwaldPro – new view mode: collapse peaks

Collapse peak view – other examples





moving crystal

multicrystal

incommensurate

Typical data reduction sequence

Unit cell finding

↓

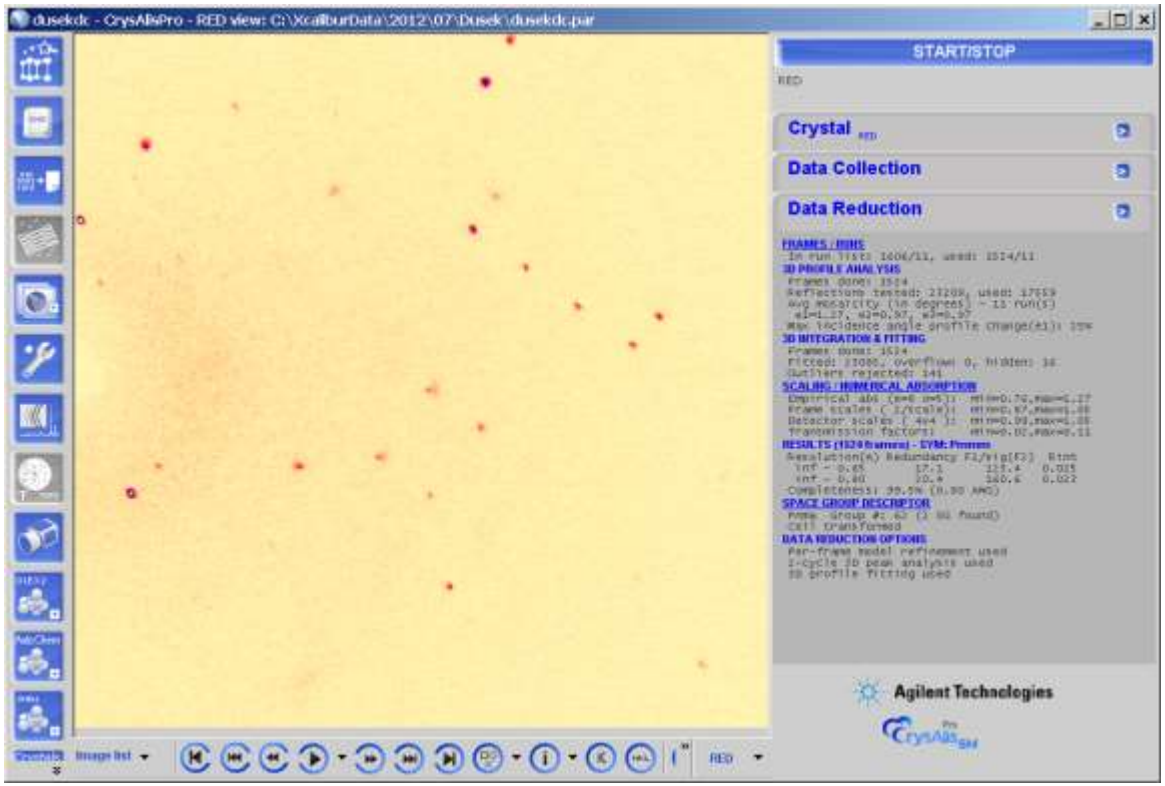
Profile learning

↓

Integration

↓

Post correction

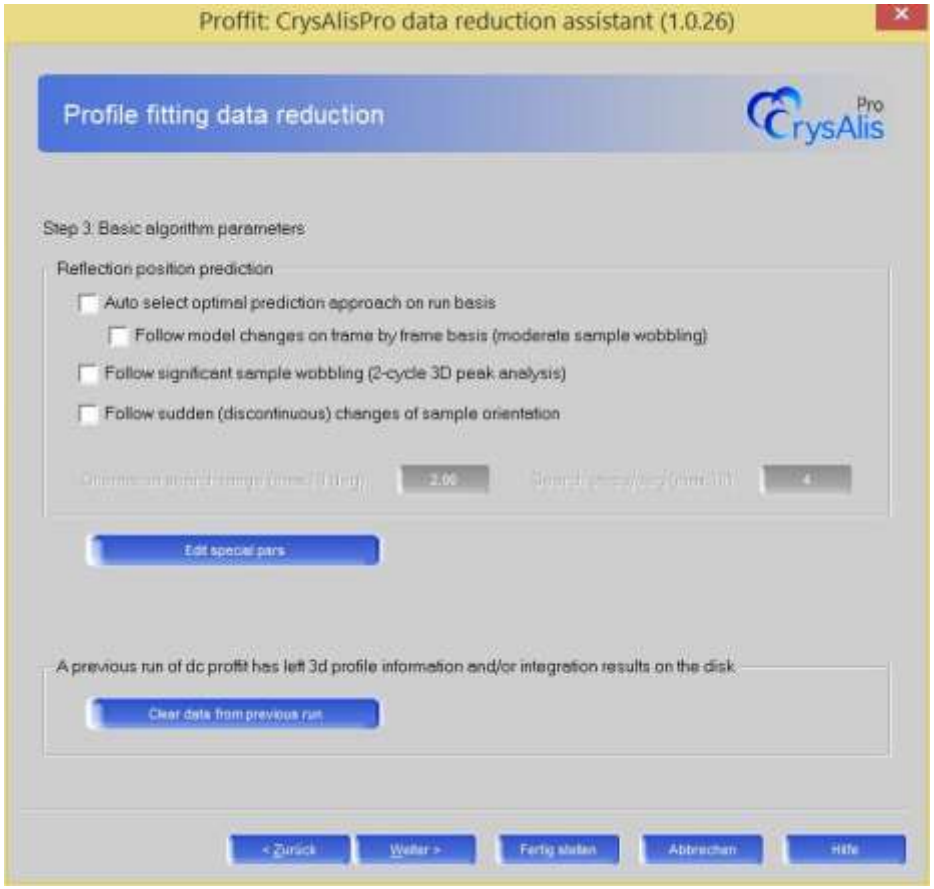


Data reduction wizard

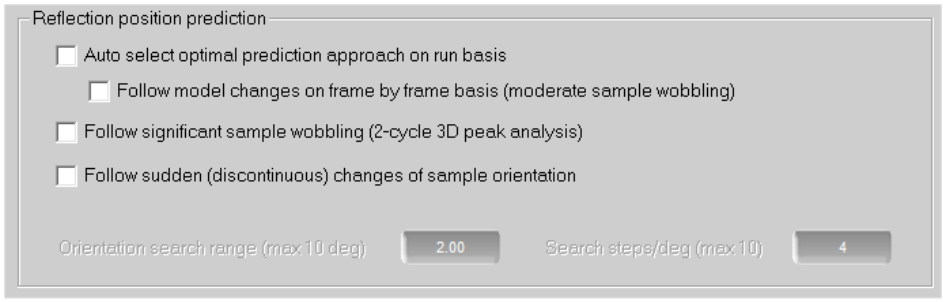
- Collecting information
- Approaches of reflection finding/profile learning/integration
- Approaches to background

Data reduction wizard – options

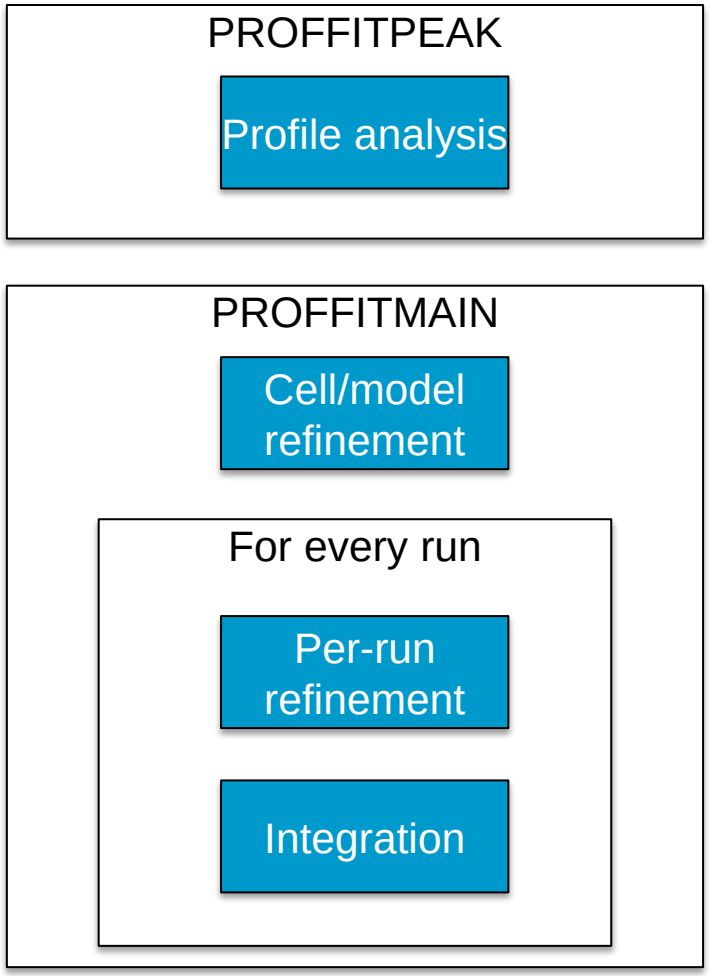
- Model refinement options – for optimal prediction accuracy
- Special data reduction parameters



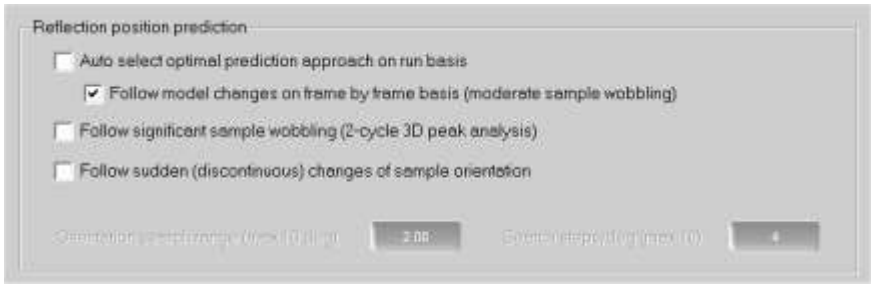
Model refinement options



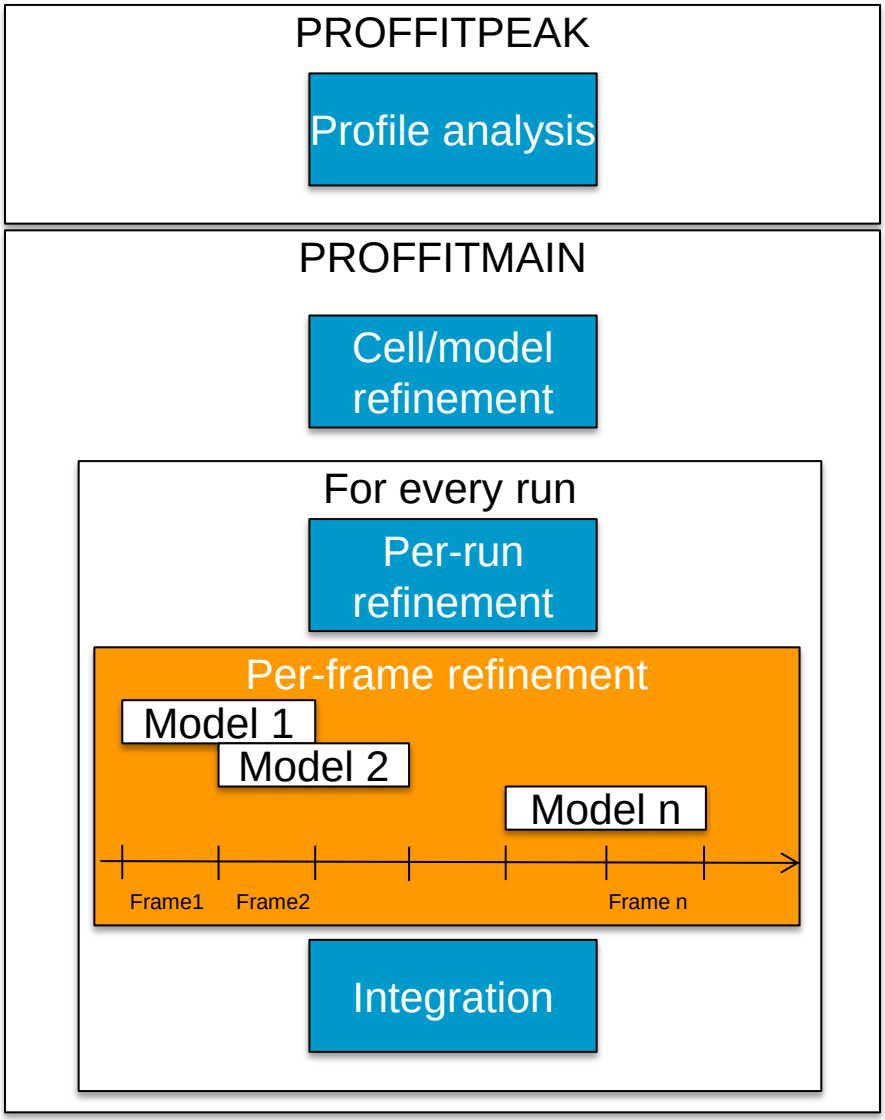
- Basic mode
- Single model for all data from one run
- Usually best choice for properly centered samples on a stiff holder



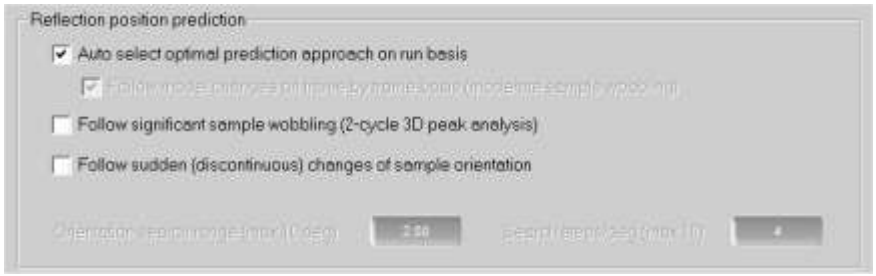
Model refinement options



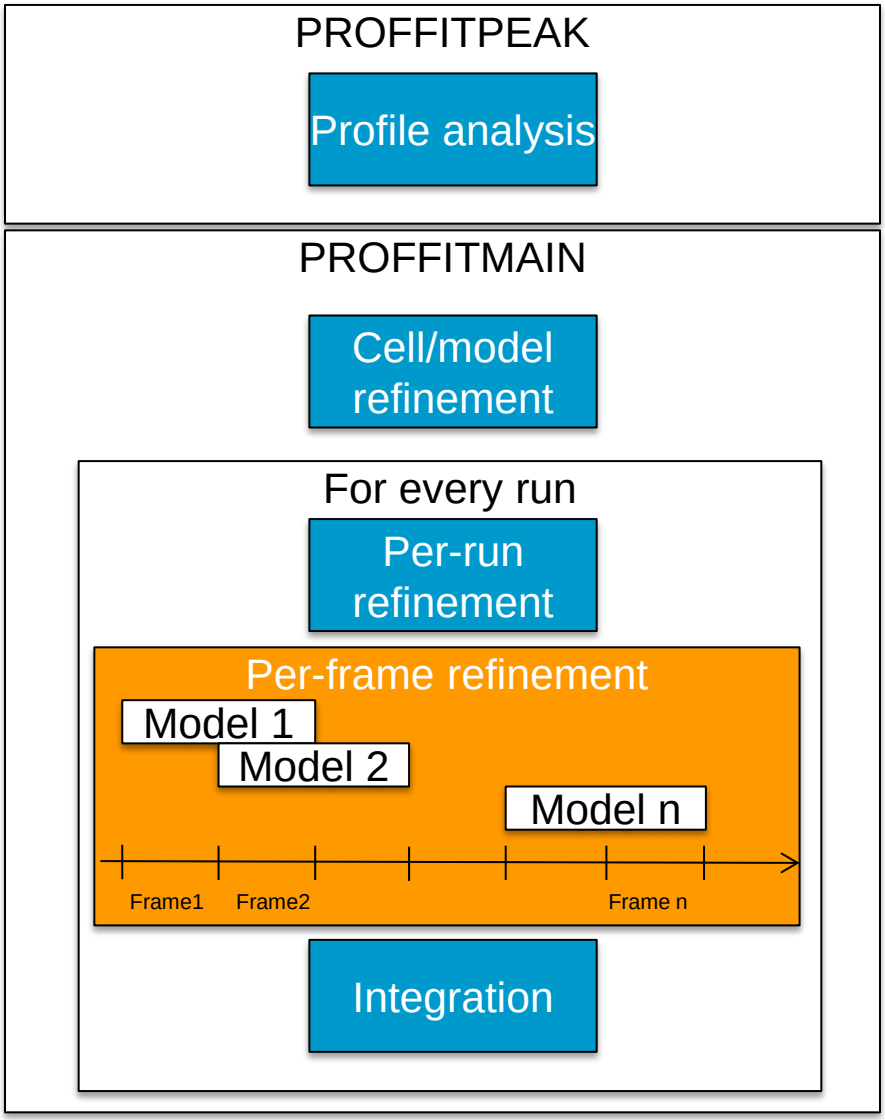
- „Per-frame" models
- Follows smooth crystal moving ("wobbling") with respect to the beam/detector
- Use if the indexation after peak hunting is above 90%, but spots are not fully covered by masks during integration



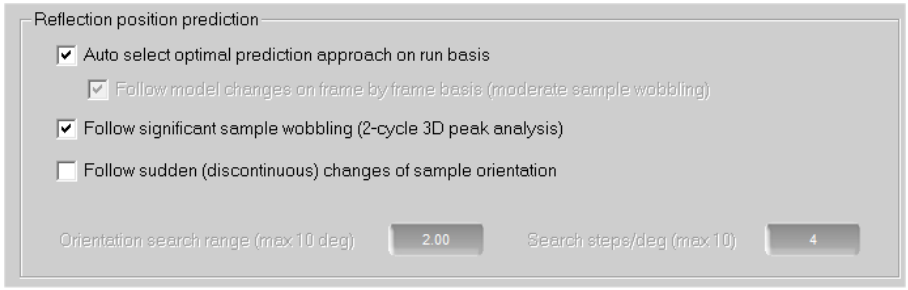
Model refinement options



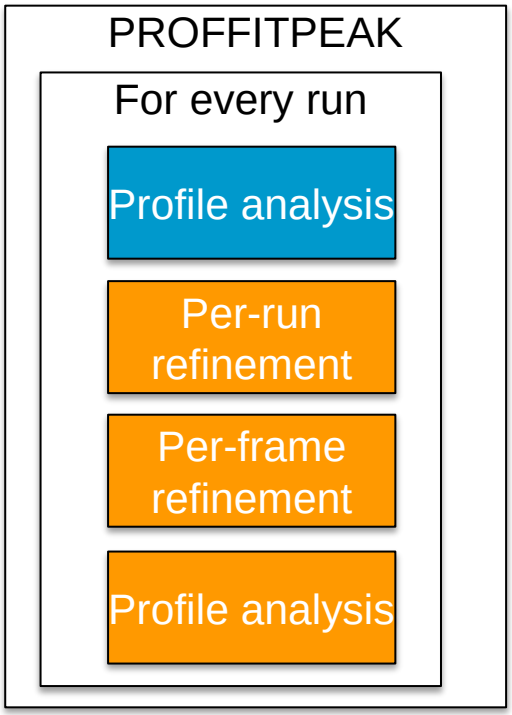
- Auto selects the optimal approach on run basis! Best default option

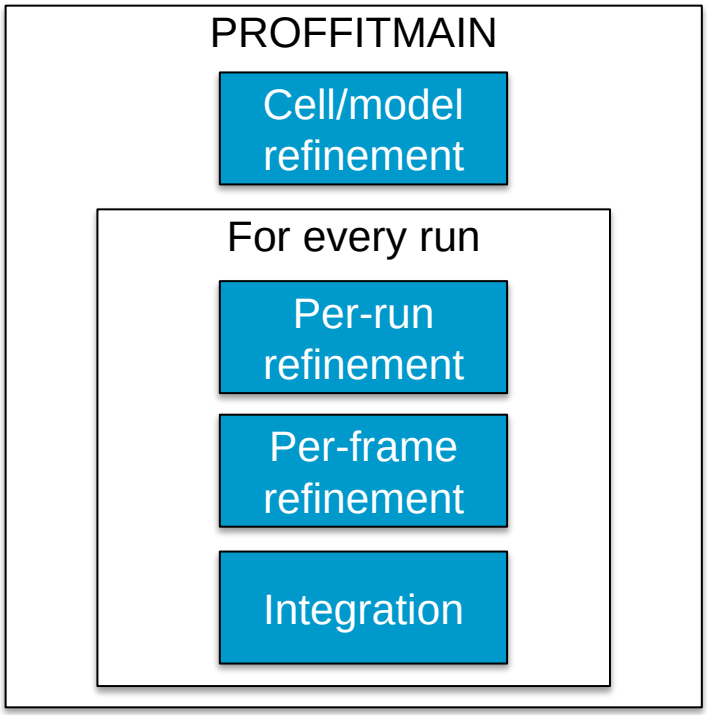


Model refinement options



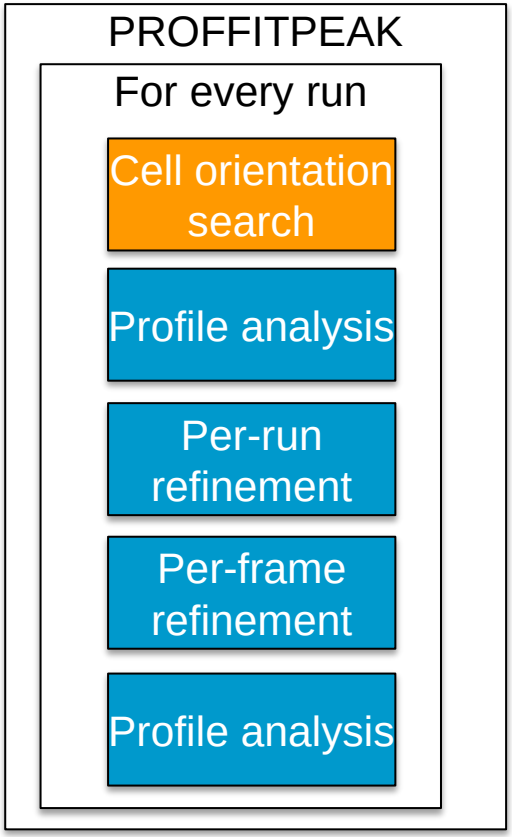
- 2-cycle PROFFITPEAK
- Covers more difficult “wobbling” cases

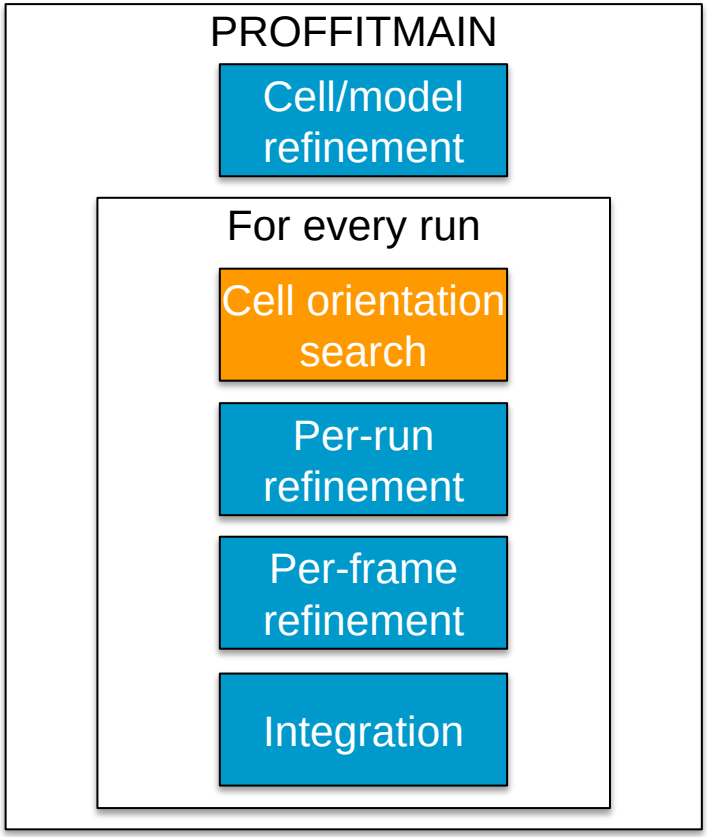




Model refinement options

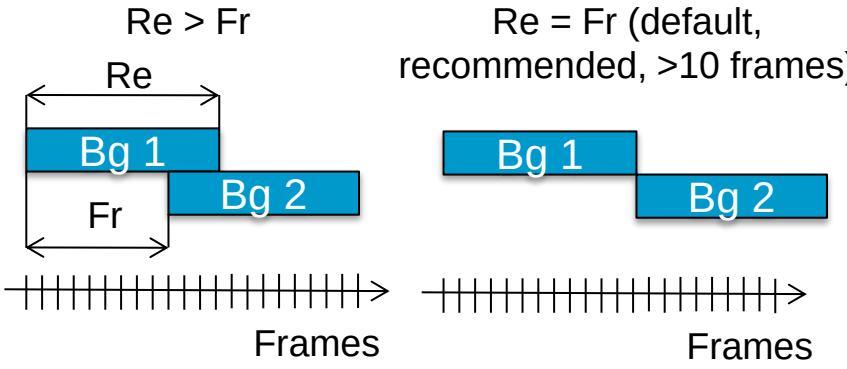
- Model refinement with matrix orientation search
- For discontinous sample jumps (LT, flexible holder, not fixed properly, etc.)





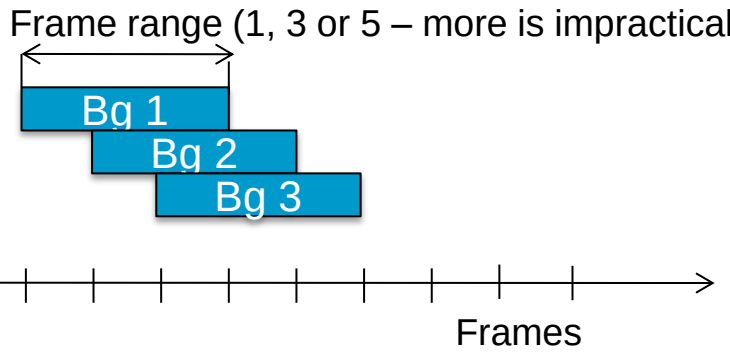
Data reduction wizard – background

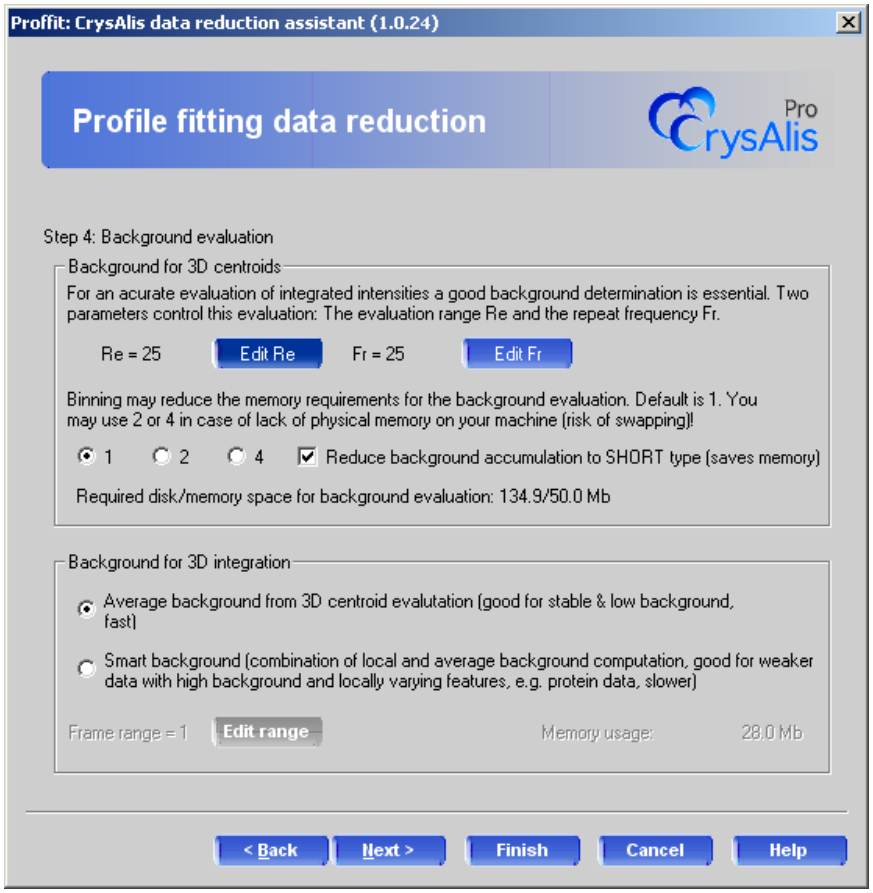
- Average background (Re>10 frames)



- Smart background:

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

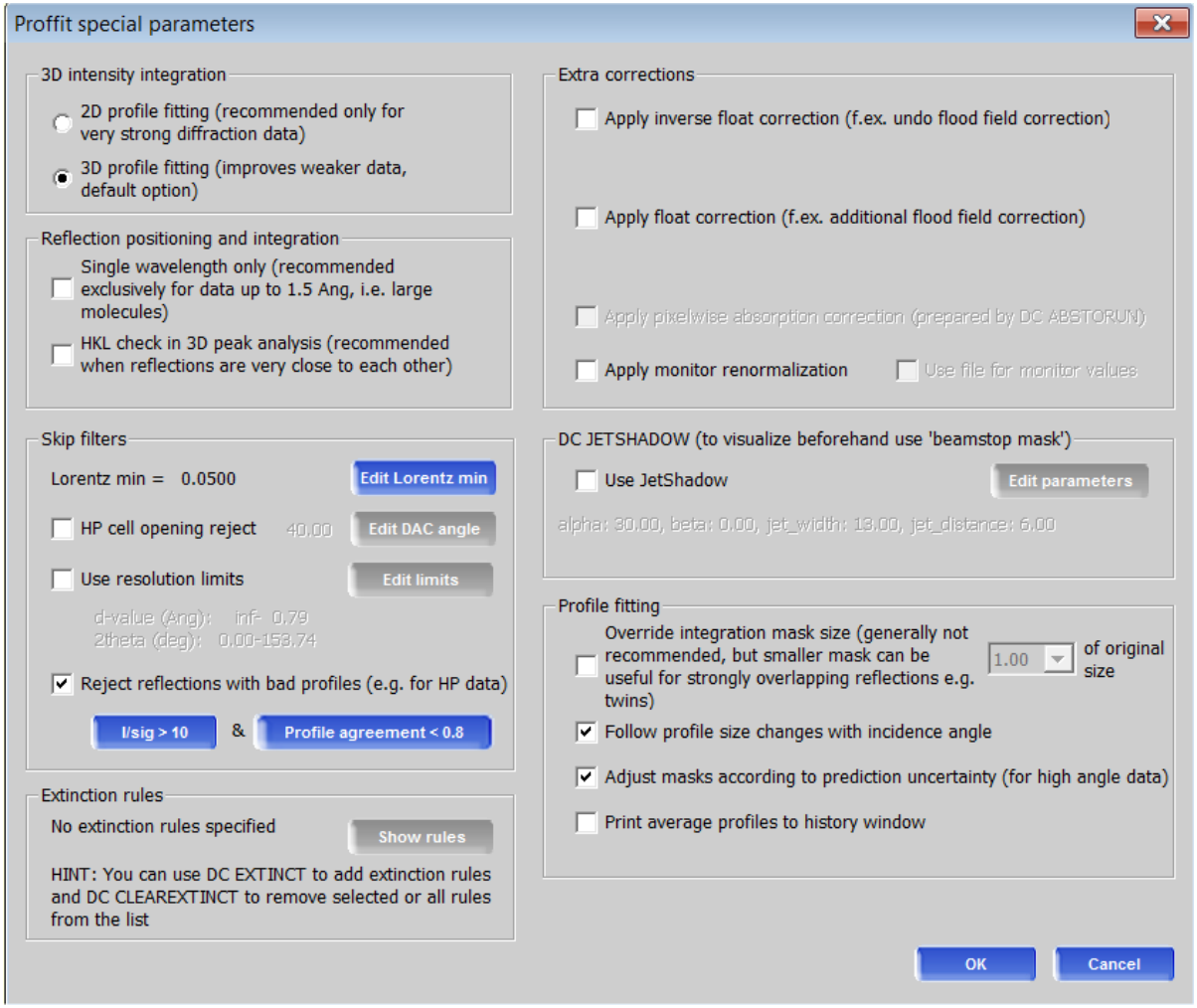




Optimal data – hints at data reduction time

- 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

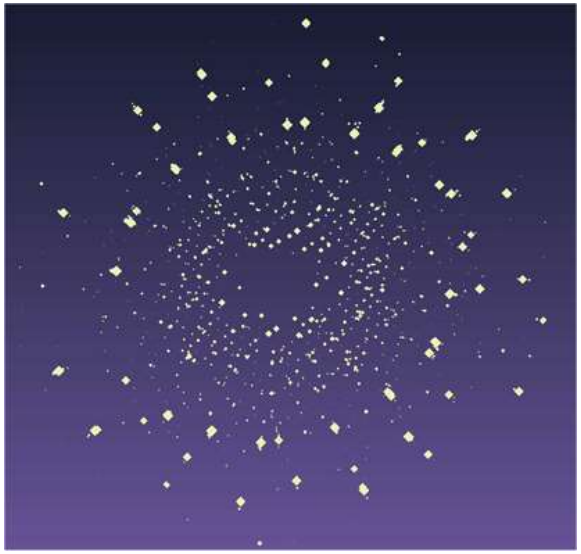


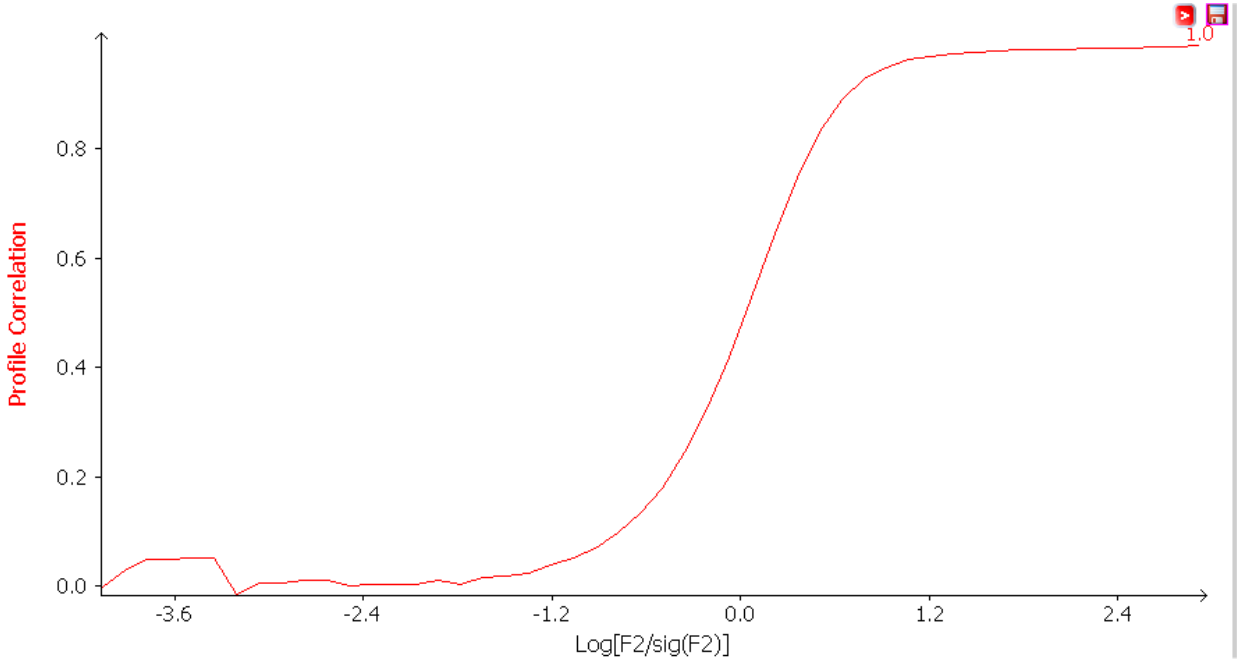
3D profile fitting

- 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

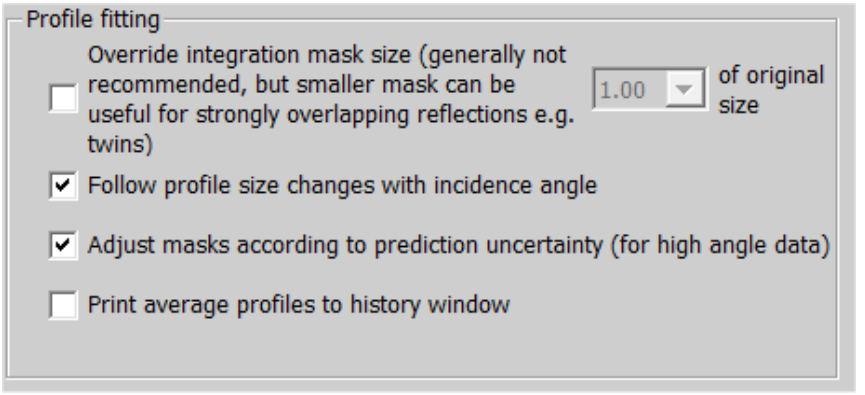
Special pars- outliers

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





Special pars - incidence



Profile size analysis (per incidence angle)

Incidence angle (deg)	# of peaks	Average profile size - assuming Gaussian shape (in degrees) 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

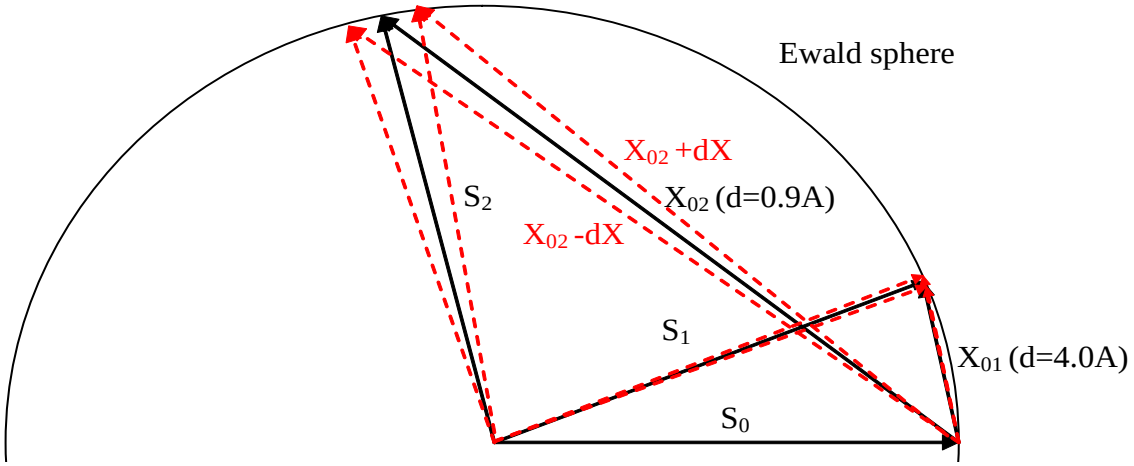
e1 dimension: a=0.0022 b=0.99

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

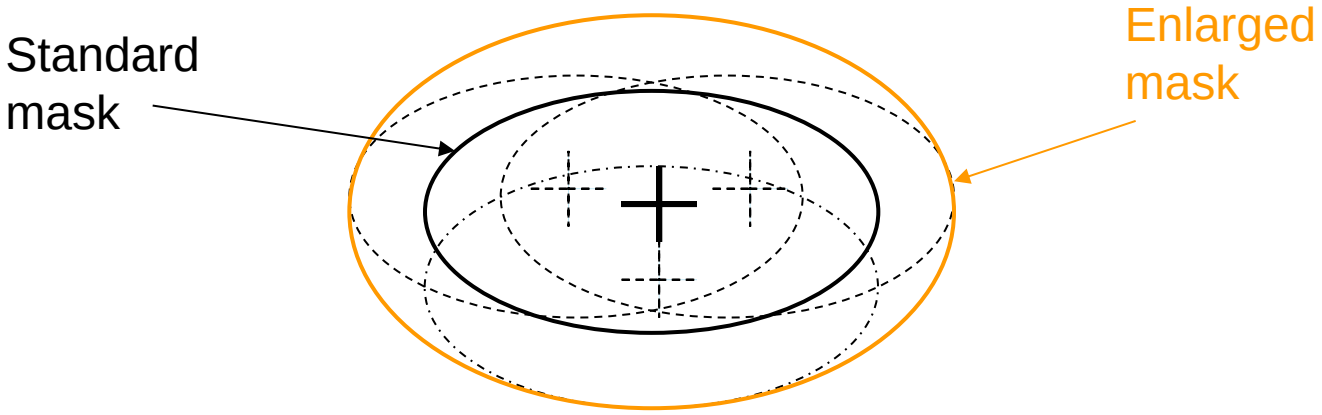
Prediction uncertainty – solution

- Estimate prediction uncertainty at given theta

Prediction accuracy statistics (in degrees)

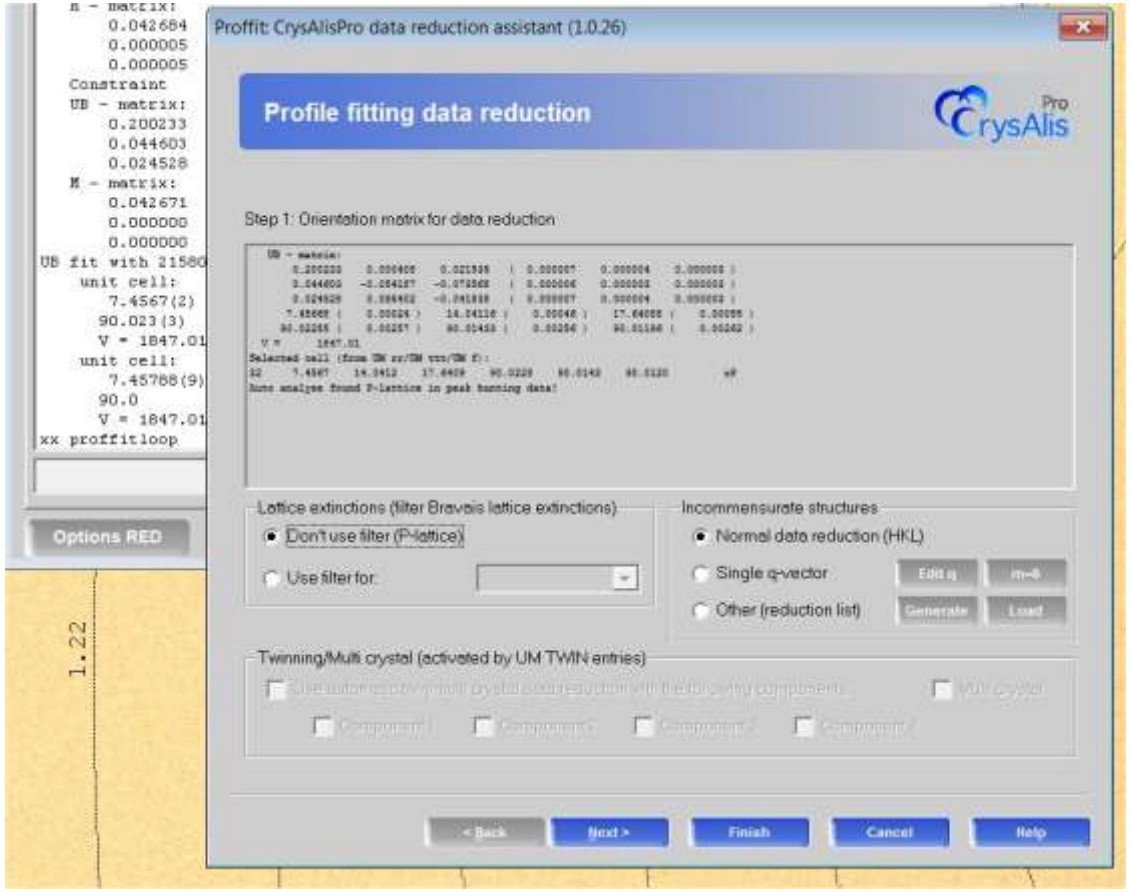
		Average prediction error (in degrees)		
Resolution	# of peaks	delta_e1 (stddev)	delta_e2 (stddev)	delta_e3 (stddev)
1.35–1.22	82	0.002 (0.020)	0.001 (0.013)	0.003 (0.170)
1.22–1.14	82	-0.002 (0.025)	0.009 (0.016)	0.020 (0.165)
1.14–1.08	82	0.003 (0.024)	0.004 (0.020)	0.020 (0.175)
1.08–1.04	82	-0.012 (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



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Typical data reduction sequence

Unit cell finding

↓

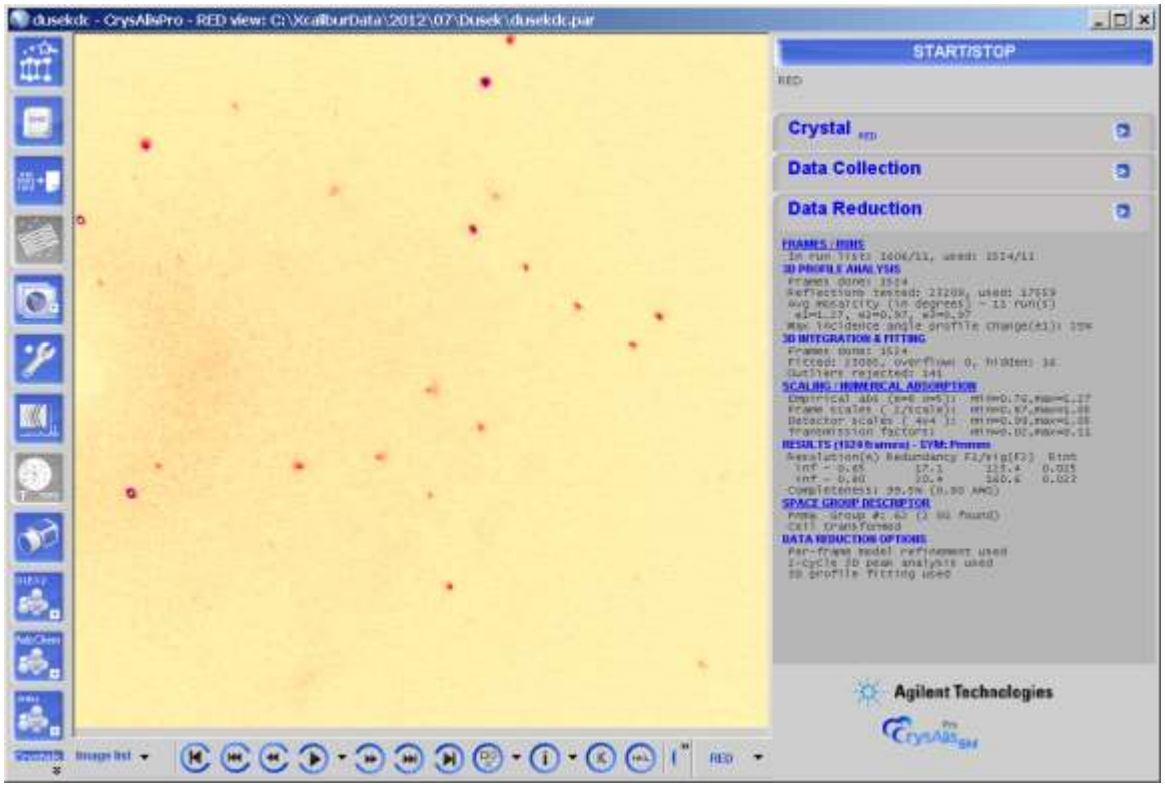
Profile learning

↓

Integration

↓

Post correction



Data reduction (reflection integration) vs. data finalization

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

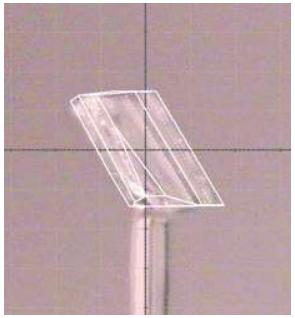
Post correction motivation

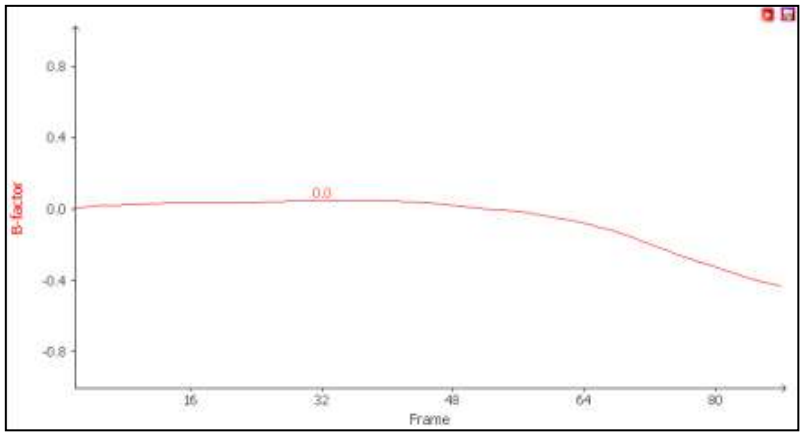
- Frame information to HKL file information
- Improve I/σ of redundant information
- Reduce 'systematic effects'

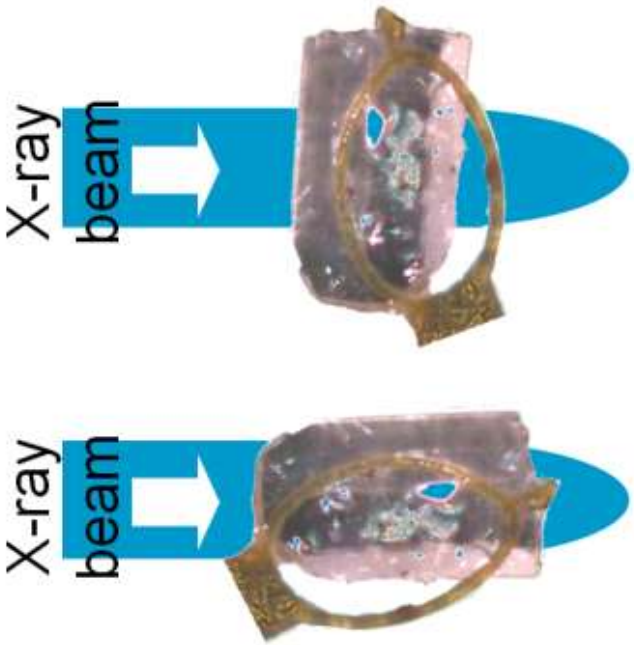
Note:
Post corrections can only correct observed data!

Systematic effects/Noise sources

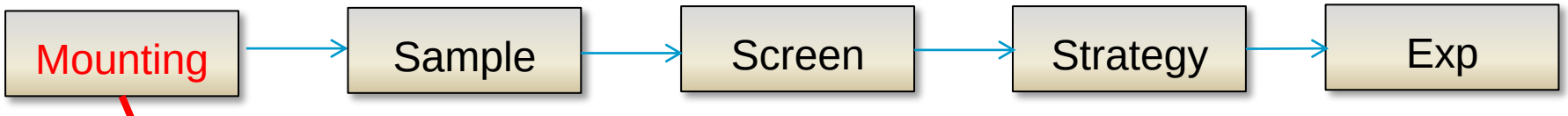
- Absorption (sample/holder)
- Beam illumination effects
- Sample centering
- Sample decay
- Experiment effects: cryo, generator
- Mounting technique

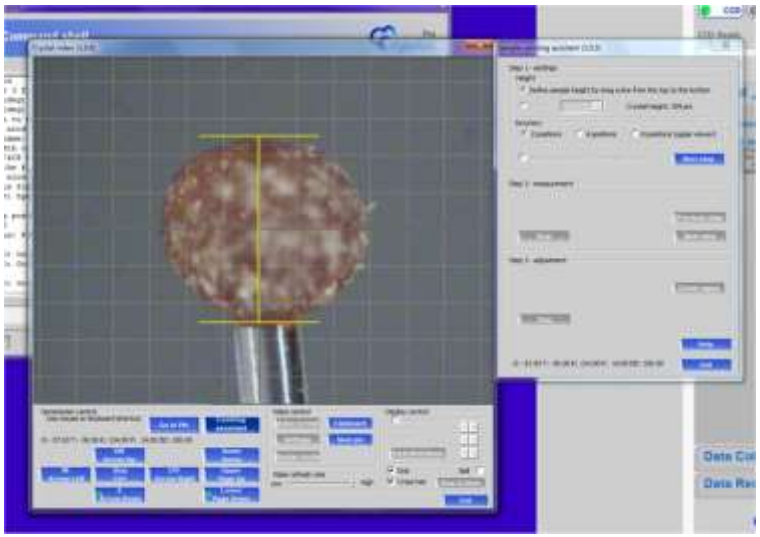




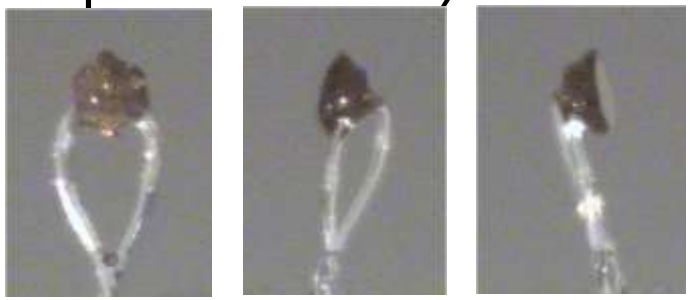


Data acquisition sequence: correction requisites



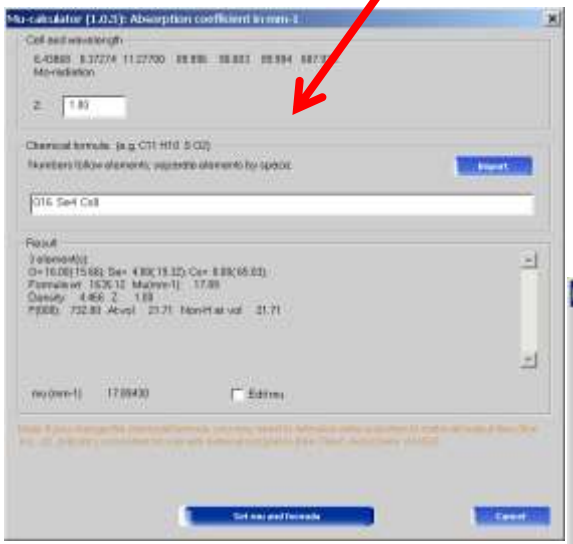


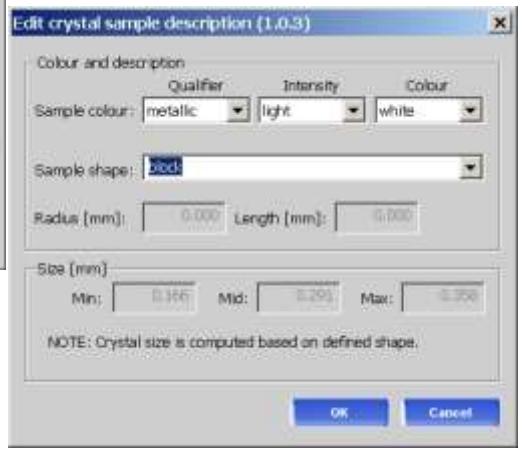
- CrysAlis^{Pro} ‘Centering assistant’ reduces centering error for anisotropic samples
- Height estimator ‘remembered’ for automatic shape generator
- Movie registers sample shape for later evaluation (triggered at experiment time)



Data acquisition sequence: correction requisites

- Sample description
- Chemical compound





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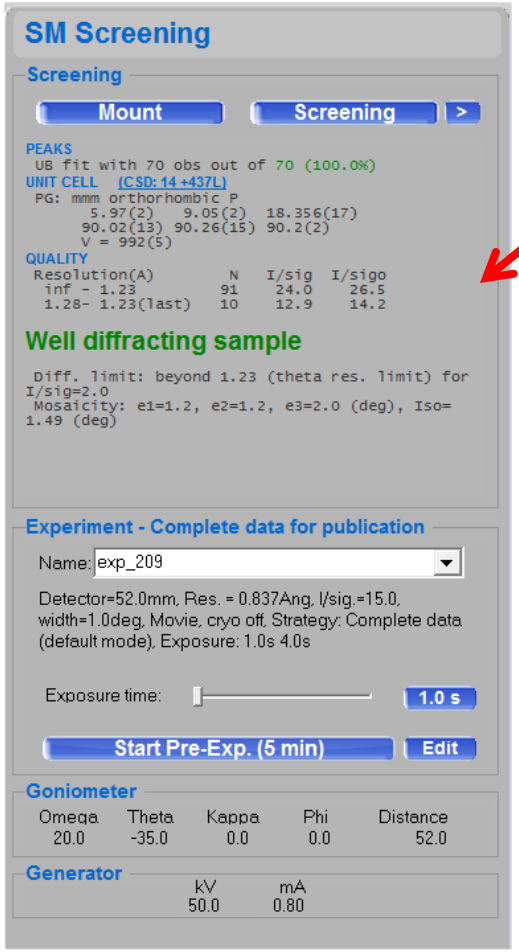
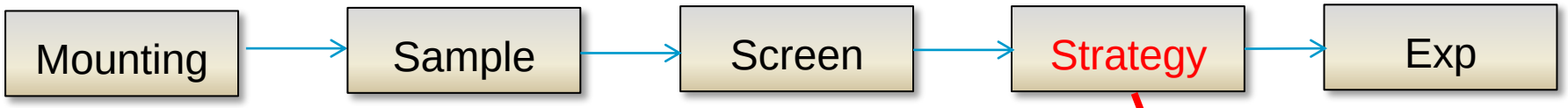
Data acquisition sequence: correction requisites

Mounting → Sample → Screen → Strategy → Exp

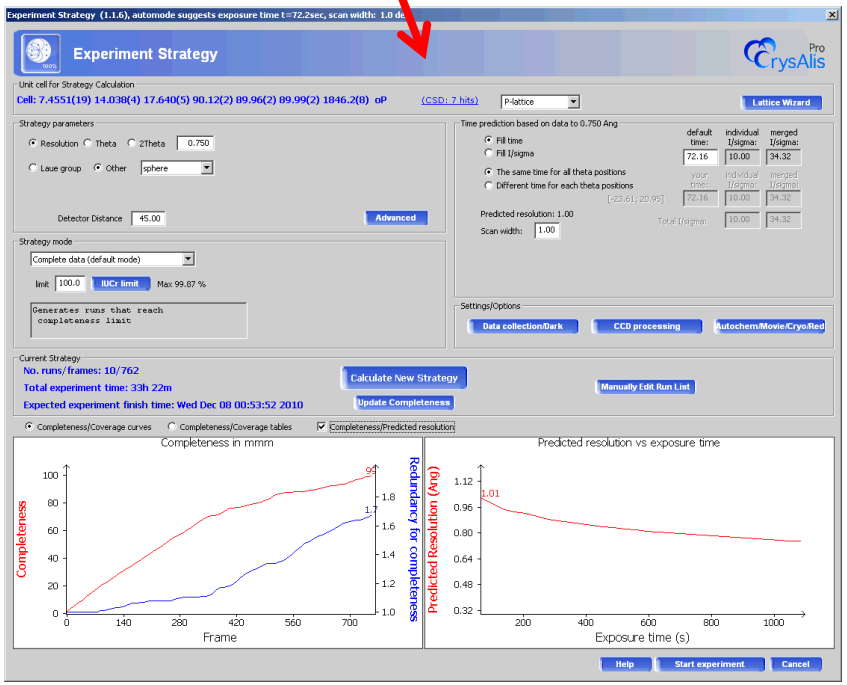


- CrysAlis^{Pro} screening tool gives unit cell in <10sec
- Selection of sample with proper diffraction power and sample quality

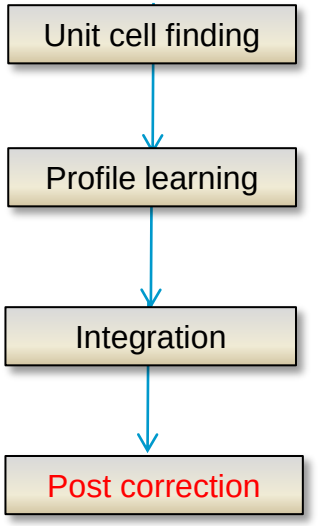
Data acquisition sequence: correction requisites

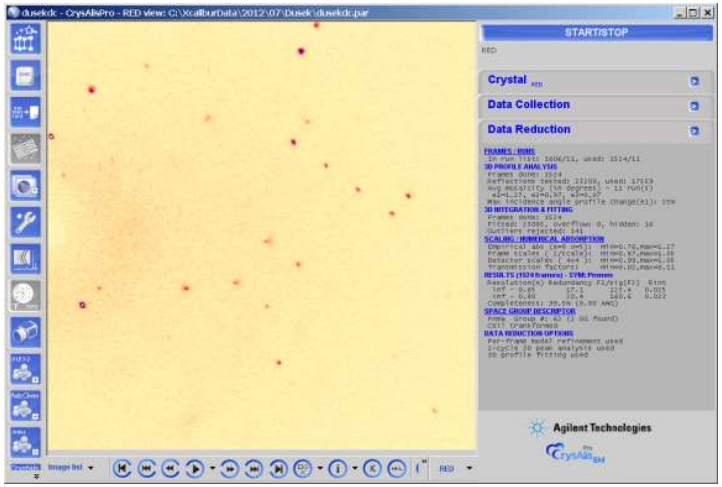


- Based on unit cell, Laue symmetry and chemical constraints the proper strategy is computed
- I/σ calculator helps to find proper 'detectivity level' for the sample
- Target redundancy can be set

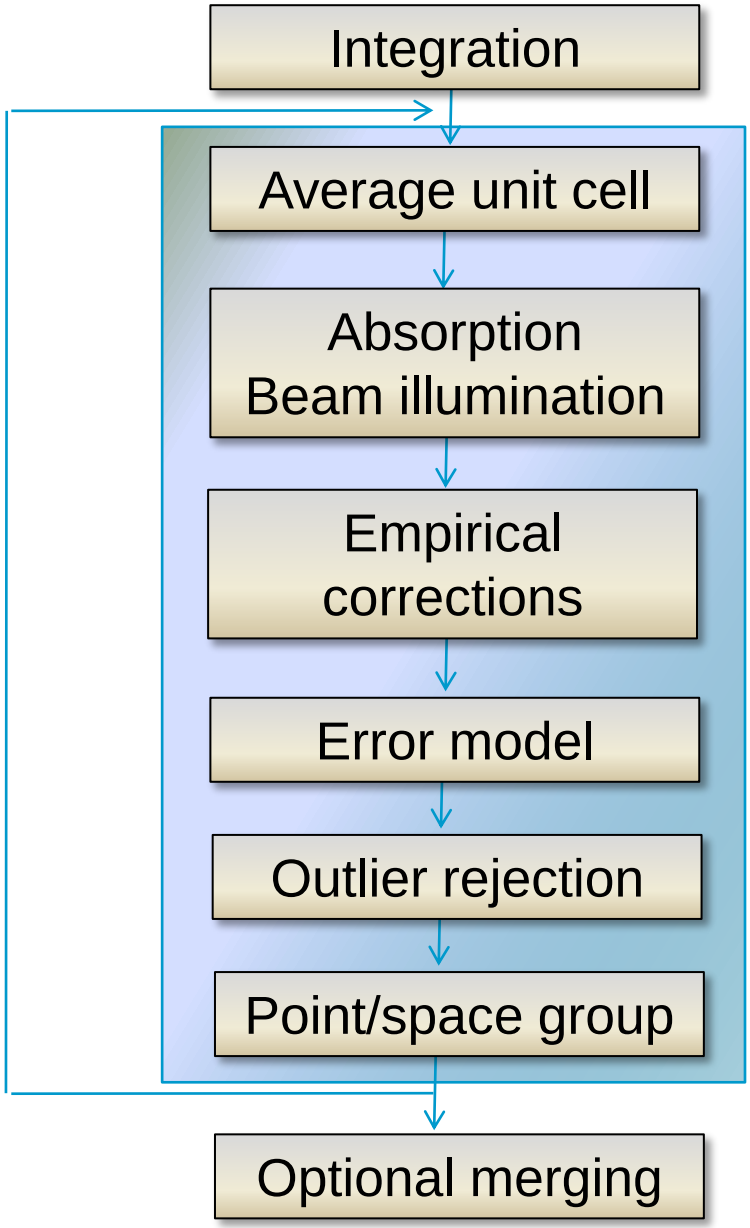


Post correction layout

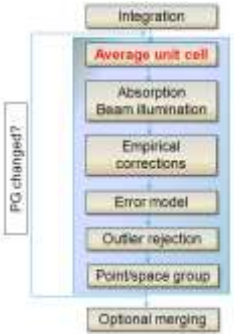




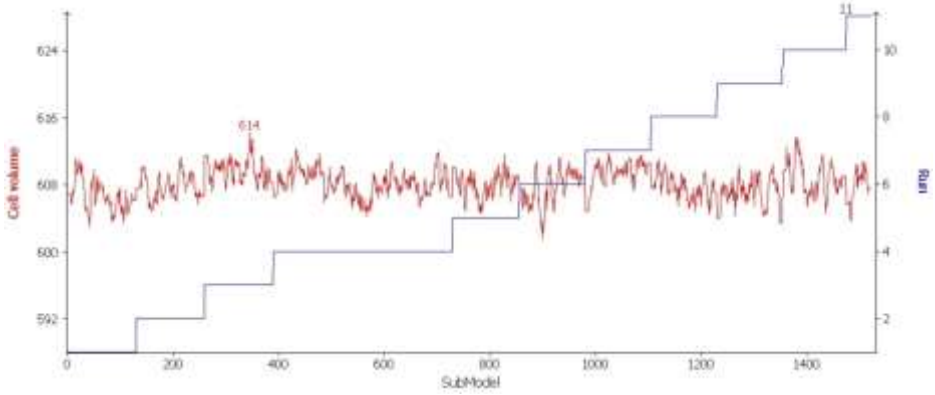
PG changed?



Average unit cell from all data



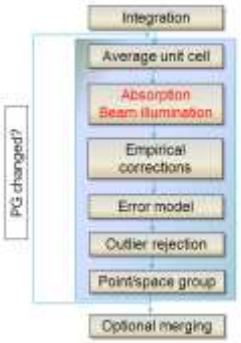
- Post integration: most accurate model description available
- 3D profiles known
- All experimental aberrations are corrected for



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

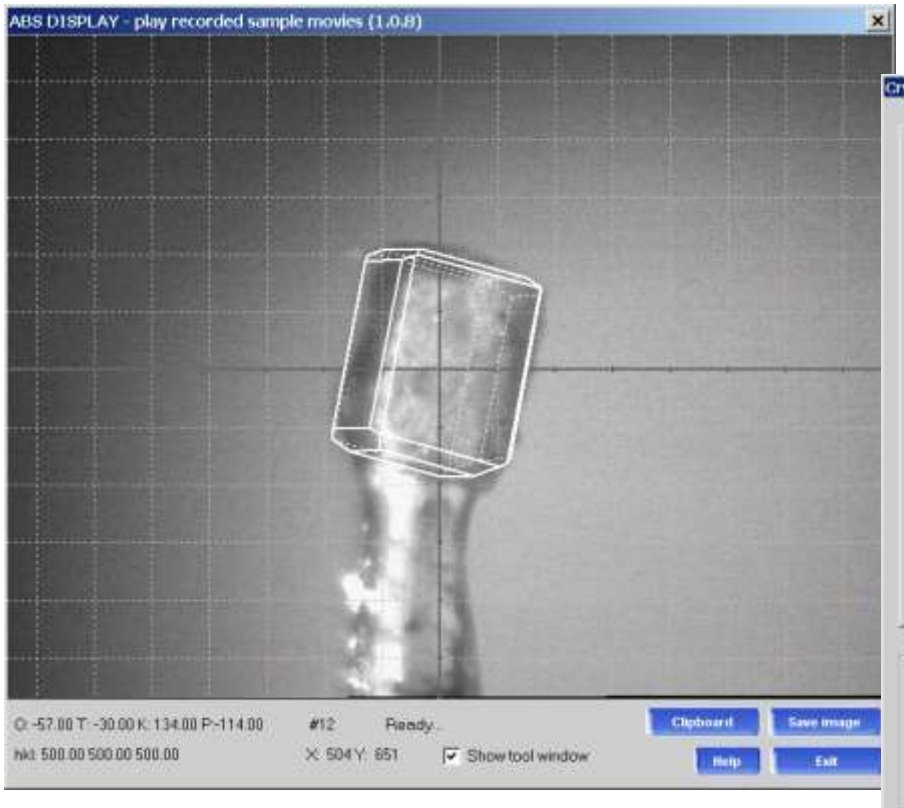


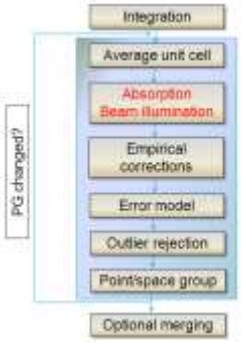
- Clark & Reid analytical formulation (allows shape optimization)[1]
- Gaussian grid, numerical integration[2]
- Beam illumination
- High Pressure: Gasket shadowing, diamond correction[3]

Note:
Shape description and chemical composition have to be known!

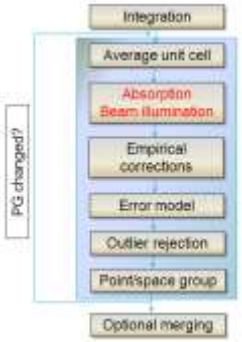
[1]Clark, R. C. & Reid, J. S. (1995). Acta Cryst. A51, 887-897; [2]Busing, W. R. & Levy, H. A. (1957). Acta Cryst. 10, 180-182;
[3]Angel, R. J. (2004). J. Appl. Cryst.. 37, 486-492

‘Face-based’: Absorption, beam illumination

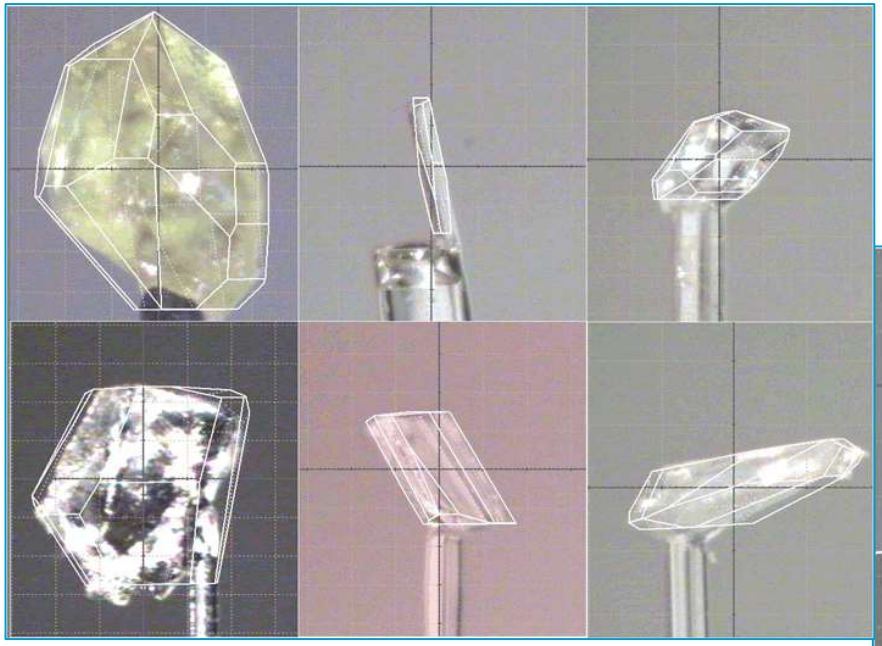


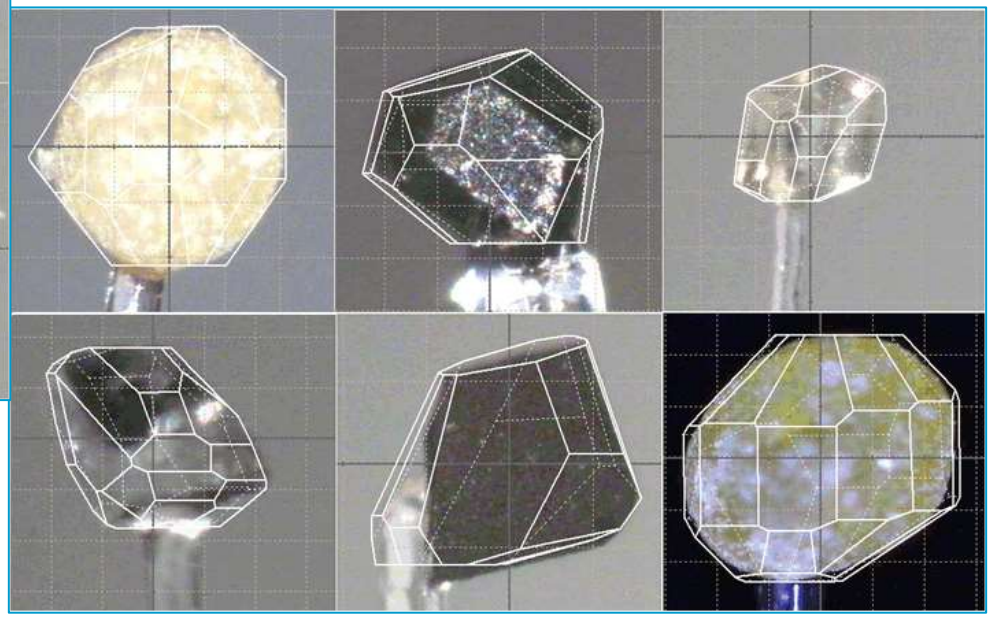


Shape building takes time… Automatic?

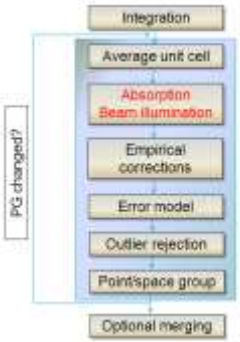


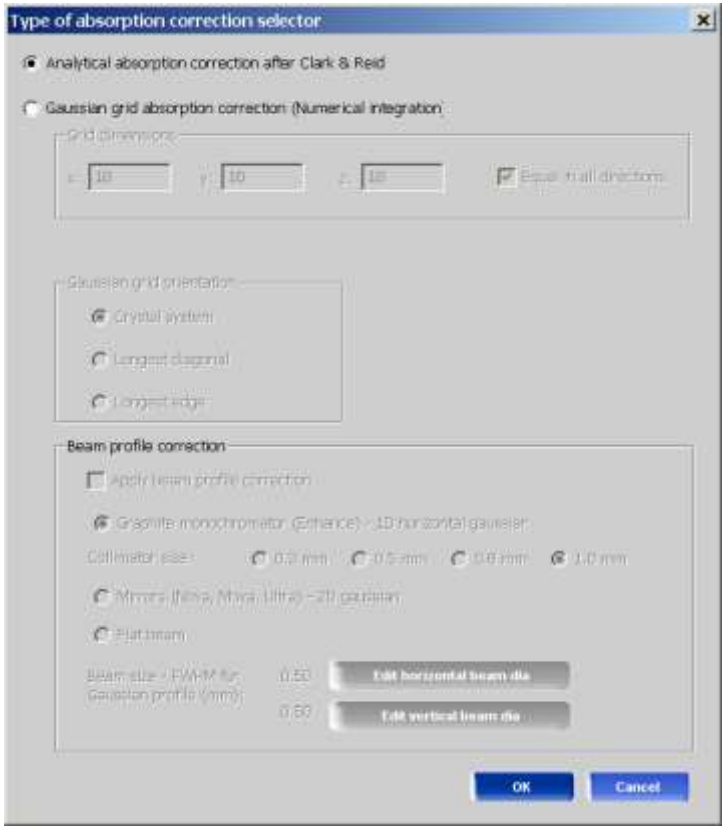
- Auto shape





‘Face-based’: Absorption, beam illumination





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

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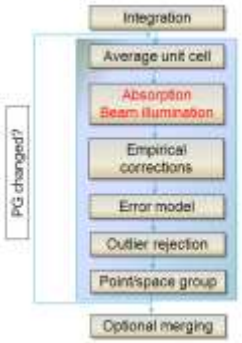
‘Face-based’: Absorption, beam illumination



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

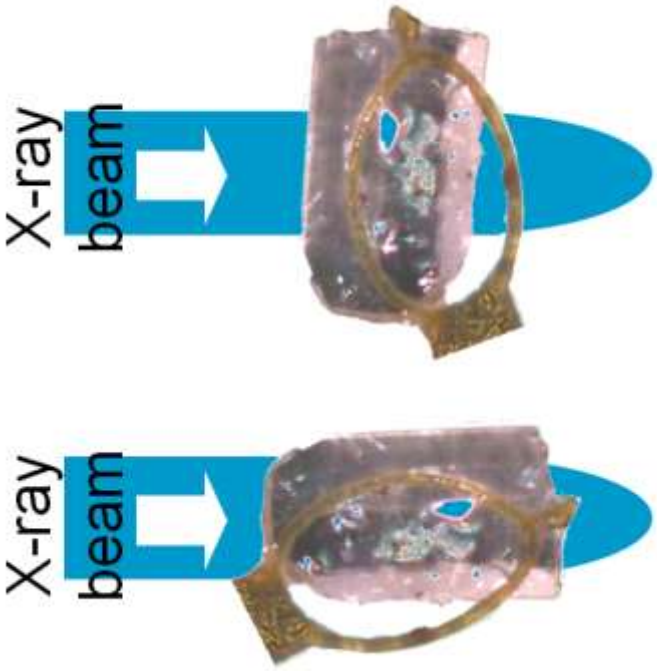
Beam profile correction

☑ Apply beam profile correction

- ○ Graphite monochromator (Enhance) - 1D horizontal gaussiar
- 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 [Edit horizontal beam dia] 0.50 [Edit vertical beam dia]

[OK] [Cancel]



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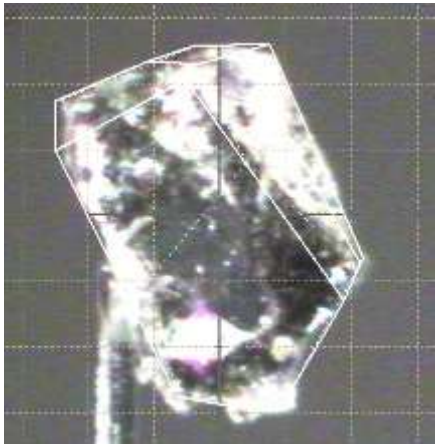
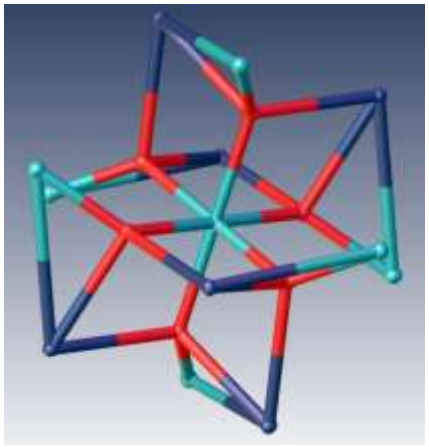
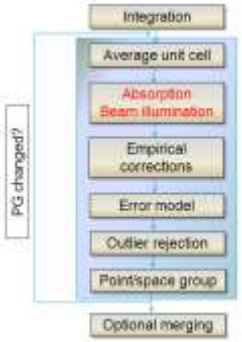
‘Face-based’: Absorption, beam illumination



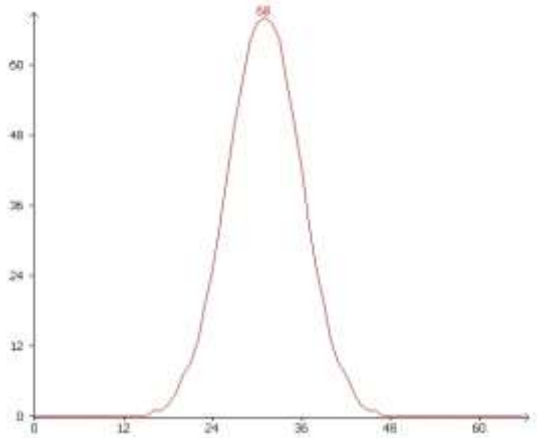




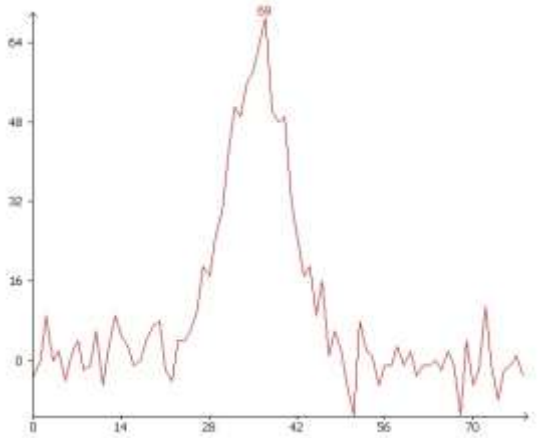
ytrium_mo – inorganic ($Y_3Al_5O_{12}$),
Mo, μ=20.3mm^{-1}, redundancy=30 (Ia-3d) , Nova micro-source

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

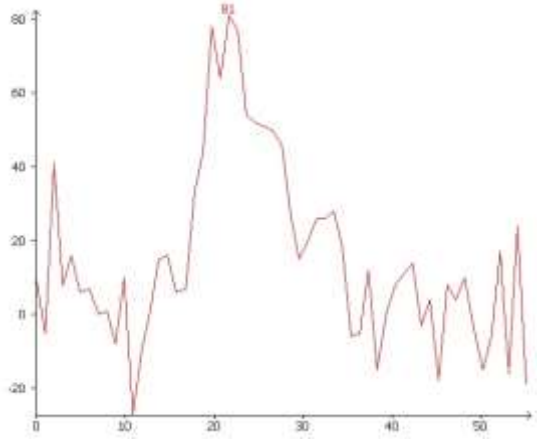
Post corrections can only correct observed data!



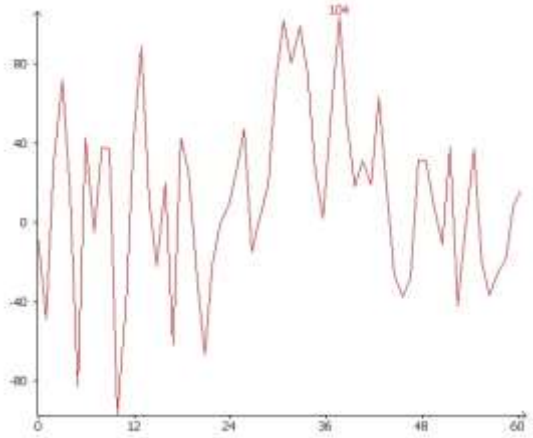
No noise - excellent



Low noise - good

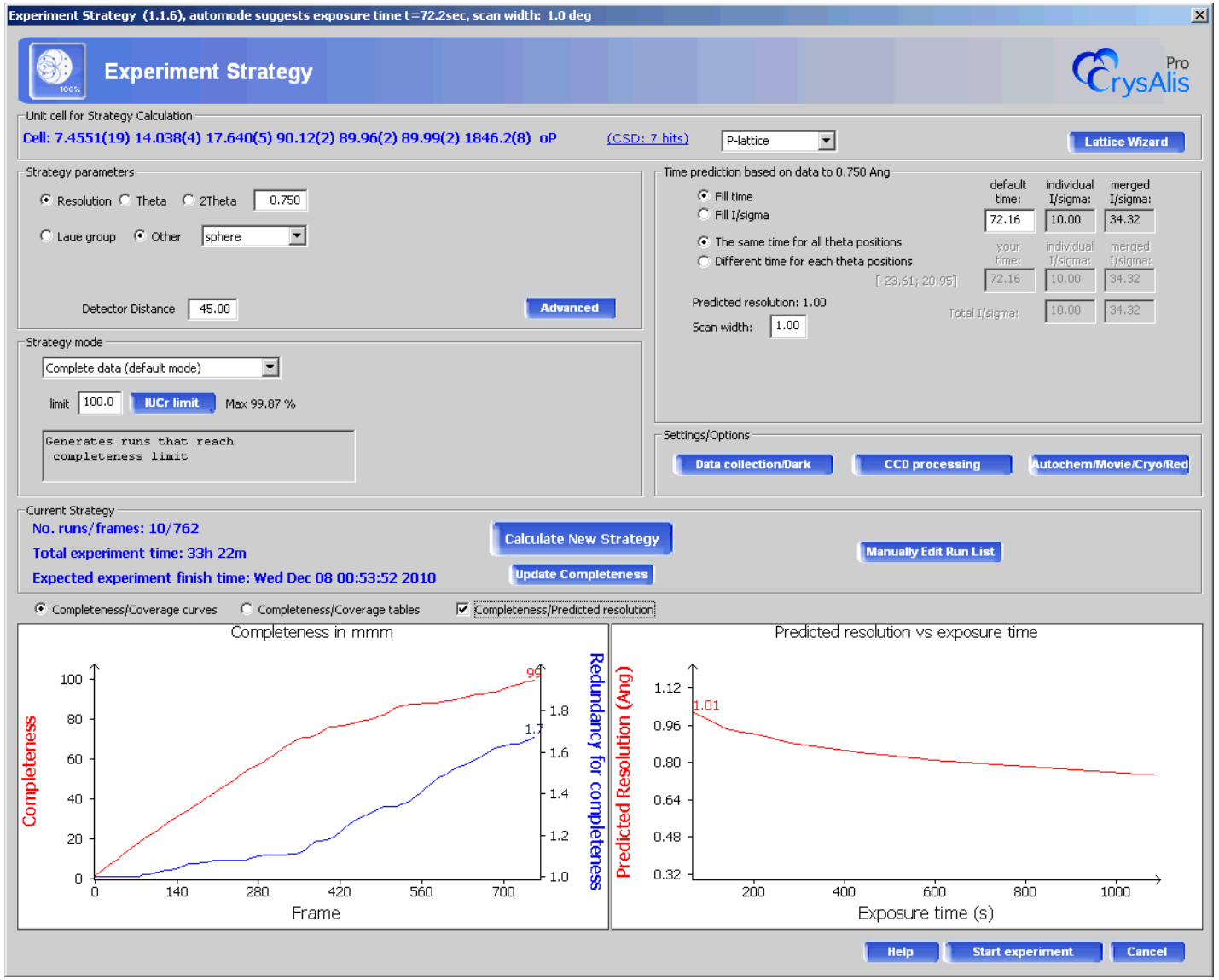


Medium noise - ok

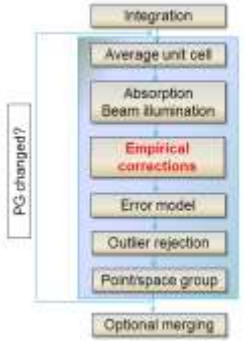


High noise – hopeless!

Strategy dilemma: redundancy vs. time



Empirical correction: logic



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

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

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

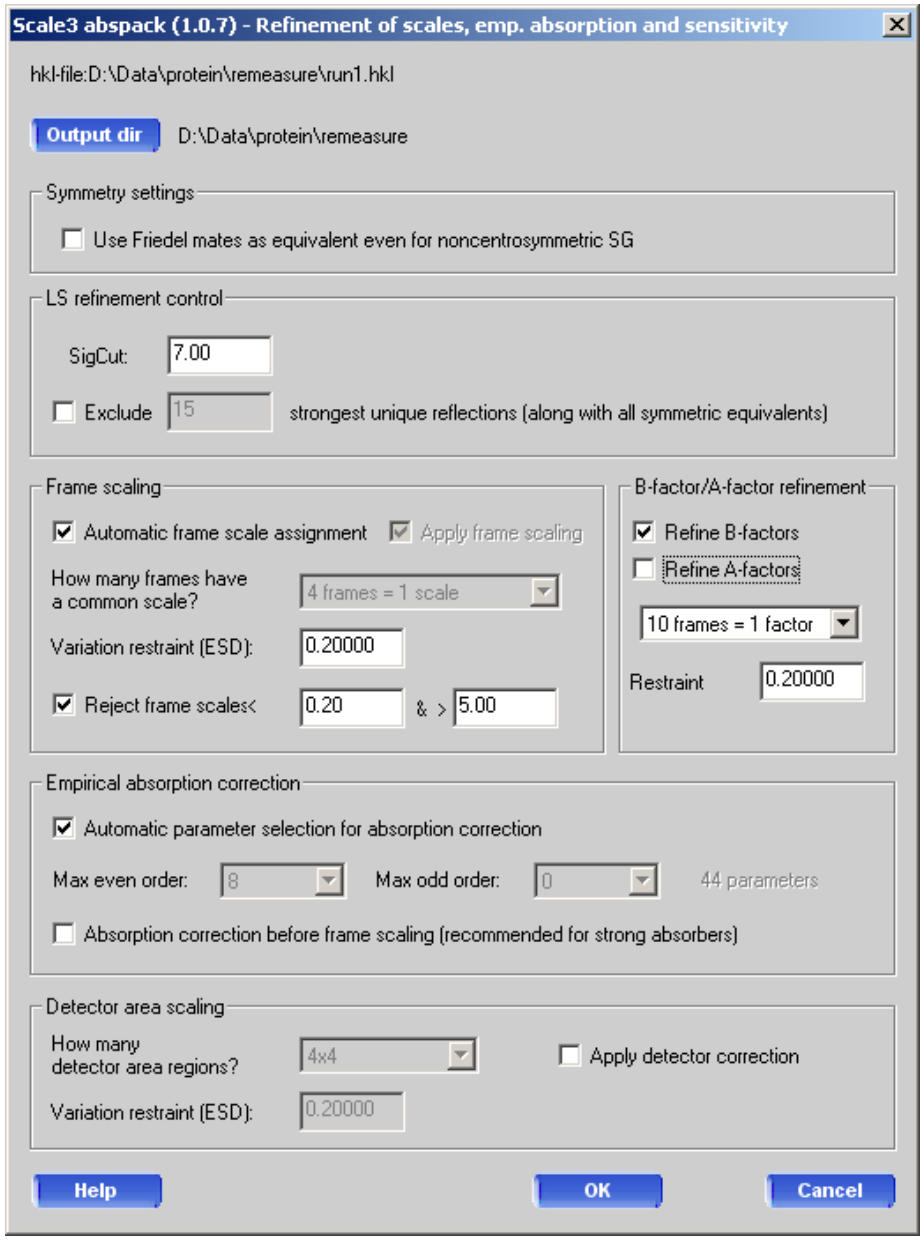
$$I_{aver} = \sum I_{corr\_prev,i}$$

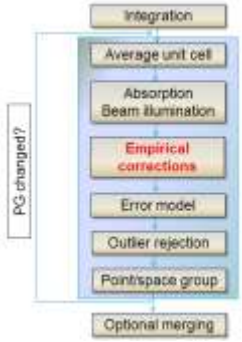
$$I_{corr,n} = C_n I_n \qquad \Delta_n = I_{aver} - I_{corr,n}$$

Note:
Redundancy is the key ingredient!

Play modes

- Scales
- Empirical
- Decay A, B
- Sensitivity





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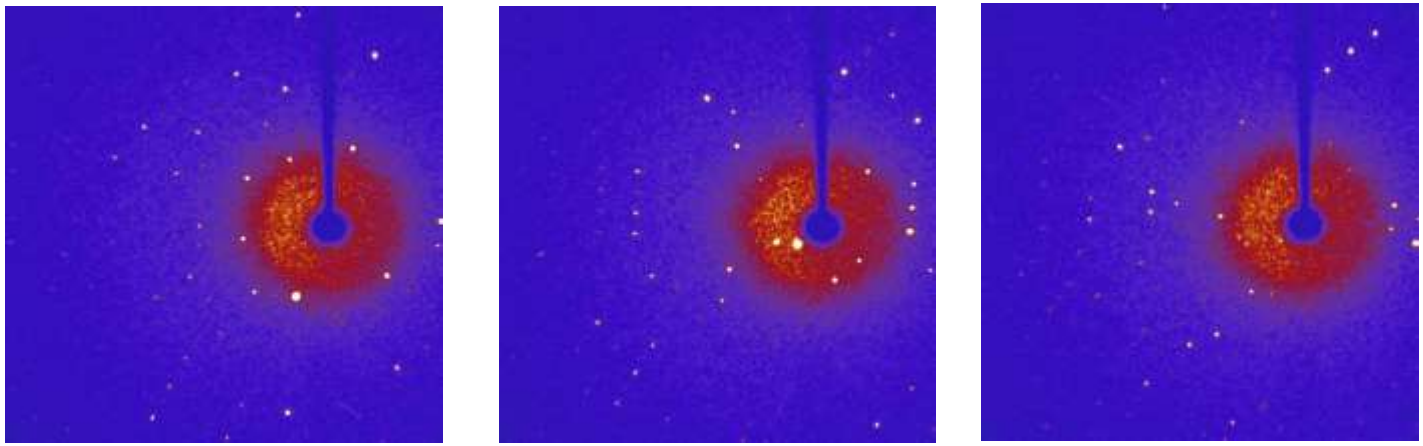
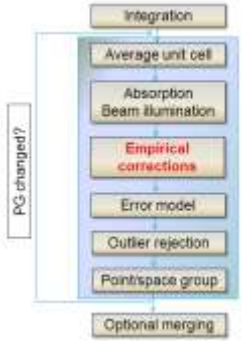
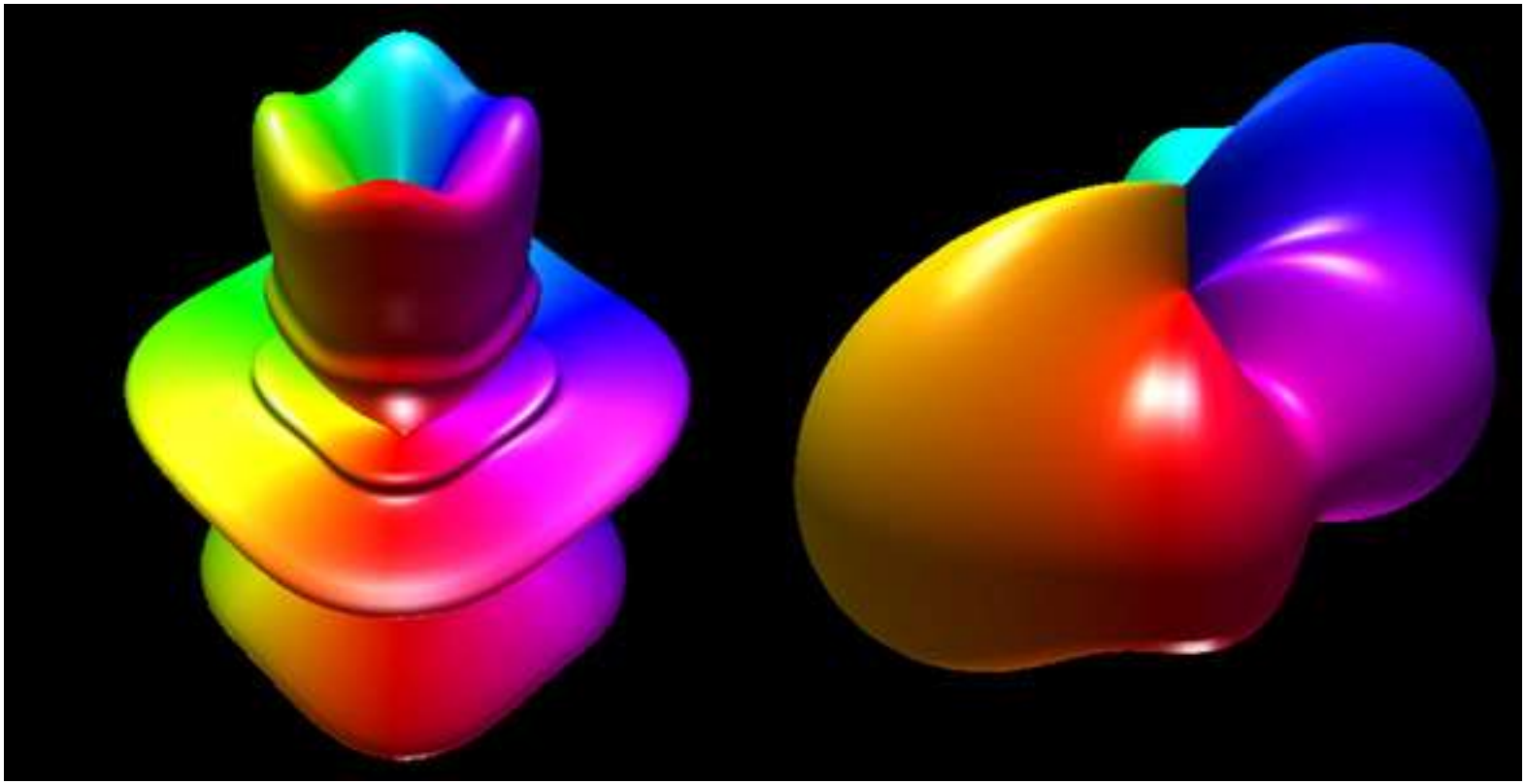
Play modes: scales

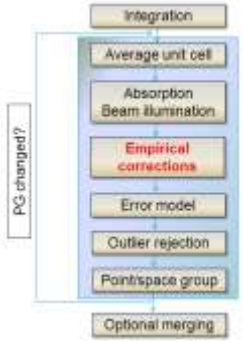


Batch



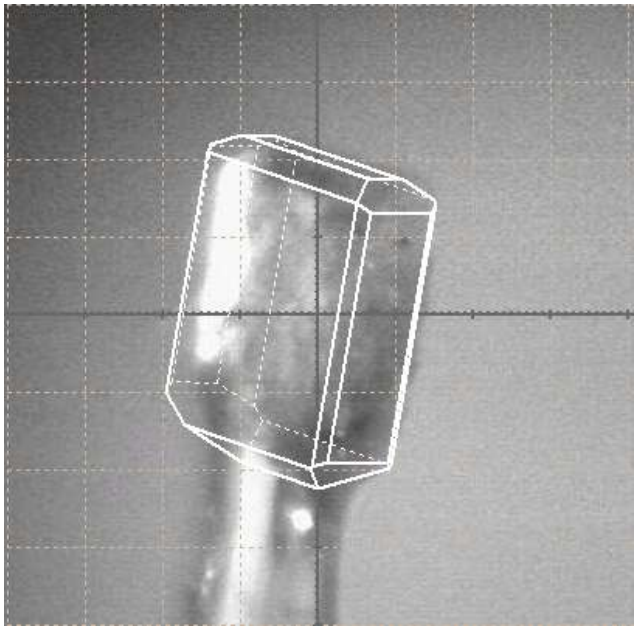
Play modes: Spherical harmonics

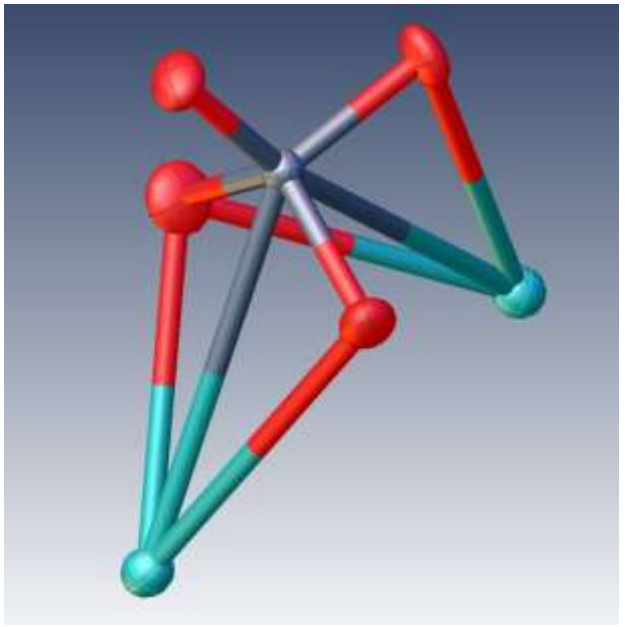


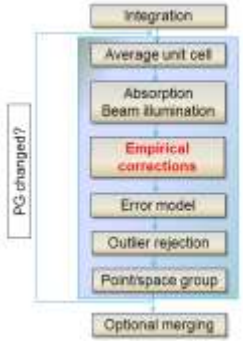


http://paulbourke.net/geometry/sphericalh/ Blessing, R.H. (1995). Acta Cryst. A51, 33-38

Empirical vs. Numeric Absorption



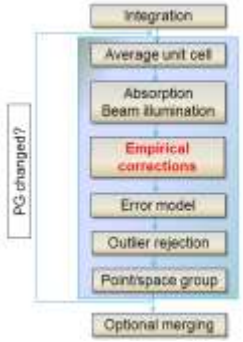




dusekdc (Michal Dusek) – inorganic (Cs_2SeO_4), Mo, μ=17.9mm^{-1}, redundancy = 4.5 ($Pna2_1$)

	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, μ=17.9mm^{-1}, redundancy = 4.5 ($Pna2_1$)			
	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?

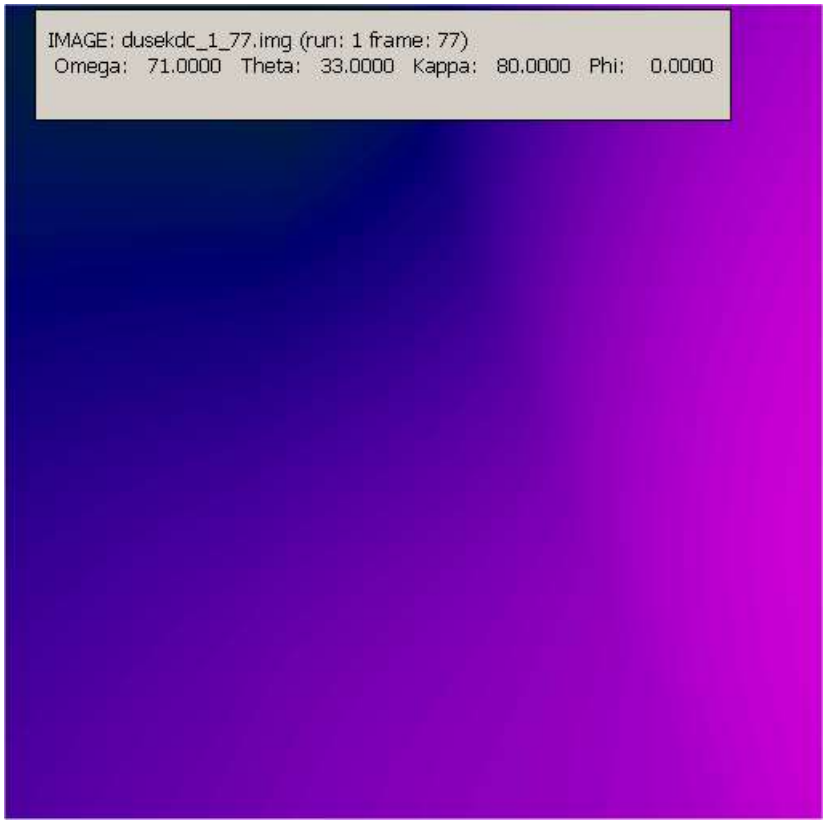
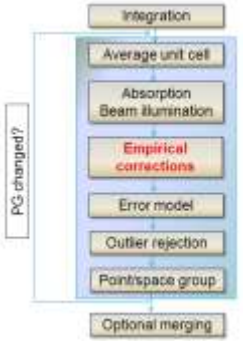
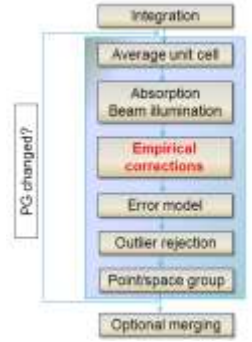


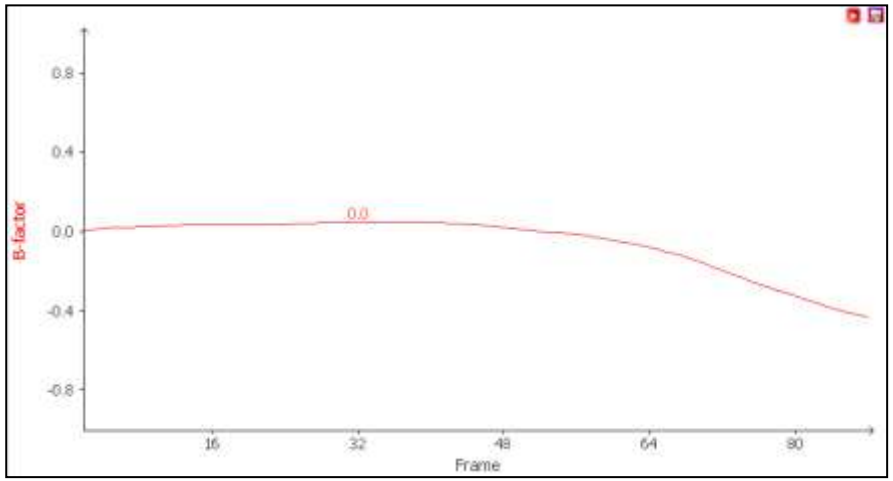
IMAGE: dusekdc_1_77.img (run: 1 frame: 77)
Omega: 71.0000 Theta: 33.0000 Kappa: 80.0000 Phi: 0.0000

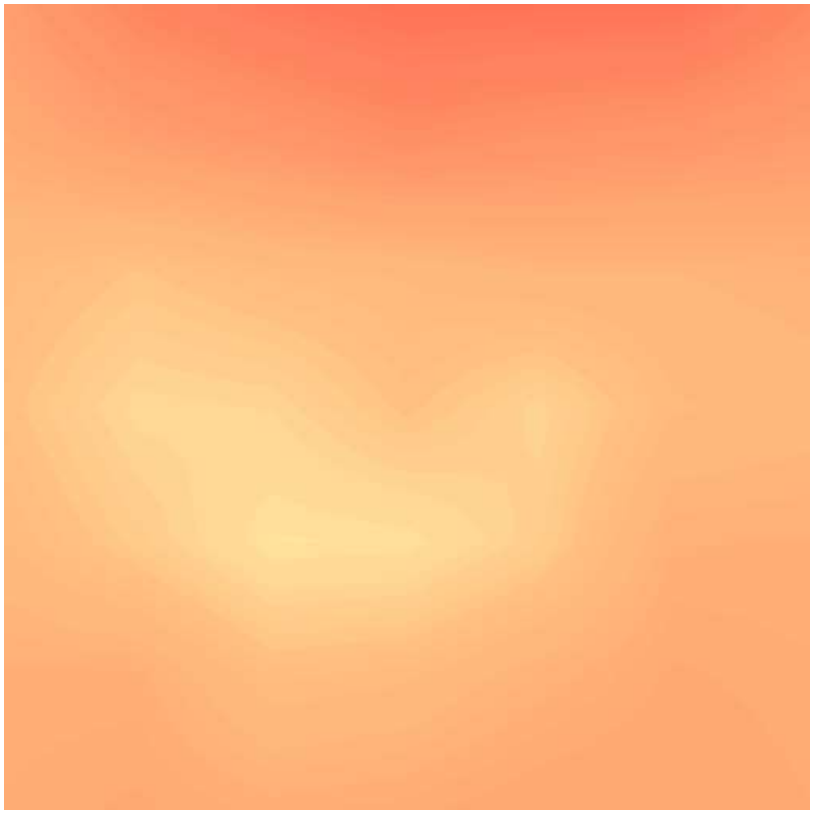

Integration
Average unit cell
Absorption
Beam illumination
Empirical corrections
Error model
Outlier rejection
Point/space group
Optional merging
PG changed?

Play modes: Decay and detector sensitivity

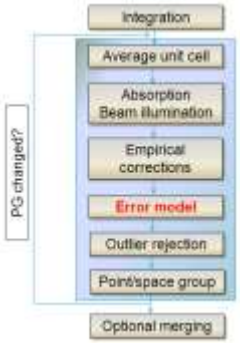
$$K_B = e^{-2\left[B_i\left(\frac{sin\theta}{\lambda}\right)^2 + A_i\left(\frac{sin\theta}{\lambda}\right)\right]}$$

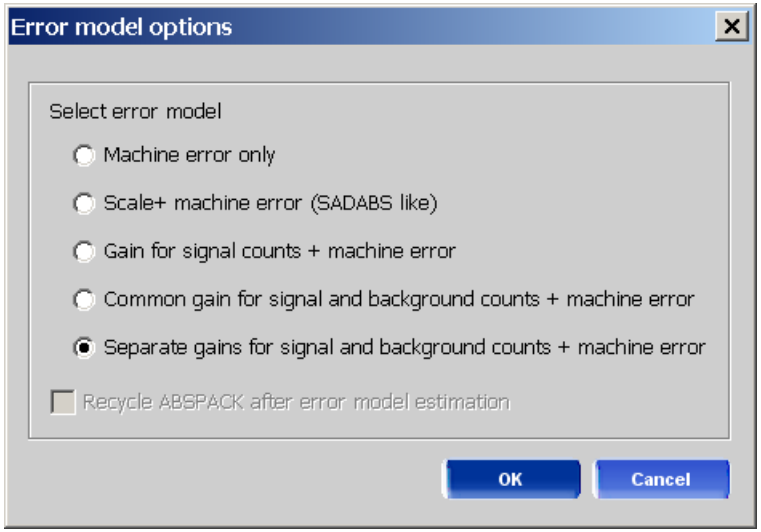


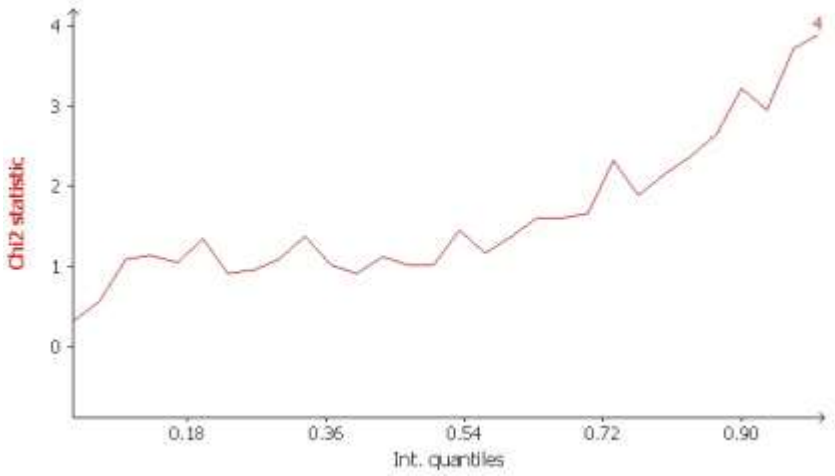


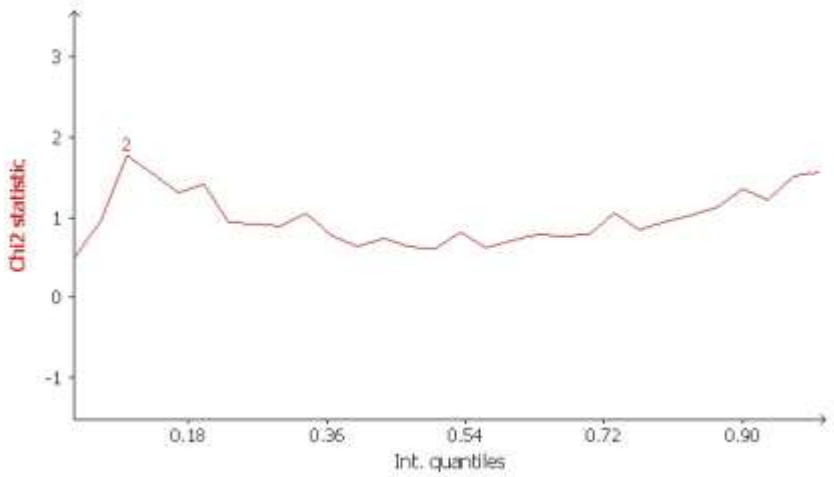


Error model fitting

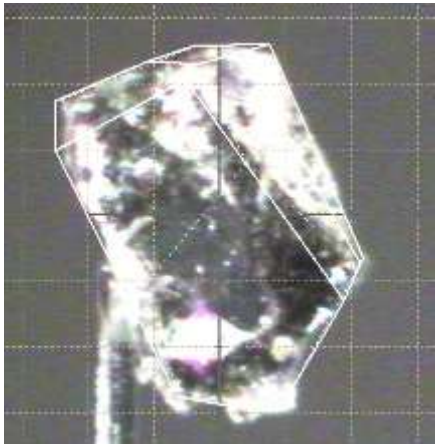


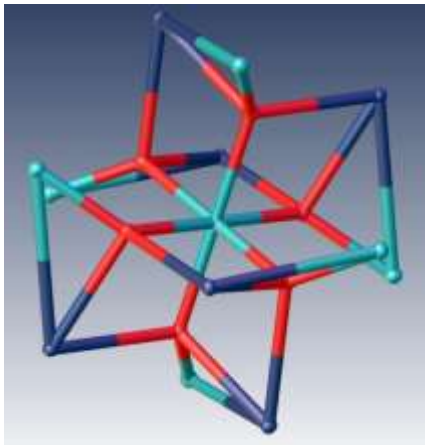


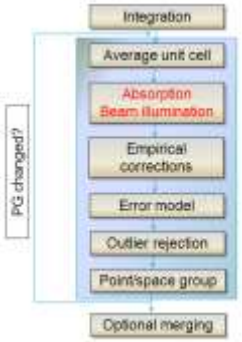




All corrections together…





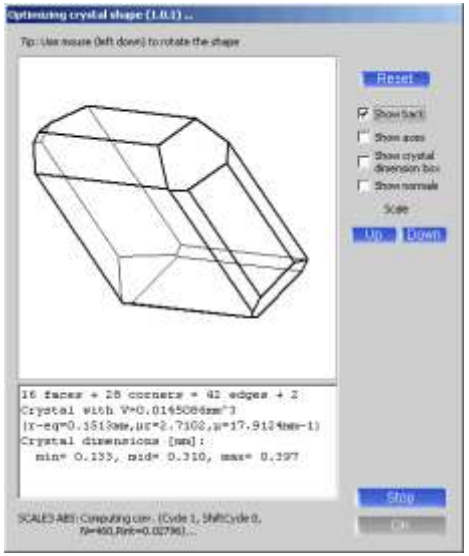


ytrium_mo – inorganic ($Y_3Al_5O_{12}$),
Mo, μ=20.3mm^{-1}, redundancy=30 (Ia-3d) , Nova micro-source

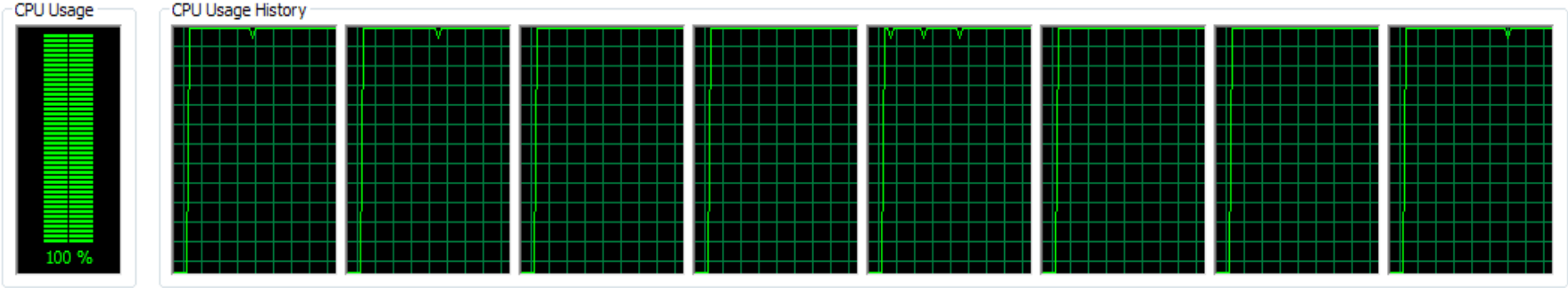
	R_{int}	I/σ	R1
No correction	50%	7.8	8.9%
Empirical	35%	8.5	7%
Absorption + beam illumination	20%	9.8	3.4%
Abs. + beam ill. + empirical	13.6%	11.6	2.4%

Multi – core

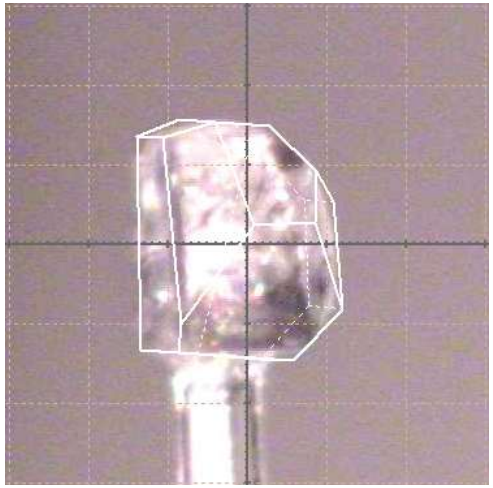
- All absorption and beam illumination computation
- All empirical corrections
- Auto shape generation
- Shape optimizer
- All matrix operations for Least squares

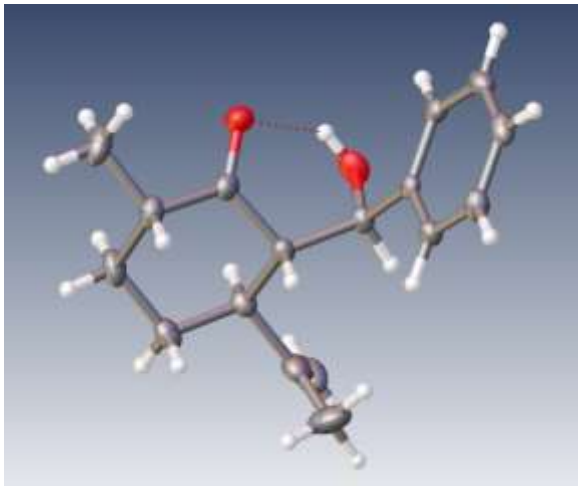


- What a blessing…



A very normal Cu data set

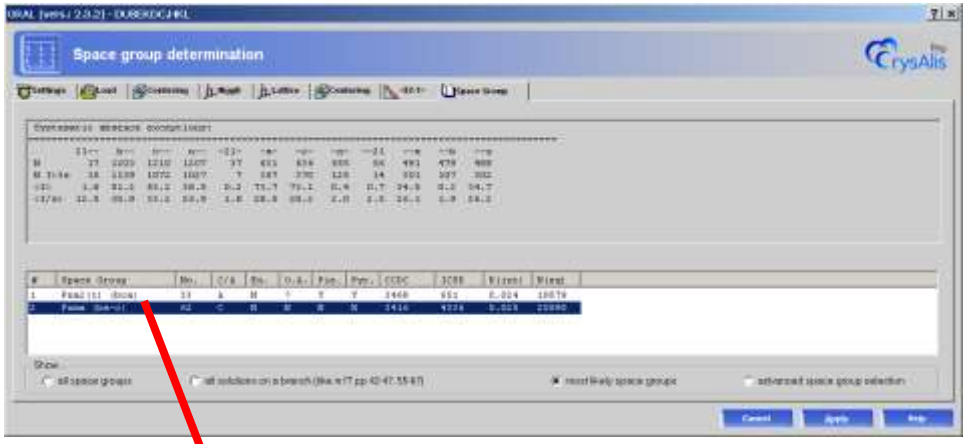




Schott (Peter Jones) – organic (C_{17} H_{22} O_2),
Cu, μ=0.58mm^{-1}, redundancy=7.4 ($P2_12_12_1$)

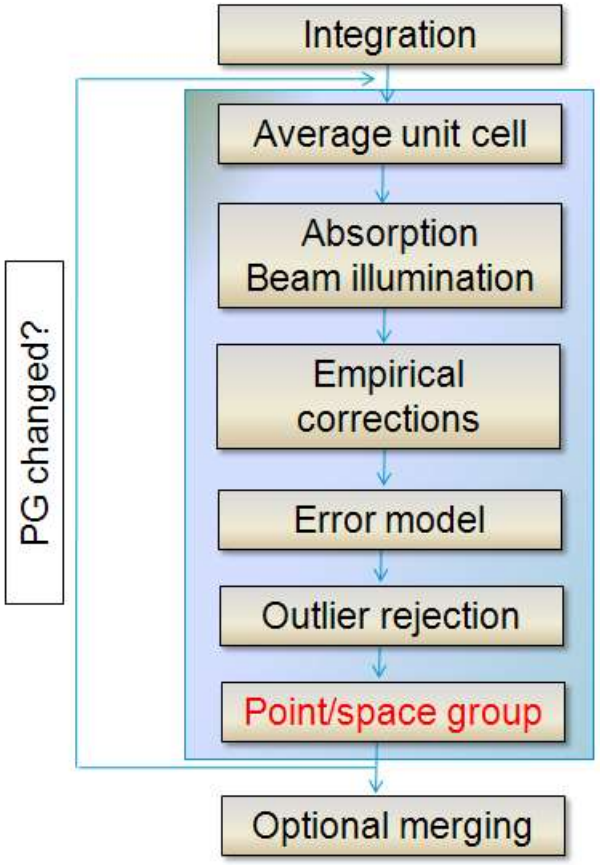
	R_{int}	I/σ	R1
No correction	10%	23	3.73%
Empirical	2%	34	2.93%
Empirical + Numeric	2%	34	2.93%

Proper space group for empirical corrections

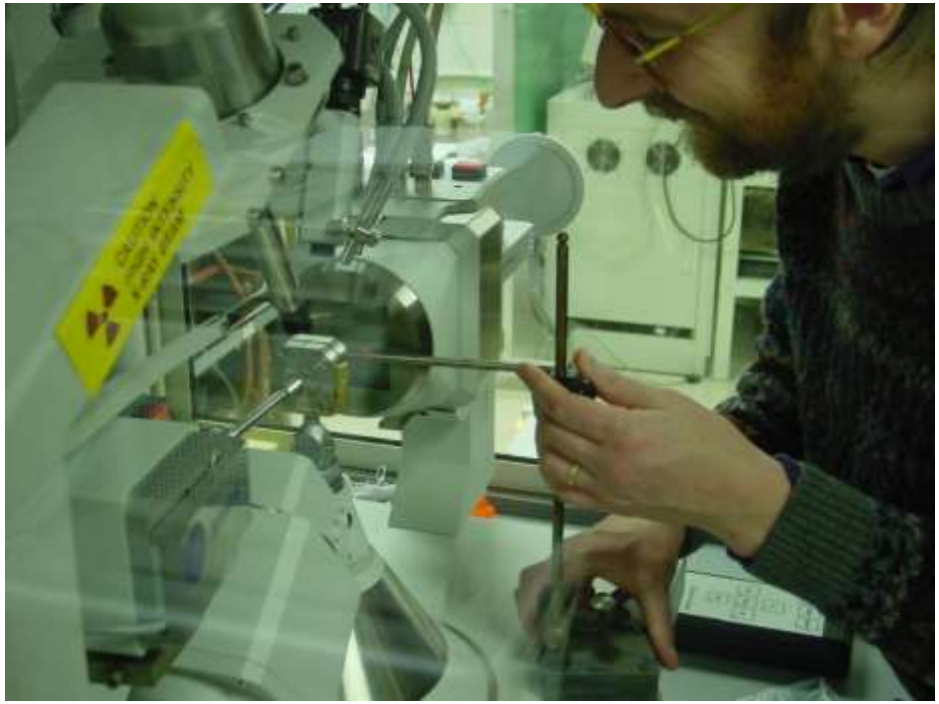


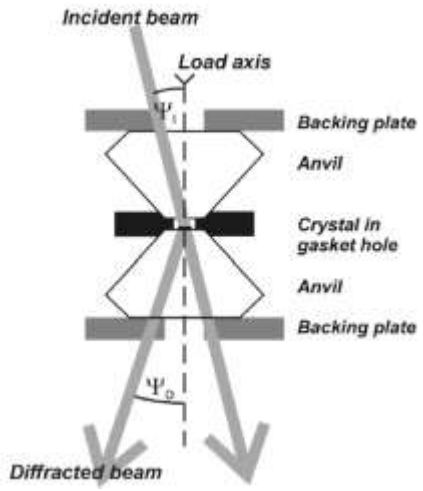
#	Space Group	No.	C/A	En.	O.A.	Pie.	Pyr.	CCDC	ICSD	R(int
1	Pna2(1) (bca)	33	A	N	?	Y	Y	3468	651	0.024
2	Pnma (ba-c)	62	C	N	N	N	N	3416	4336	0.025

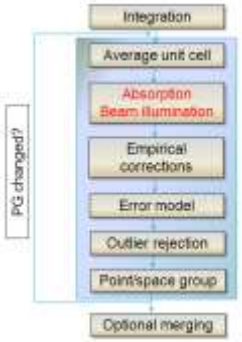
Show ...
- all space groups
- all solutions on a branch (like in IT pp 42-47, 55-67)



‘Face-based’: Diamond anvil cell correction



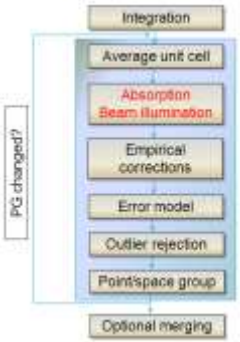


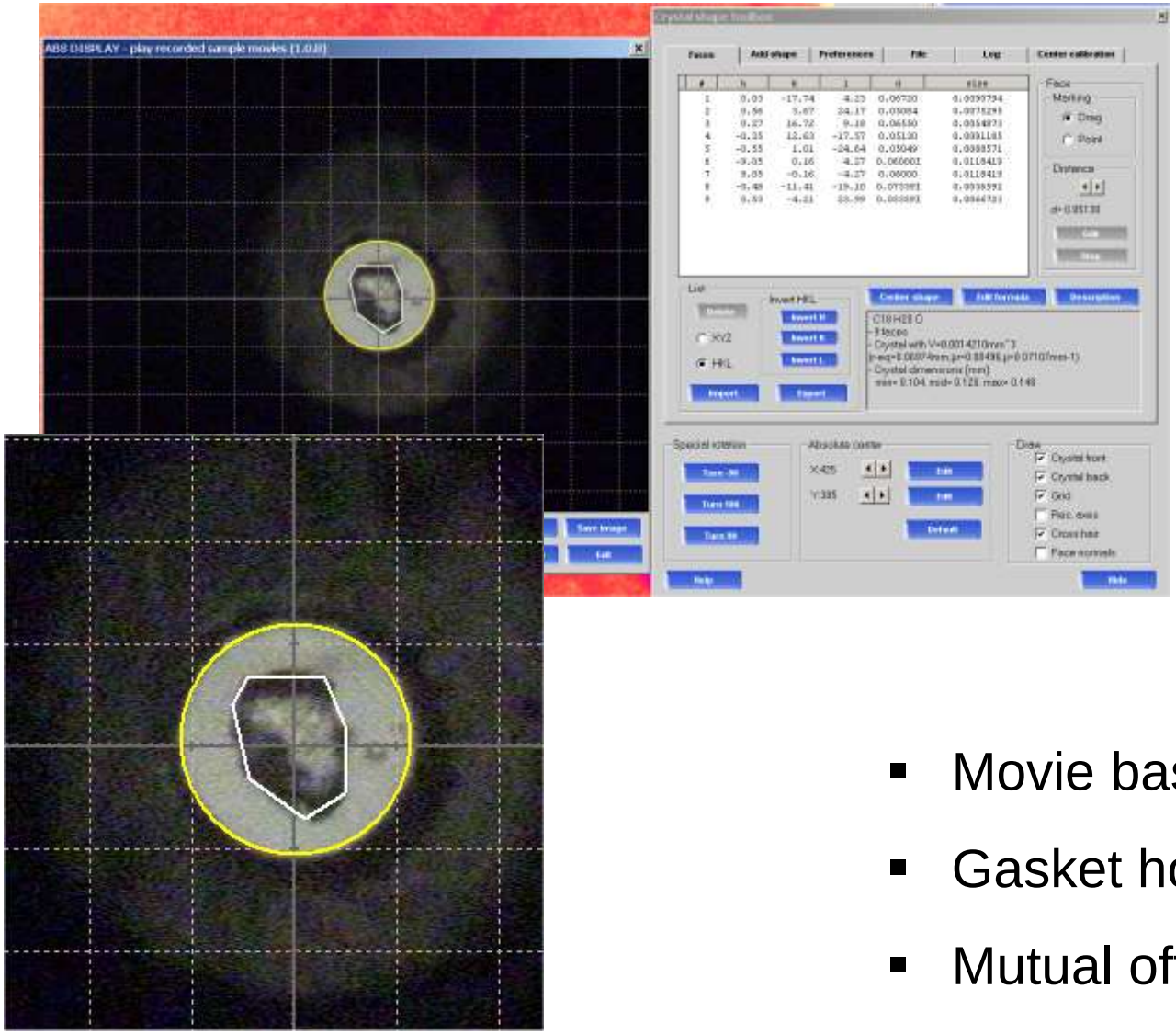


- Absorption
- Gasket shadowing
- Backing plates

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

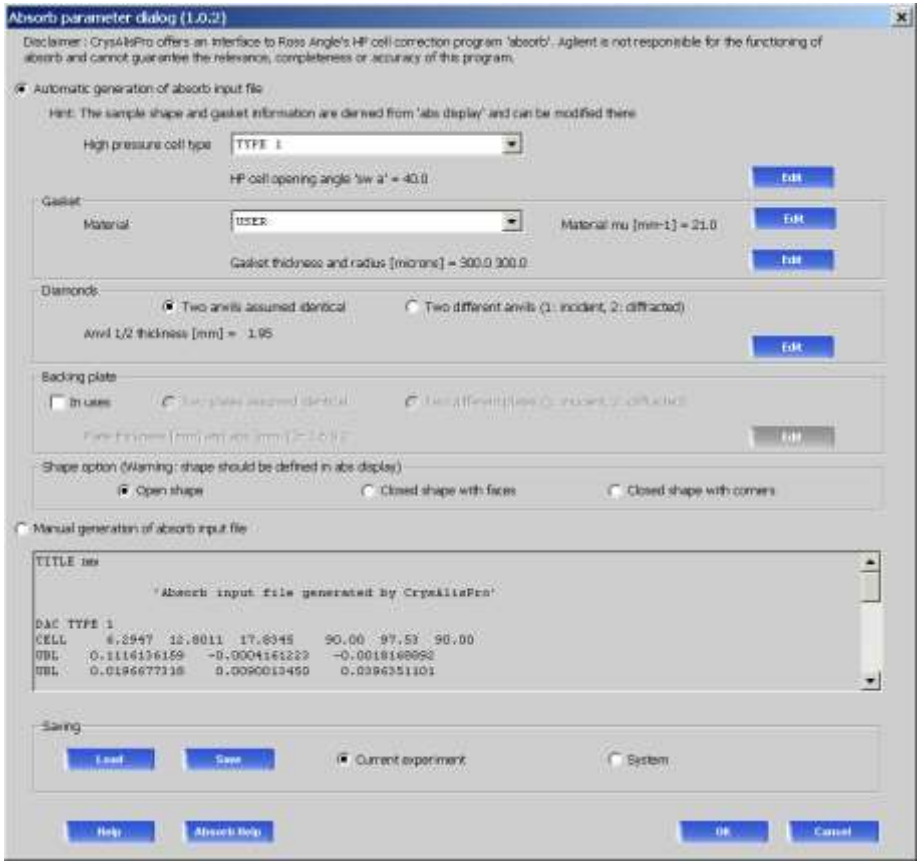
‘Face-based’: Diamond anvil cell correction

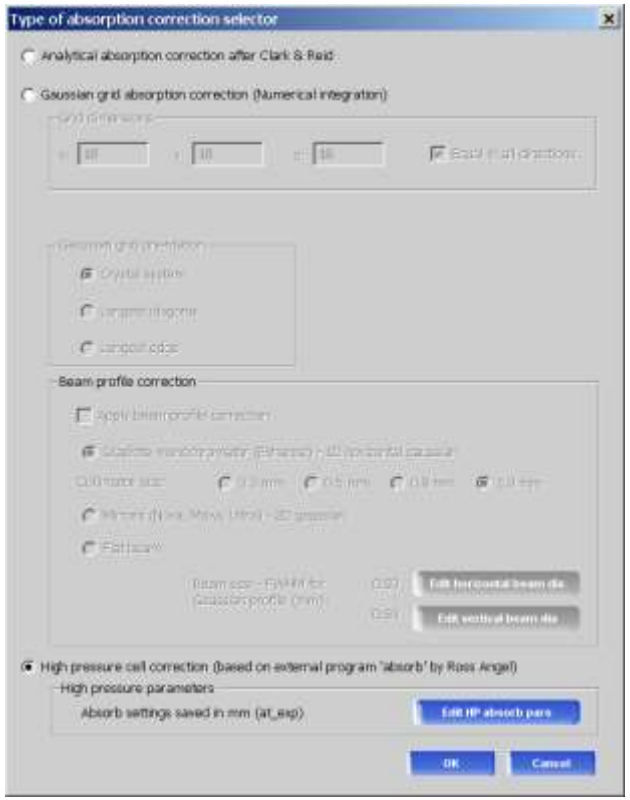


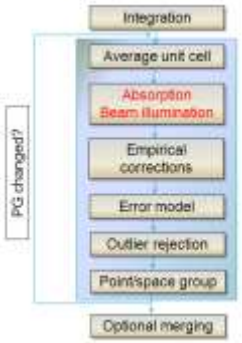


- Movie based sample shape
- Gasket hole
- Mutual offsets

‘Face-based’: Diamond anvil cell correction

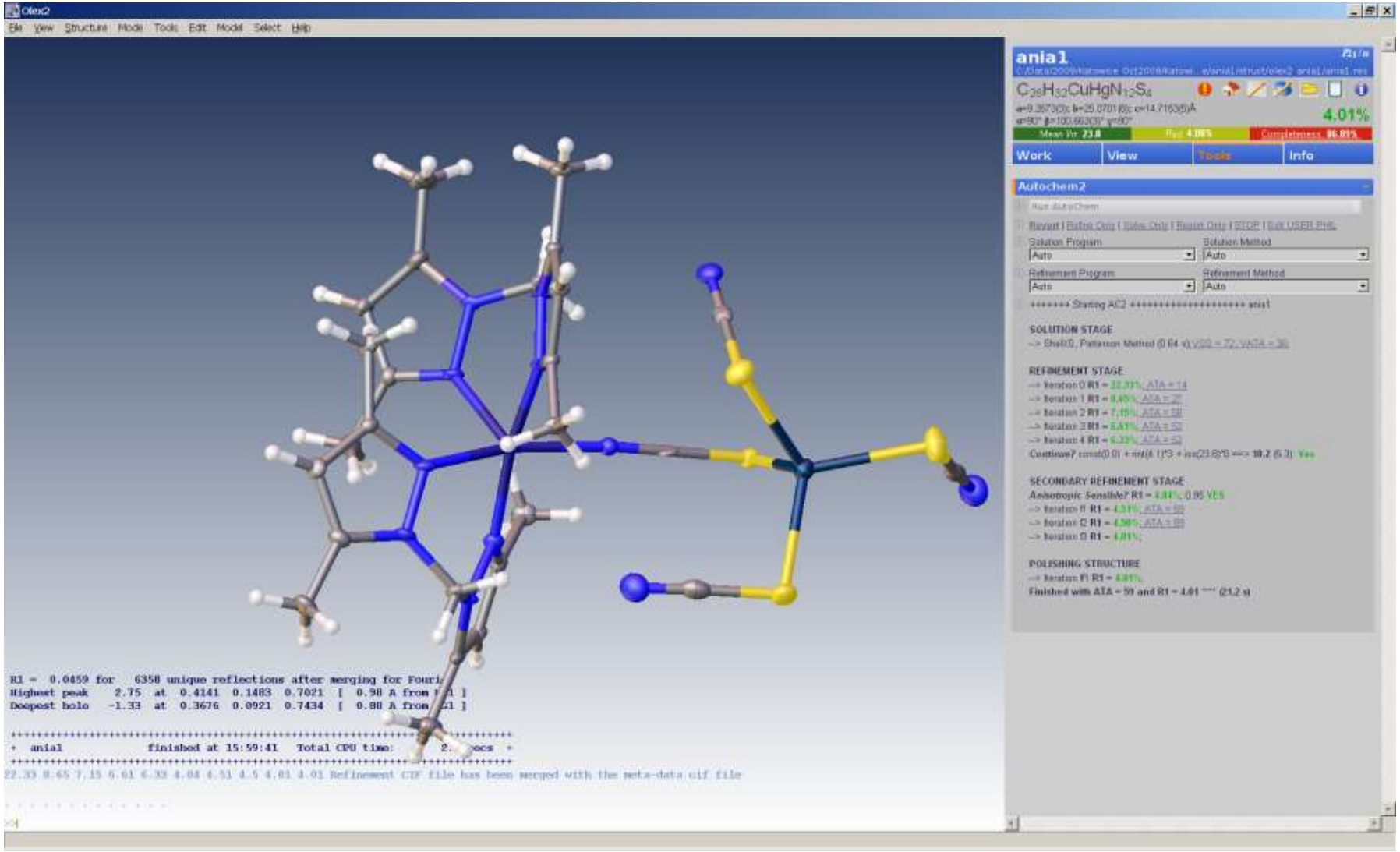






- Automatically generated input files for Absorb 7.0
- Fully integrated to workflow

Integration with external programs



Agilent Technologies

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 (μ>5mm^{-1}) numeric face based corrections are very necessary. Take the effort!
- For samples with μ<5mm^{-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…

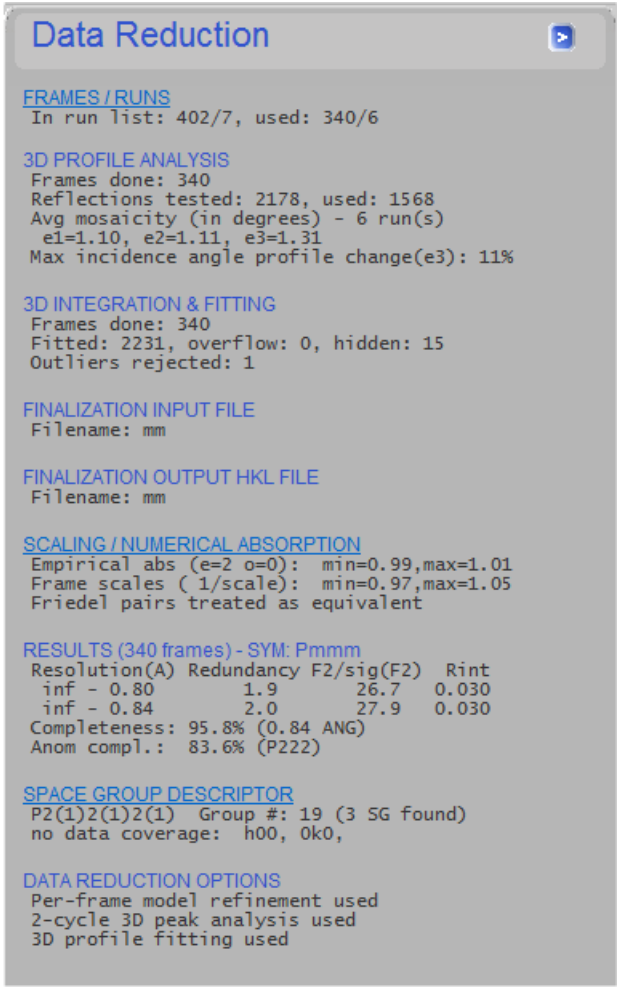
- Experiment
- Unit cell
- Data reduction
- Finalization
- Pseudo symmetry, twinning, incommensurate

Approach a data set…

- Inspect executive tab

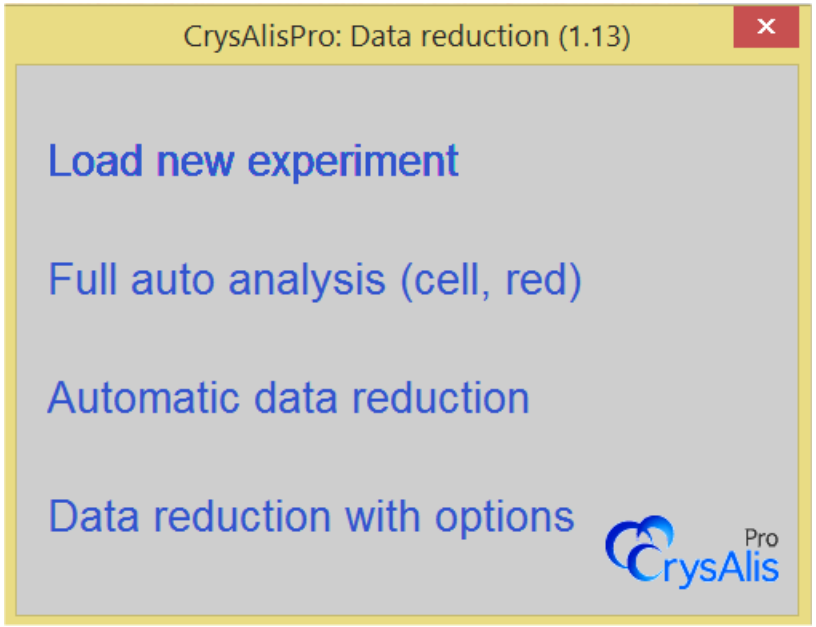
- Warning signs:

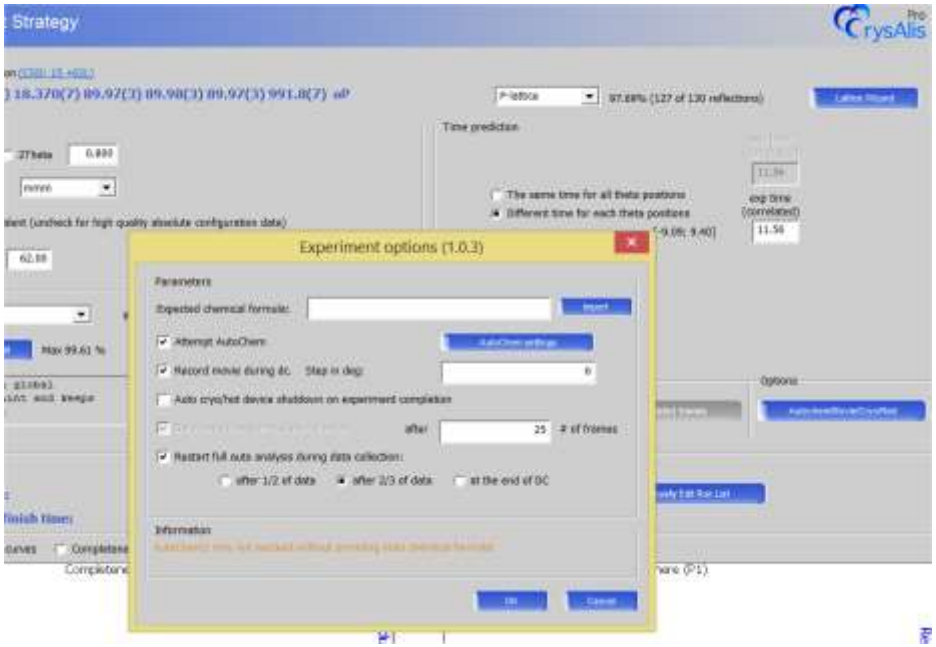
- Run list incomplete
- High mosaicity
- Scaling unusual
- I/sig low; low redundancy
- SG issues



Approach a data set…

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





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

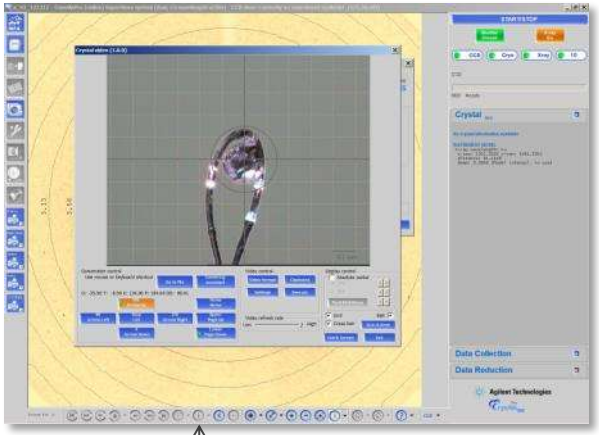
Optimal data – hints at data reduction time

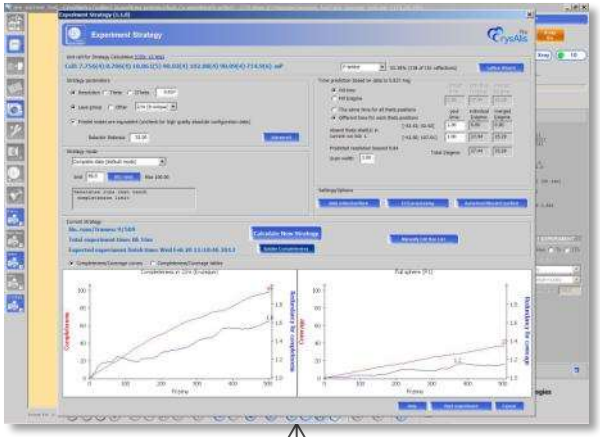
- Check for experiment artifacts (empty frames, trips)
- Apply Bravais lattice where obvious
- Special pars
  - Use bad reflection filter
  - Use reduced profile size if overlapping
  - Incidence correction, prediction accuracy
- Smart background on high background data
- xx proffitloop

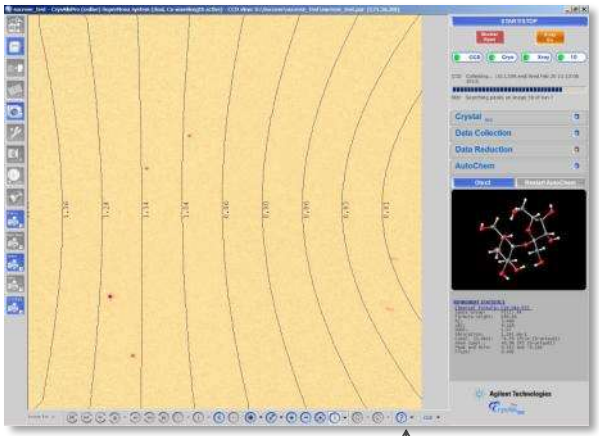
Optimal data – hints at data finalization time

- 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

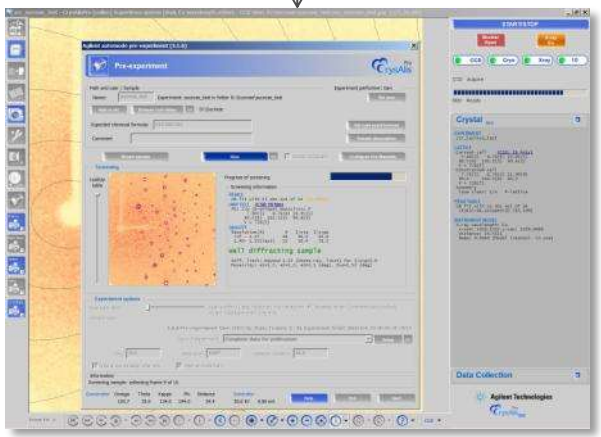
Typical Work-Flow

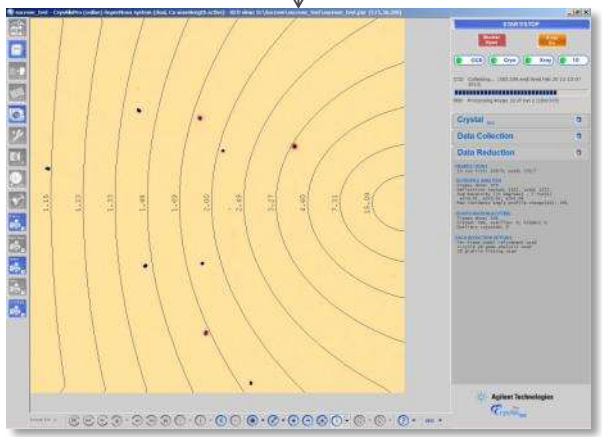






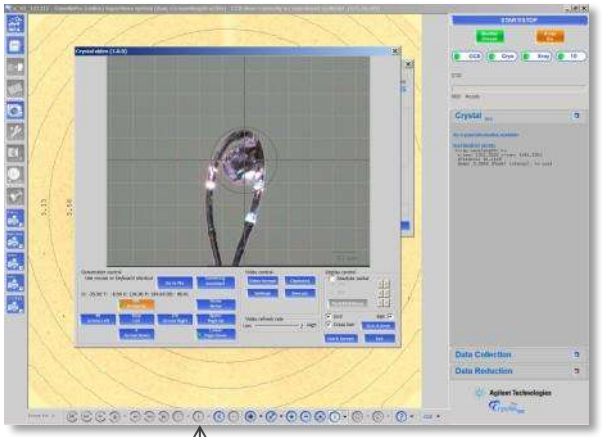
Mounting → Screen/Pre → Strategy → Experiment → Structure

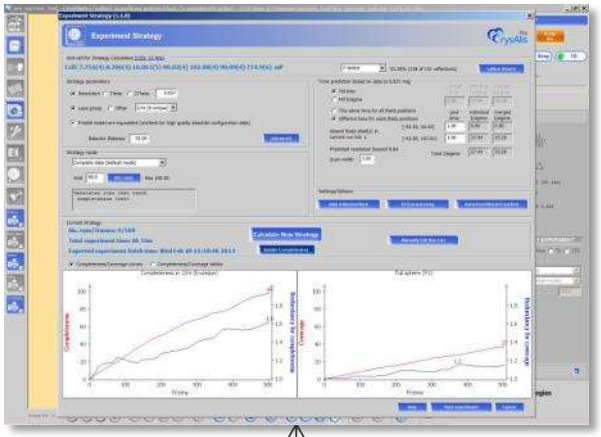


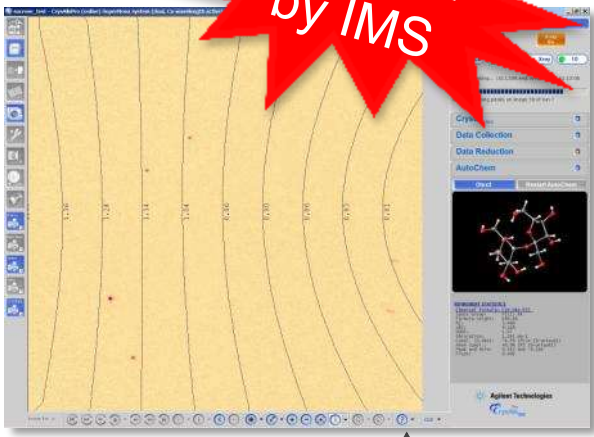


Typical Work-Flow

Enabled by IMS

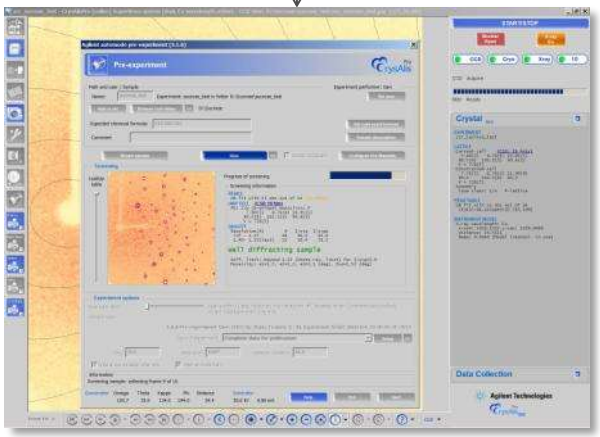


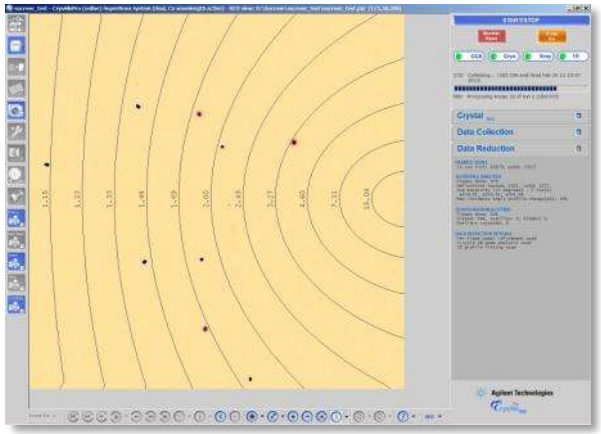




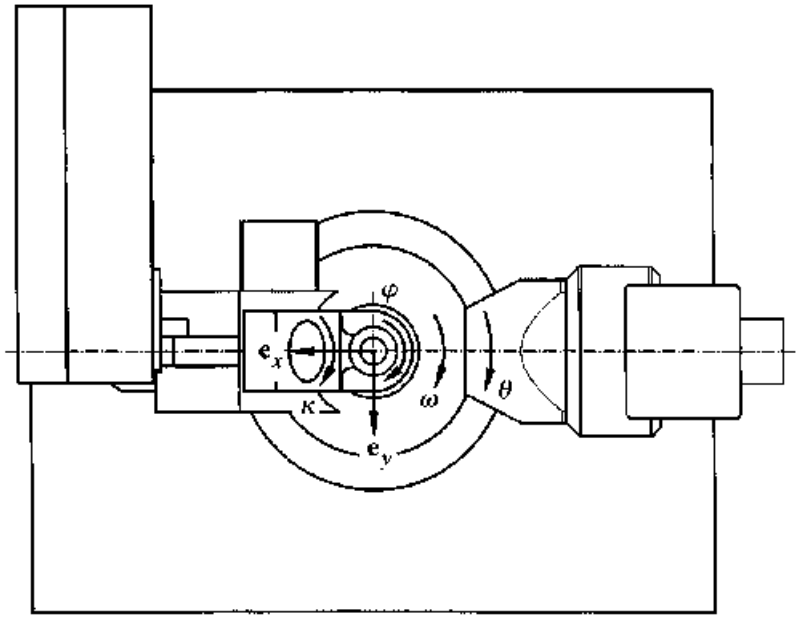
Mounting → **Screen/Pre** → **Strategy** → **Experiment** → **Structure**

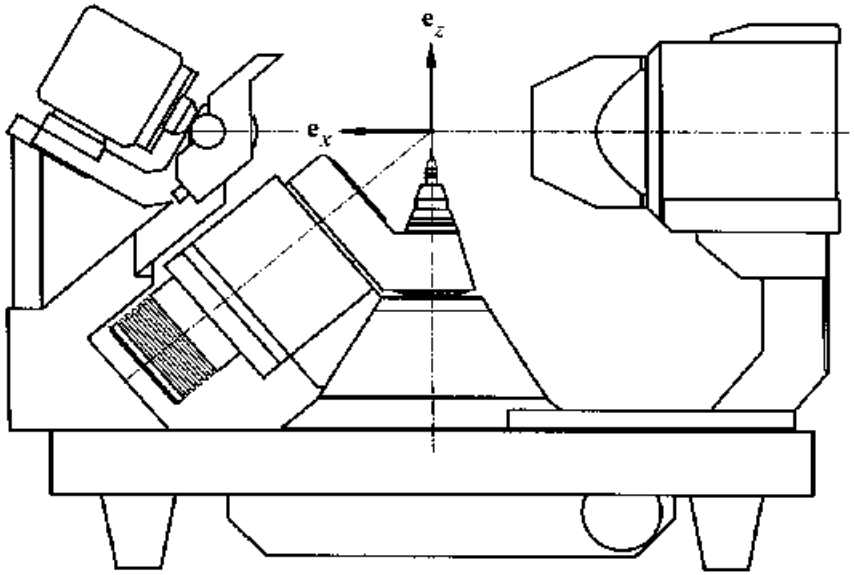
<1min | 10-60s | 1-15min | <30s | 15min-3d | concurrent



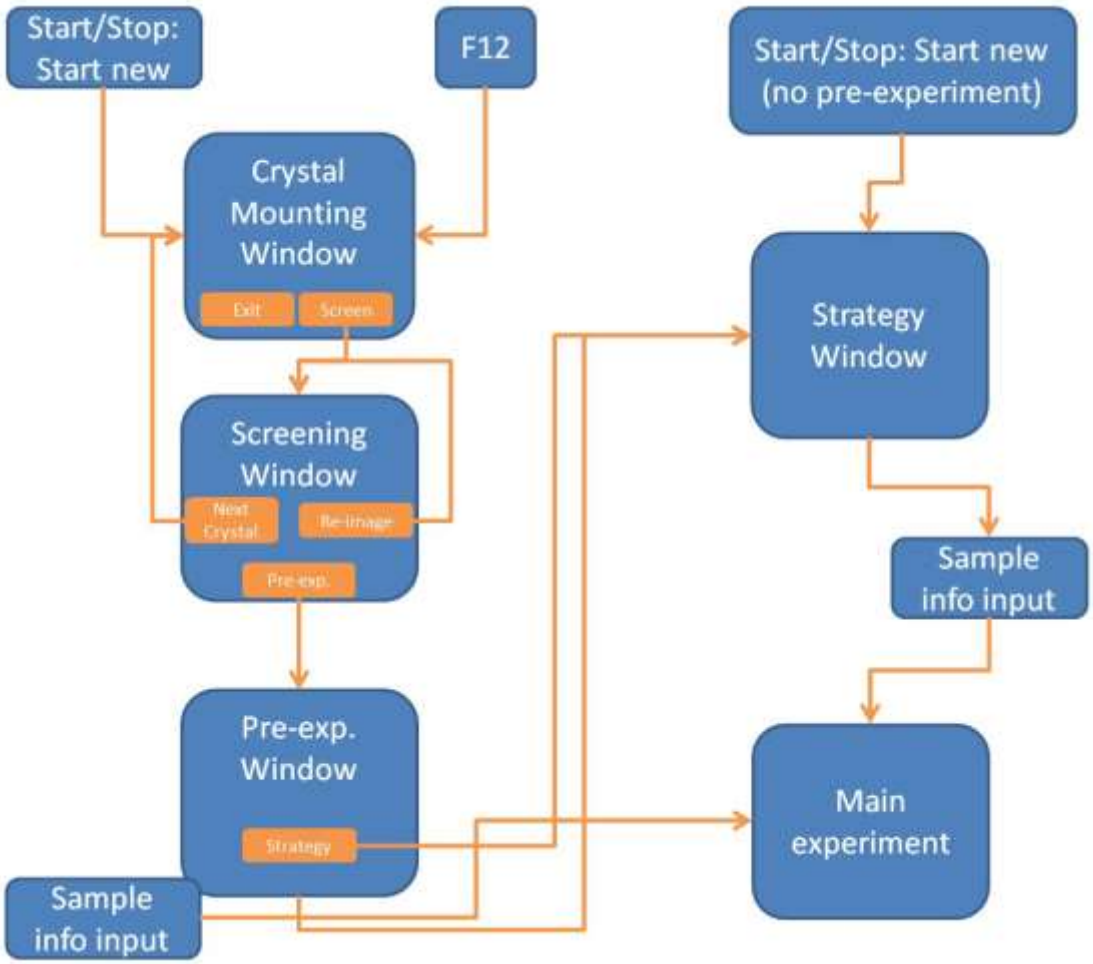


Excursion - instrument





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: 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/sigo
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

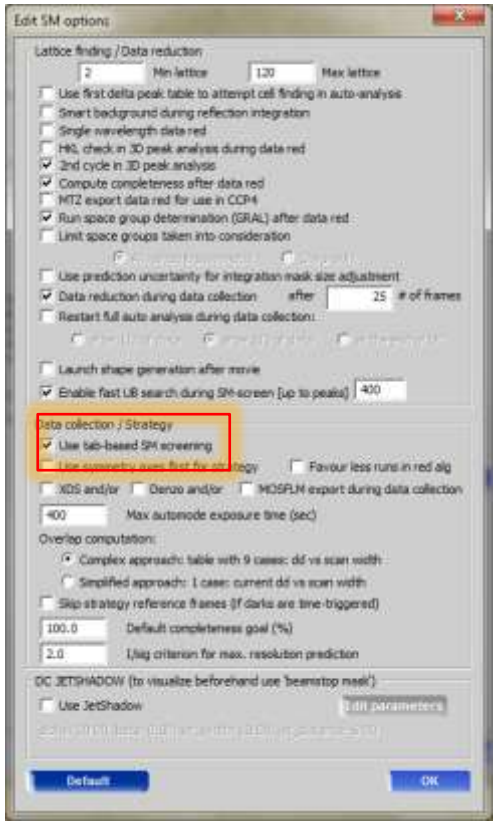
Goniometer

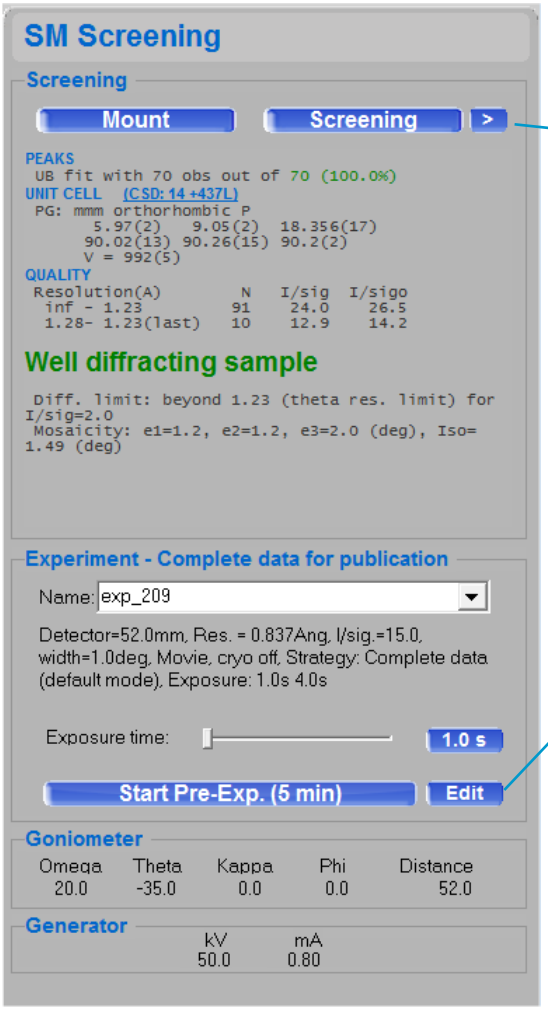
Omega	Theta	Kappa	Phi	Distance
20.0	-35.0	0.0	0.0	52.0

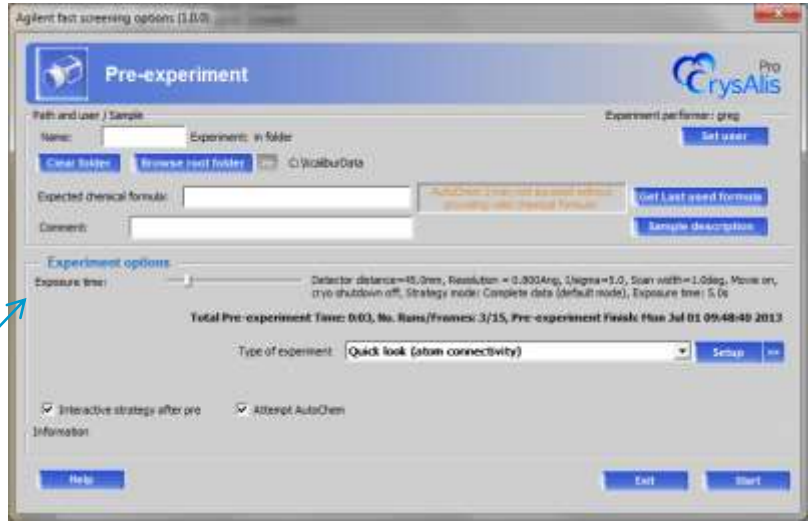
Generator

kV	mA
50.0	0.80

Tab Based Screening 2







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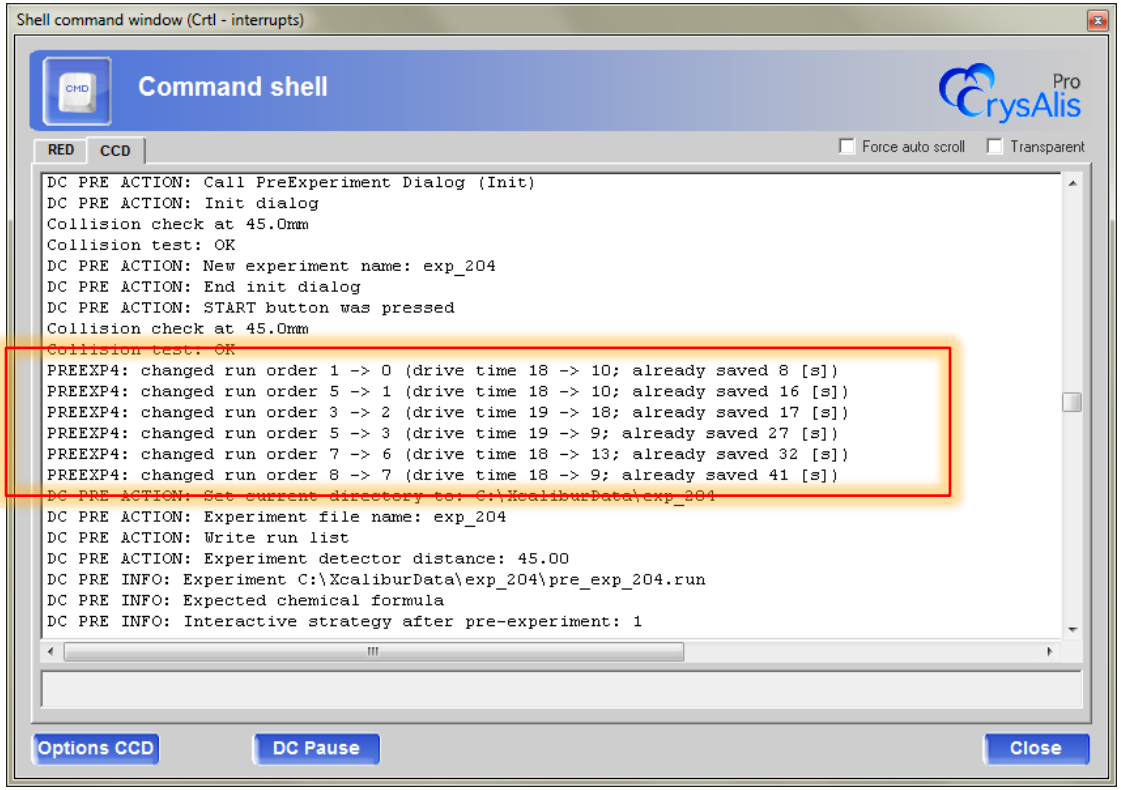
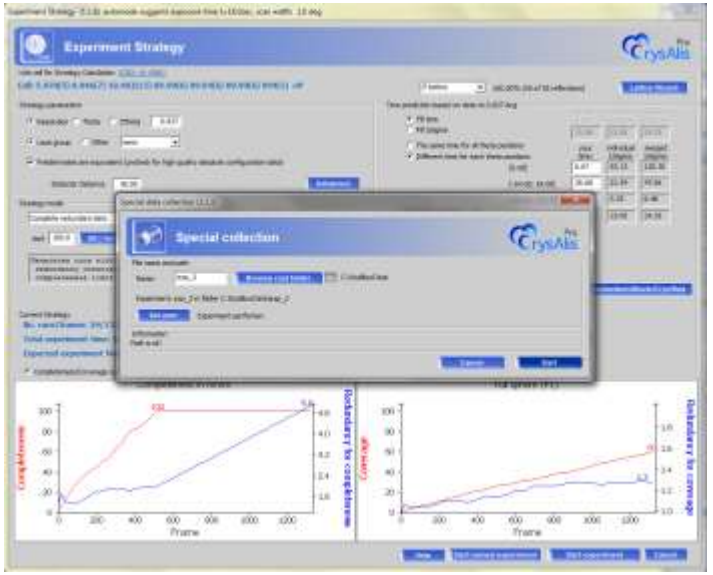
Tab Based Screening 3

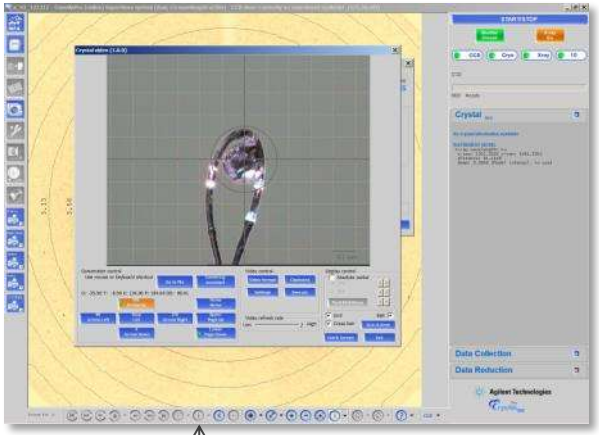


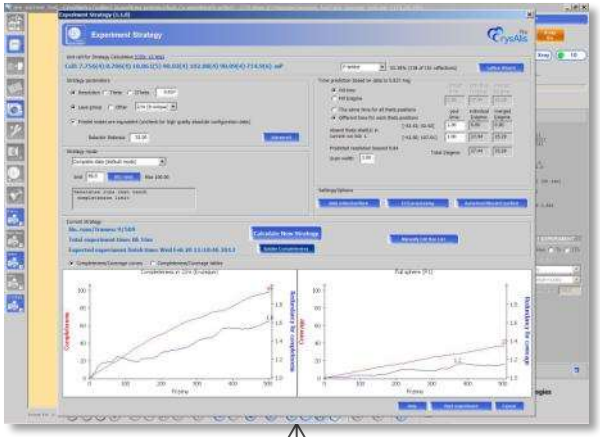


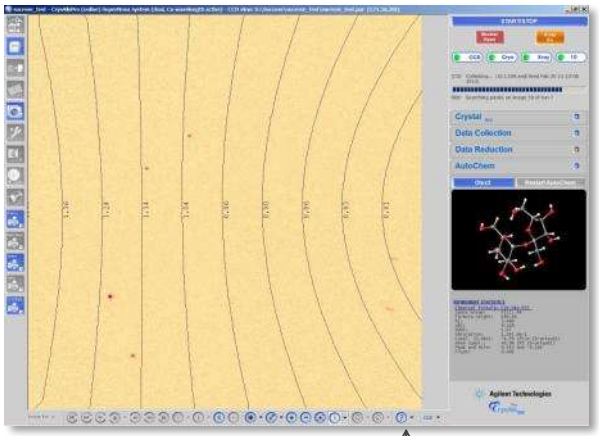
Screening

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

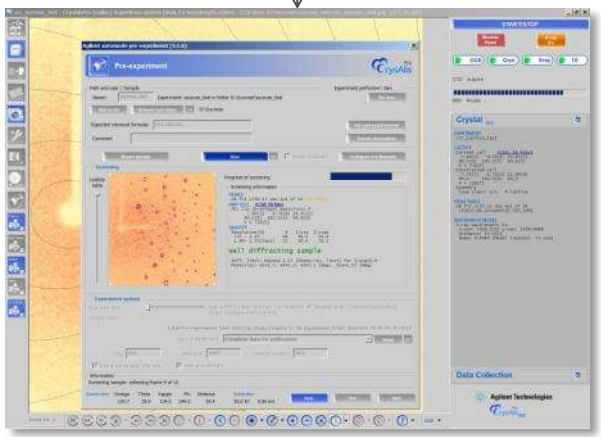
Typical Work-Flow

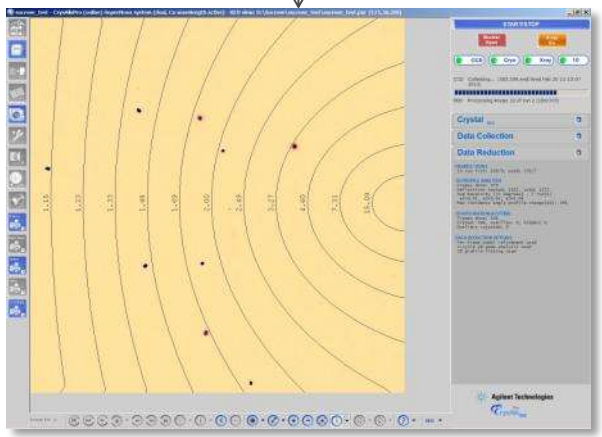






Mounting → Screen/Pre → Strategy → Experiment → Structure





Pre-experiment

- Careful experiment planning = same theta range as final experiment: Mo = 0.8; 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/sig

HW – instrumentation

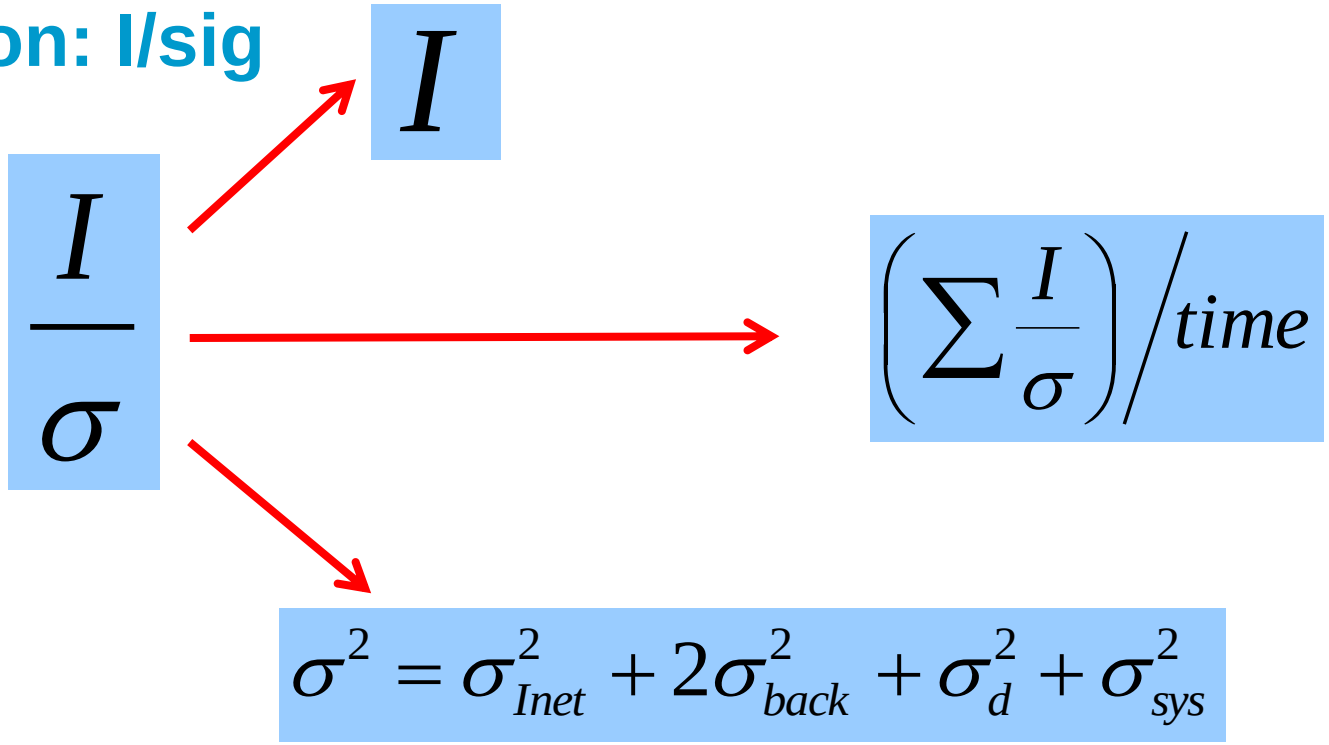
$$\frac{I}{\sigma}$$

Procedure

Data reduction
Corrections

- X-ray diffraction experiment are governed by Poisson statistics: I; sig(I)=sqrt(I)
- Rule of thumb: Inverse of I/sig is your R1

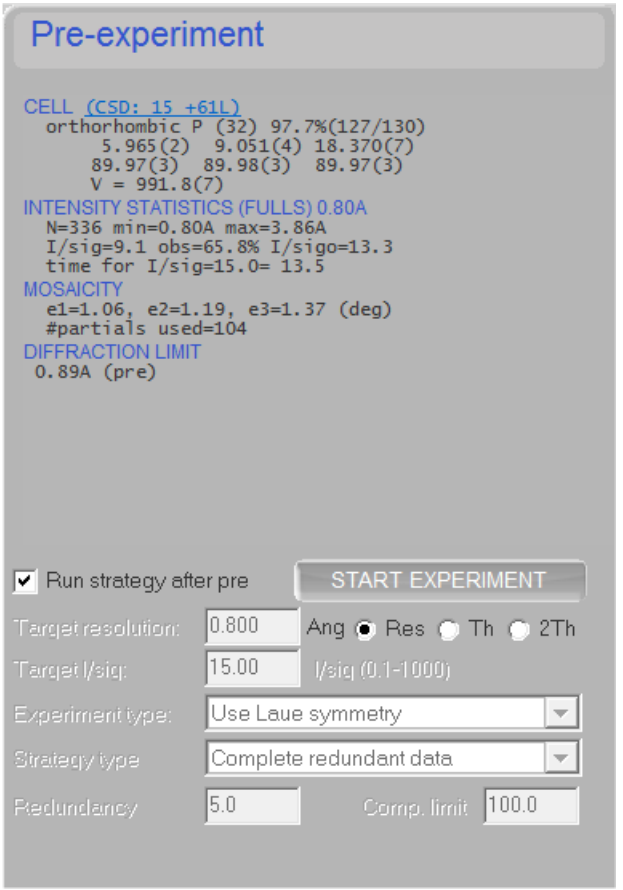
Excursion: I/sig



- 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



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

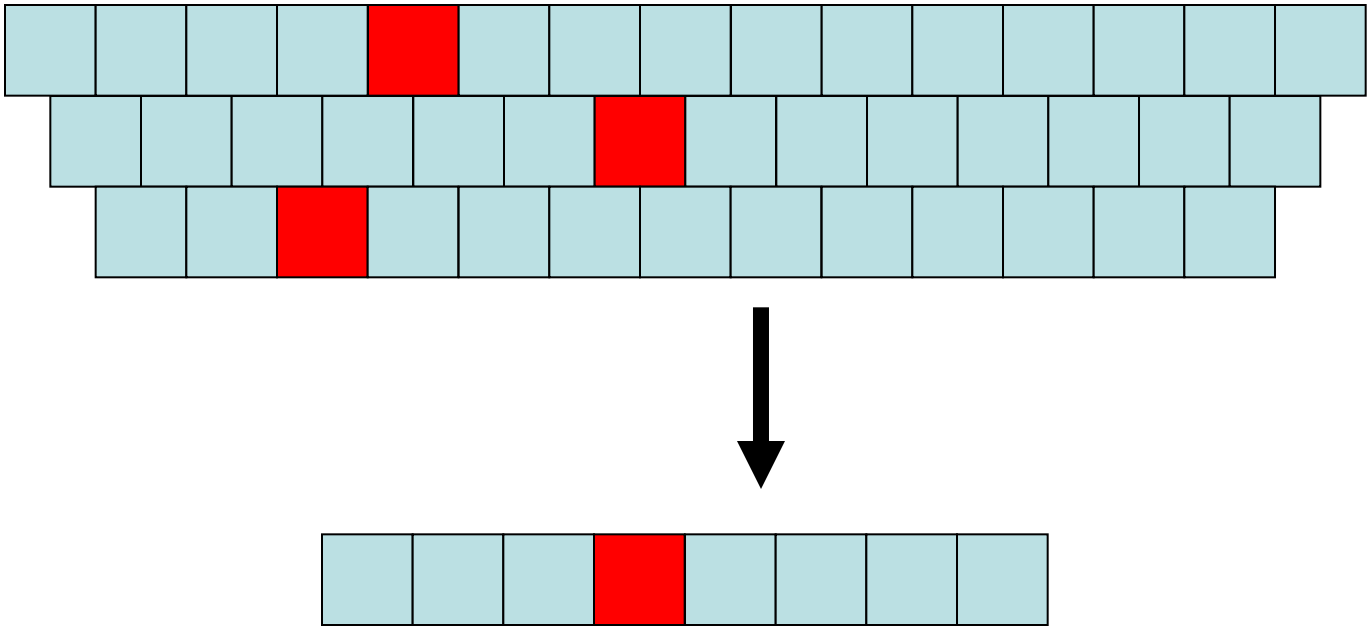
The strategy computation question

user constraints

distance, detector, collisions

Pool of runs
Typically 150-2000

The strategy computation question 2



Level 1 25%

Level 2 49%

Level 3 67%

Level n 100%

What is efficient?

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

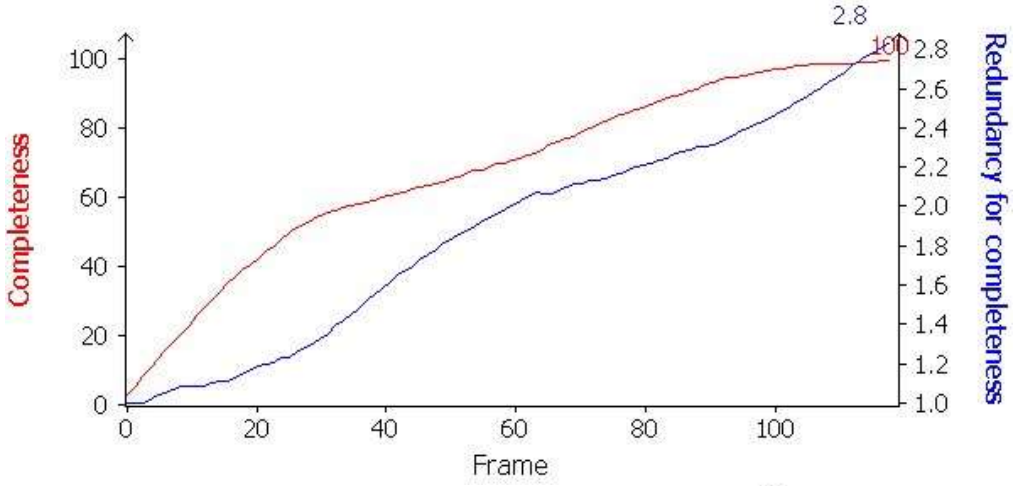
Laue: 2/m; Friedel off, Target 0.800Ang

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
18.50-	0.80	4146	4152	99.86%	1.9	7977	5560	8282	67.13%	1.4	7977

Rule of thumb:

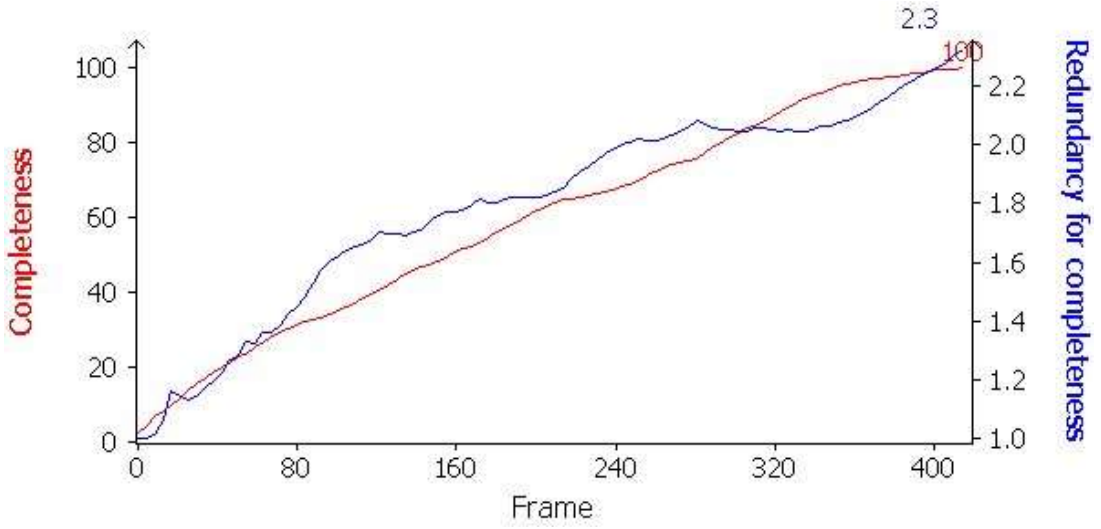
"When requiring 100% completeness the minimum achievable redundancy is about 2"

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

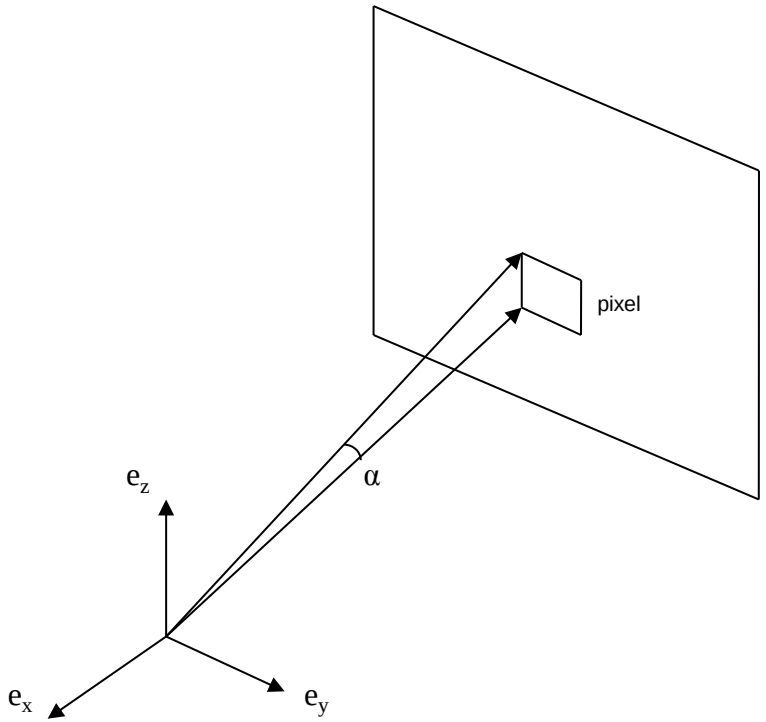
Sapphire, res =0.8, complete data

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)

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

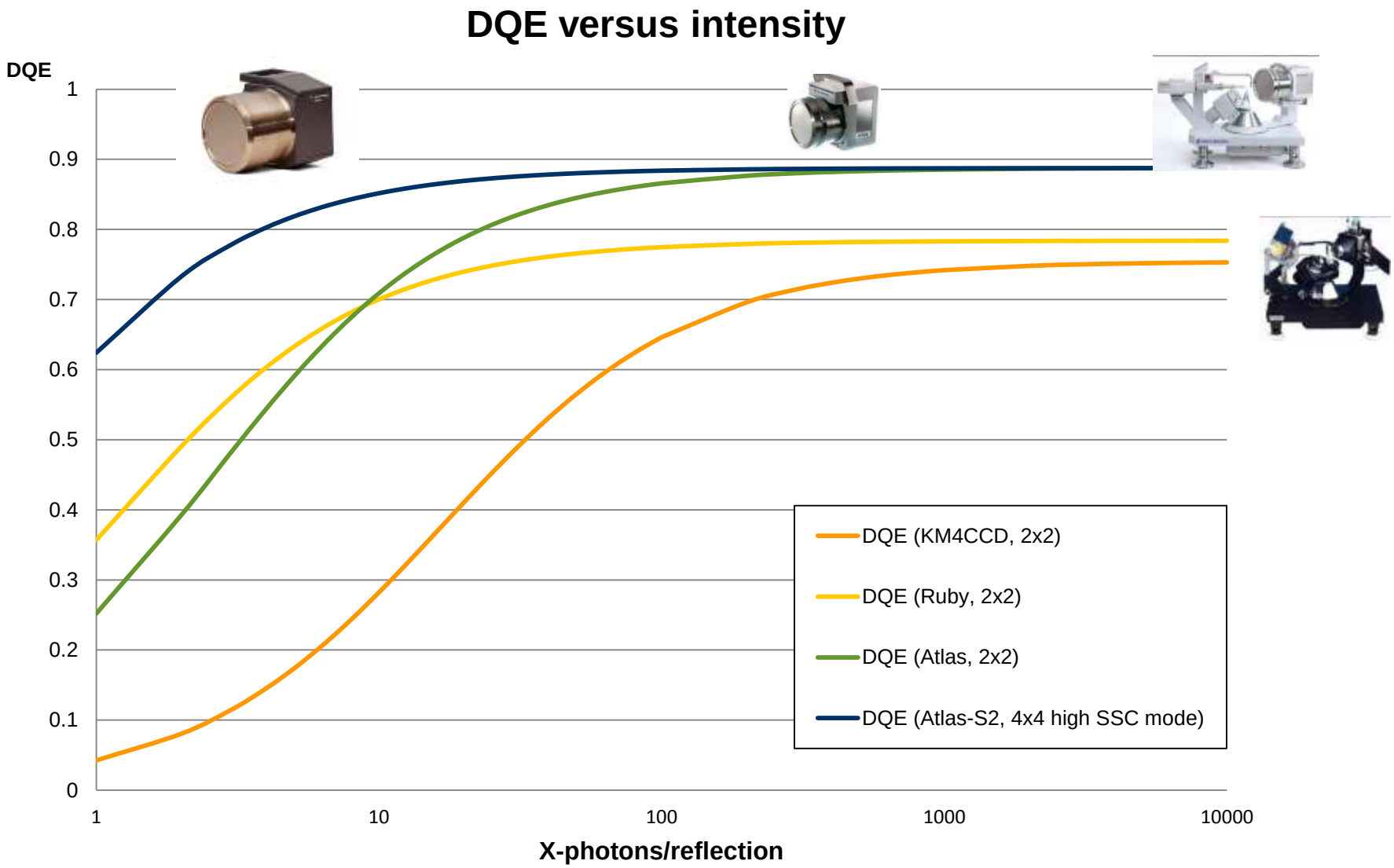
Window
Scintillator

Gain

Noise:
Read and dark

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

Detective Quantum Efficiency (DQE) advances

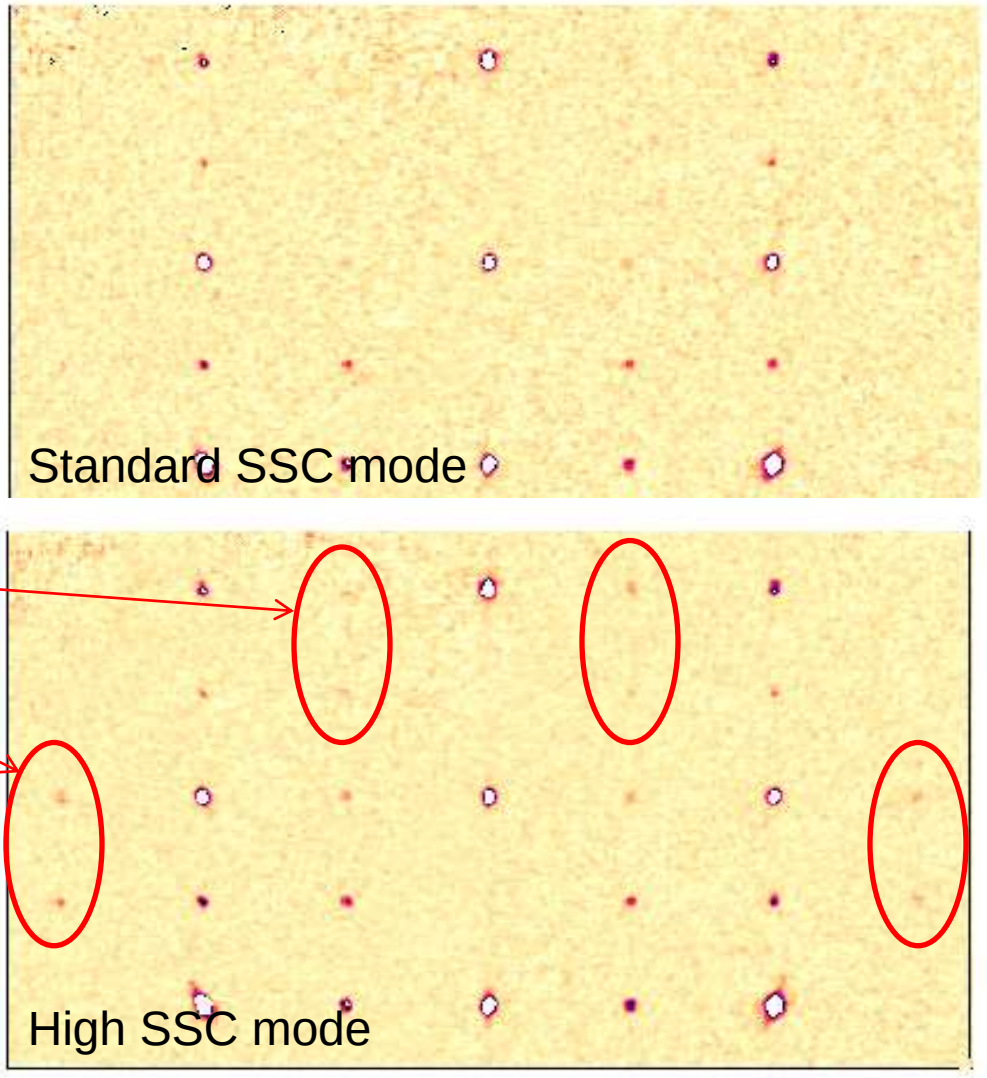


DQE for Mo radiation, peak dia 1mm at 60mm

What is ‘Smart sensitivity control’ SSC

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

Precession image (Fe formate, 0kl plane)

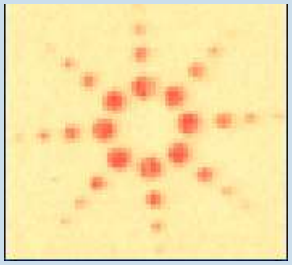
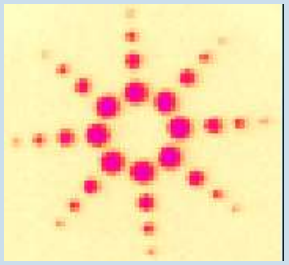
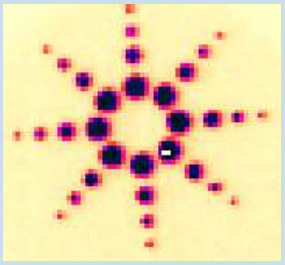
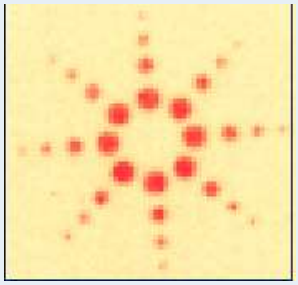
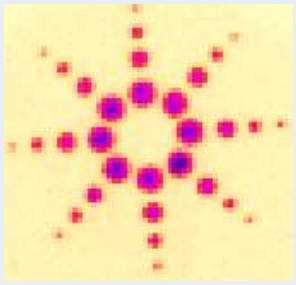
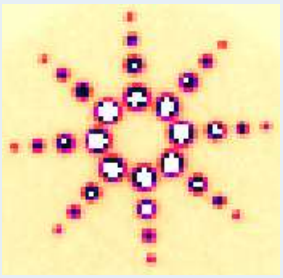


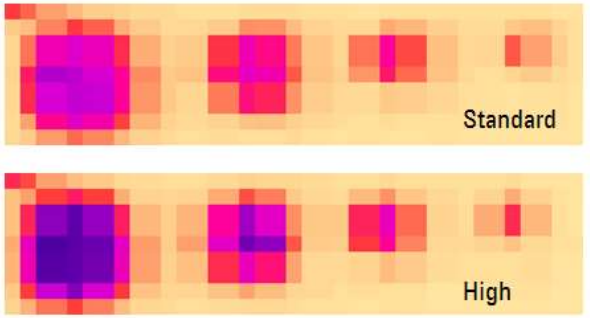
These spots became visible only in high SSC mode!

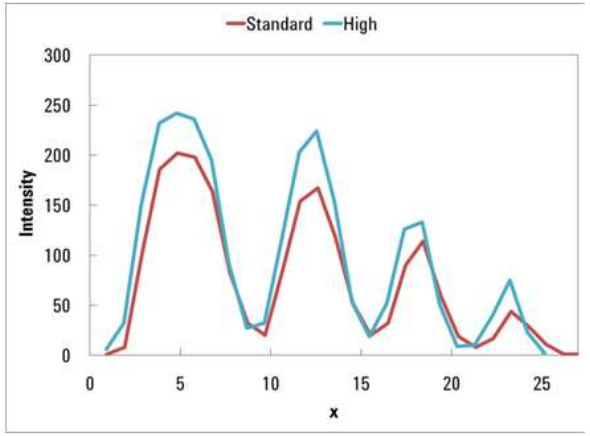
State-of-the-art electronics provide:
- Detectivity

Titan S2 camera SSC functionality

4x4 binning

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





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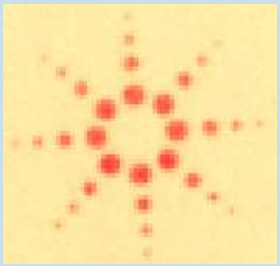
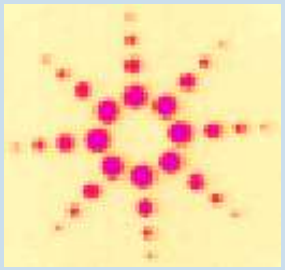
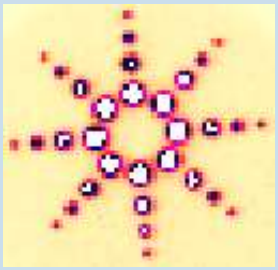
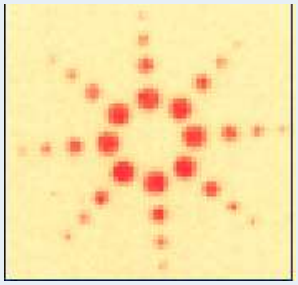
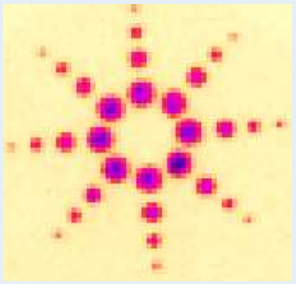
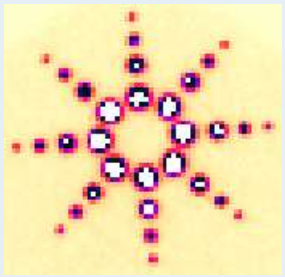
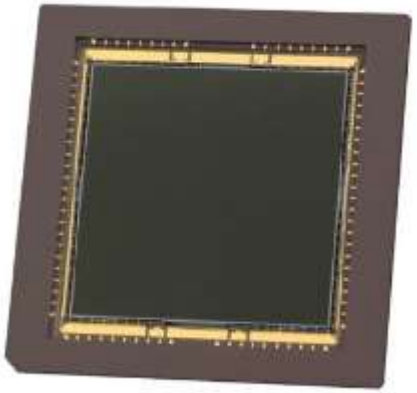
Titan S2 camera SSC functionality

2x2 vs. 4x4 binning

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

HW – Detector tech: Full well/Dynamic

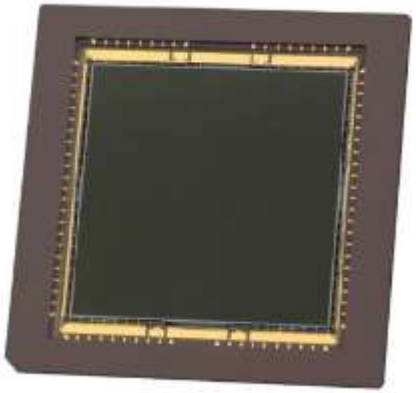


Detector generation	Full well with respect to base binning pix (e^-)	Relative
KM4CCD, Sapphire 2x2	**256’000**	**1**
Ruby 2x2	**128’000**	**0.5**
Atlas 2x2	**550’000**	**2.1**
Atlas – S2 4x4	**8’800’000**	**34.3**

State-of-the-art electronics provide:

- Speed
- Instant binning switch
- Full well

HW – Detector tech: Read-out times

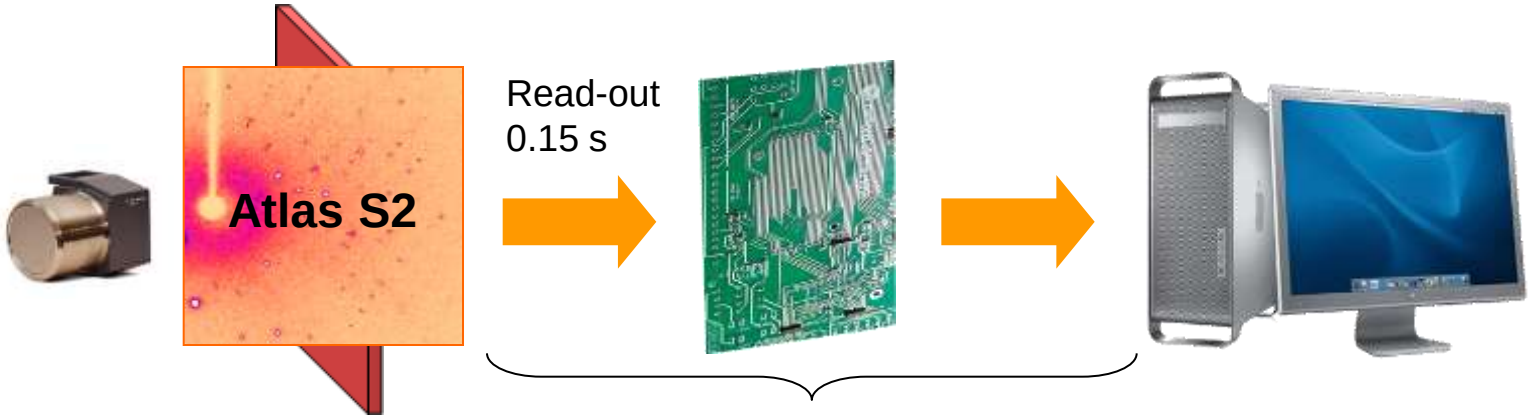


Detector generation	Readout frequency for 1x1 binned image	Relative
KM4CCD, Sapphire 1M	**0.075Mhz**	**1**
Ruby 4M	**0.420Mhz**	**5.6**
Atlas 4M	**2.100Mhz**	**28**
Atlas – S2 4M	**4.400Mhz**	**56.7**

State-of-the-art electronics provide:

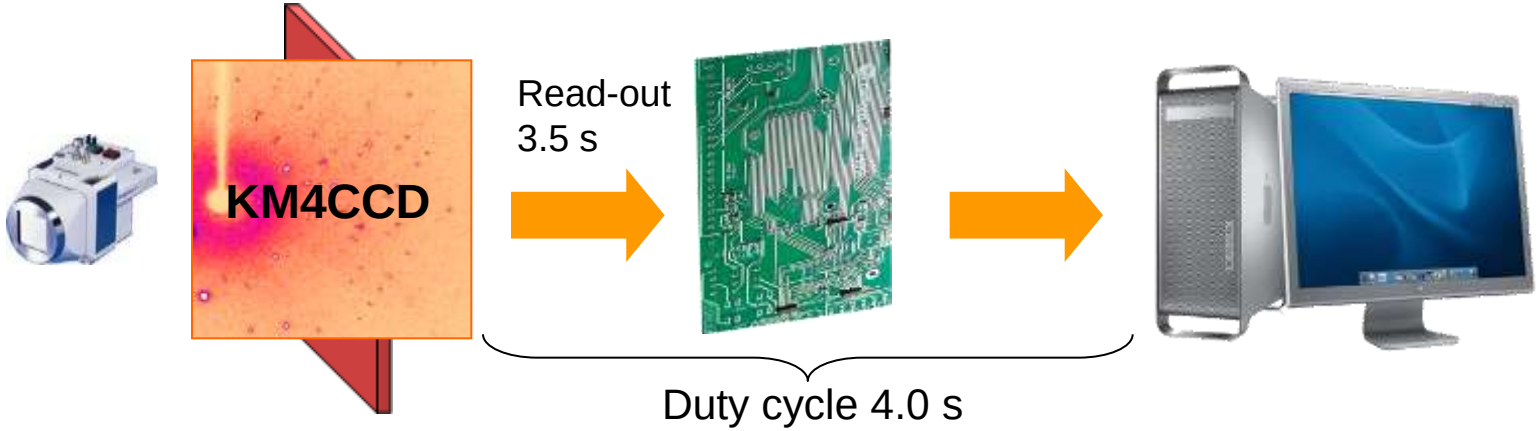
- Speed

HW – Detector tech: Read-out times



Frames
1 2 3 4 5 6 7 8 9 10

Total time
= <u>2.2 sec</u>



Frames
1 2 3 4 5 6 7 8 9 10

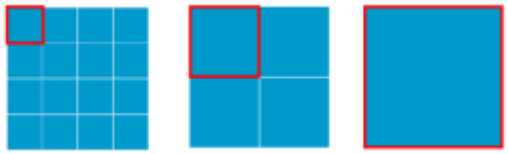
Total time
= <u>40 sec</u>

HW – Sources

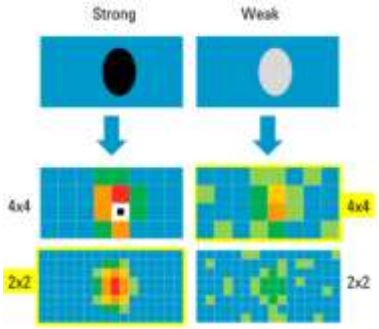
Same sample 0.3mm, normal tubes (2kW, 0.5mm collimator), micro-focus (50W), GV1000 (1000W)

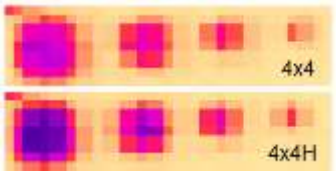
Source type	Integral intensity relative Enhance Mo
Enhance Mo	**1**
Enhance Cu	**5**
Ultra Cu	**40**
Nova Cu	**120**
Nova Cu 2nd gen	**240**
GV1000	**Up to 2400**

Intelligent measurement system - IMS



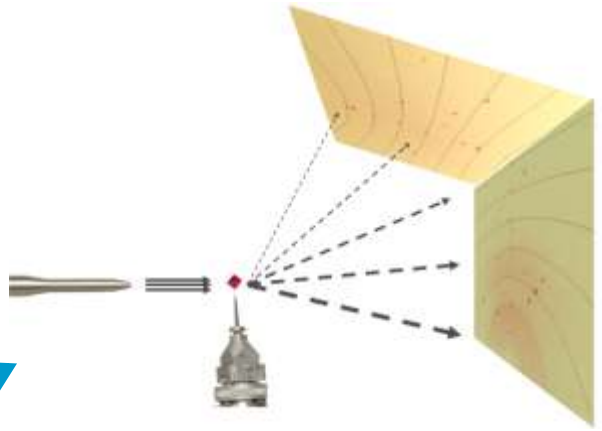
Base binning





Smart sensitivity control

IMS



Theta dependent binning

Gonio/scan settings correlation

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”

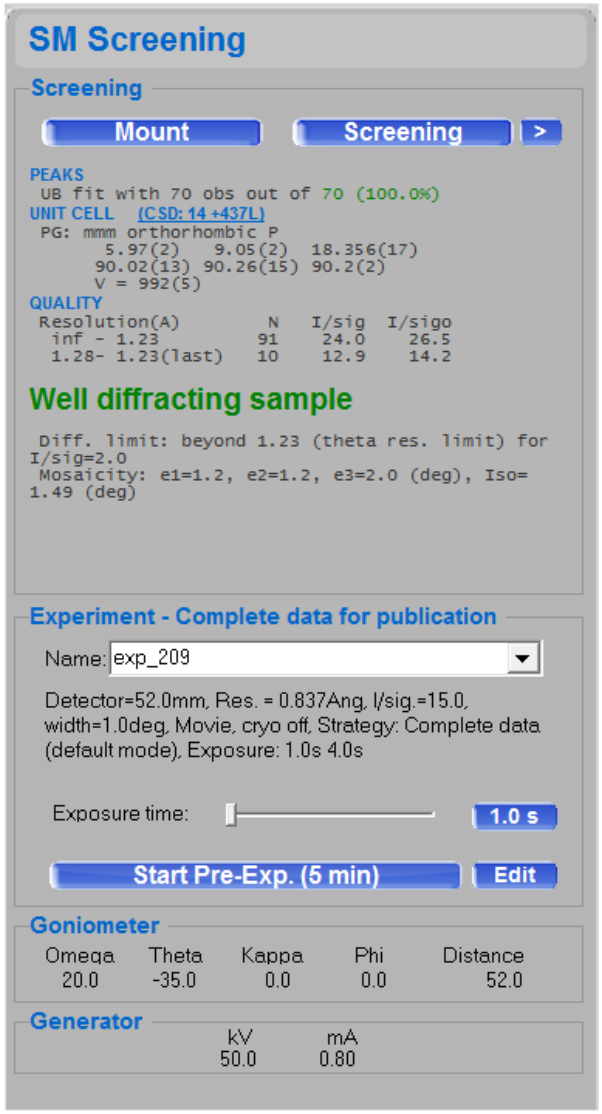
„Increase redundancy for good scaling and absorption correction”

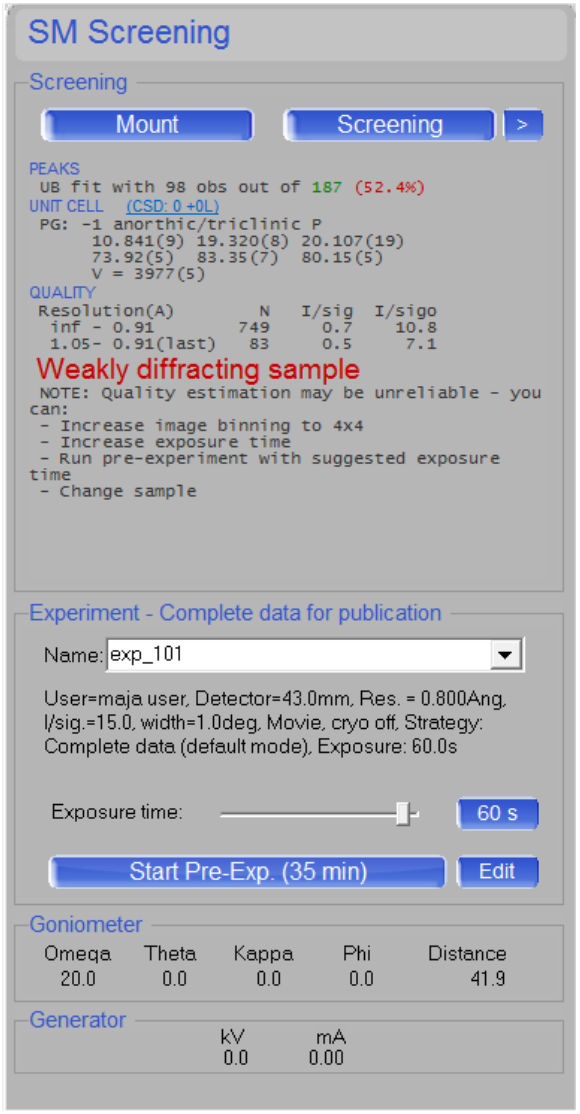
Twinning*: Challenges for the crystallographer

- 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

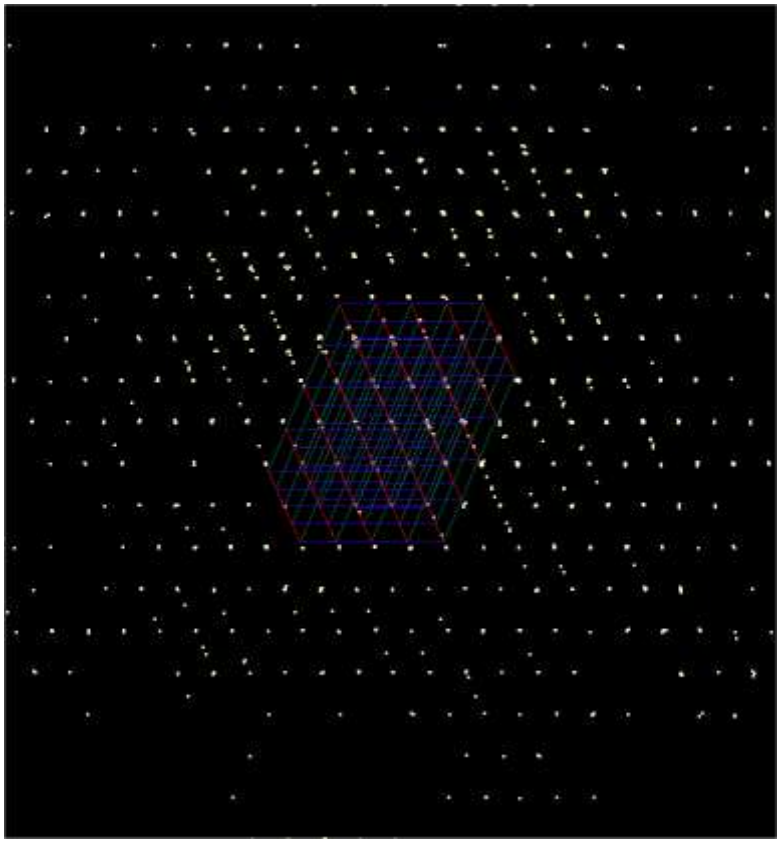
*non-merohedric

Screening tool for quickly judging sample quality

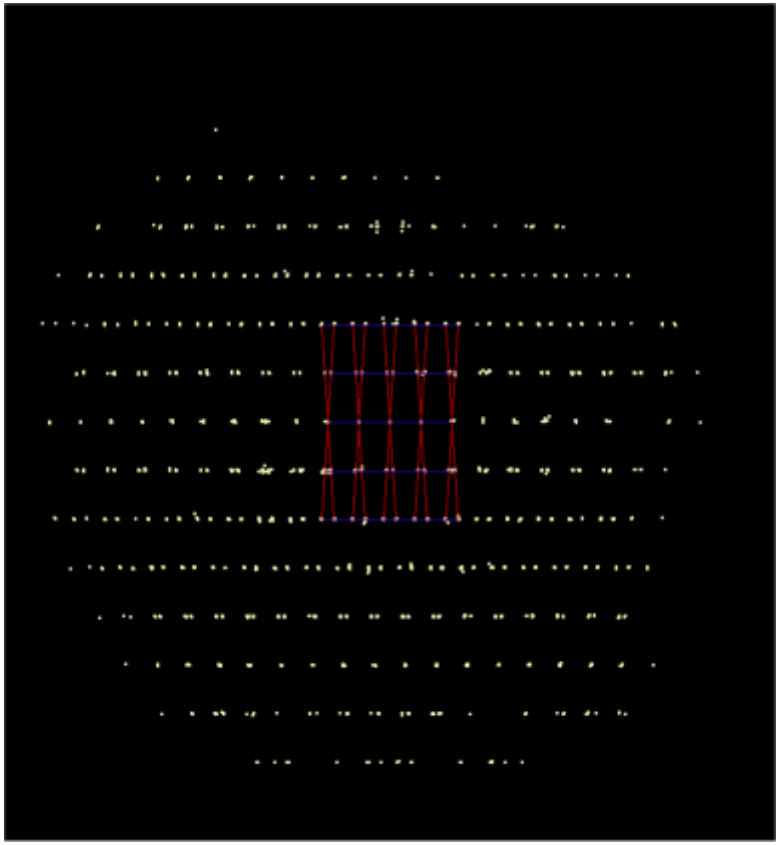




Twin types

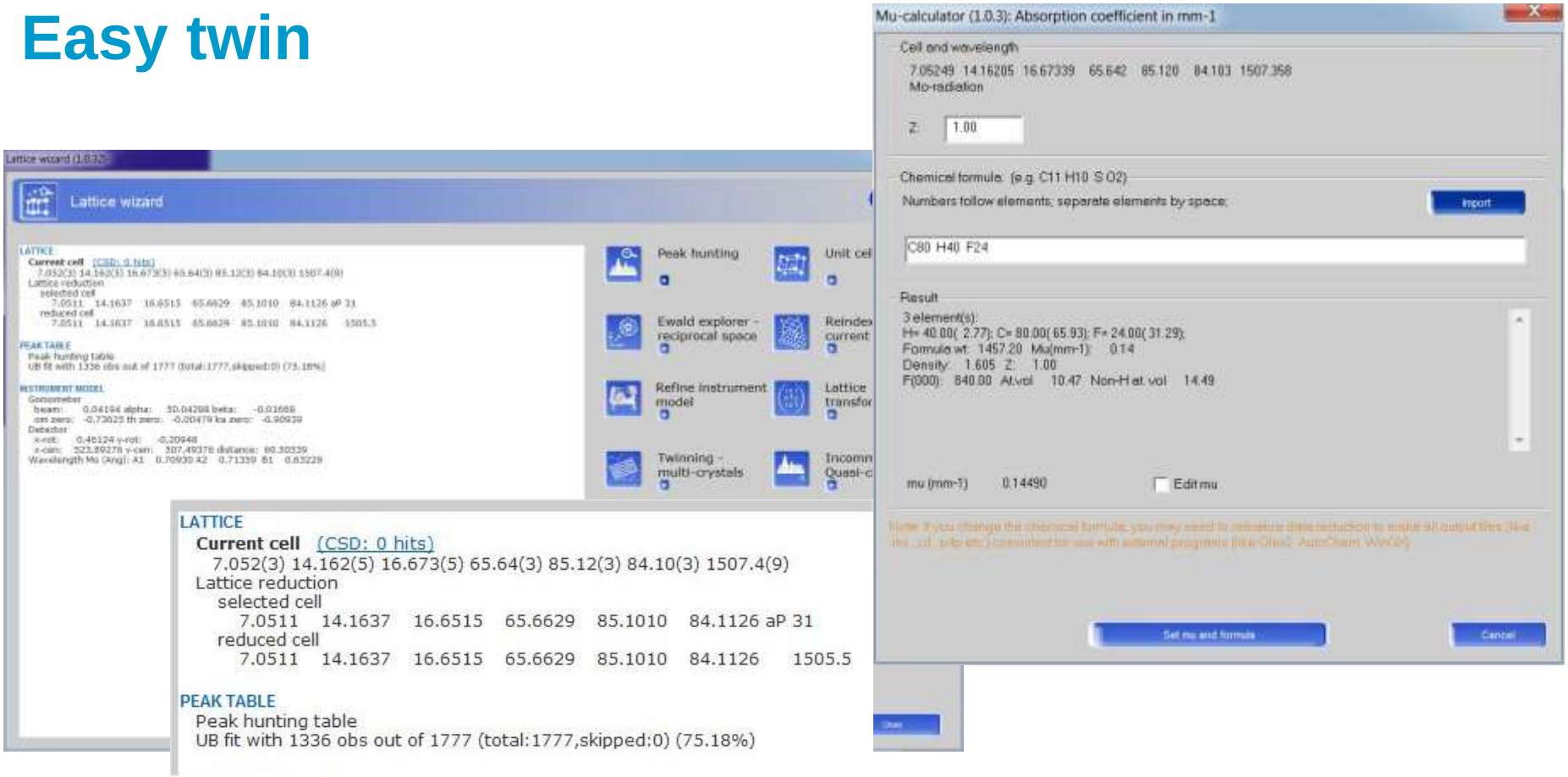


- Easy twin



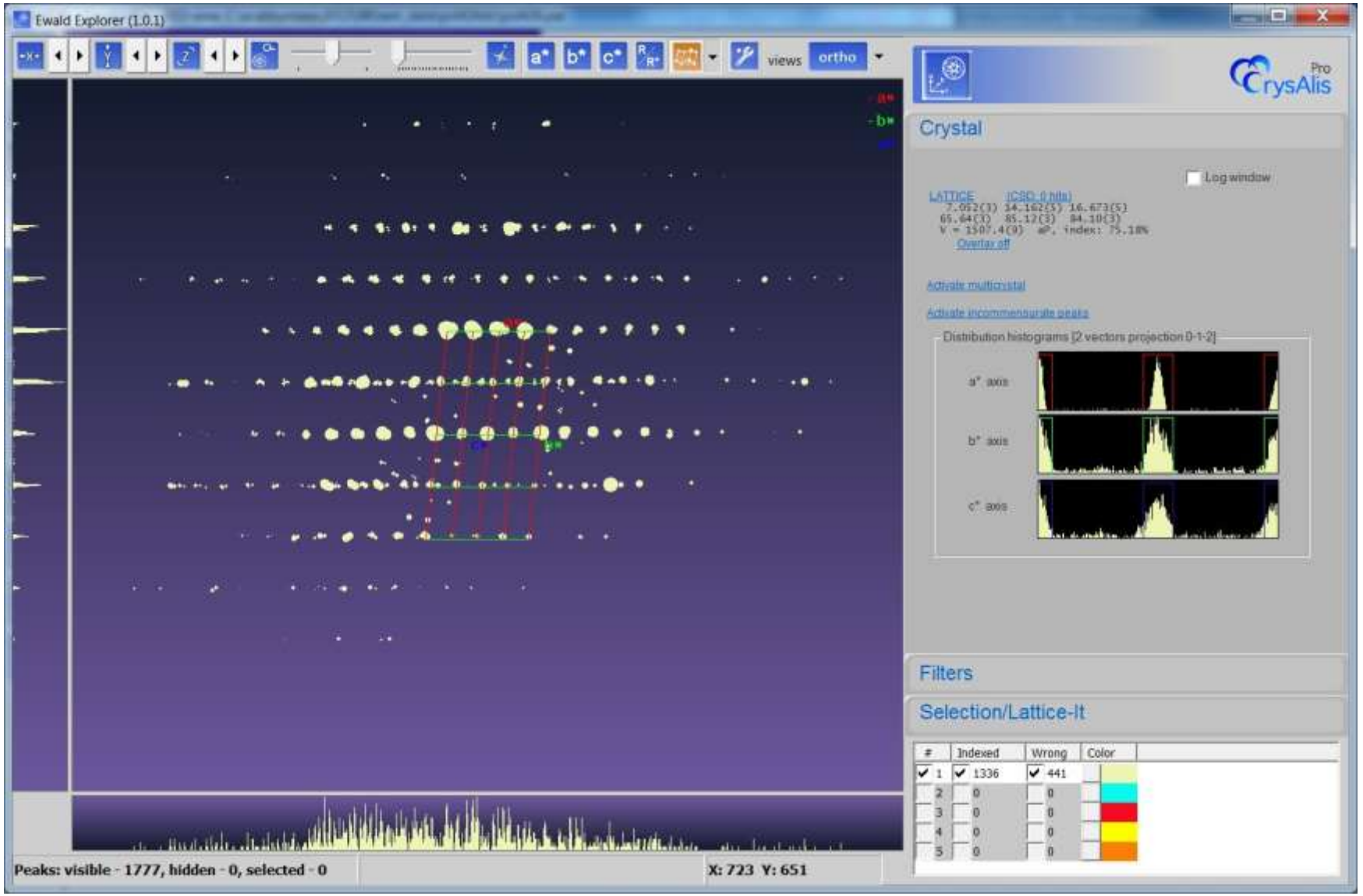
- Super lattice “faker”

Easy twin



- Indexation <90%
- Chemical content consistent

Easy twin in new Ewald^{Pro}



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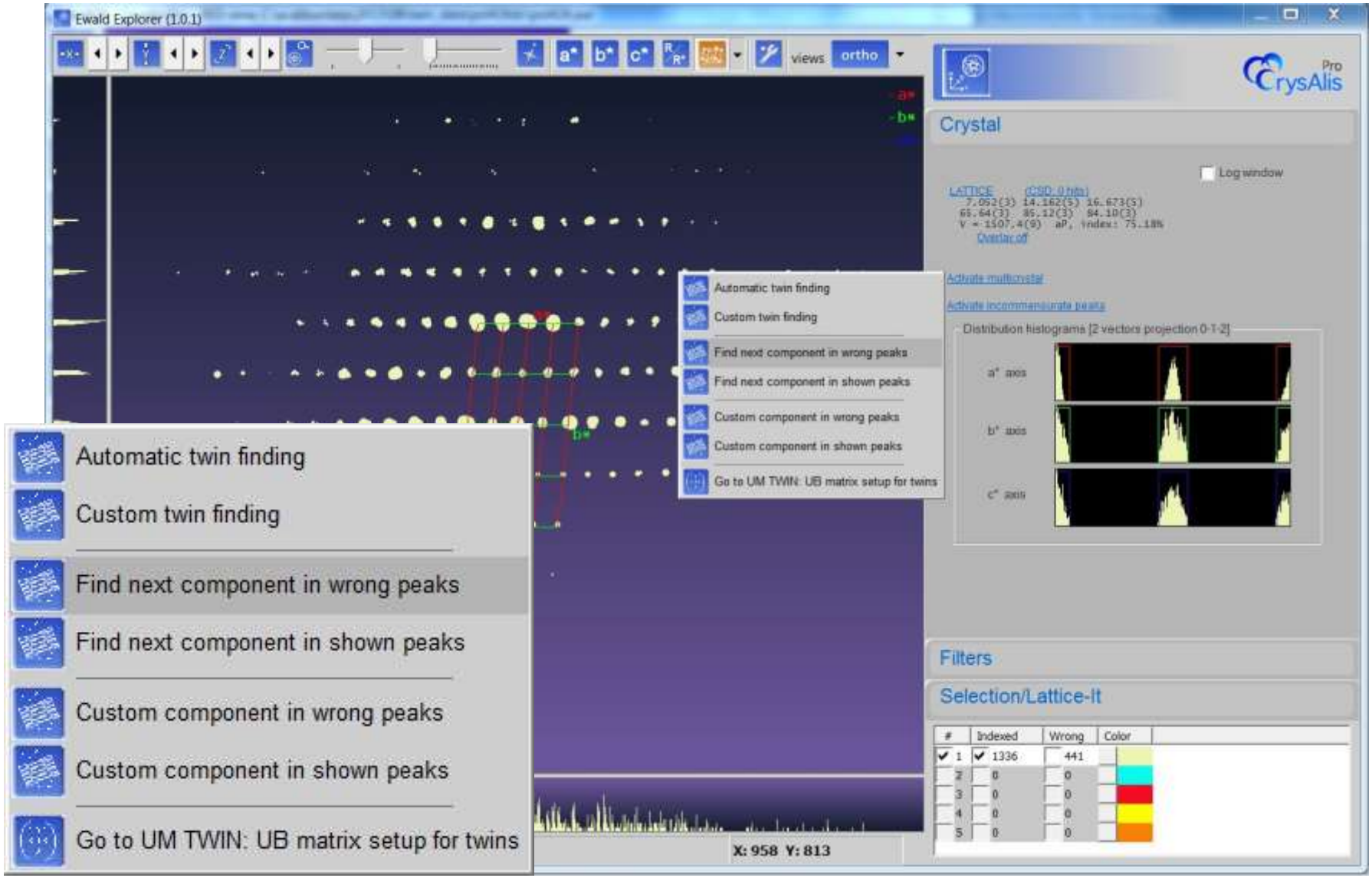
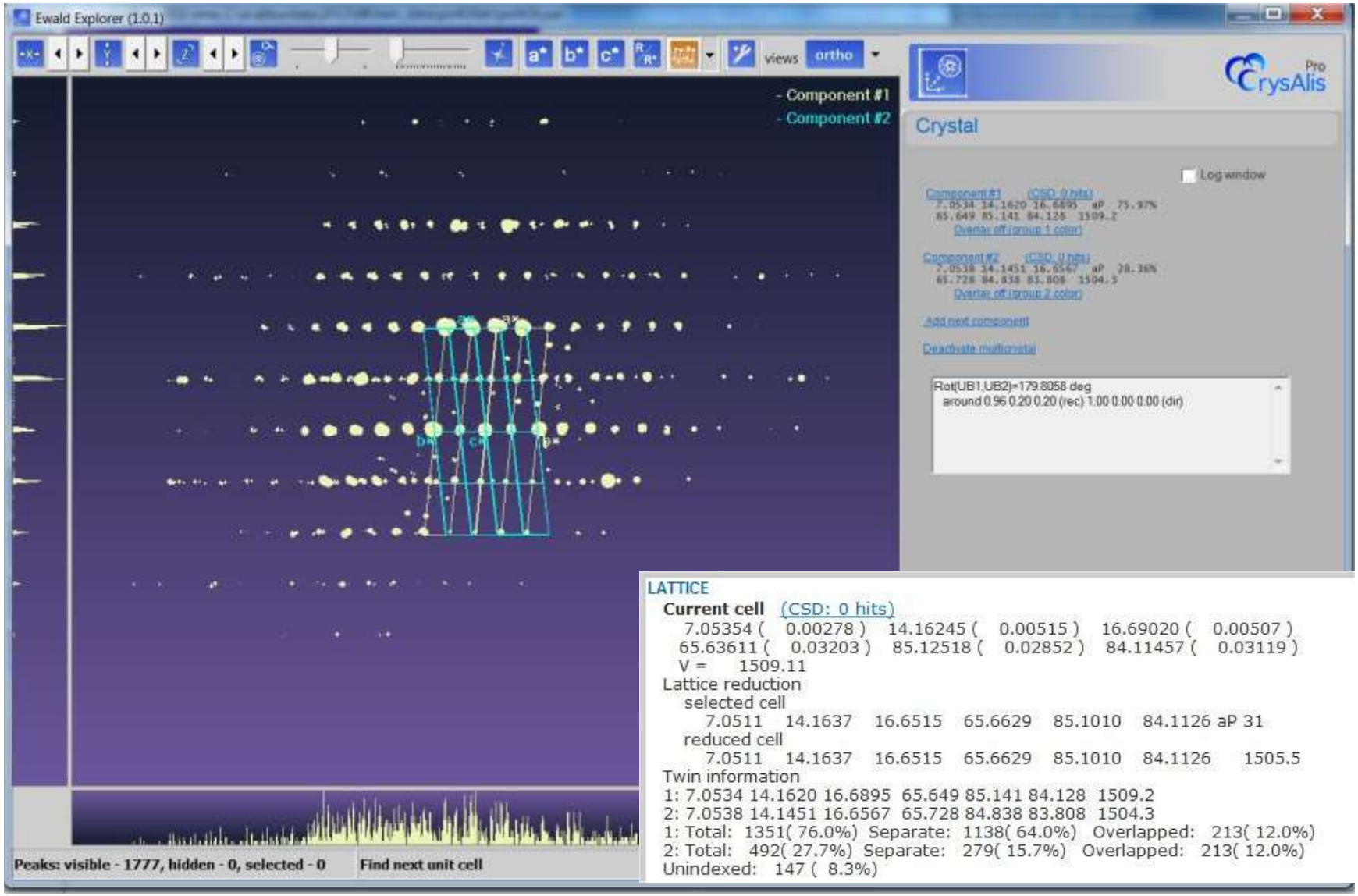
Easy twin in new Ewald^{Pro}



Easy twin in new Ewald^{Pro}



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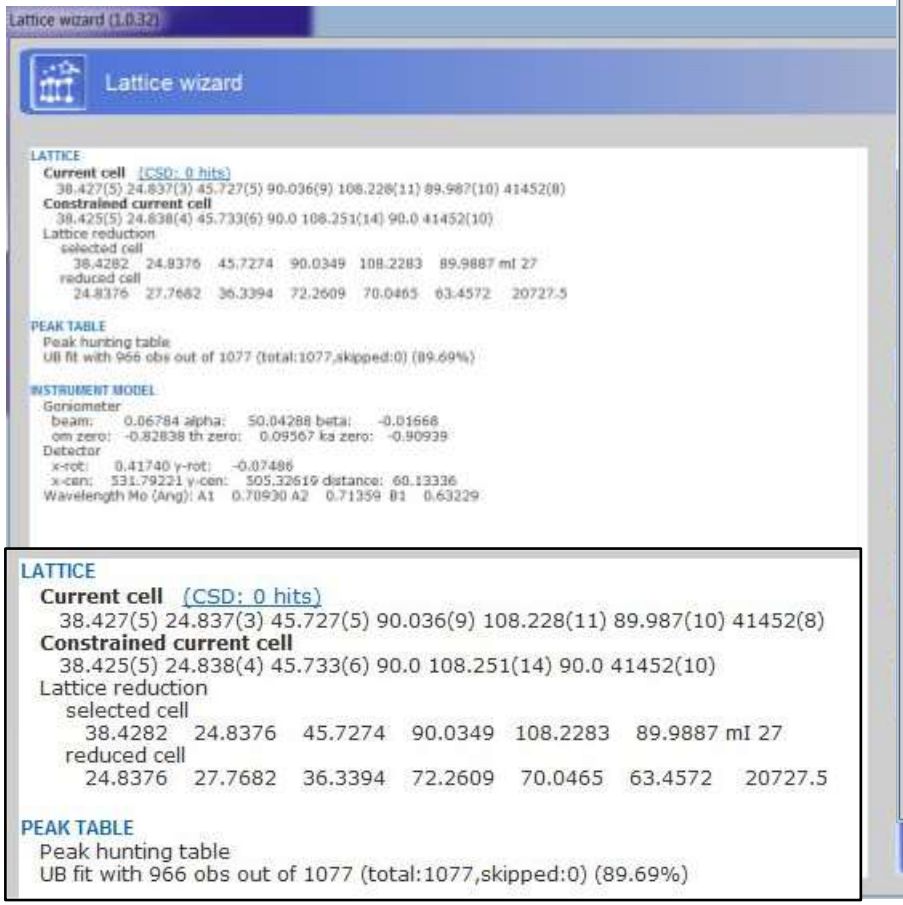
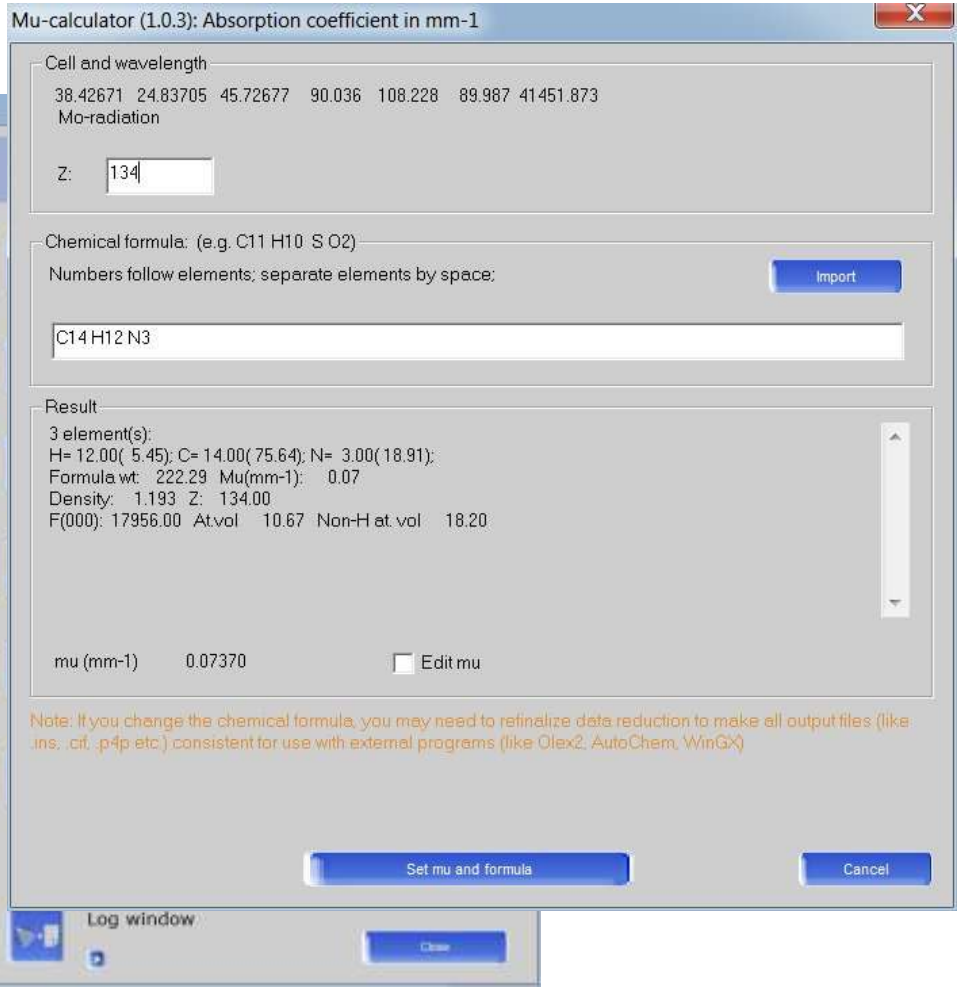
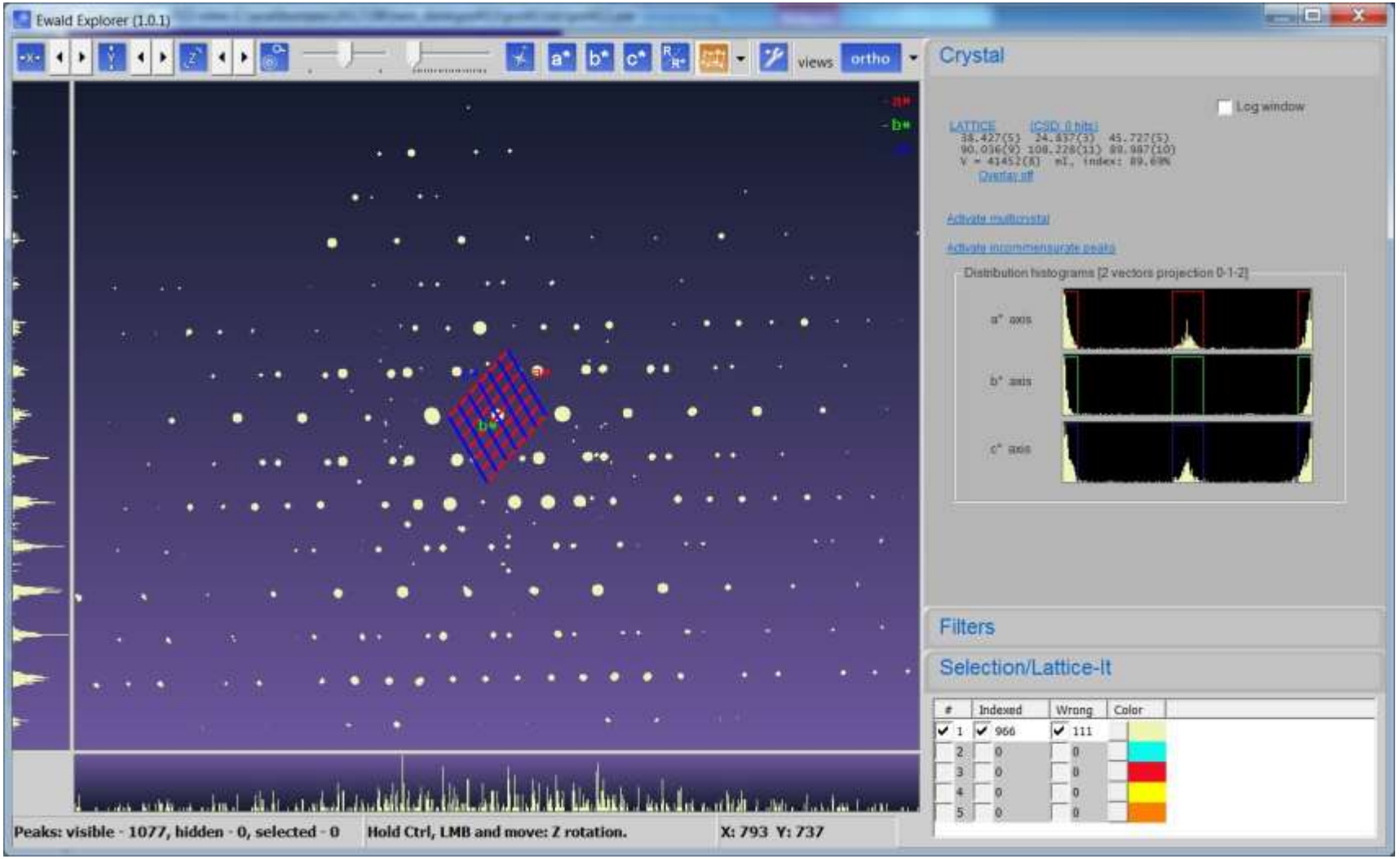
Super lattice faker





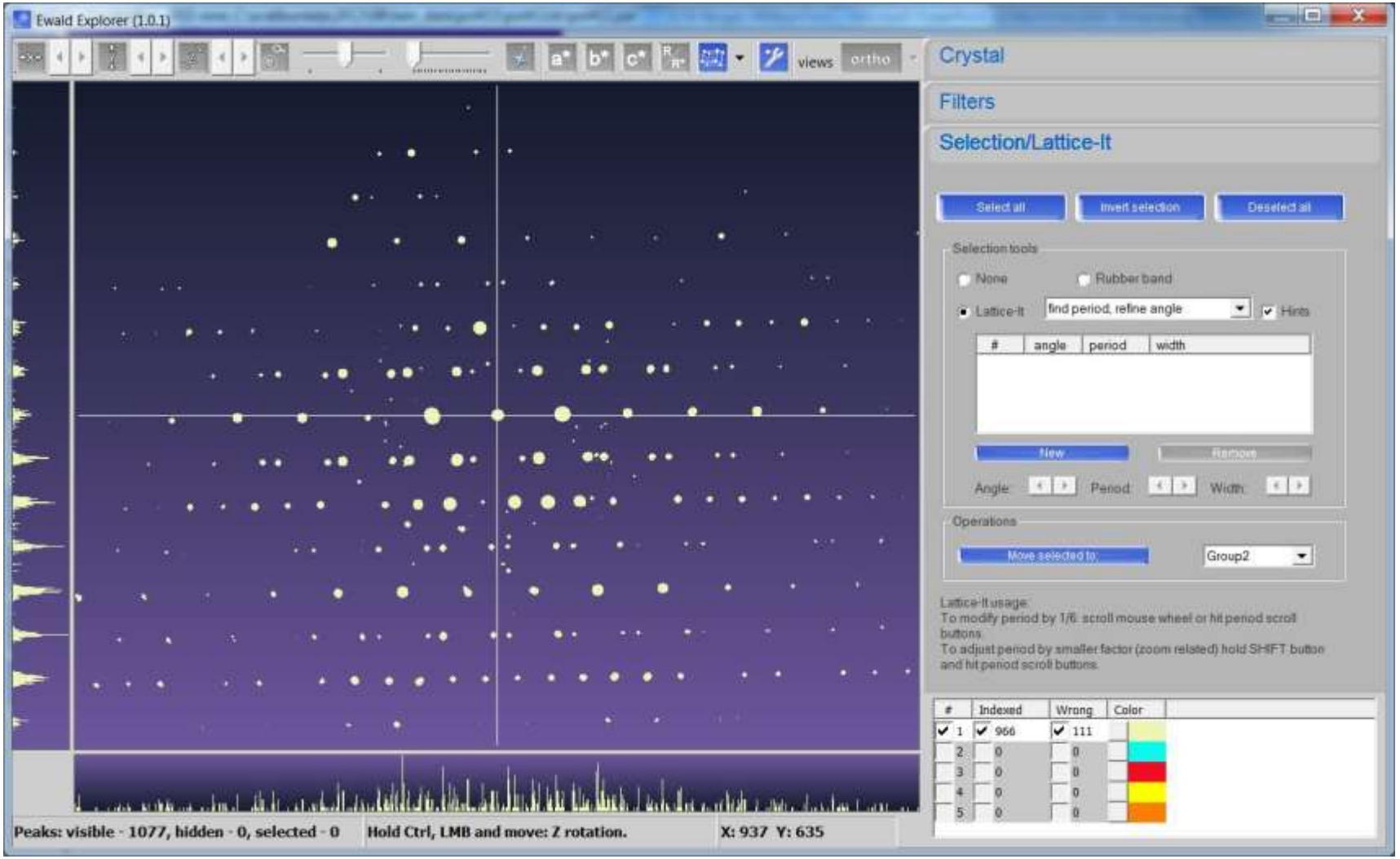
- Indexation good/high
- Chemical content inconsistent (here Z=134)

Super lattice faker in new Ewald^{Pro}

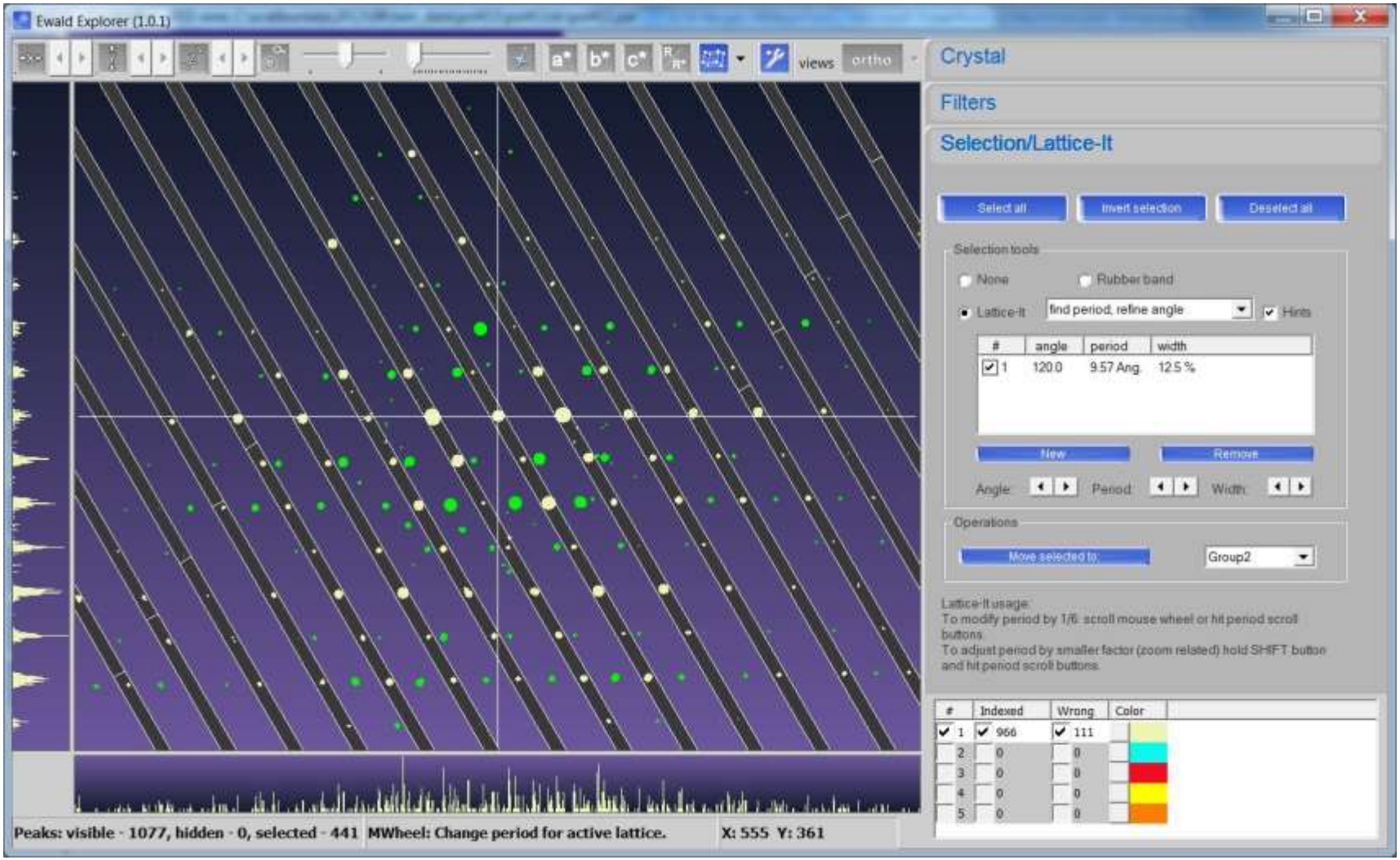


Agilent Technologies

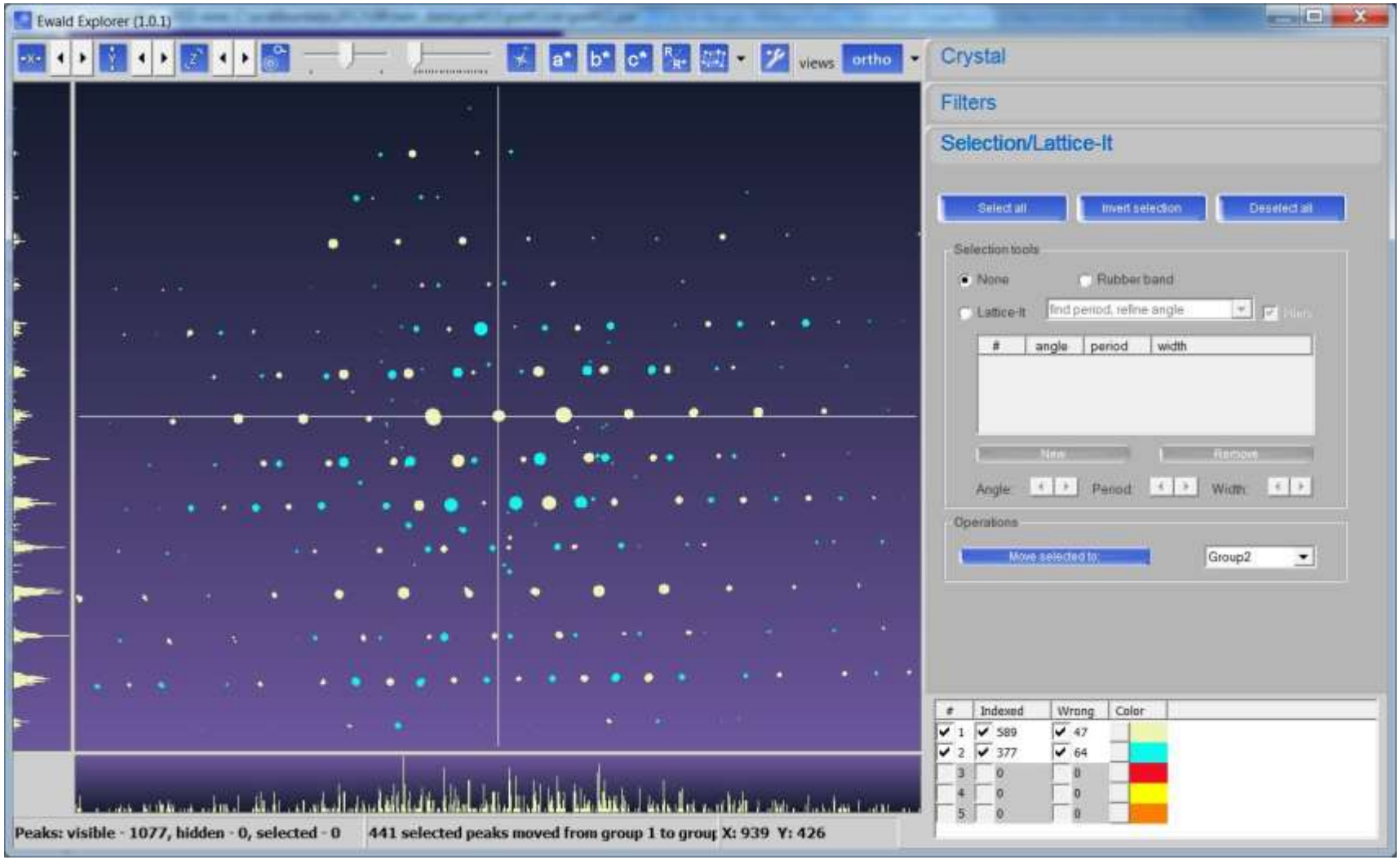
Super lattice faker



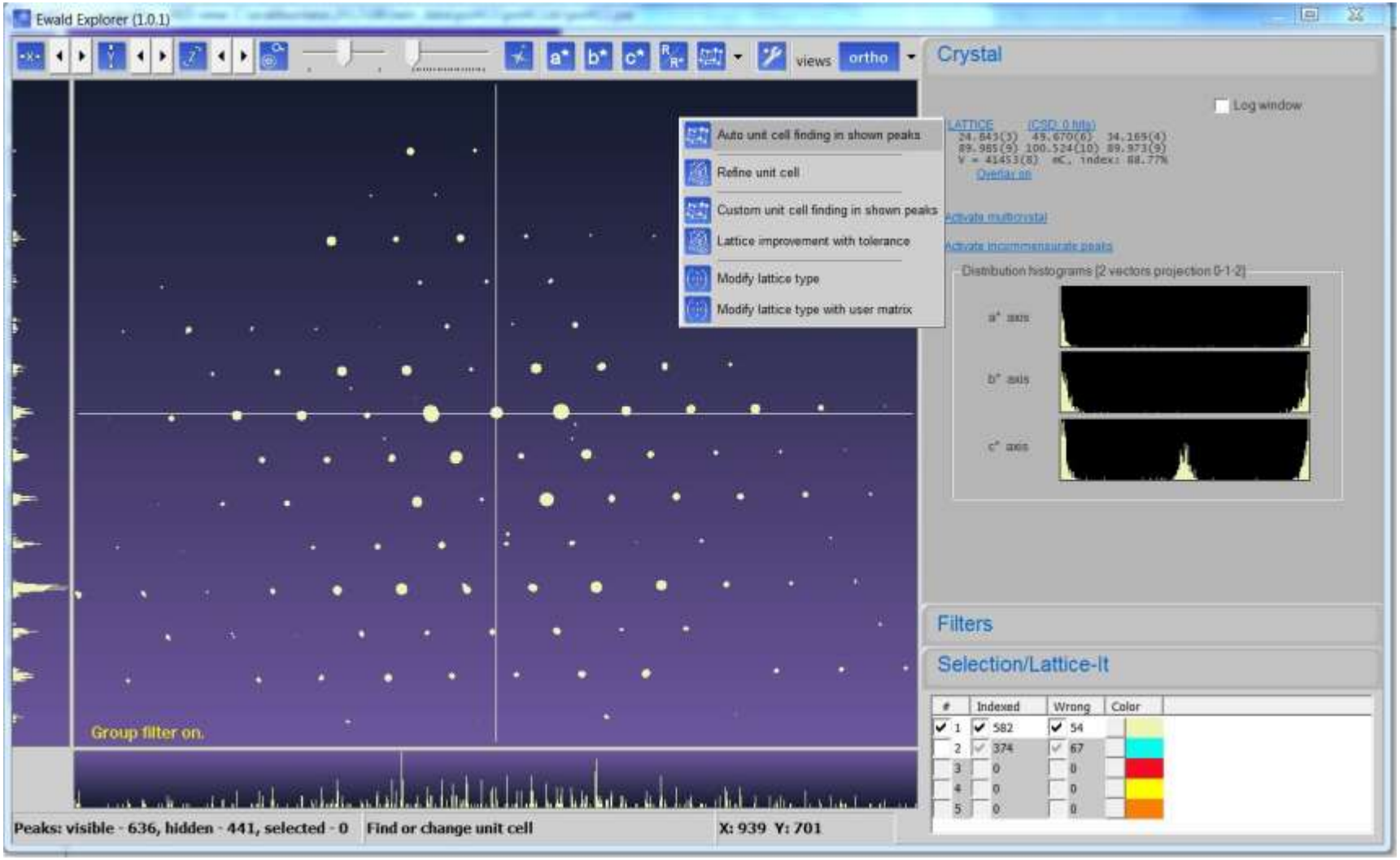
Super lattice faker



Super lattice faker

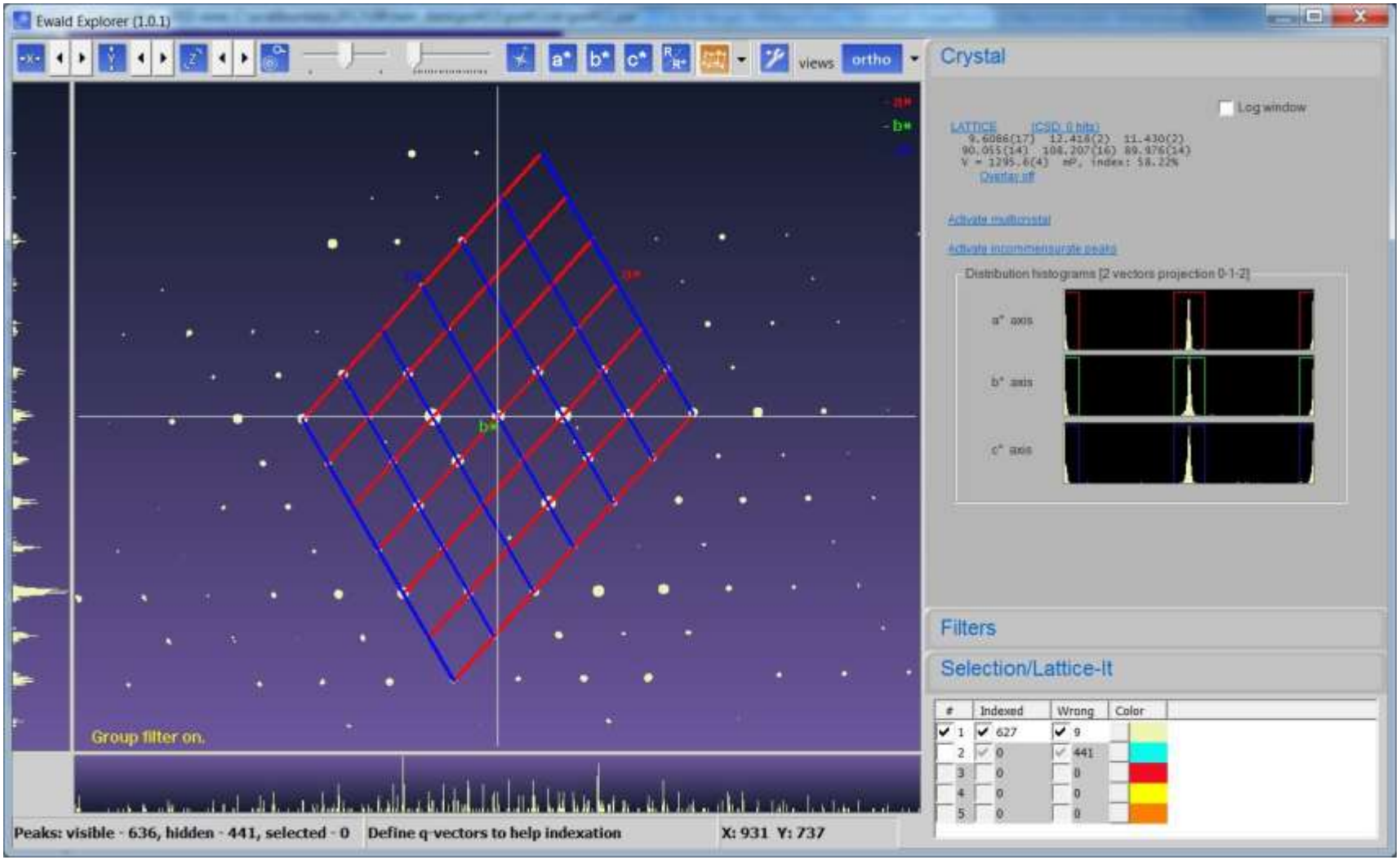


Super lattice faker



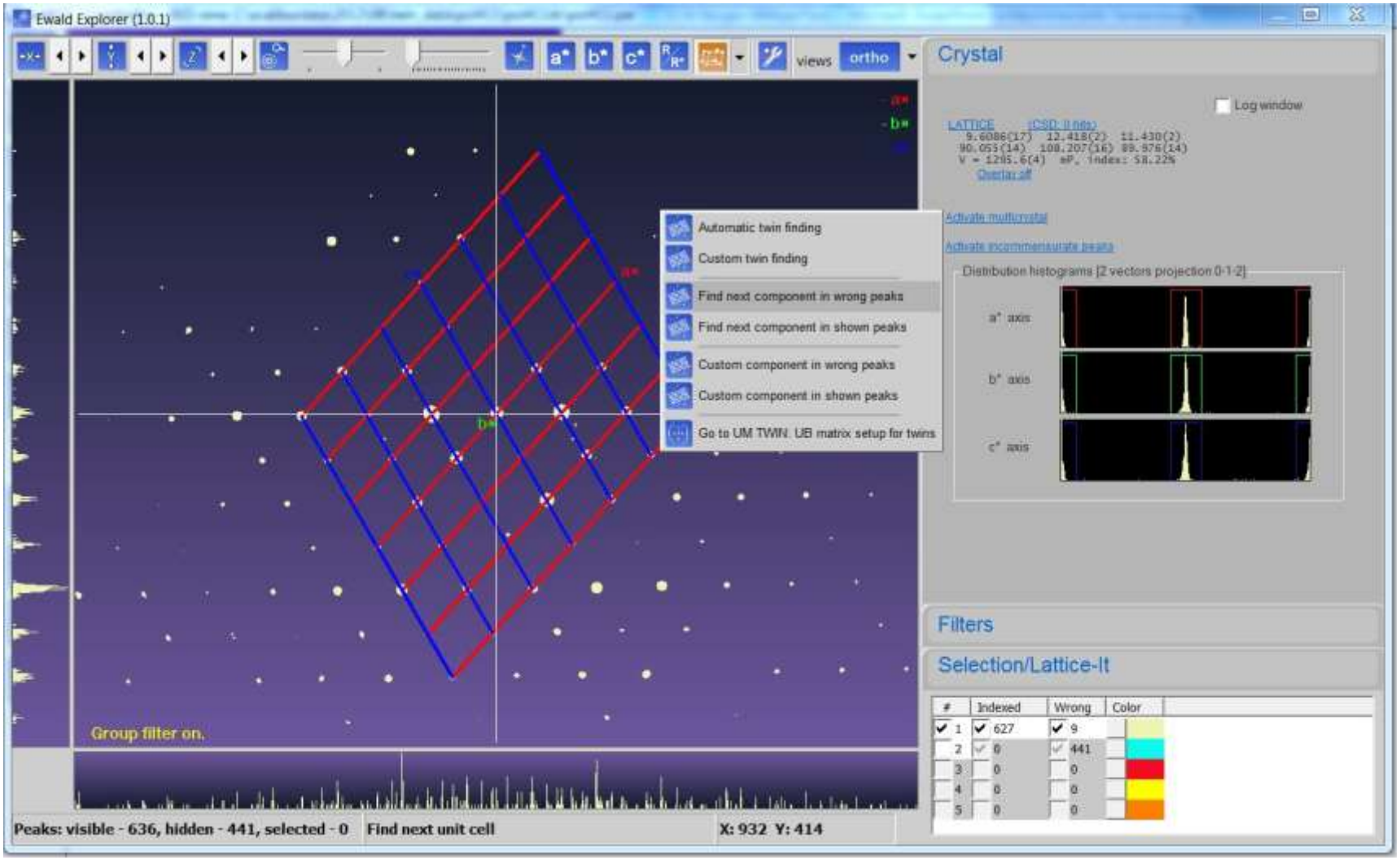
Agilent Technologies

Super lattice faker

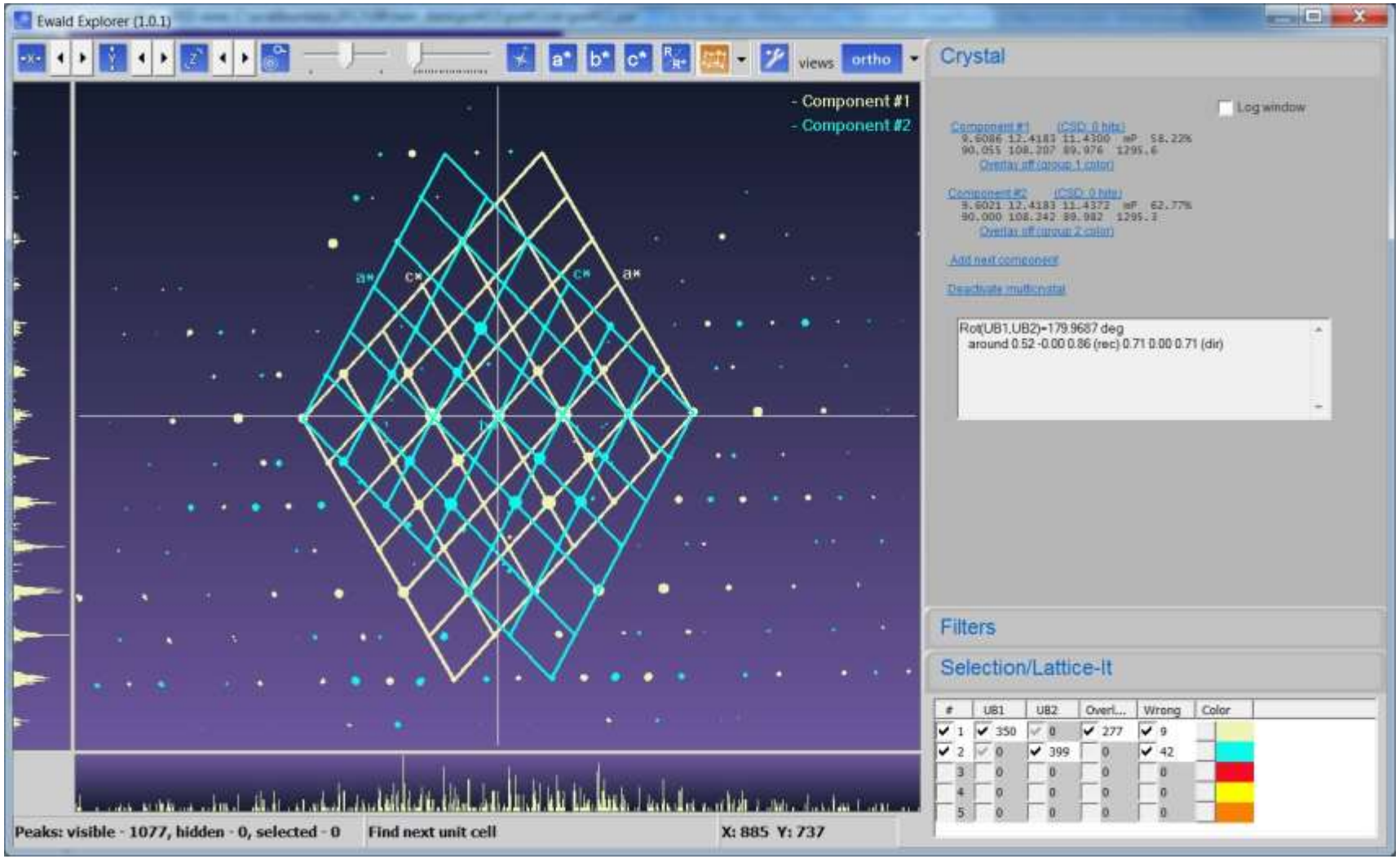


Agilent Technologies

Super lattice faker

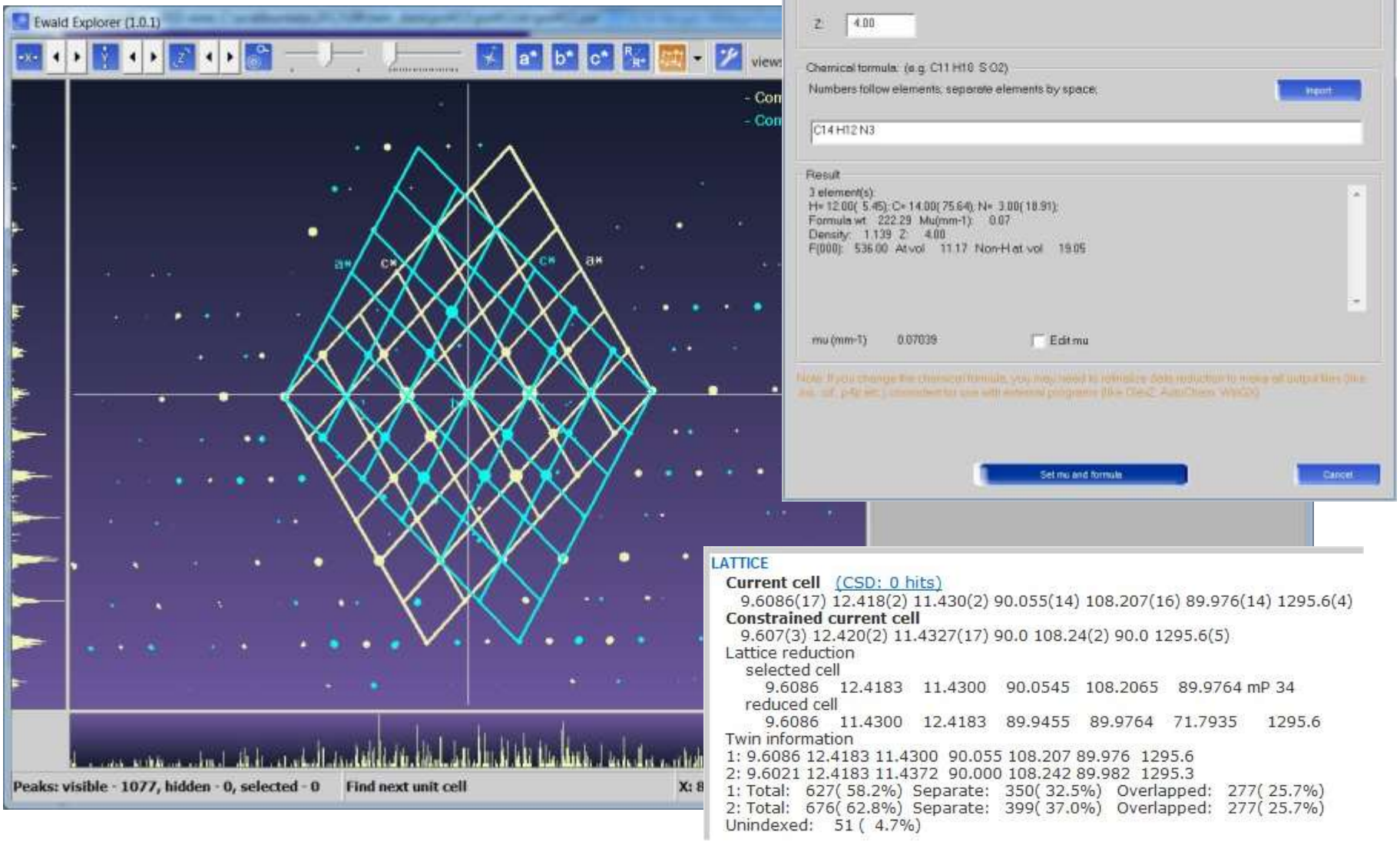


Super lattice faker



Agilent Technologies

Super lattice faker



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

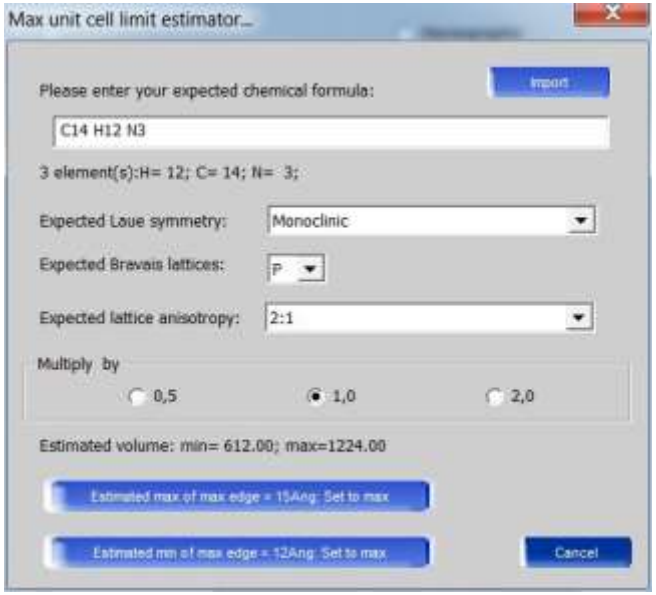
1: Total: 627(58.2%) Separate: 350(32.5%) Overlapped: 277(25.7%)

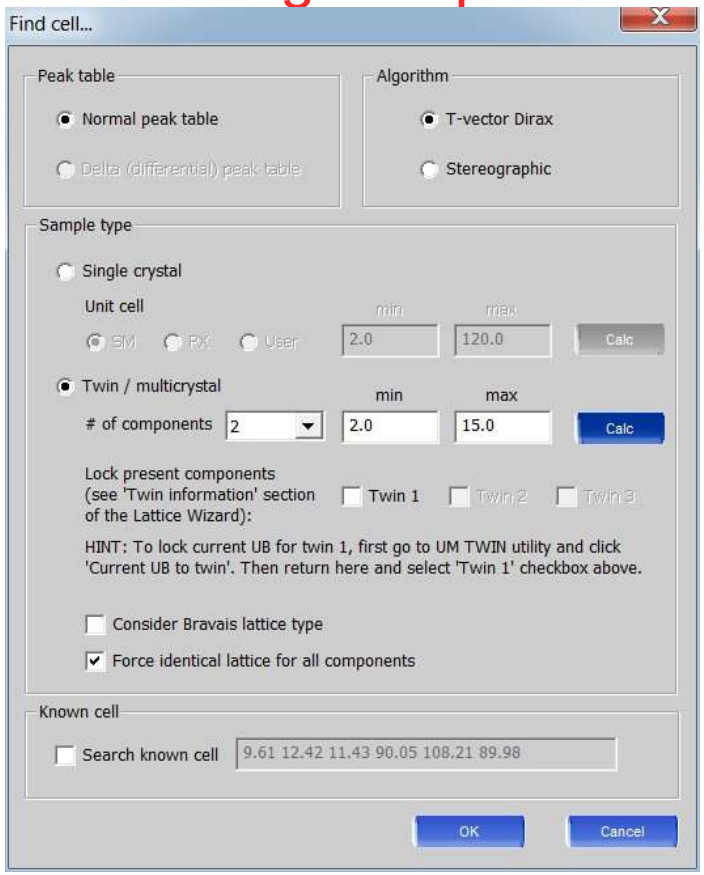
2: Total: 676(62.8%) Separate: 399(37.0%) Overlapped: 277(25.7%)

Unindexed: 51 (4.7%)

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





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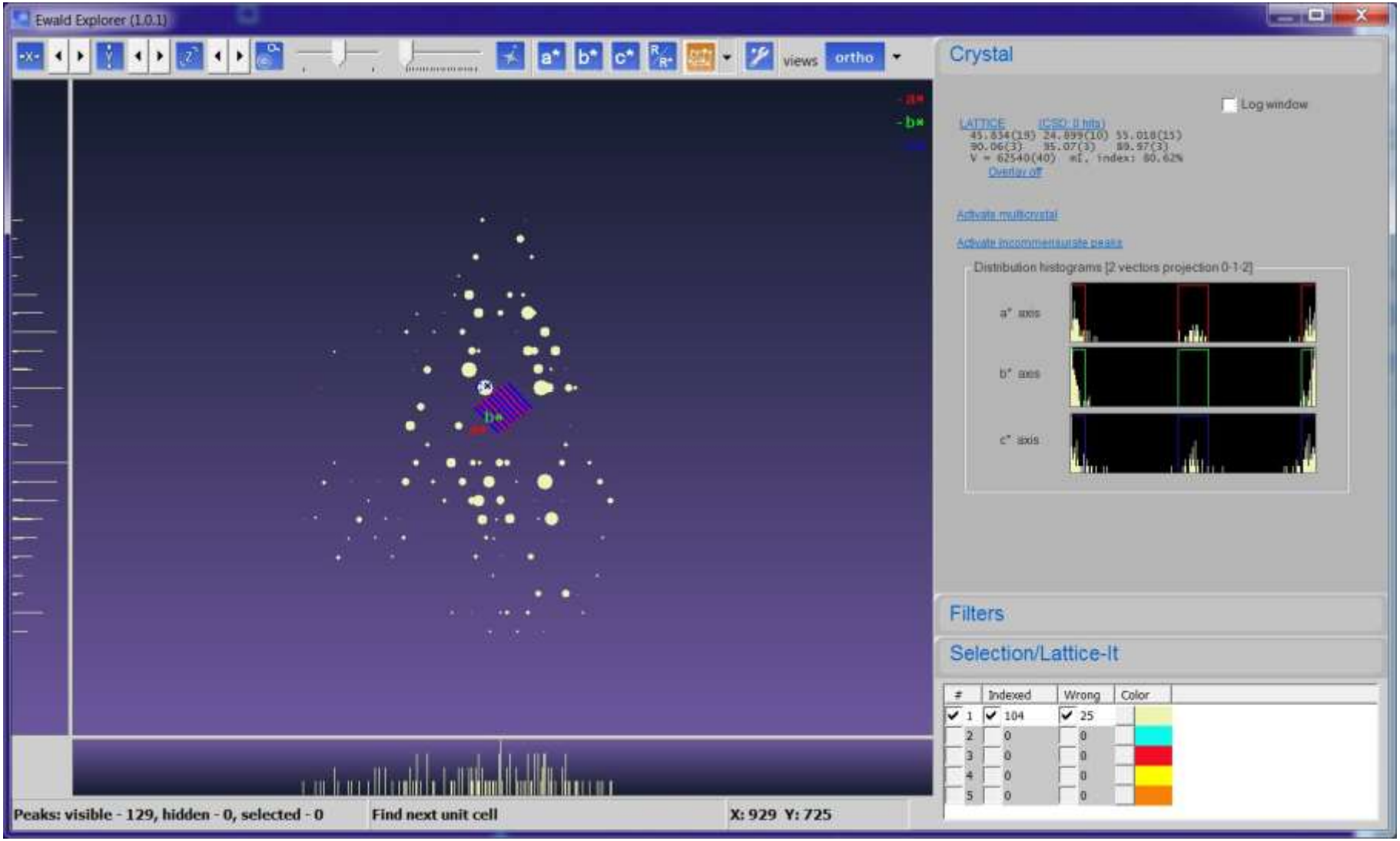
Judging a twin from few reflections can be tricky…



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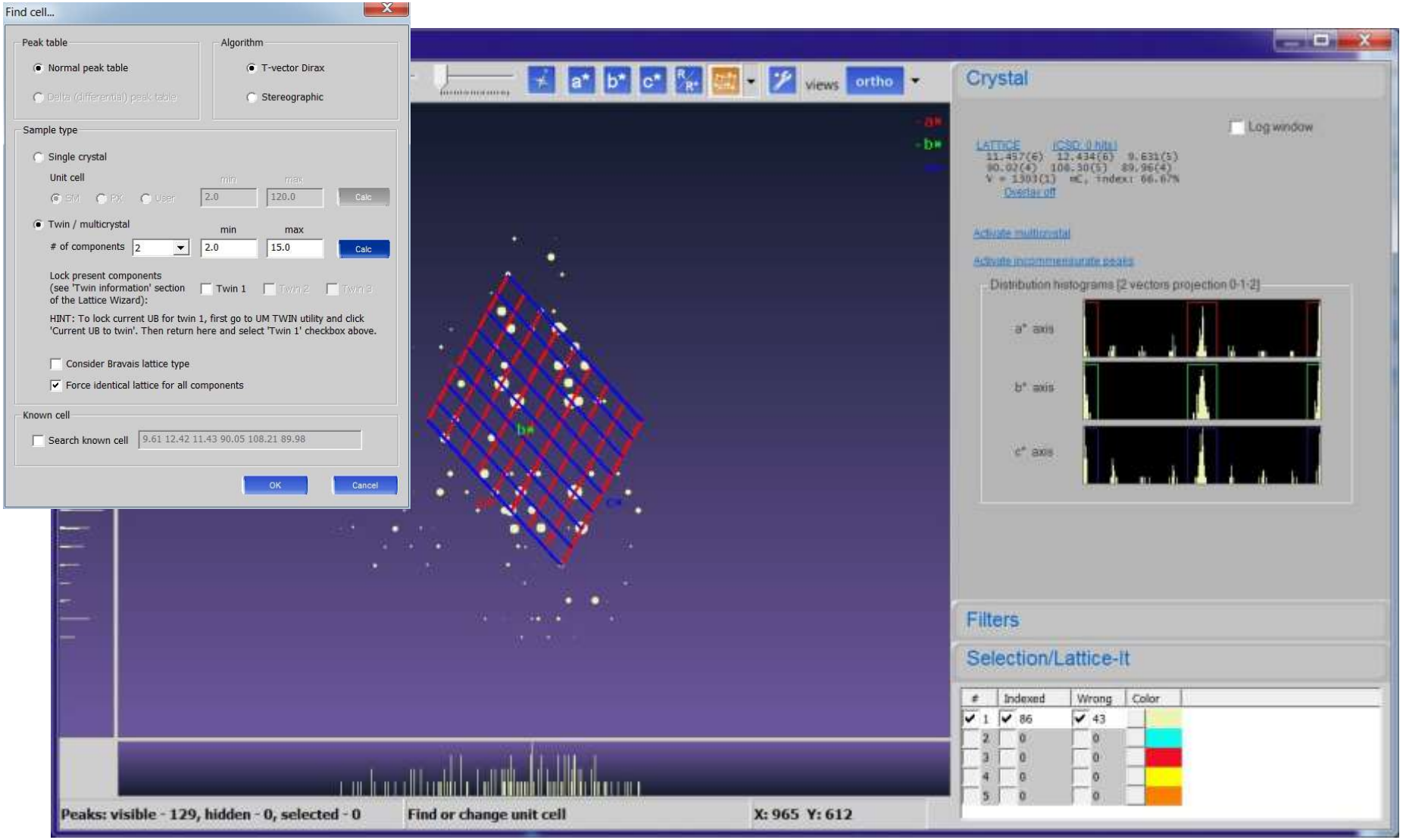
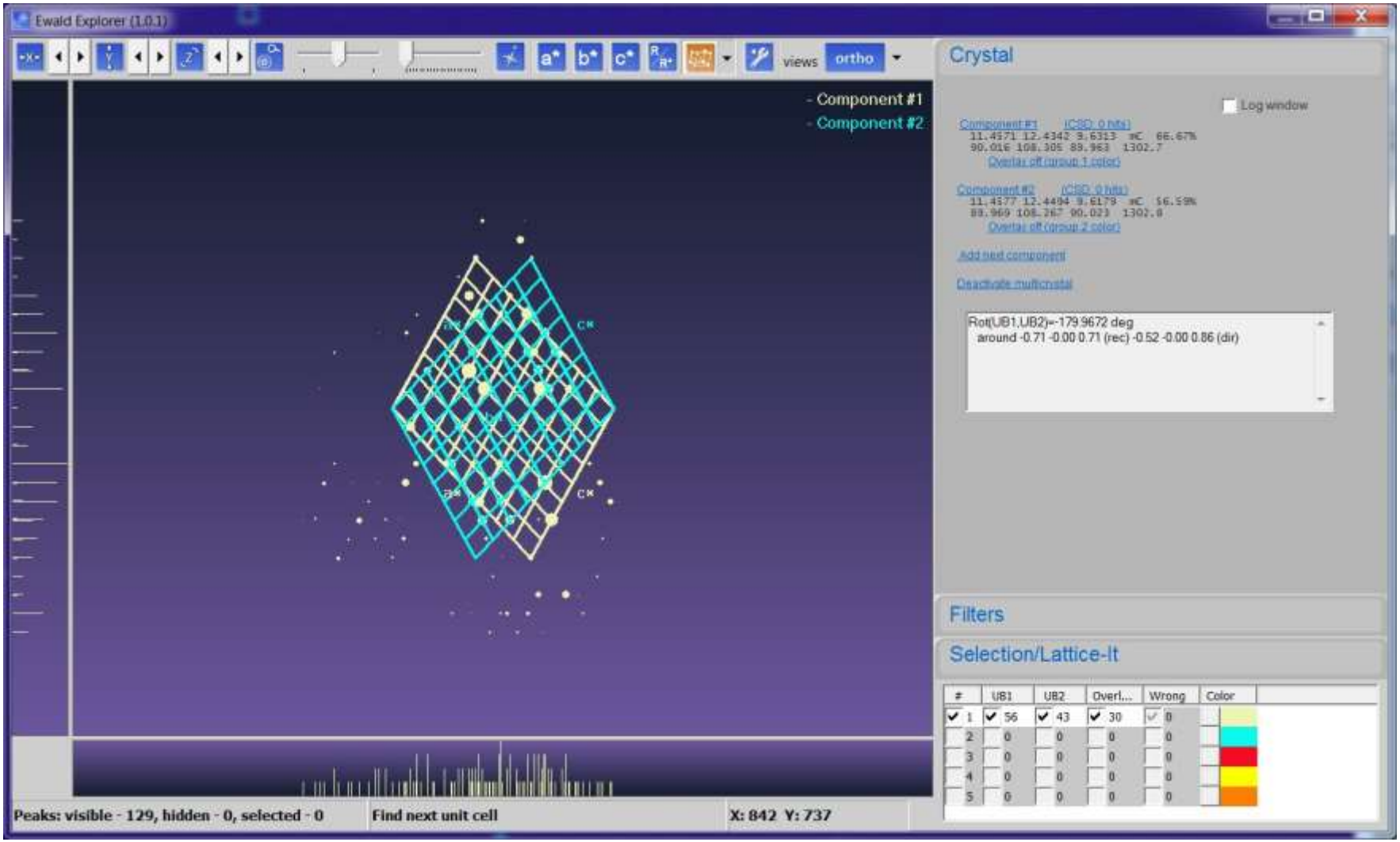
Use the custom unit cell tool…



Find the twin from the wrong peaks…



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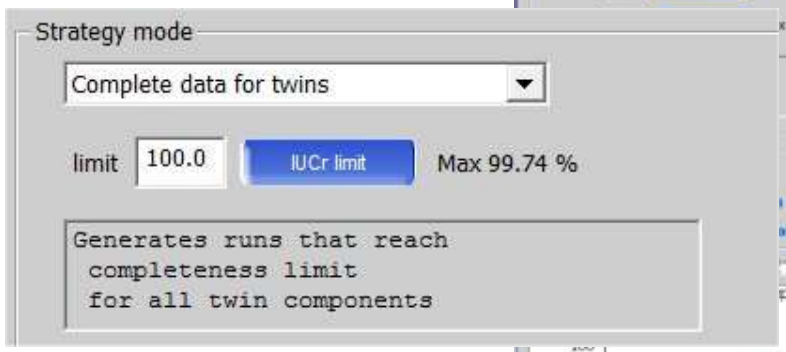
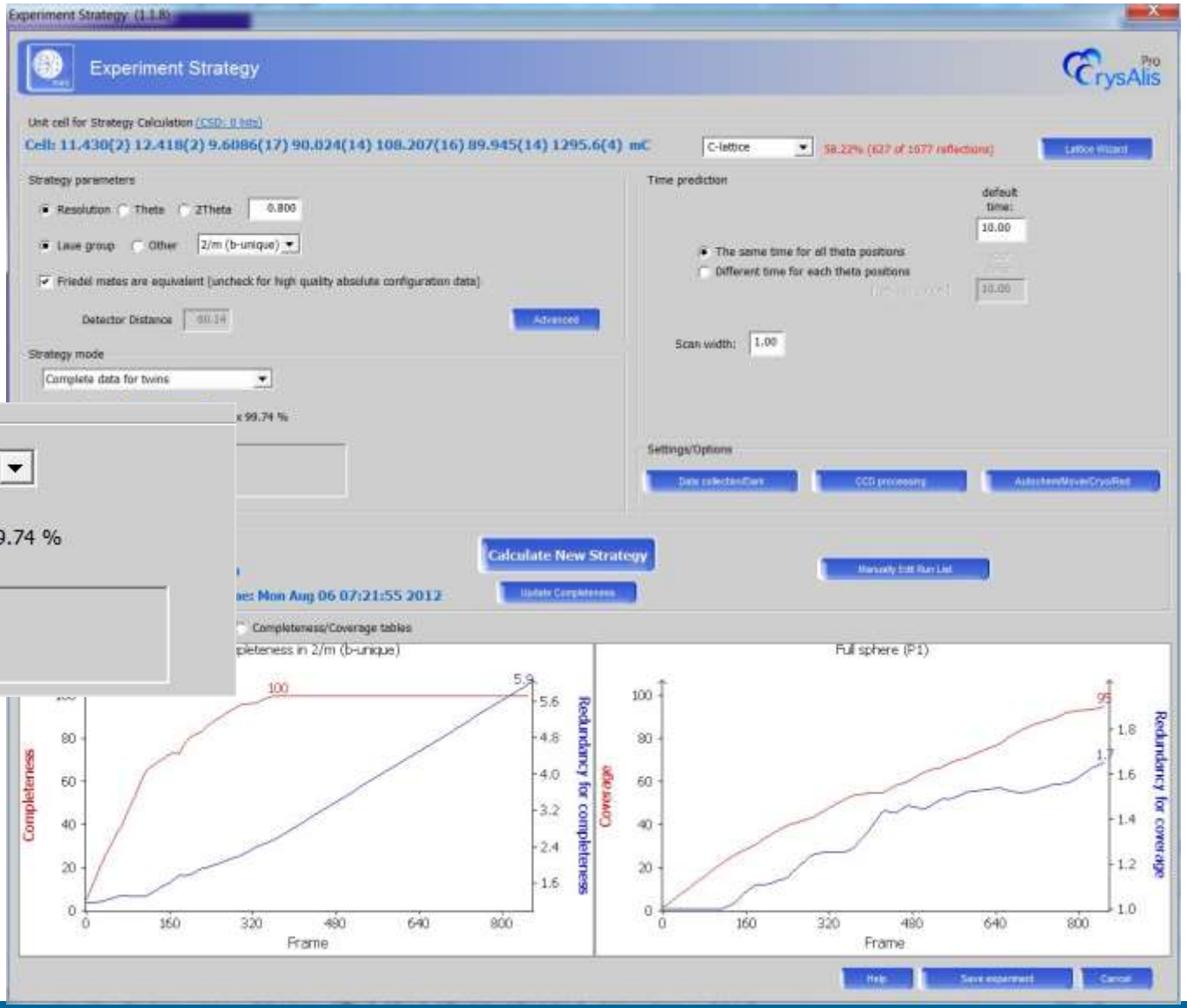
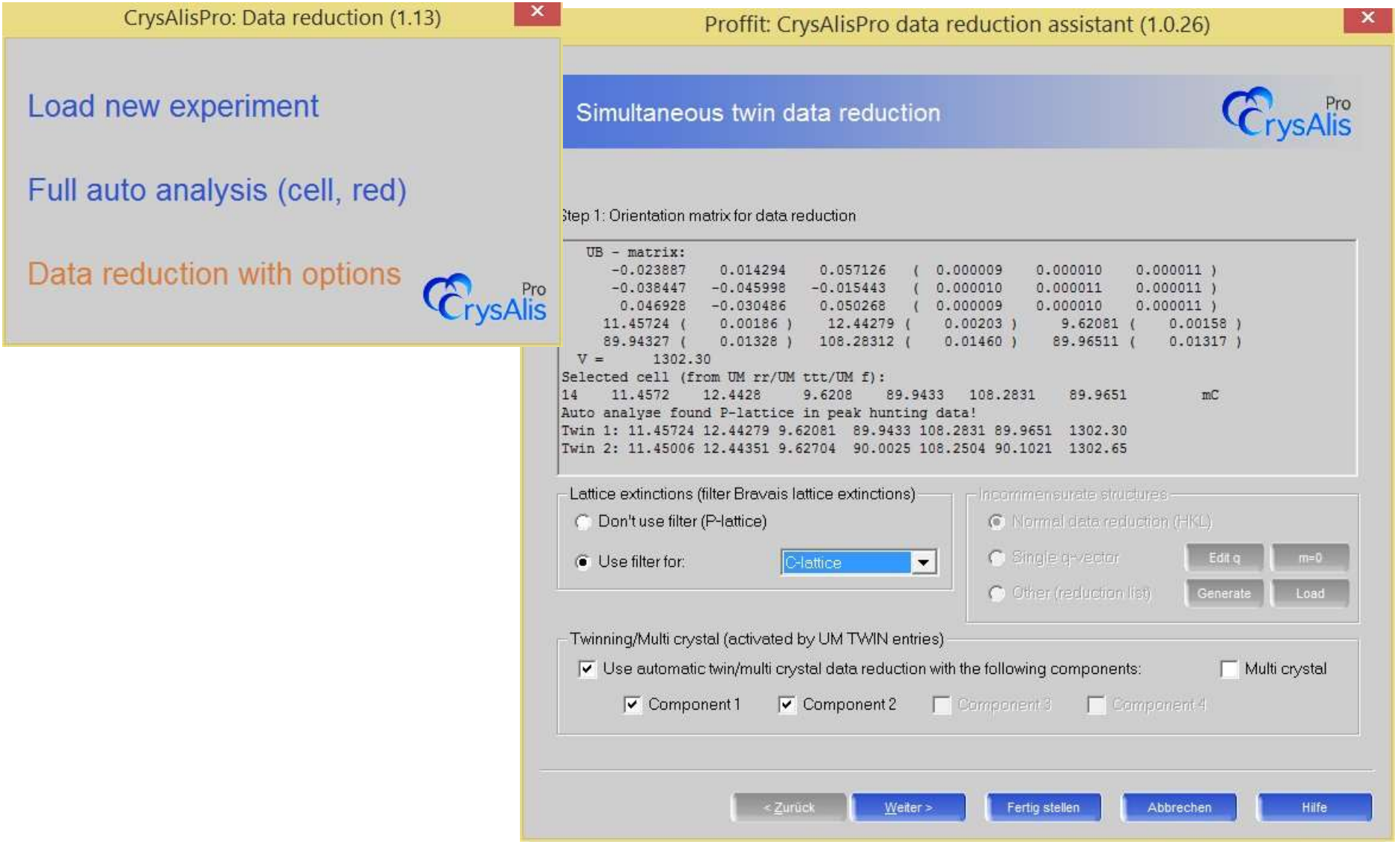
Strategy with known twin law

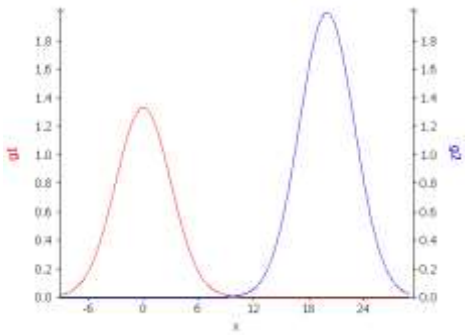




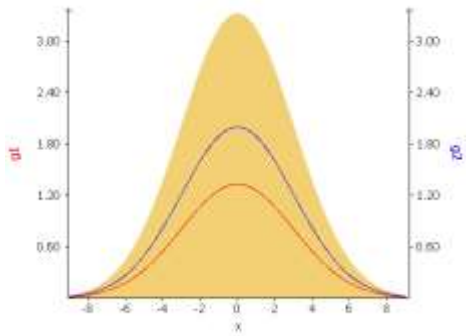
Data reduction



Twin profile data

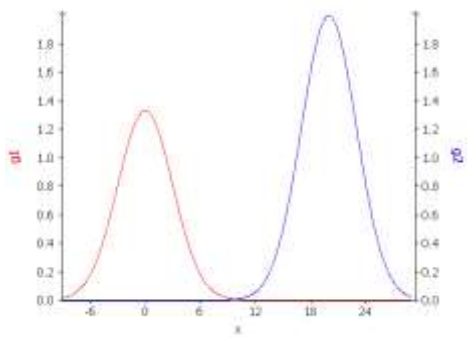


Fully separated

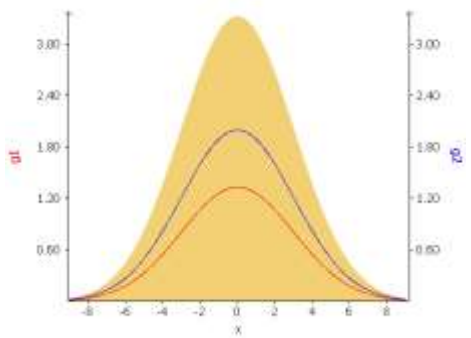


80% - 100%
Overlap data

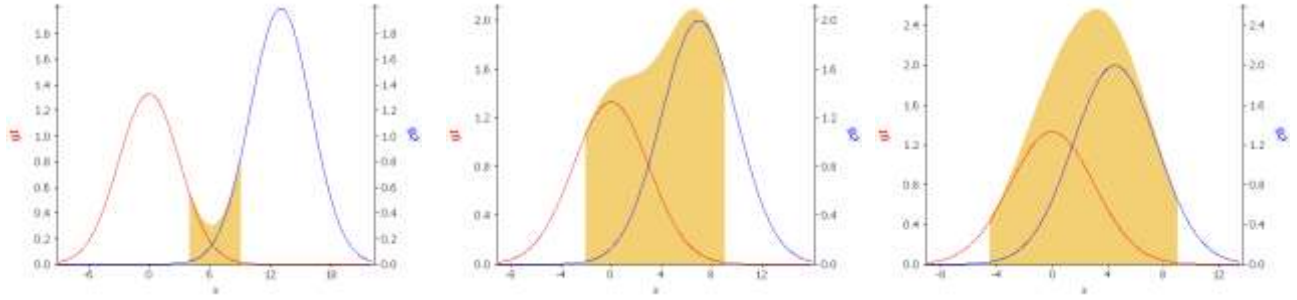
Twin profile data



Fully separated



80% - 100%
Overlap data

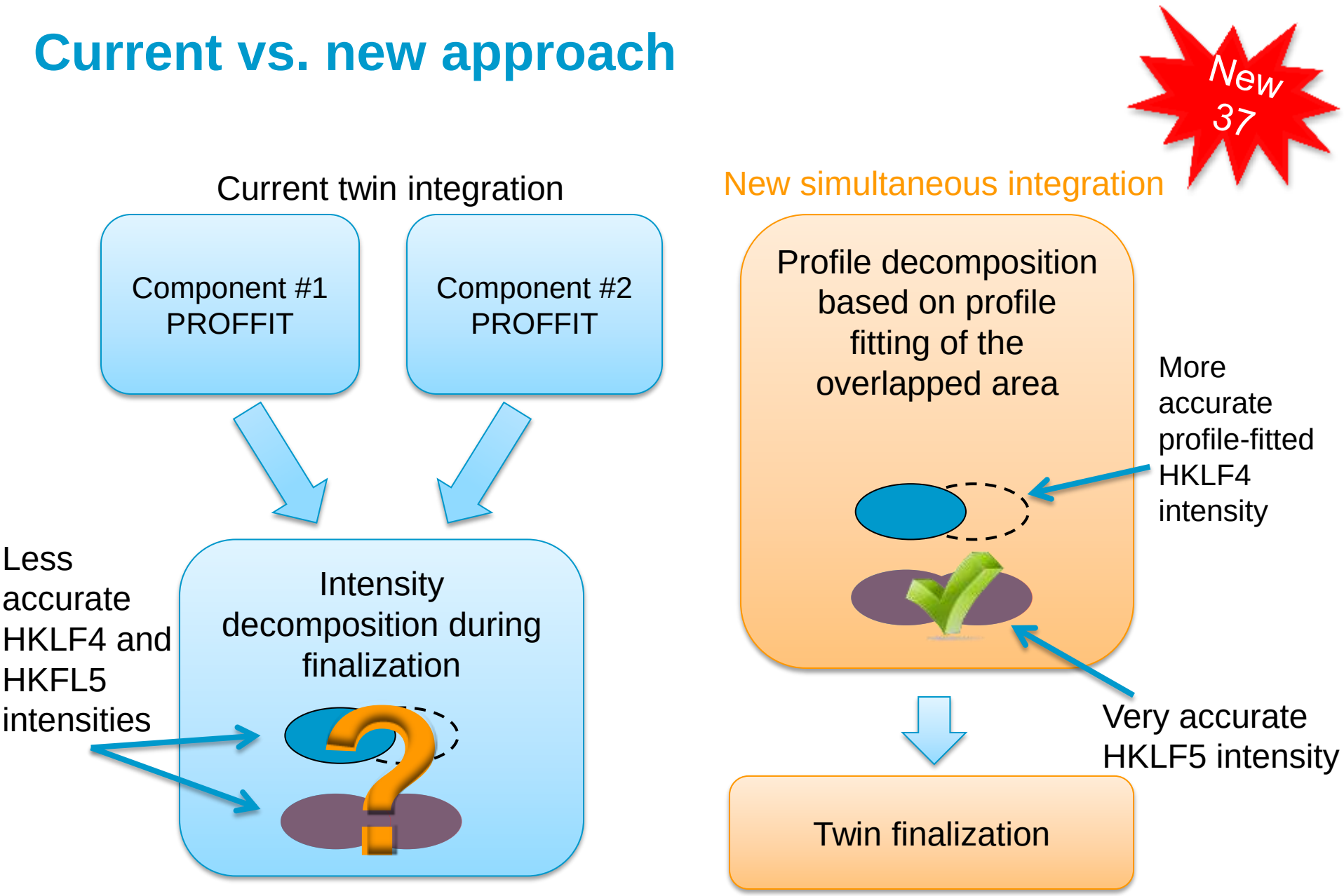


0%

Deconvolution possible

80%

Current vs. new approach

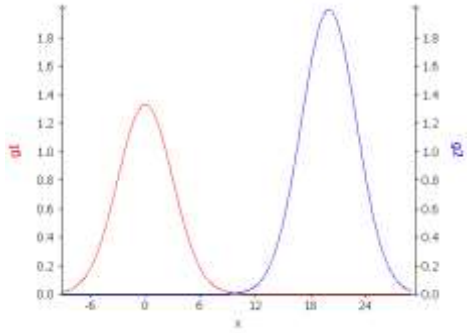


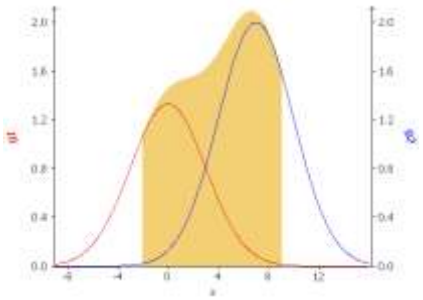
HKLF4 and HKLF5 play modes

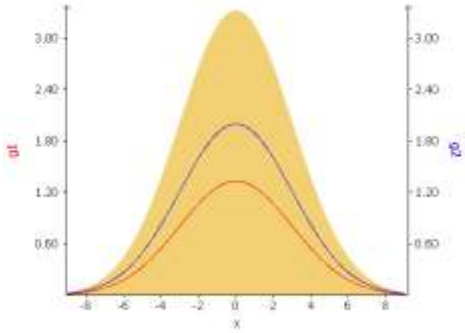
Fully separated

Partially overlapped

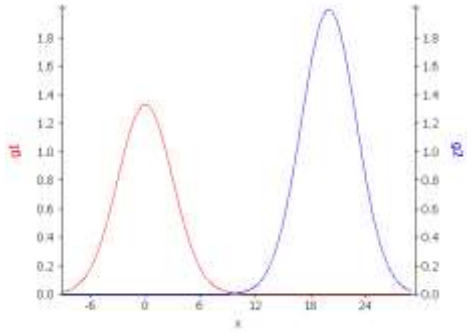
‘Full’ overlap

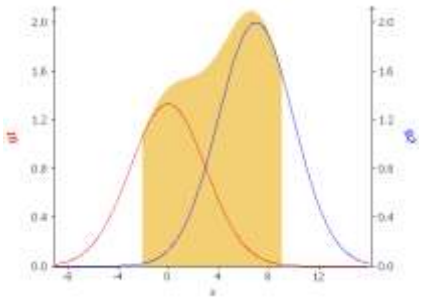


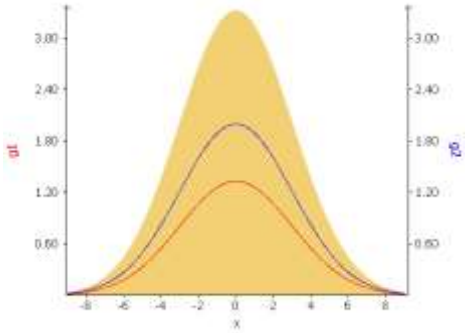




Component 1







Component 2

…

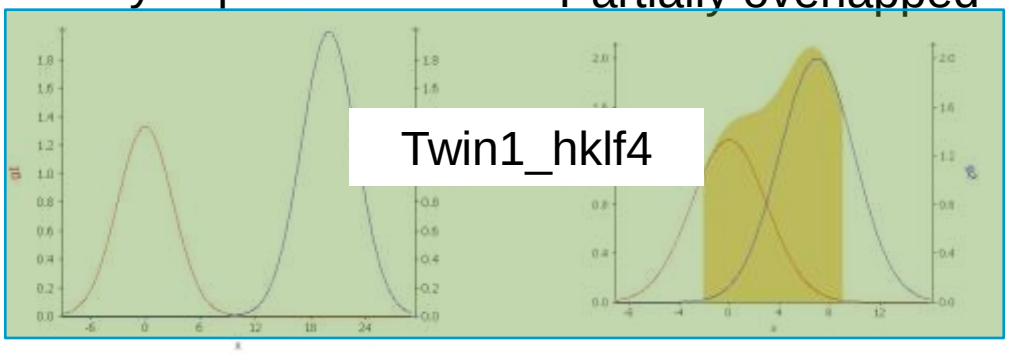
Component n

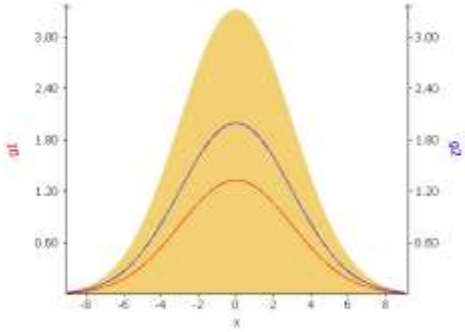
HKLF4 and HKLF5 play modes: 1st trial

Fully separated

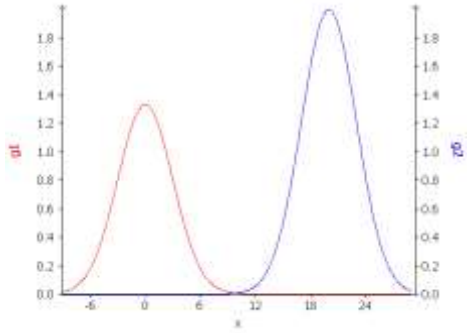
Partially overlapped

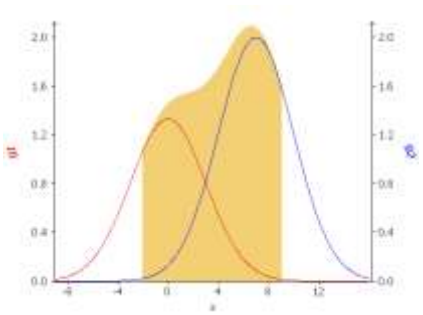
'Full' overlap

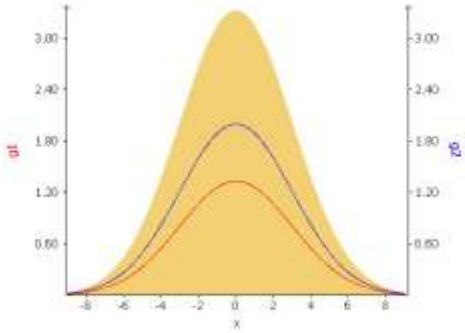




Component 1





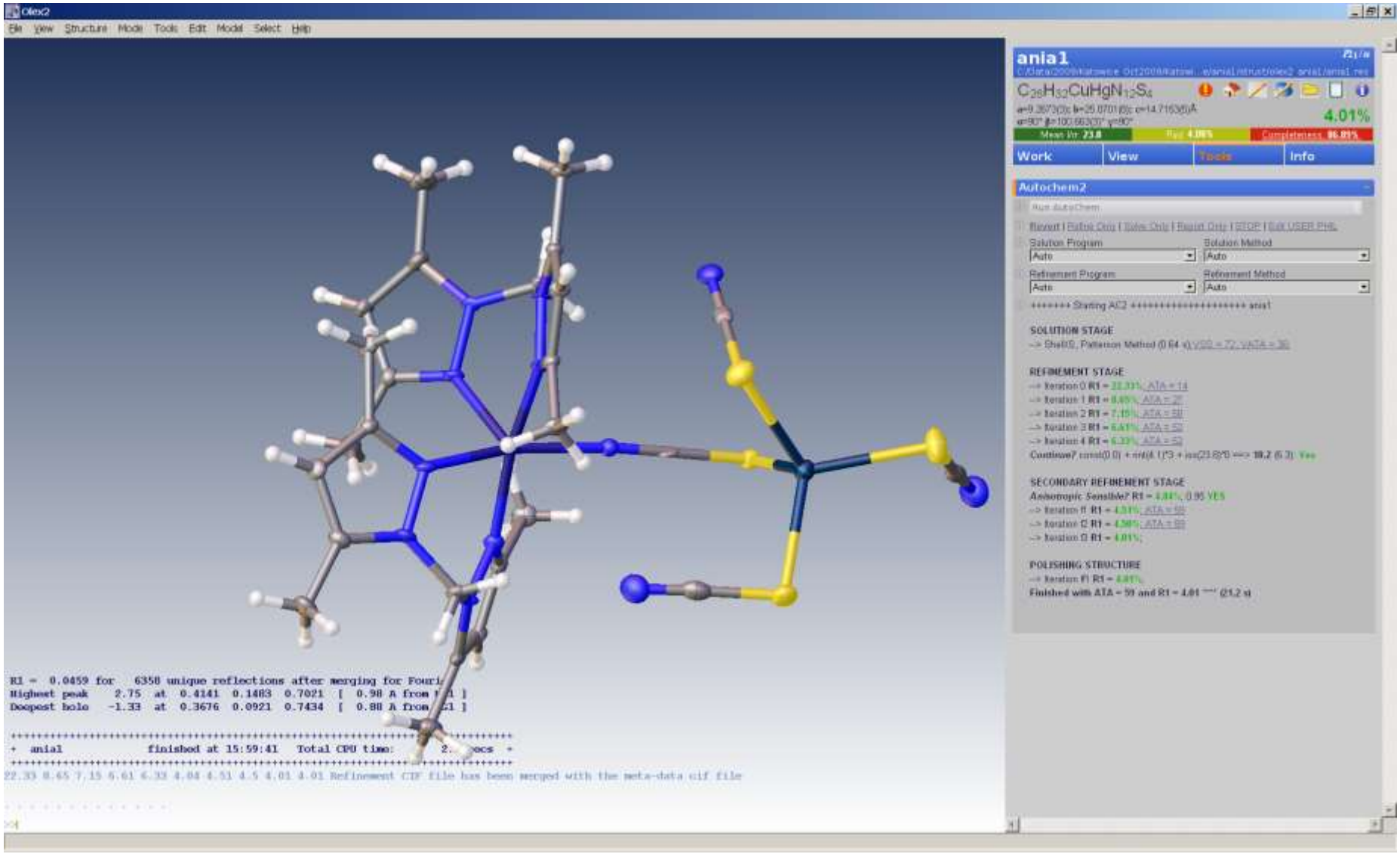


Component 2

…

Component n

Structure solve and refine using AutoChem$^{2.0}$



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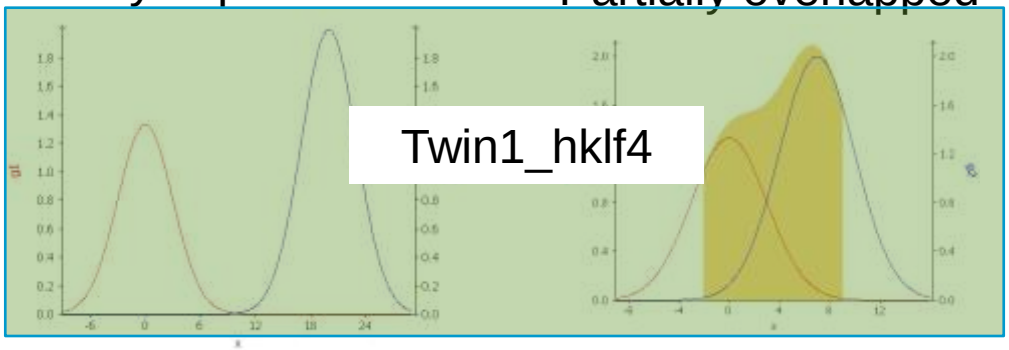
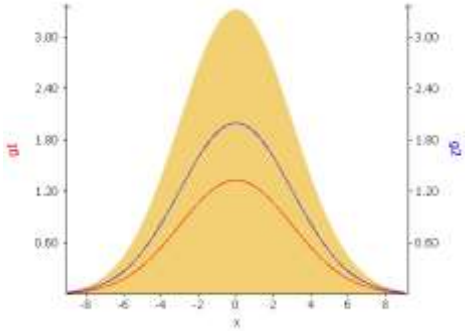
Get extra data in to solve the structure…

Fully separated

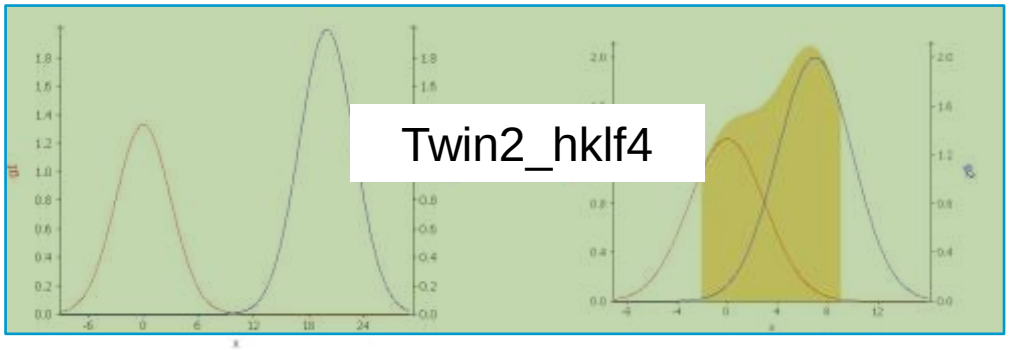
Partially overlapped

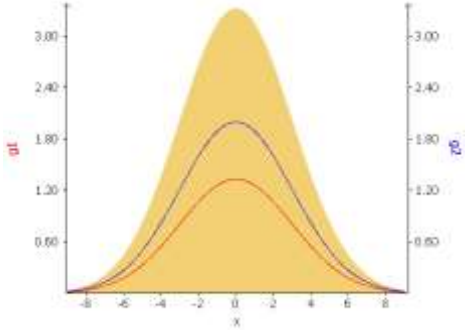
‘Full’ overlap





Component 1





Component 2

…

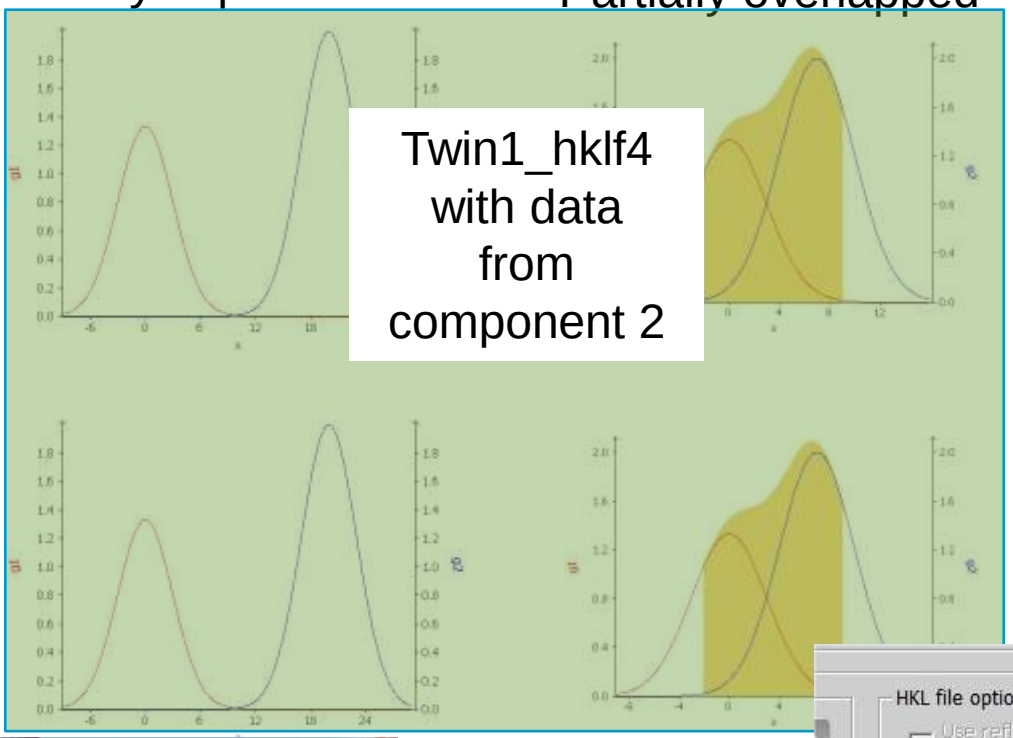
Component n

Get extra data in to solve the structure…

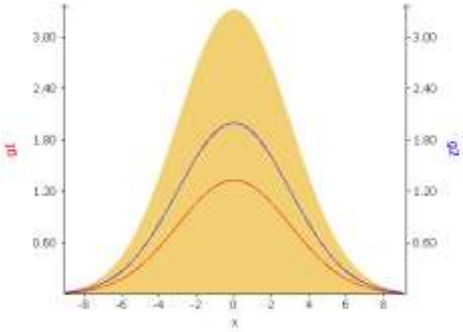
Fully separated

Partially overlapped

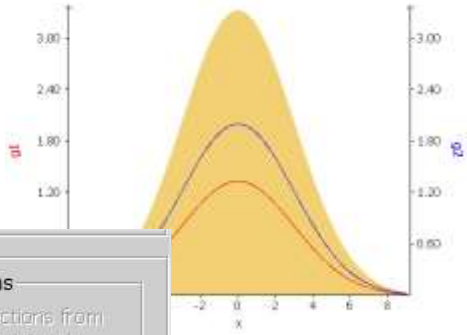
'Full' overlap



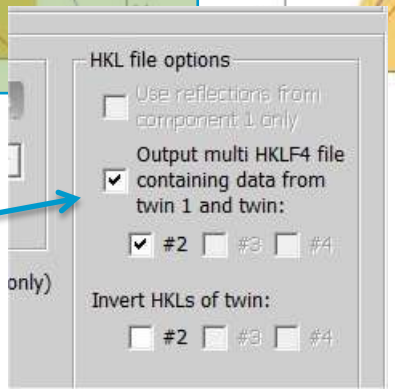
Twin1_hklf4 with data from component 2



Component 1



Component 2



…

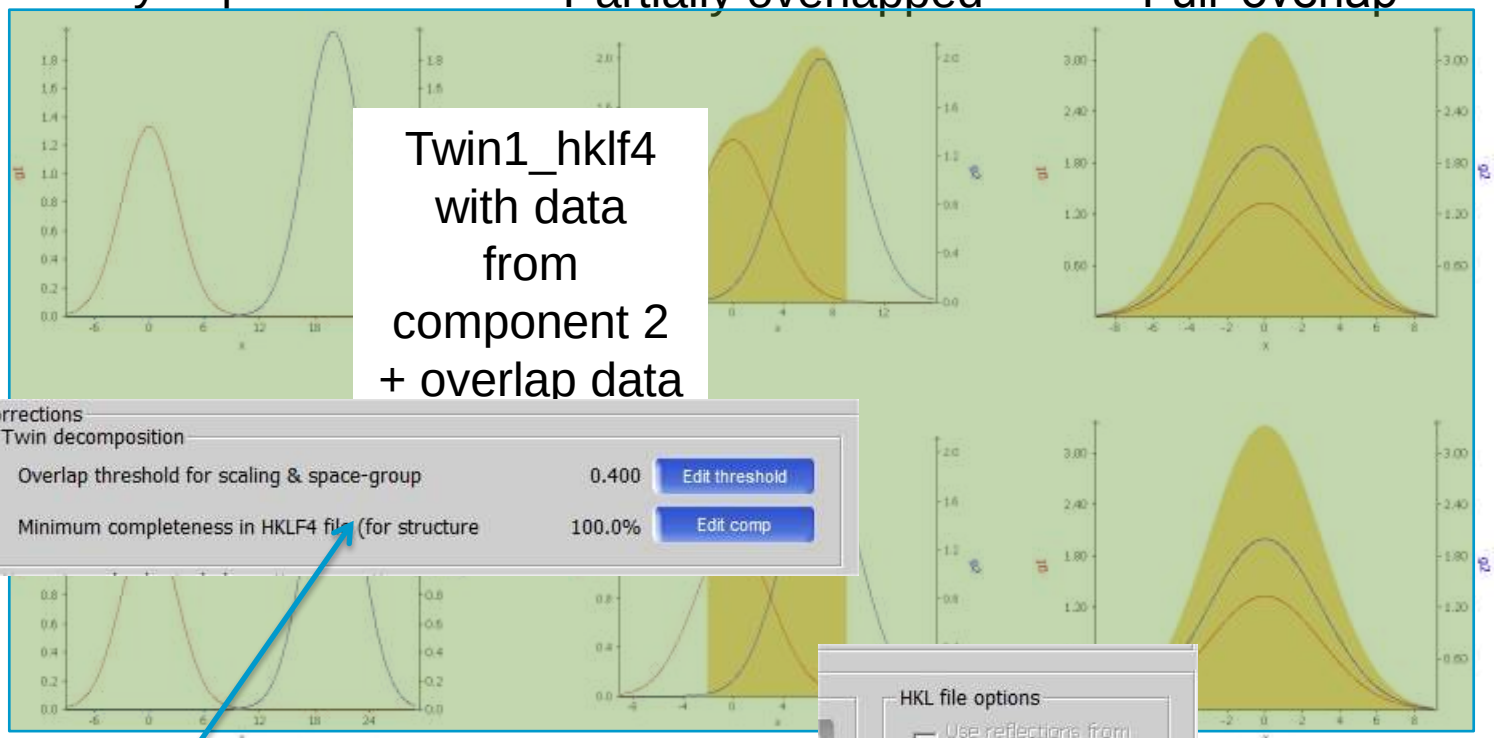
Component n

Not enough yet: Add full overlap data…

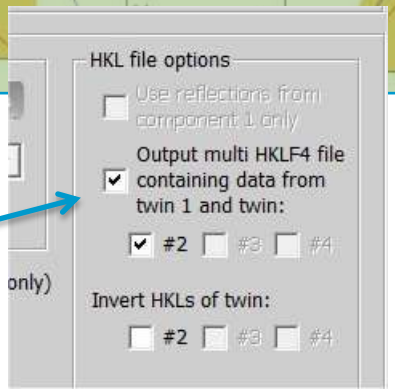
Fully separated

Partially overlapped

'Full' overlap



Twin1_hklf4 with data from component 2 + overlap data



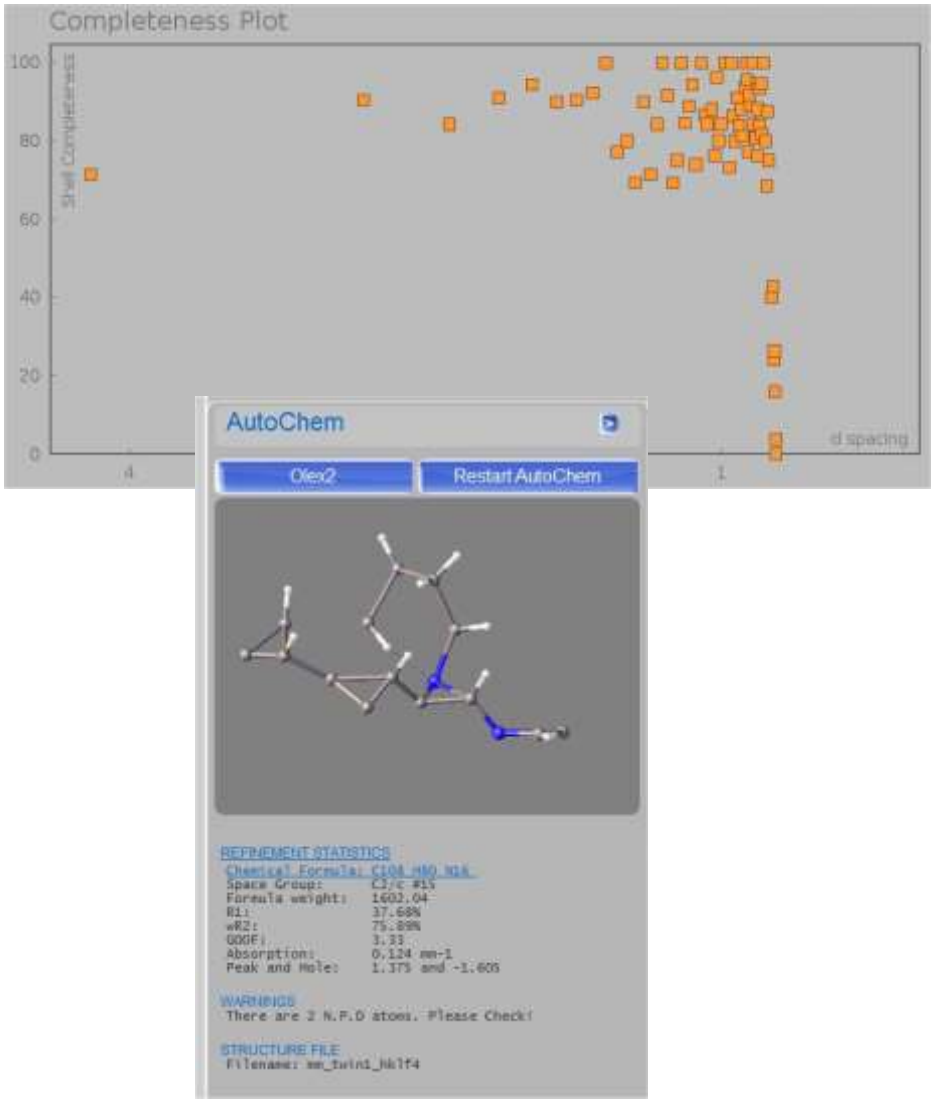
Component 1

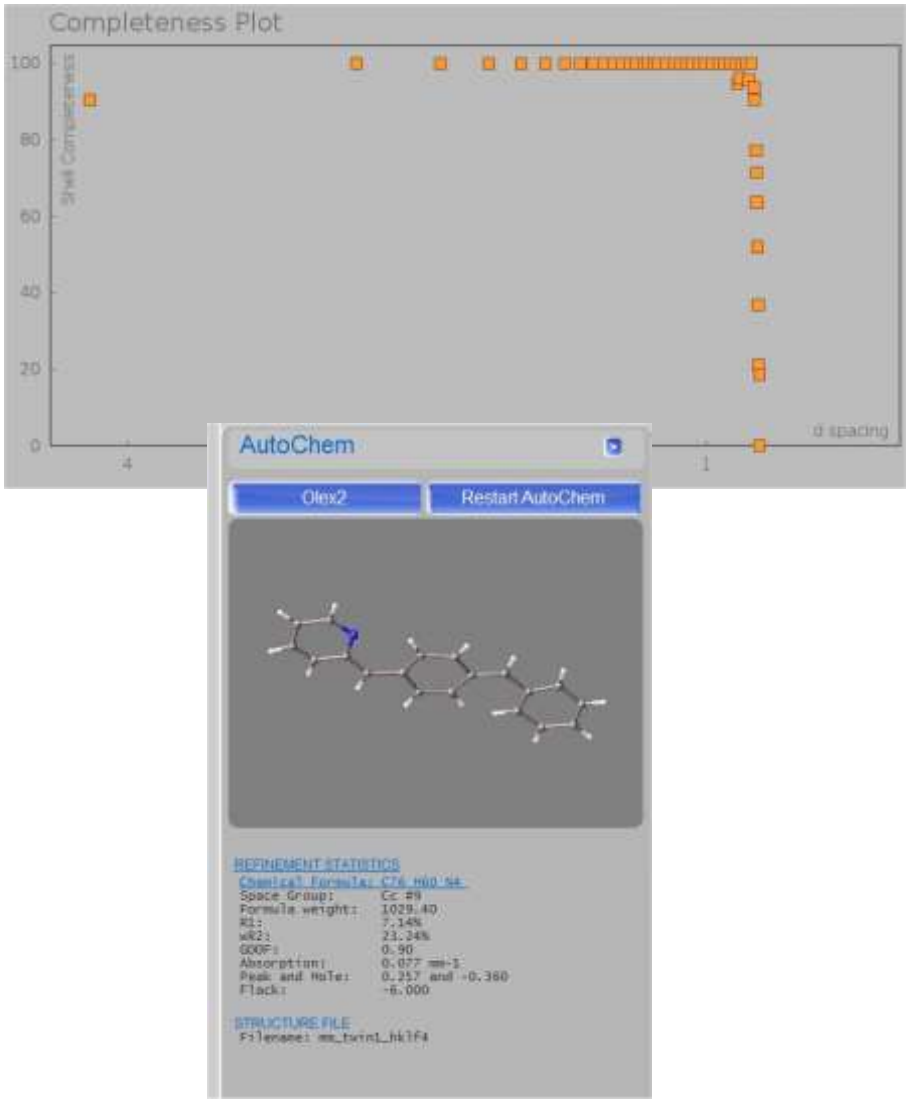
Component 2

…

Component n

Why?


Completeness Plot
Shell Completeness
100
80
60
40
20
0
4
1
d spacing
AutoChem
Olex2
Restart AutoChem
REFINEMENT STATISTICS
Chemical Formula: C104 H80 N4
Space Group: C2/c #15
Formula weight: 1602.04
R1: 17.68%
wR2: 75.89%
GOOF: 3.33
Absorption: 0.124 mm-1
Peak and Hole: 1.375 and -1.605
WARNINGS
There are 2 N.P.D atoms. Please Check!
STRUCTURE FILE
Filename: mm_twin1_hklf4


Completeness Plot
Shell Completeness
100
80
60
40
20
0
4
1
d spacing
AutoChem
Olex2
Restart AutoChem
REFINEMENT STATISTICS
Chemical Formula: C76 H60 N4
Space Group: Cc #9
Formula weight: 1029.40
R1: 7.14%
wR2: 23.34%
GOOF: 0.90
Absorption: 0.077 mm-1
Peak and Hole: 0.257 and -0.360
Flack: -6.000
STRUCTURE FILE
Filename: mm_twin1_hklf4

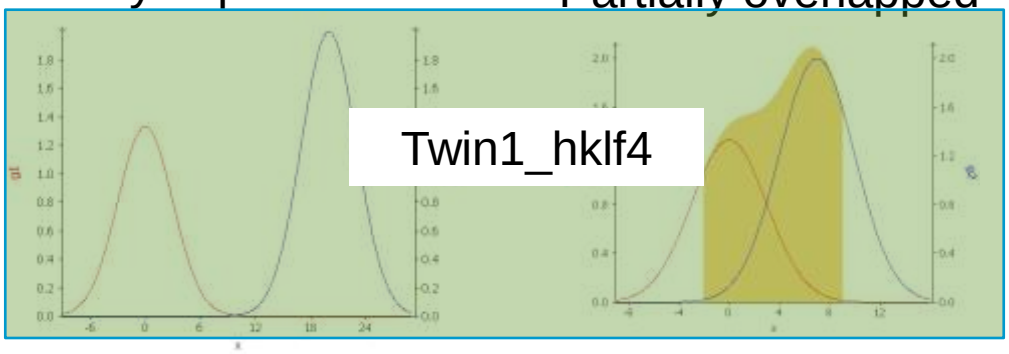
Solve done: Refine on good hklf4

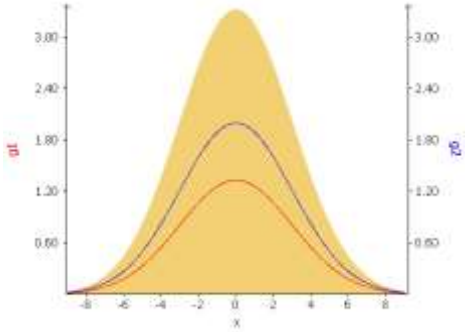
Fully separated

Partially overlapped

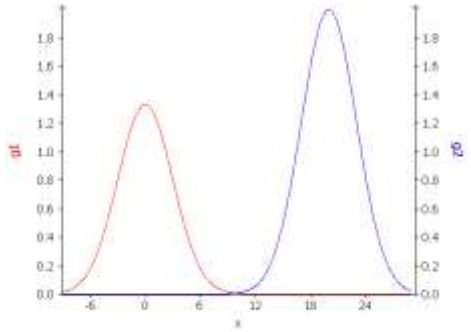
'Full' overlap

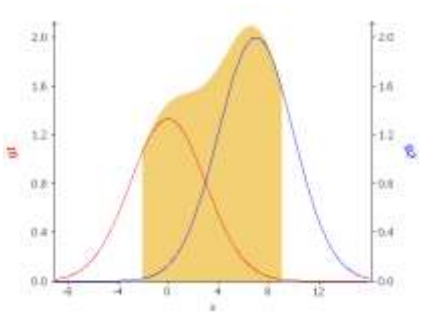
Twin1_hklf4

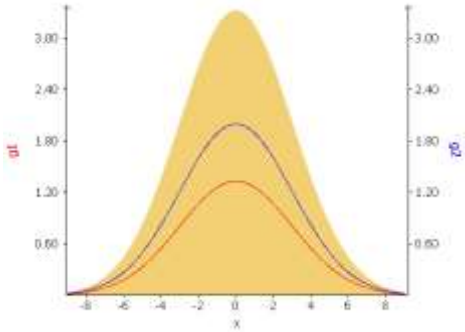




Component 1







Component 2

…

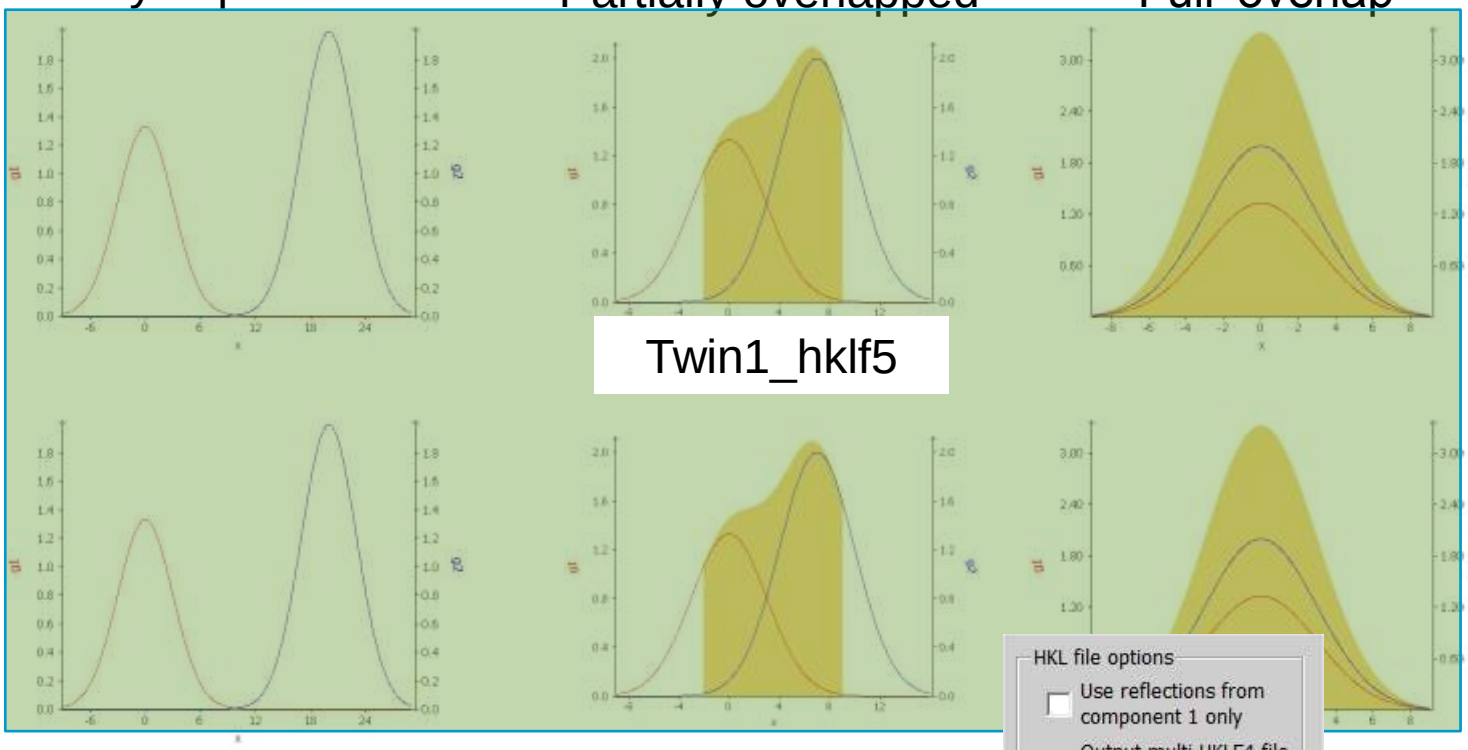
Component n

Solve done: Refine on good hklf5

Fully separated

Partially overlapped

'Full' overlap



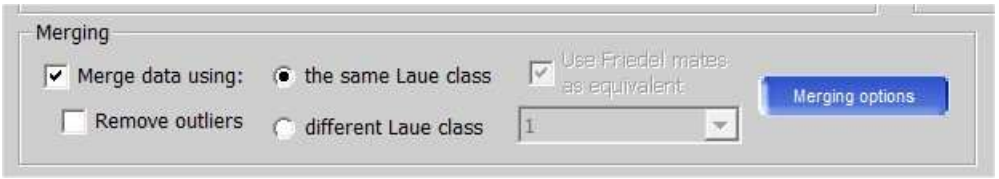
Twin1_hklf5

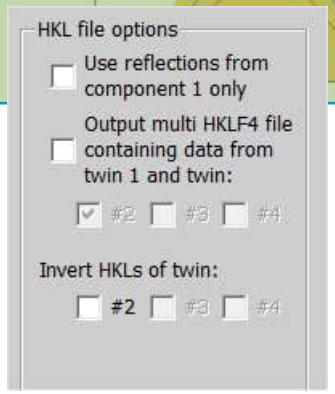
Component 1

Component 2

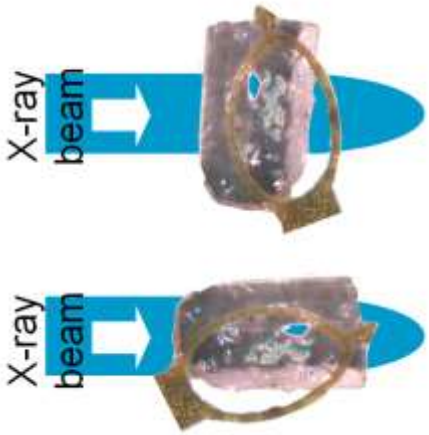
…

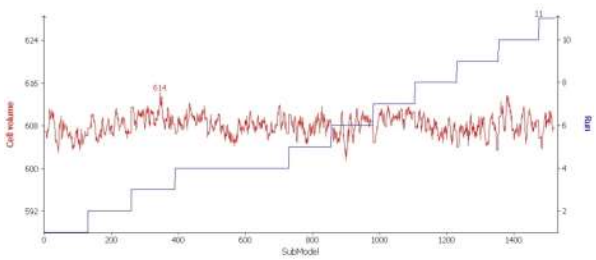
Component n

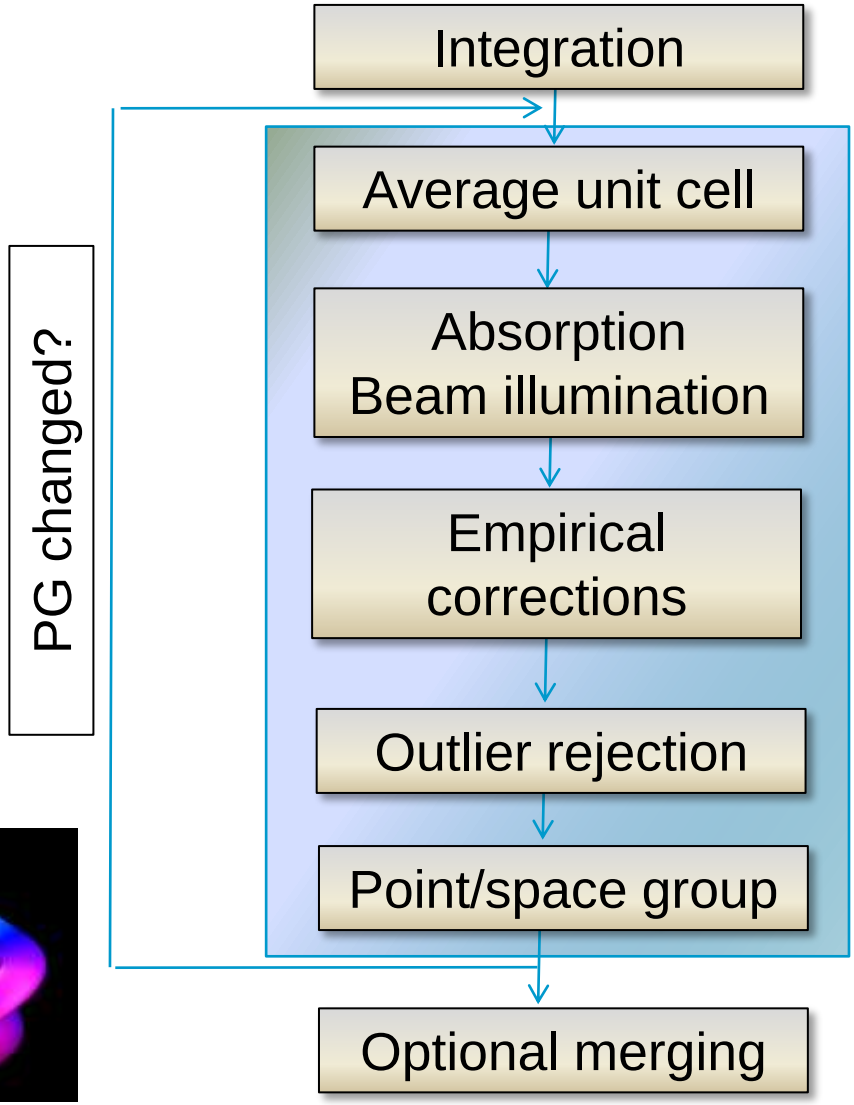




Good data quality through full post corrections





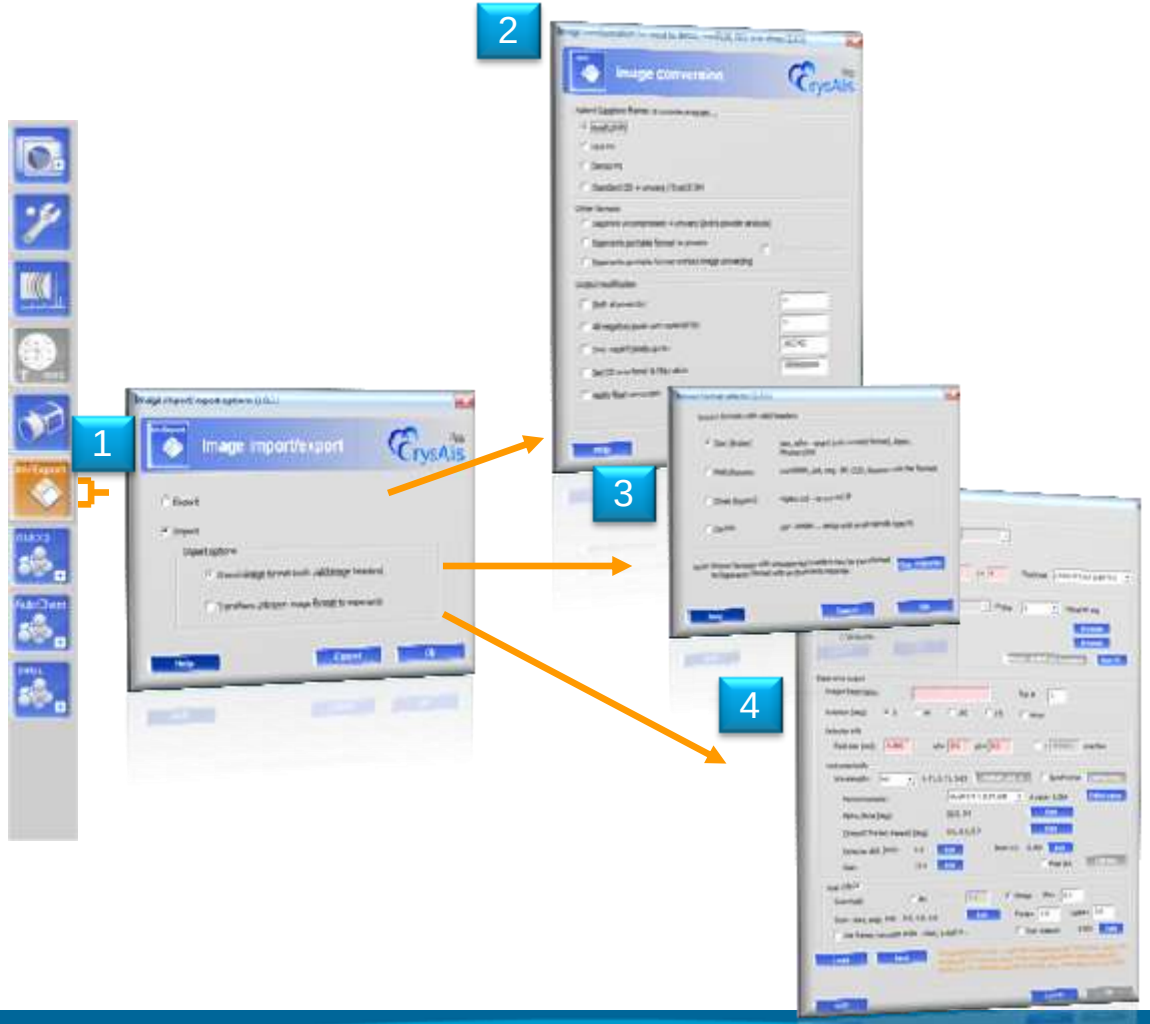


Summary - twin

- Twins can be recognized early on during screeing/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.

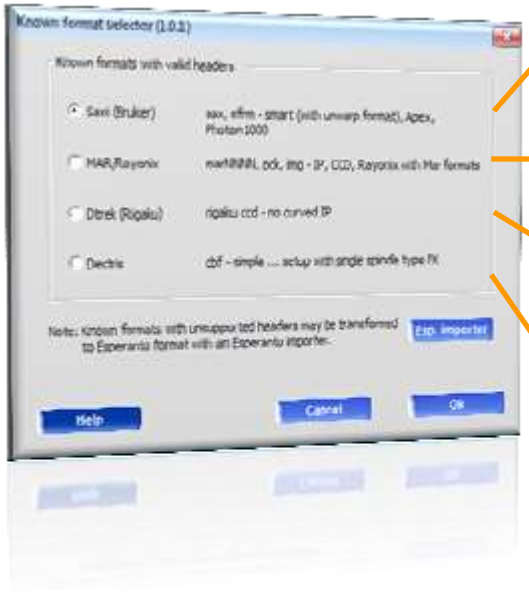
Import/export from/to external formats

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.



Known format import

1. Clean information which image types are supported.
2. No command needed for creation of the run list.
3. Access to Esperanto importer in case of incompatible format.



SAXI CREATERUNLIST

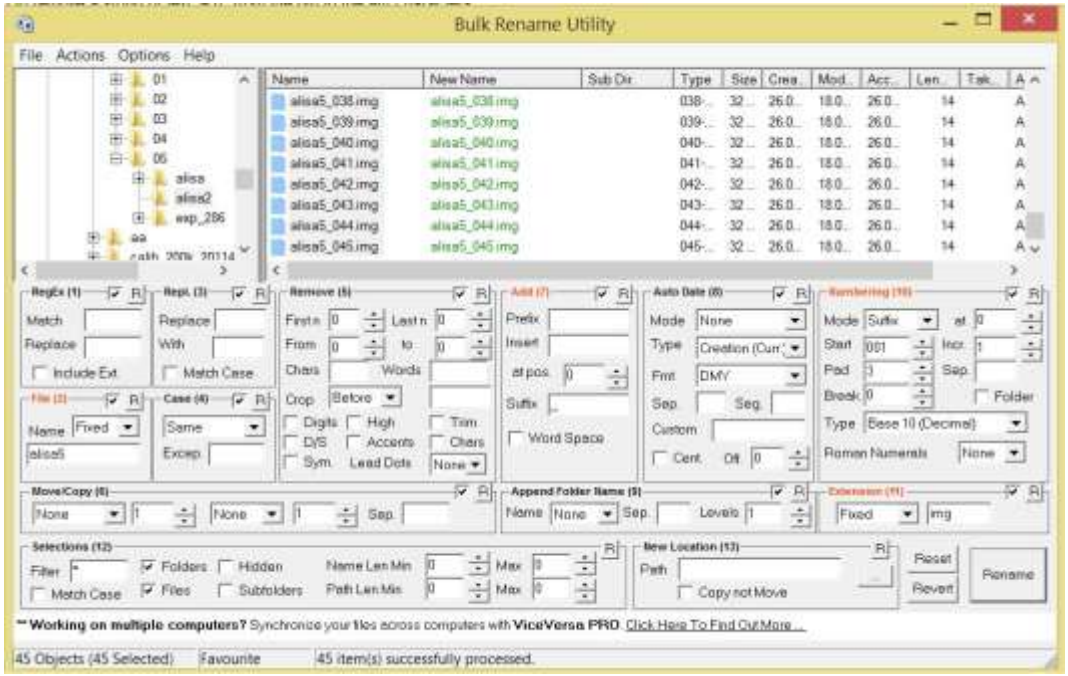
MAR EXPERIMENTSETUP

DTREK CREATERUNLIST

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)

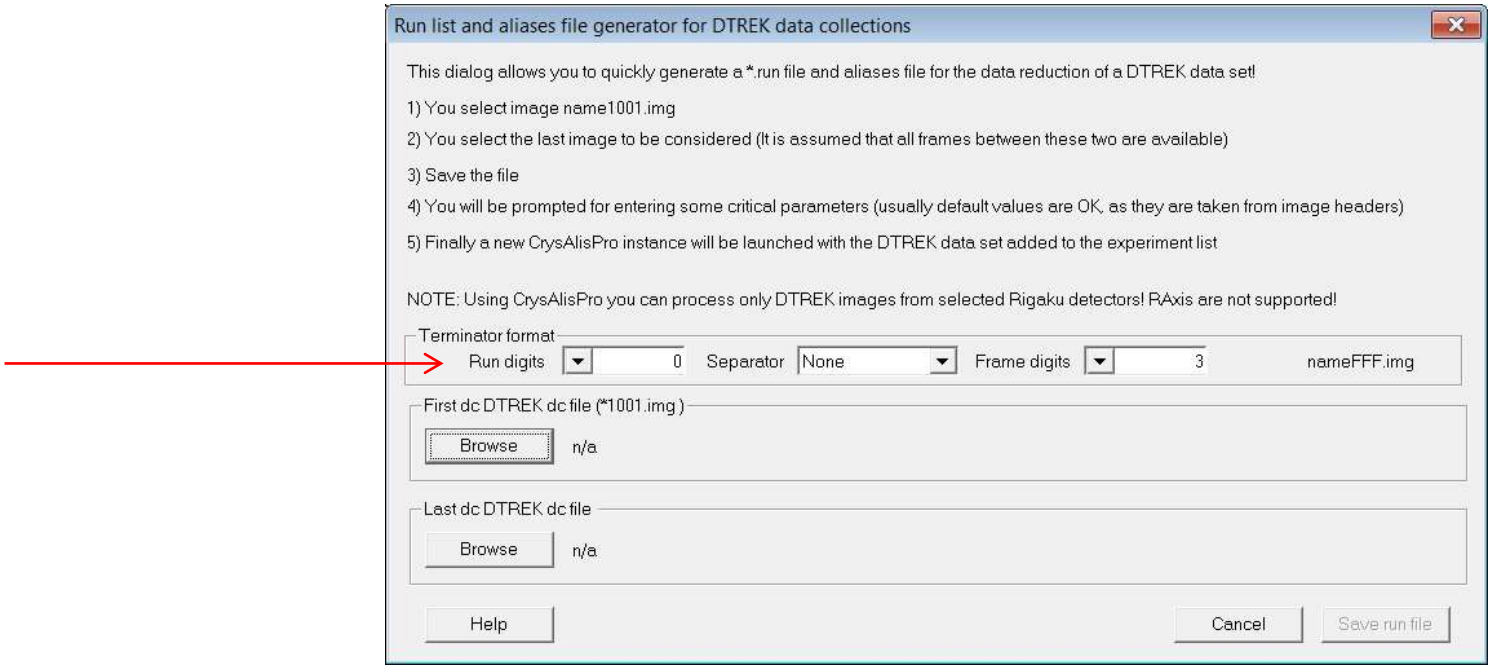
- Data from photon100 detectors can be processed.
- The following observations can be made:

- 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 (`[dsithicknessmmforpixeldetector] - 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

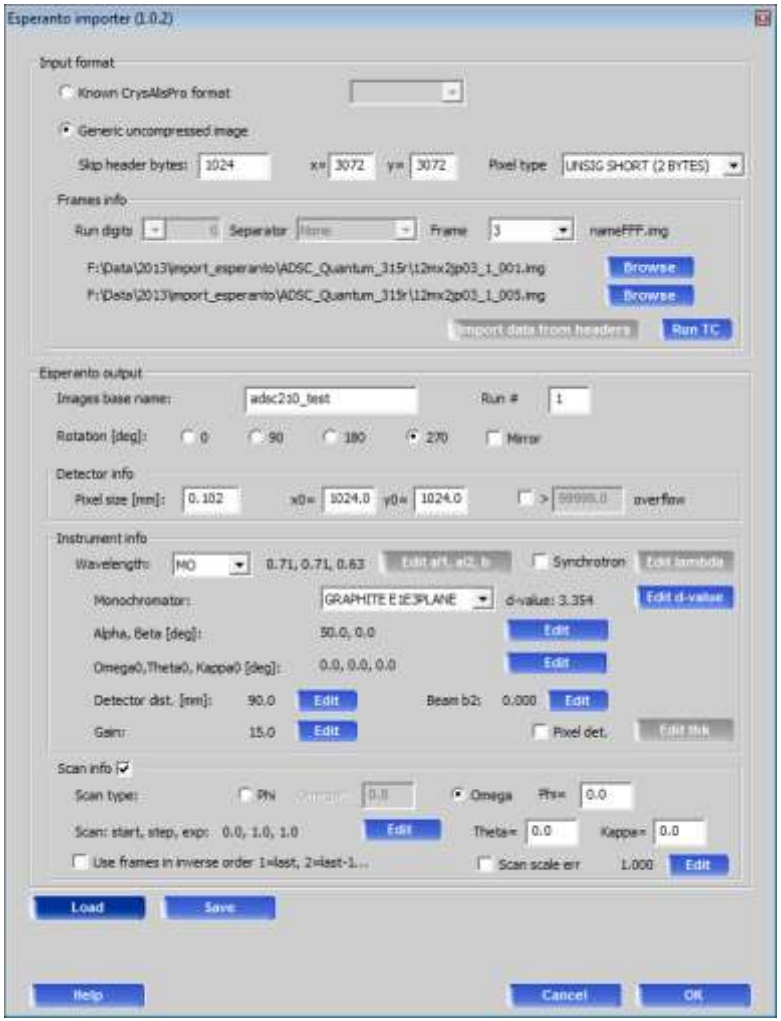
André Rothkirch, G. Diego Gatta, Mathias Meyer, Sébastien Merkel, Marco Merlini and Hanns-Peter Liermann

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



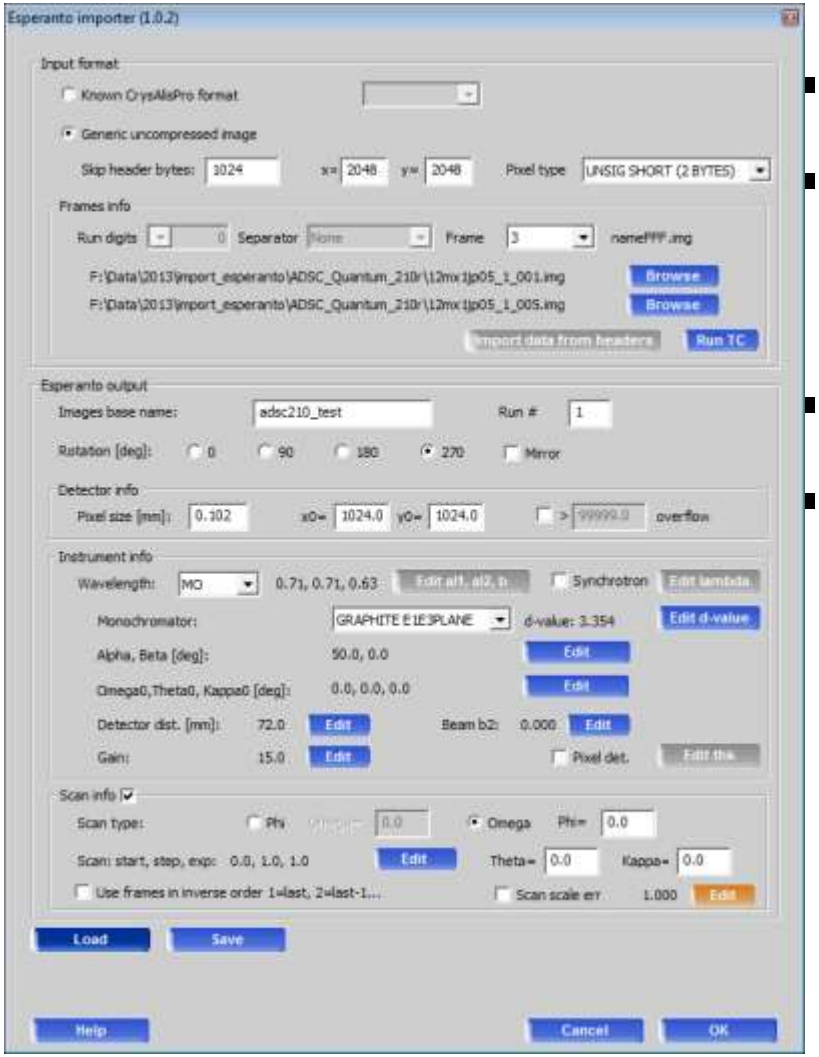
Command dc rit

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

Then esperanto createrunlist

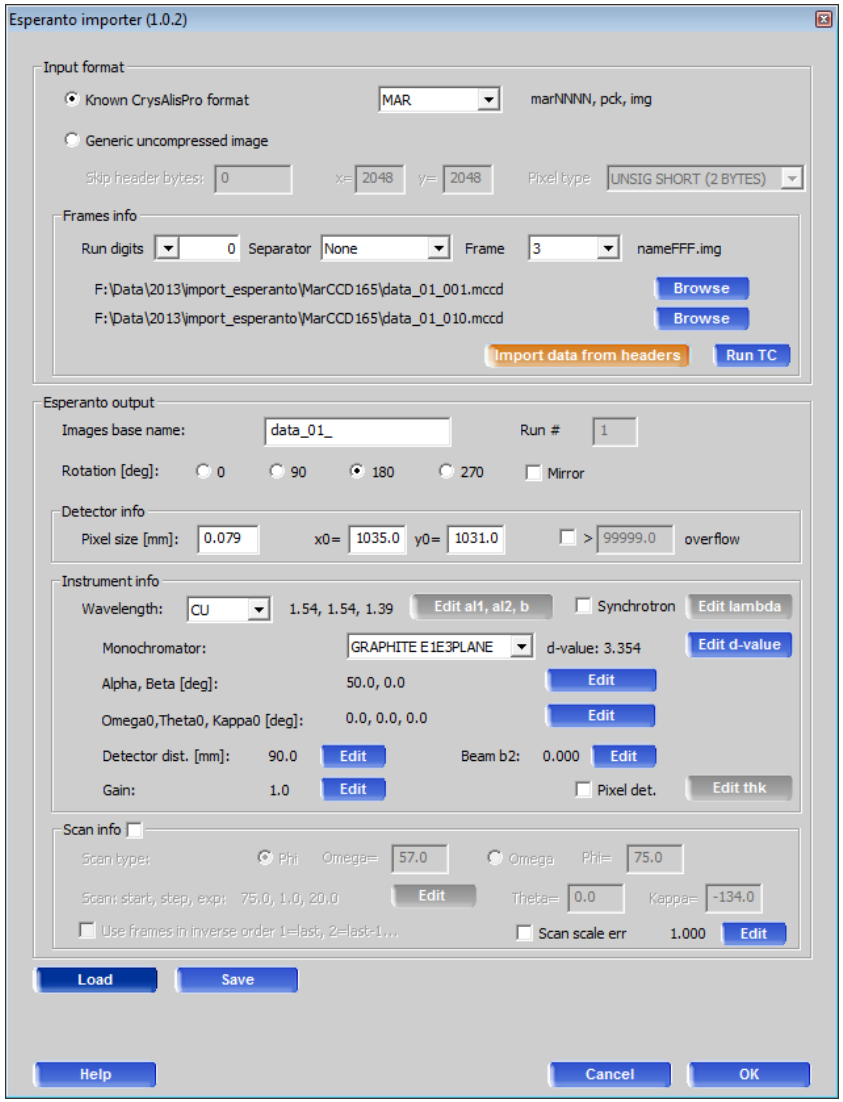
Slight play in EwaldPro to get the center right.

Dc rit: ADSC 210



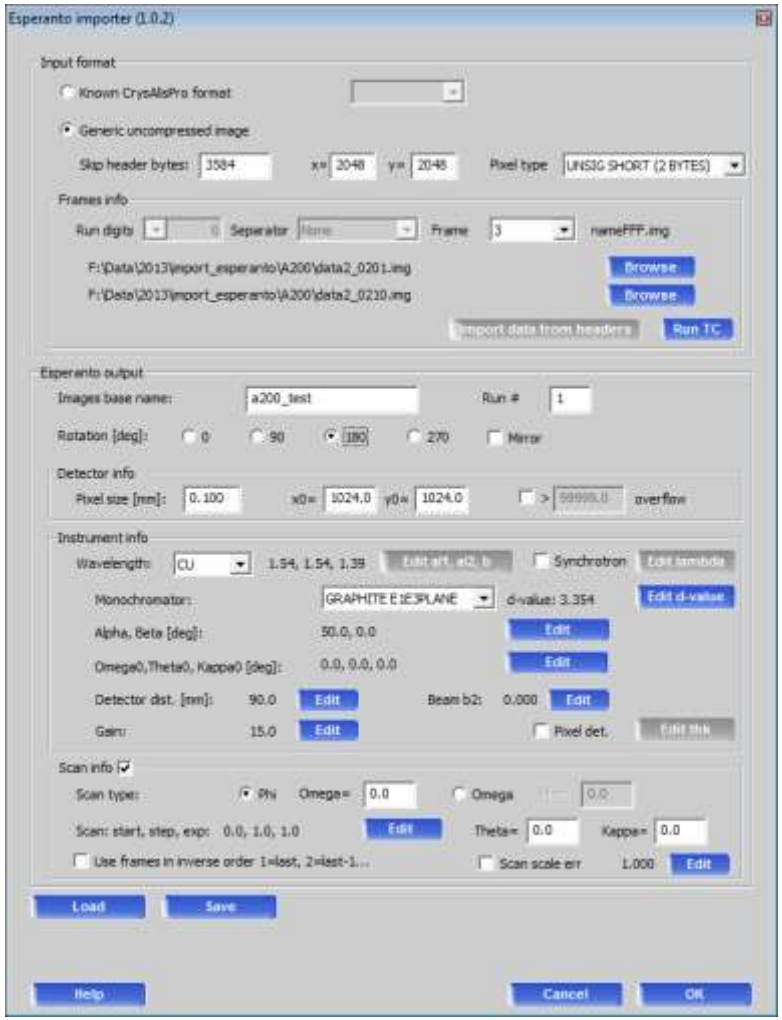
- Command dc rit
- Header bytes 1024, x 2048 y 20482 and other info from text header (f.ex with total commander)
- Then esperanto createrunlist
- Slight play in EwaldPro to get the center right.

Dc rit: MAR165ccd



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

Dc rit: A200 detector

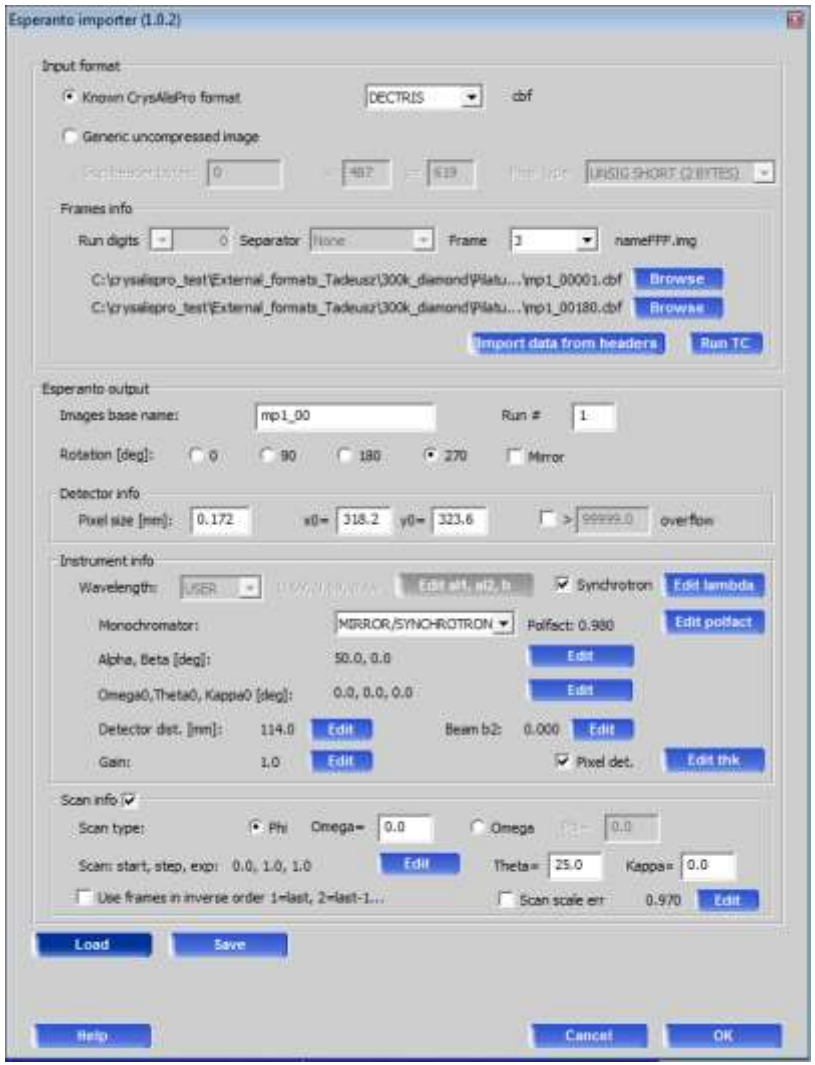


- Command dc rit
- Header bytes 3584, x 2048 y 20482 and other info from text header (f.ex with total commander)
- Then esperanto createrunlist
- Slight play in EwaldPro to get the center right.

Dc rit: Diamond ID 19 Dectris turned

- 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.

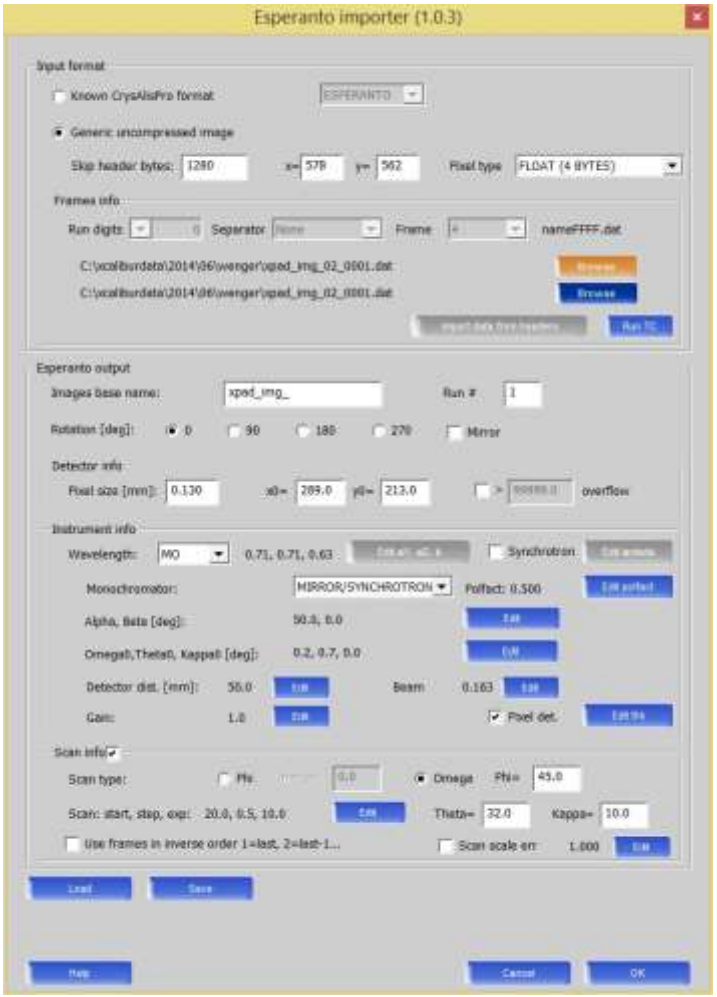
Dc rit: IPDS

- 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 handly 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 know 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

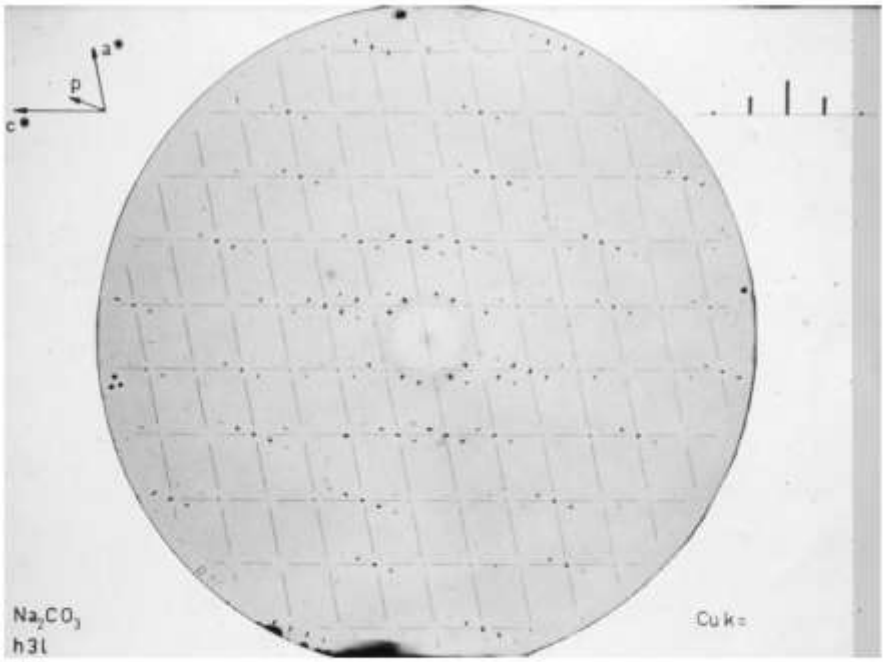
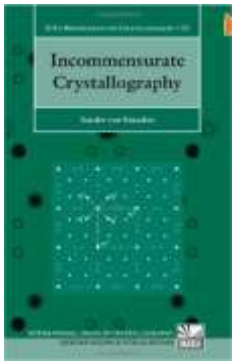


FIG. 1.11. Diffraction pattern of Na_2CO_3 (retigram; similar to a precession photograph). The measured X-ray diffraction has been overlayed with thin lines, highlighting the reciprocal lattice of the basic structure. p indicates the modulation wave vector. Reprinted from Tuinstra and Fraase Storm (1972)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

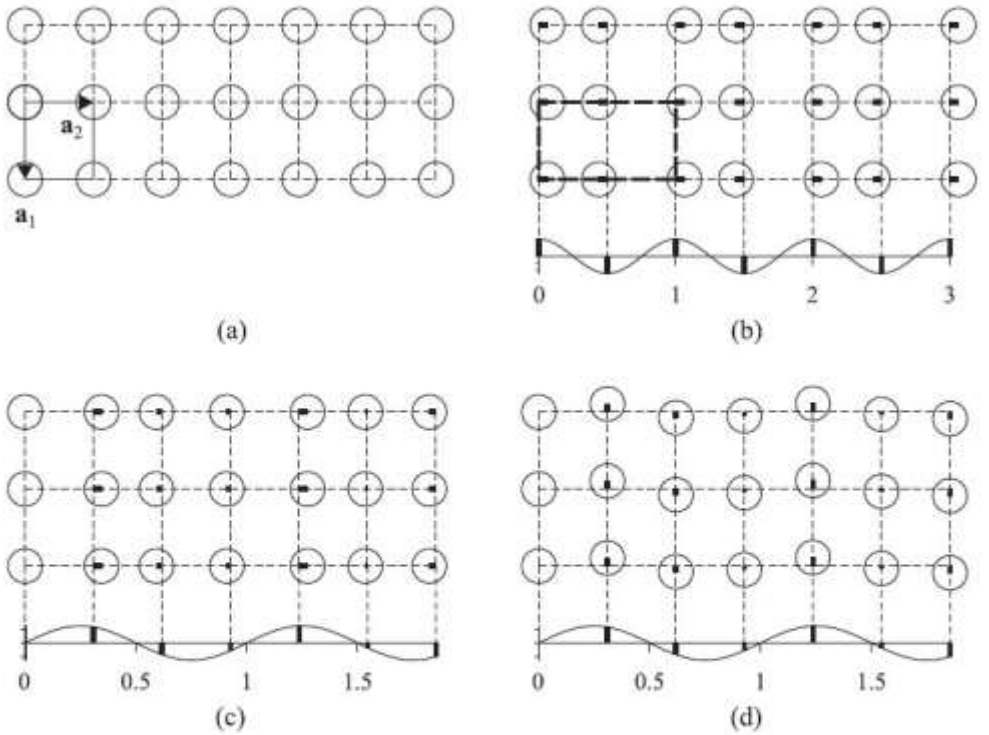


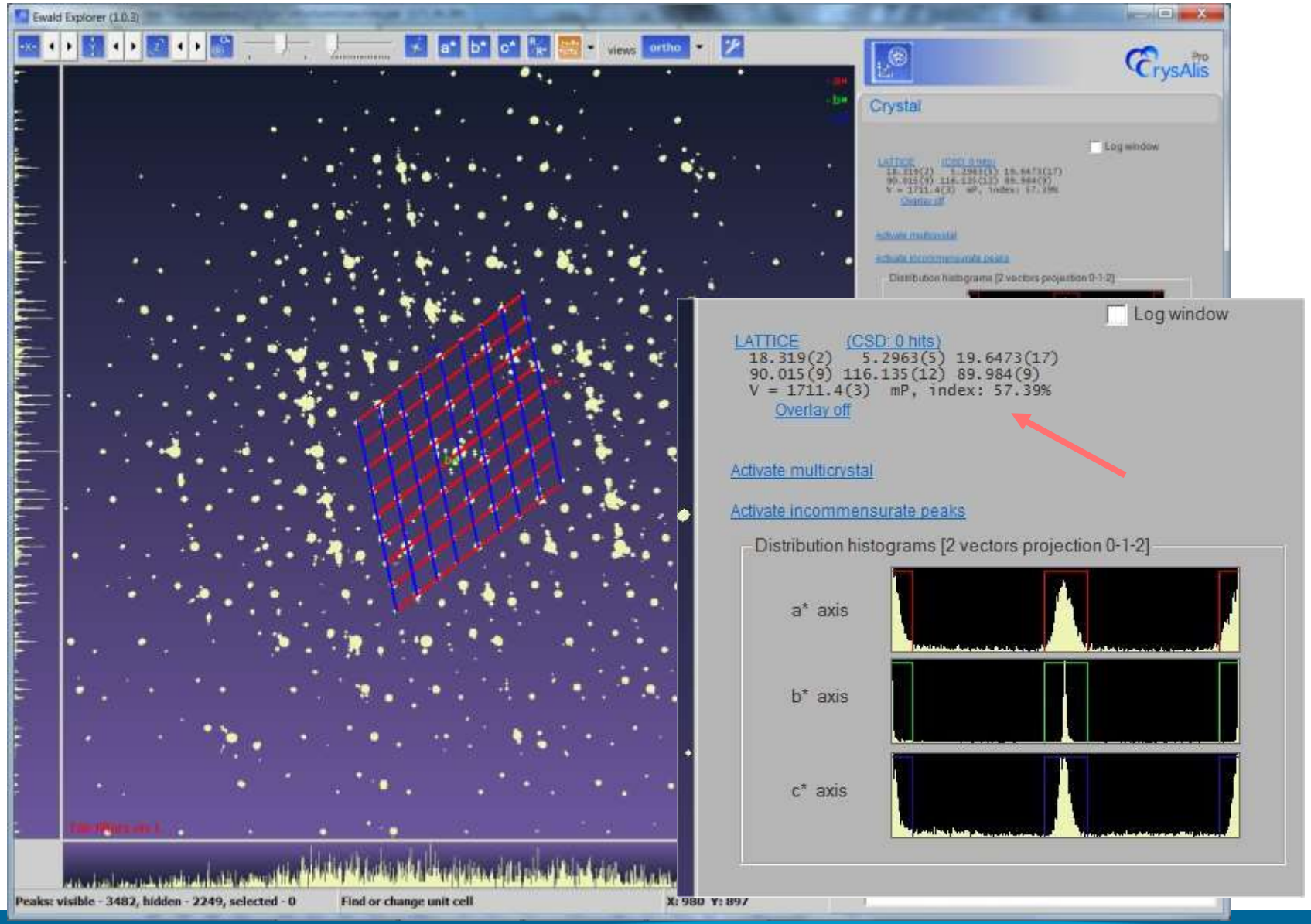
FIG. 1.2. Crystal structures with displacement modulations. (a) Basic structure. (b) Twofold superstructure with the supercell indicated by heavy dashed lines. (c) Incommensurate longitudinal modulation. (d) Incommensurate transversal modulation. Lattices of the periodic basic structures are indicated 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 $\mathbf{u}(\bar{x}_4)$ [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

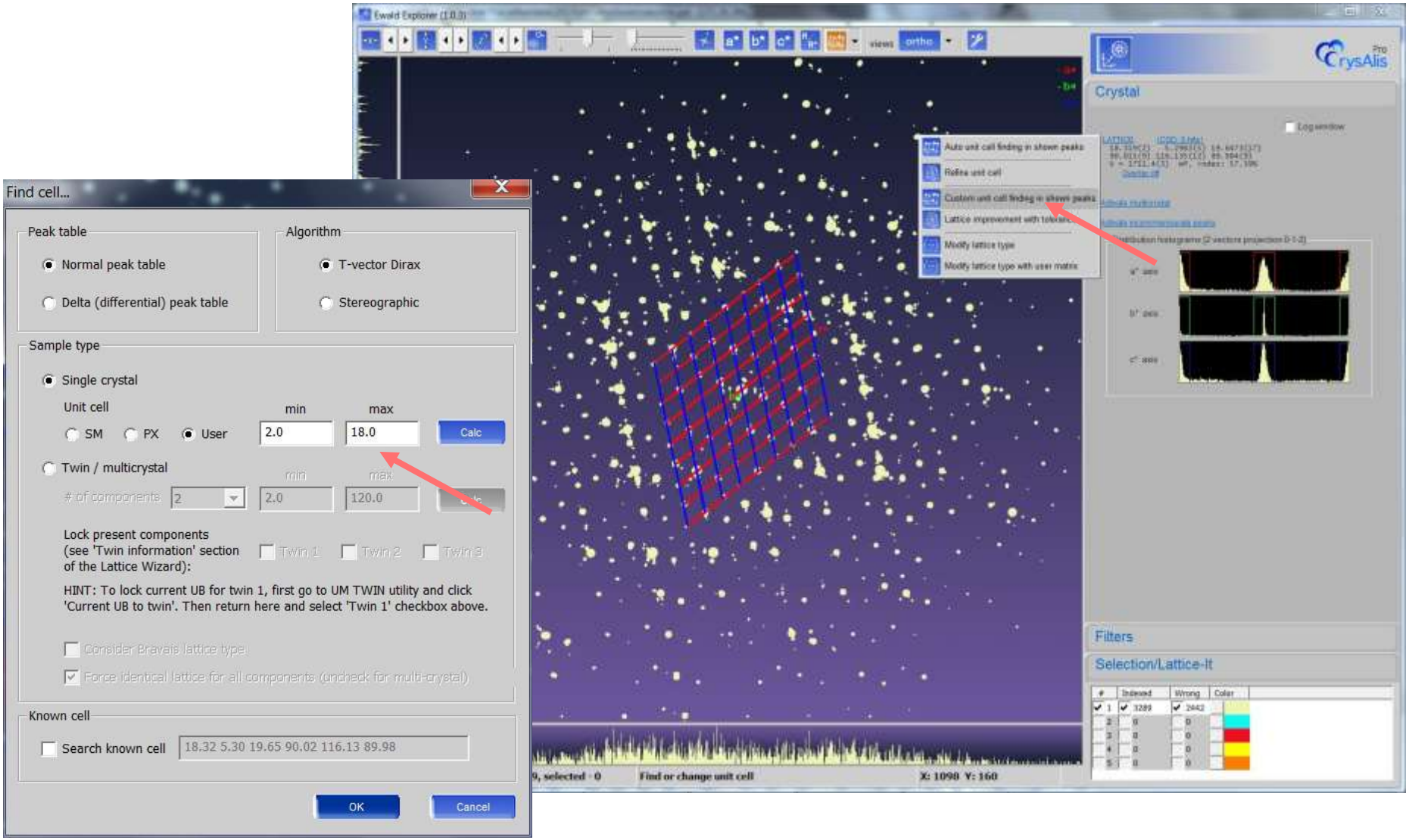
- Case Na_2CO_3
- Normal unit cell finding fails: Super cell found
- ***Health warning***: samples of Naco are water sensitive and this is the baest sample ever after IUCr Tokyo: a multi-crystal/twin with 3 components: but component 1 is 80%

Naco: Super cell found



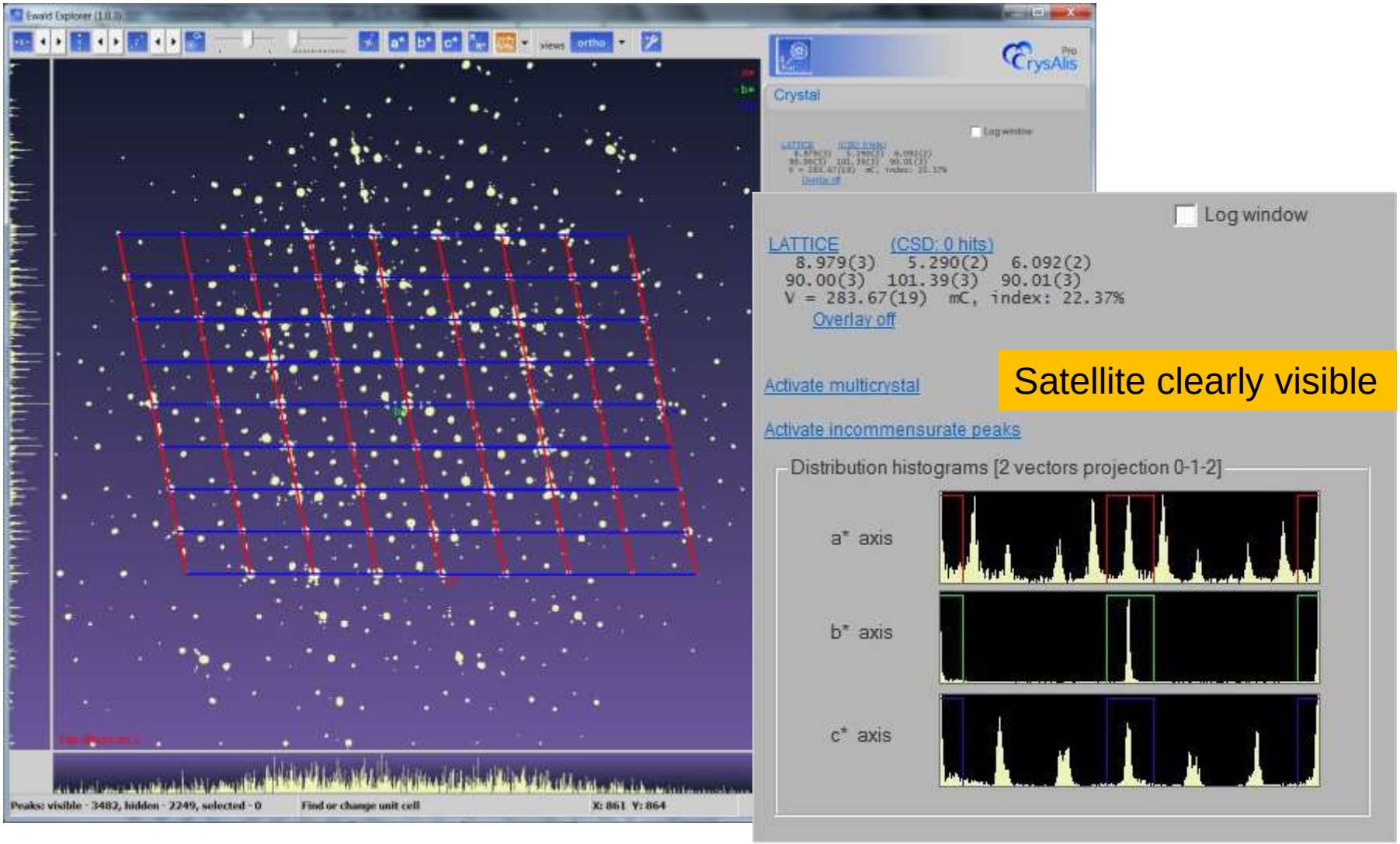
Agilent Technologies

Naco: look for basic cell – play with custom cell

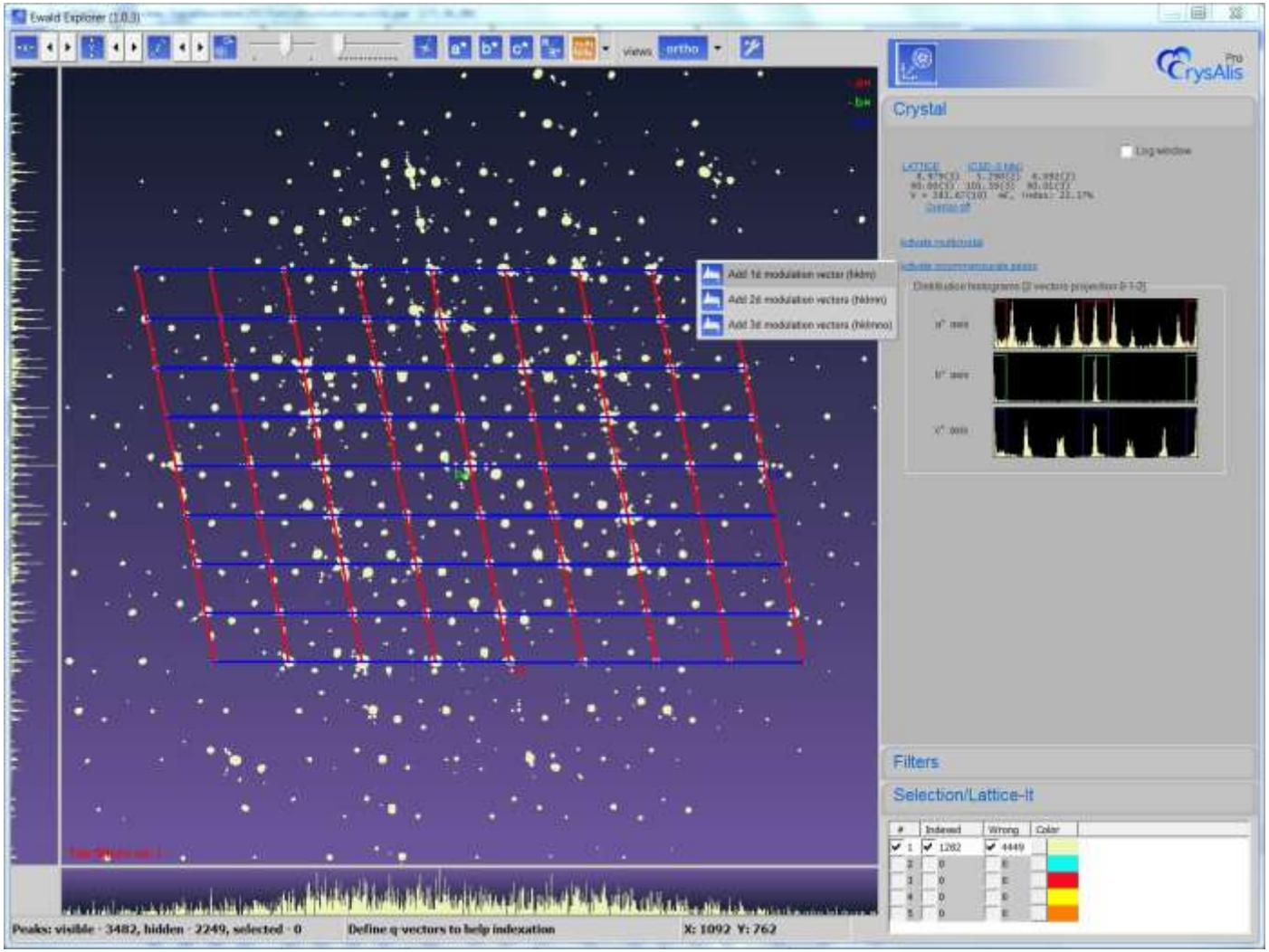


Agilent Technologies

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



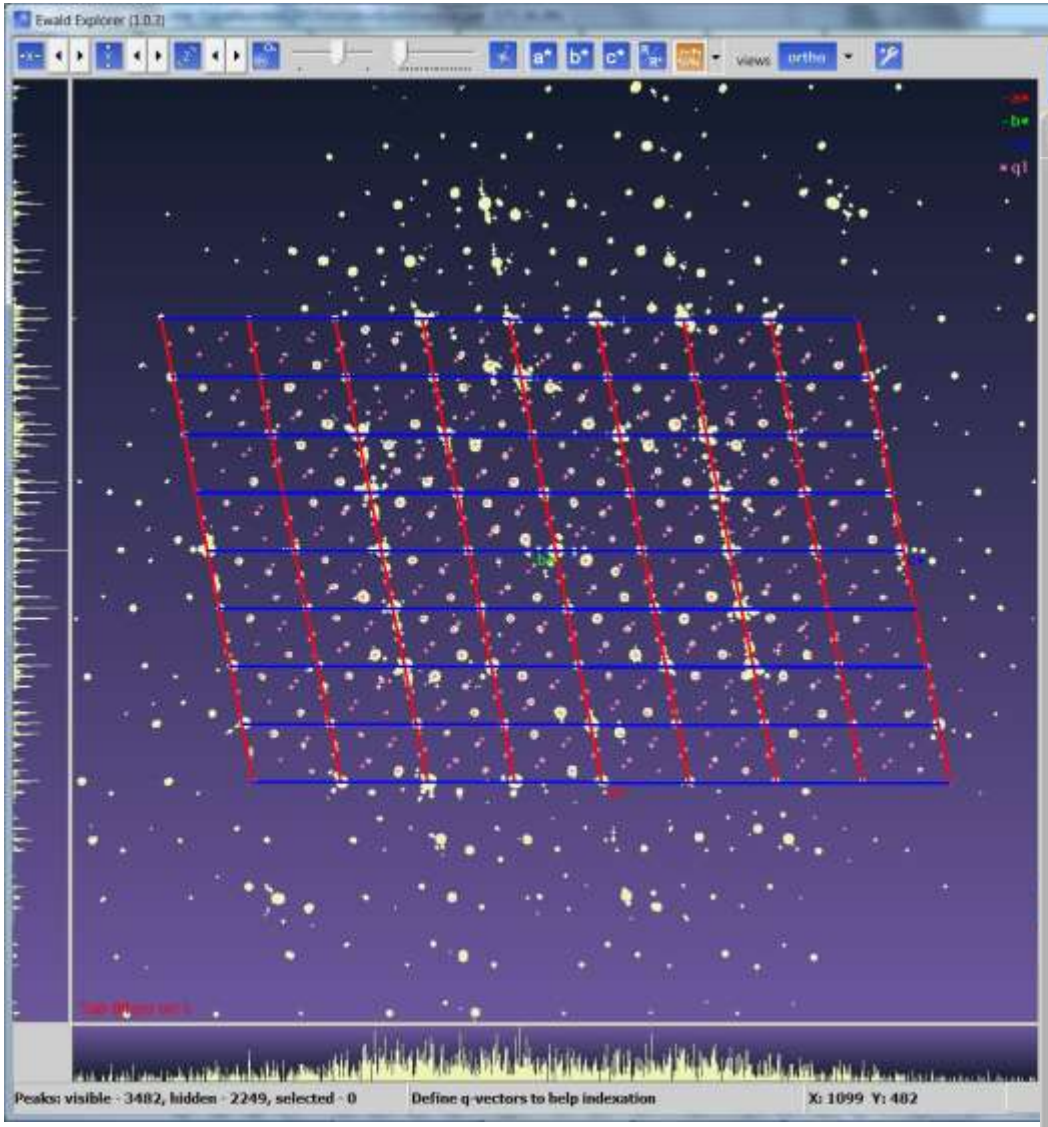
Naco: add q-vector

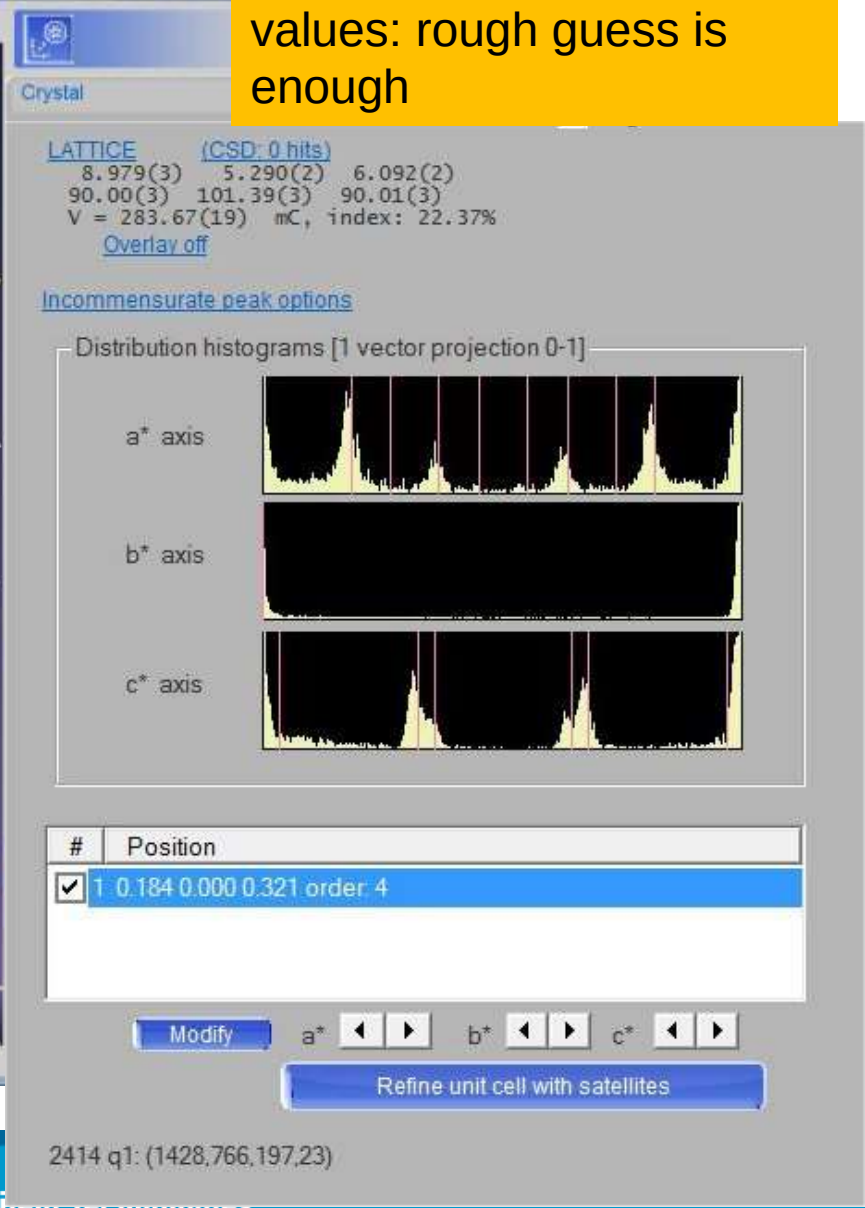


Agilent Technologies

Naco: add q-vector

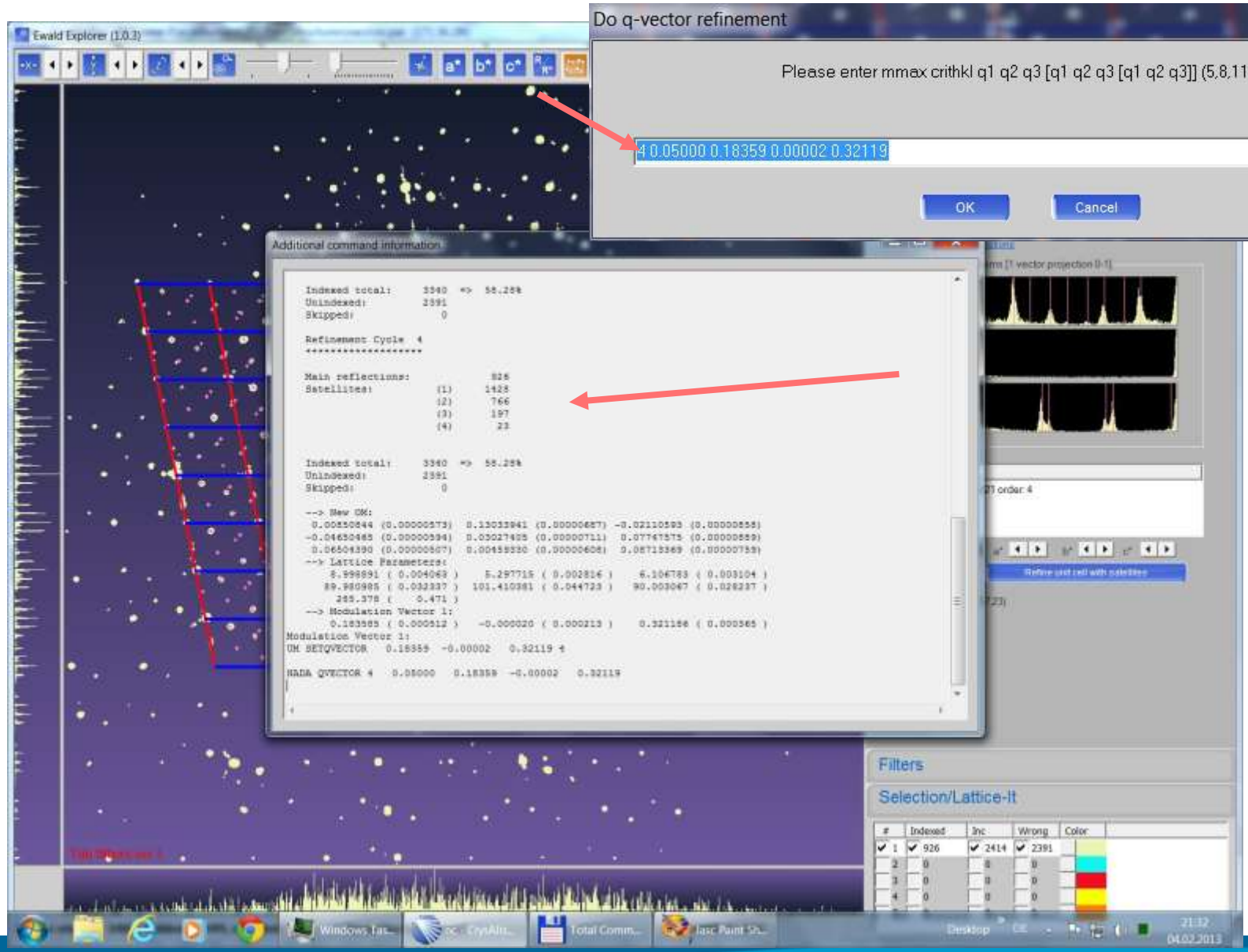
Click modify to change the values: rough guess is enough





Note: q-vectors have to confront lattice symmetry!

Naco: refine q-vector inspect results



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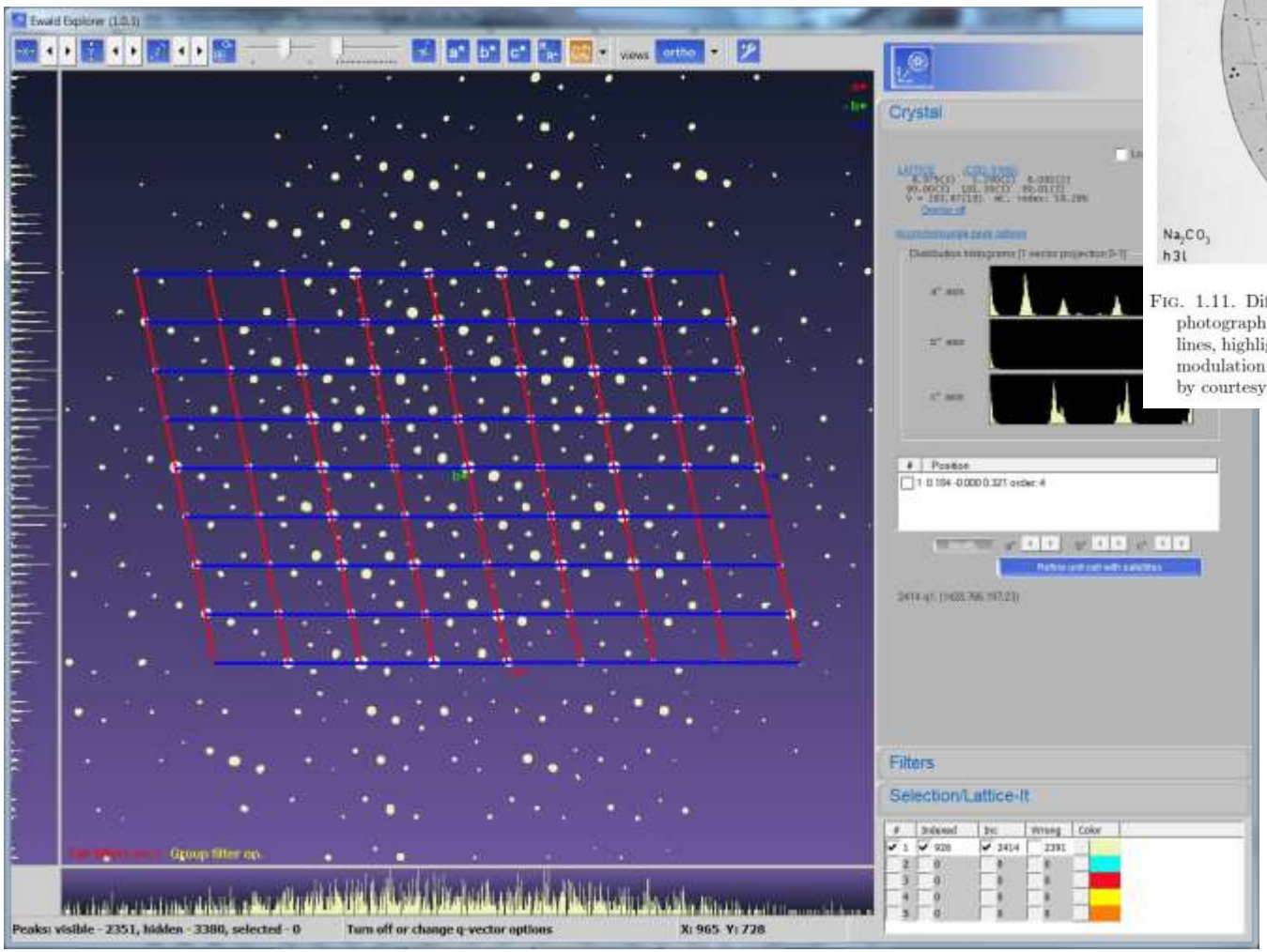
Naco: clean the picture…



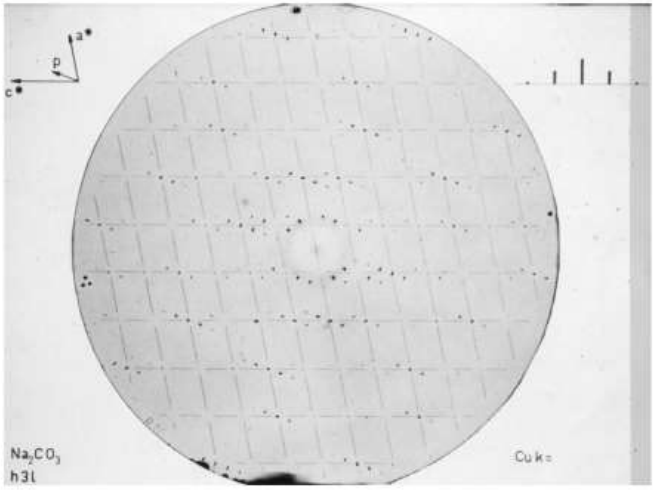


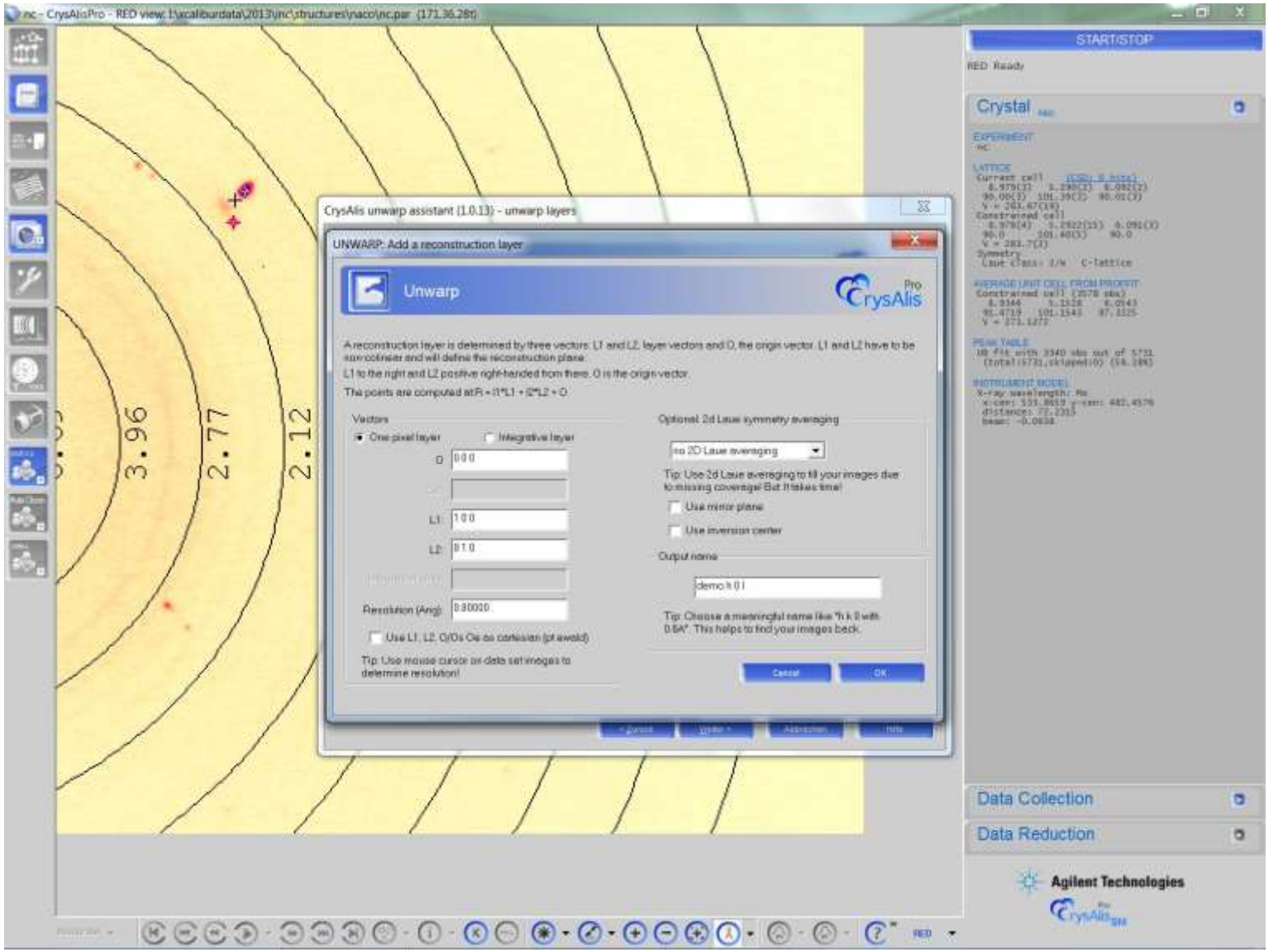
FIG. 1.11. Diffraction pattern of Na_2CO_3 (retigram; similar to a precession photograph). The measured X-ray diffraction has been overlayed with thin lines, highlighting the reciprocal lattice of the basic structure. *p* indicates the modulation wave vector. Reprinted from Tuinstra and Fraase Storm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

- Group checkmarks
- Hide wrong

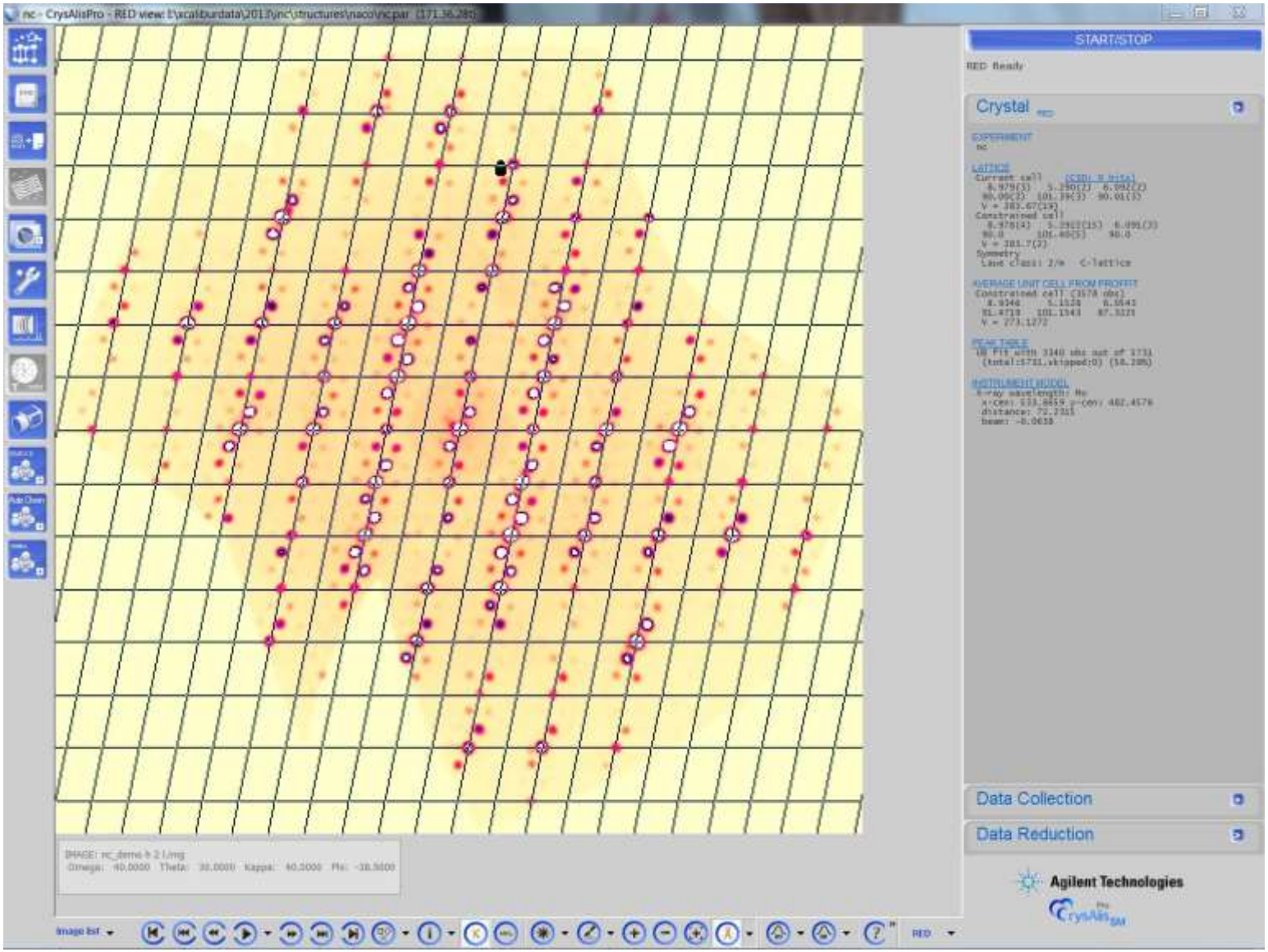
Naco: Lattice wizard

- Sorting
- Cleaning
- q-vector refinement

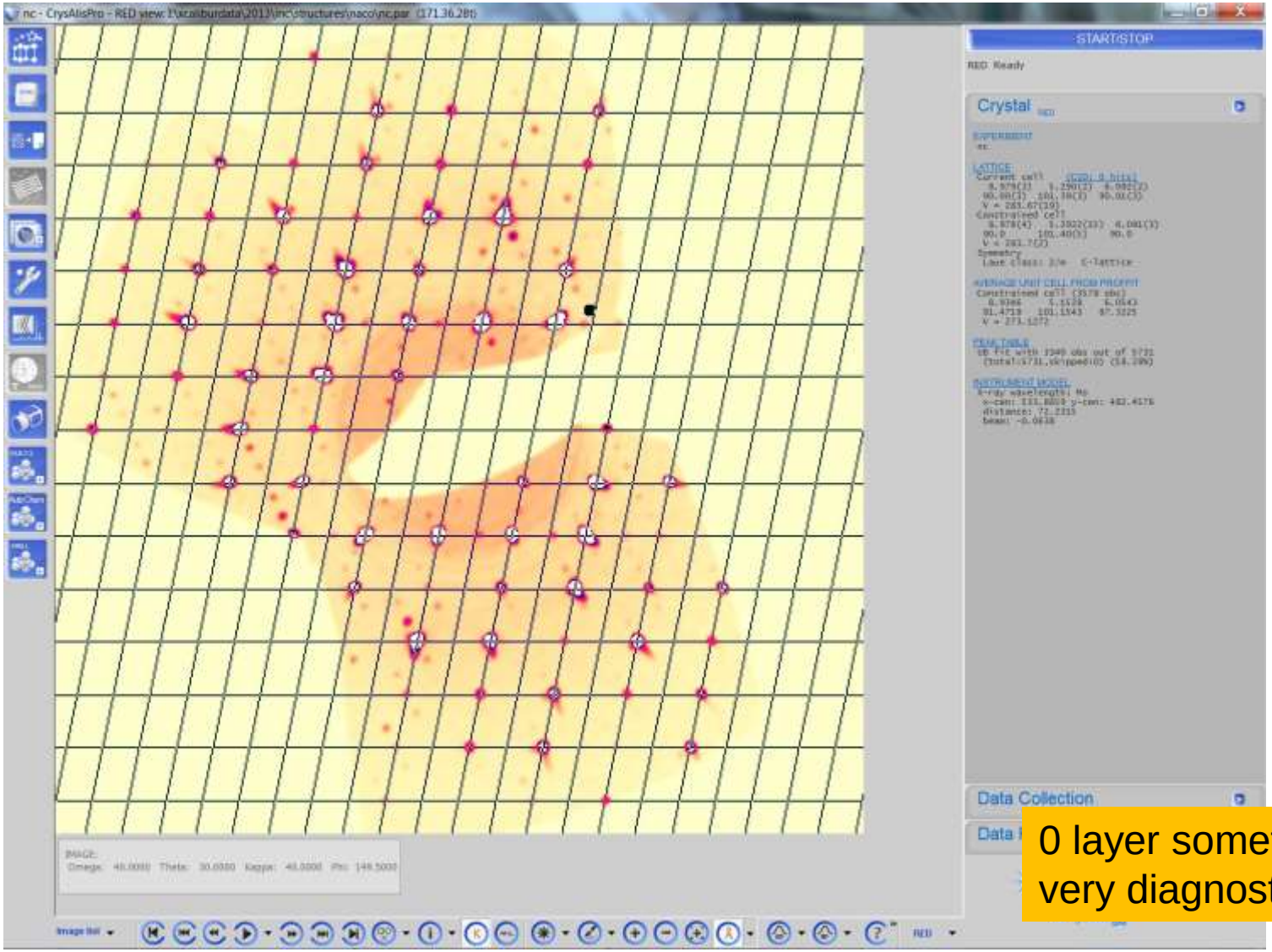
Naco: Run unwarp to gain better understanding



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

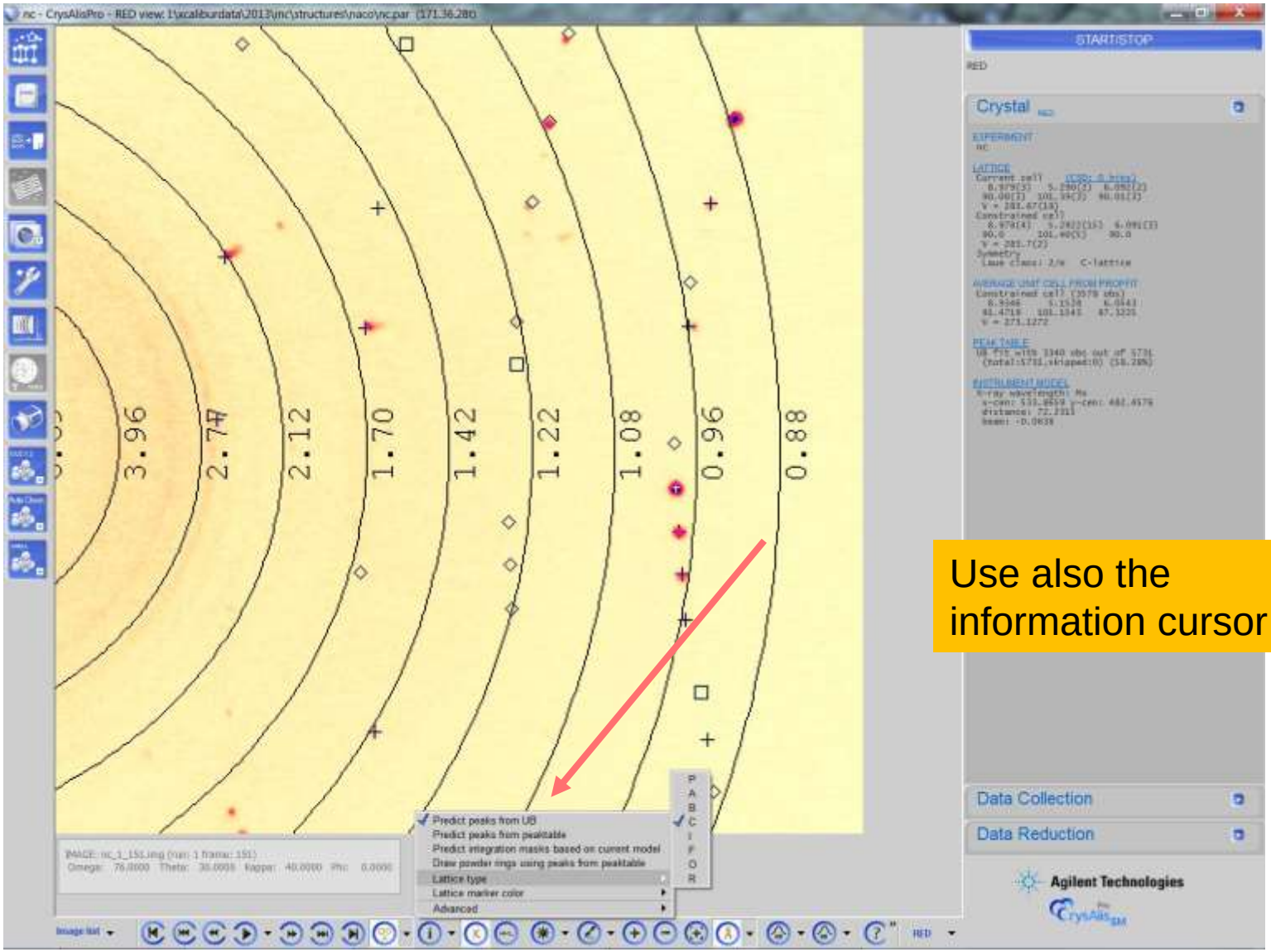


Naco: Run unwarp to gain better understanding: h 0 l

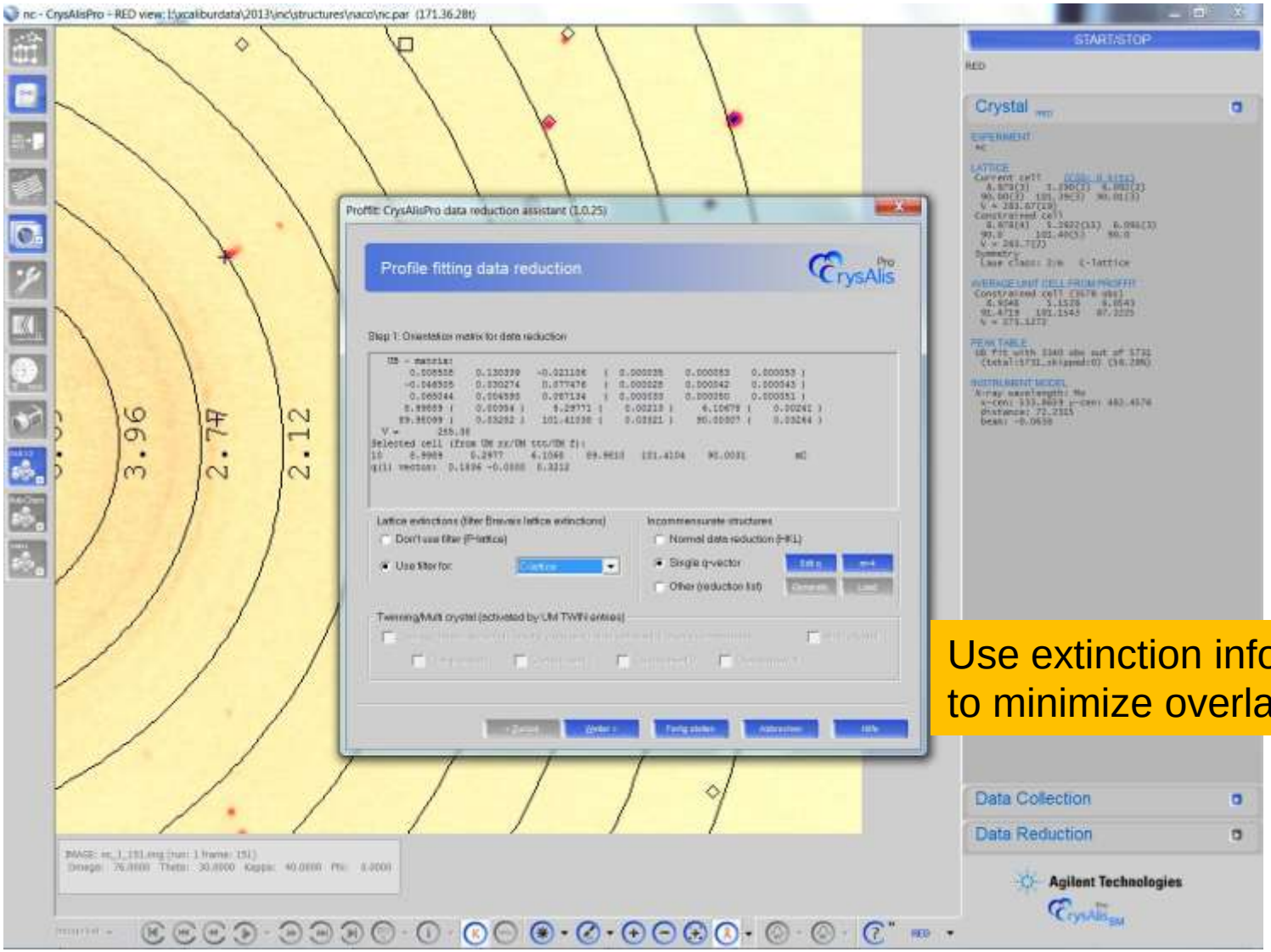


0 layer sometimes not very diagnostic

Naco: Check reflection predictions

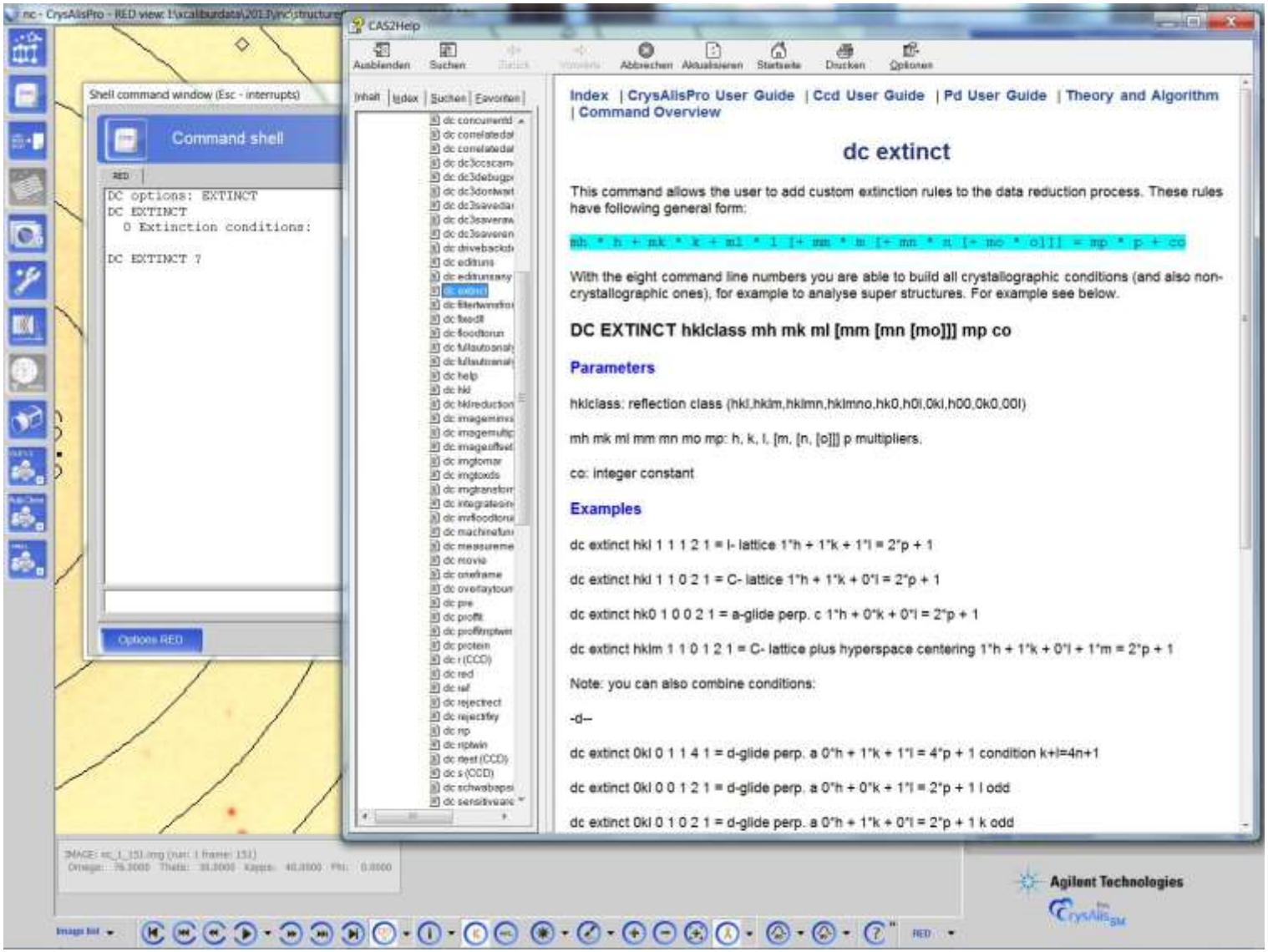


Naco: Data reduction

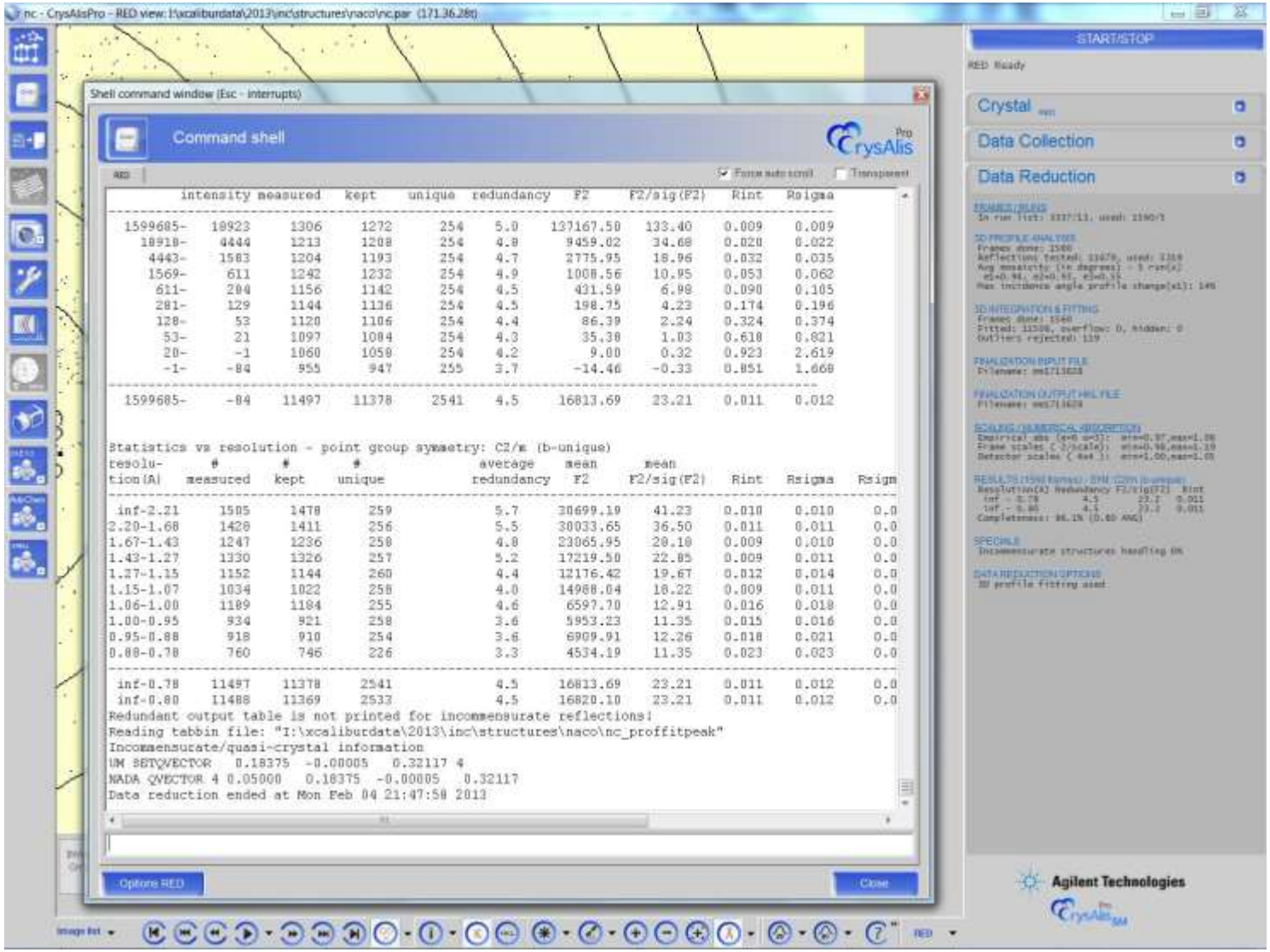


Use extinction information to minimize overlap

Naco: Alternative way to formulate extinctions



Naco: Check results



Naco: Strength of satellites

Main reflections

resolution(A)	# 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

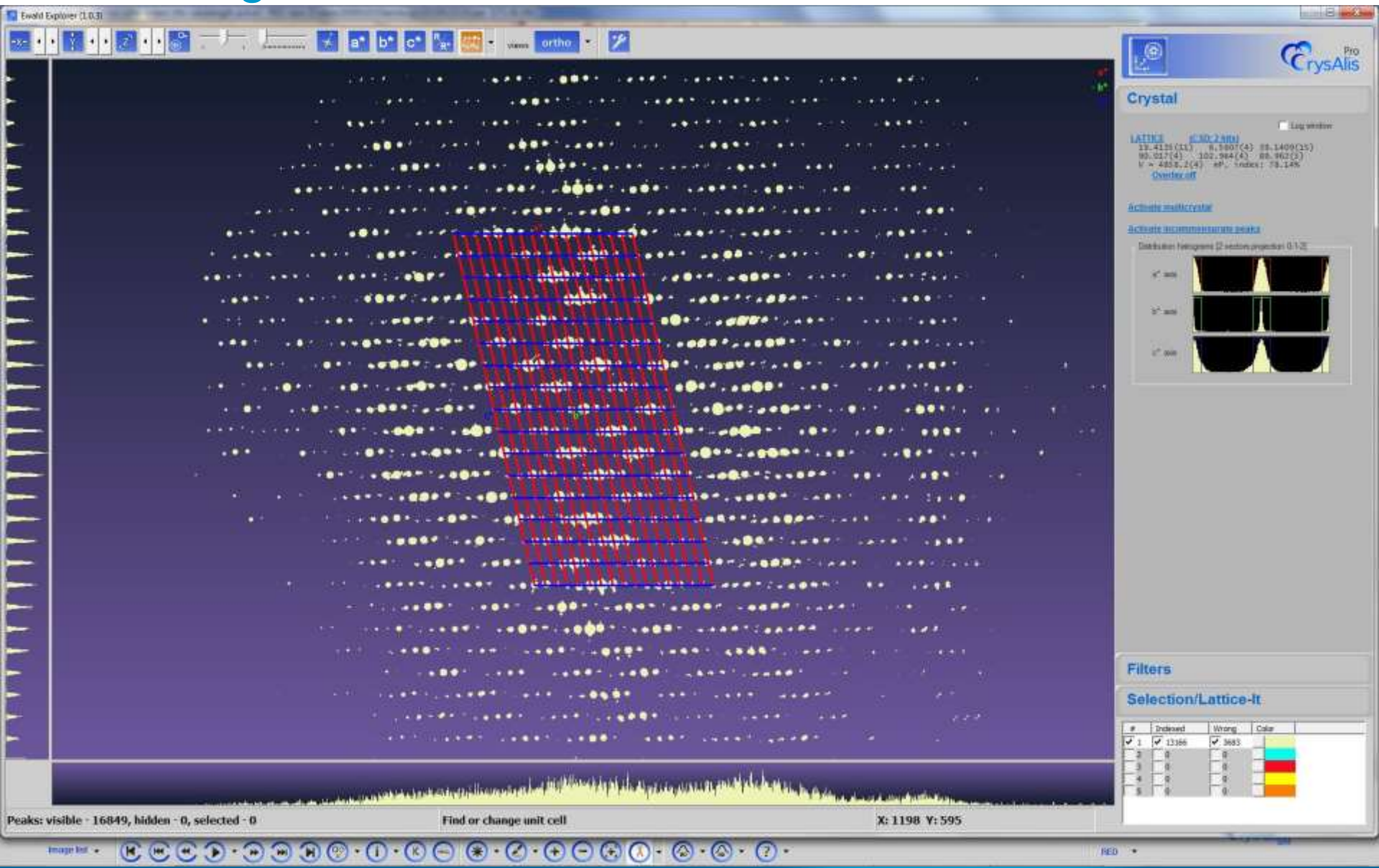
4. order satellite reflections

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:



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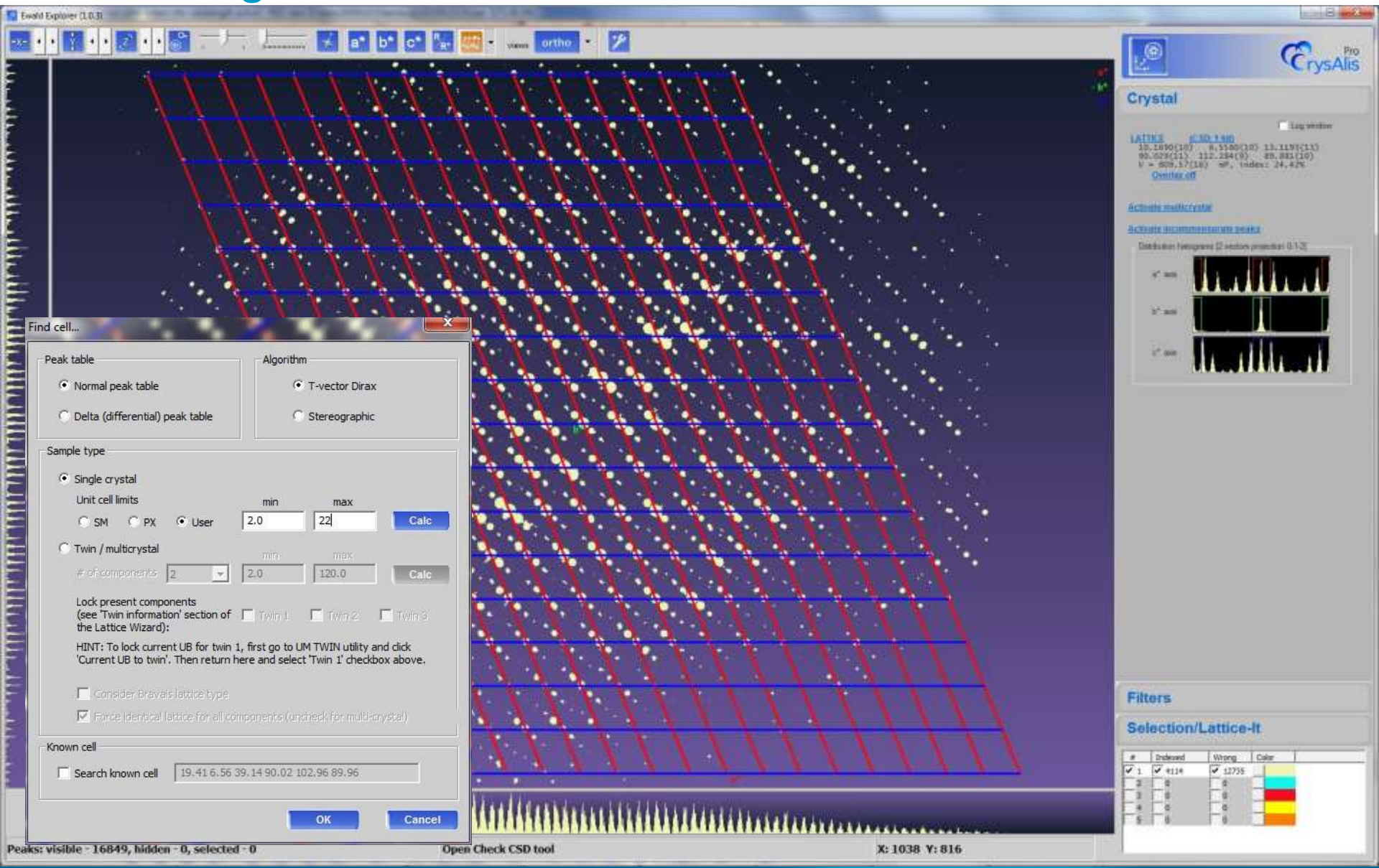
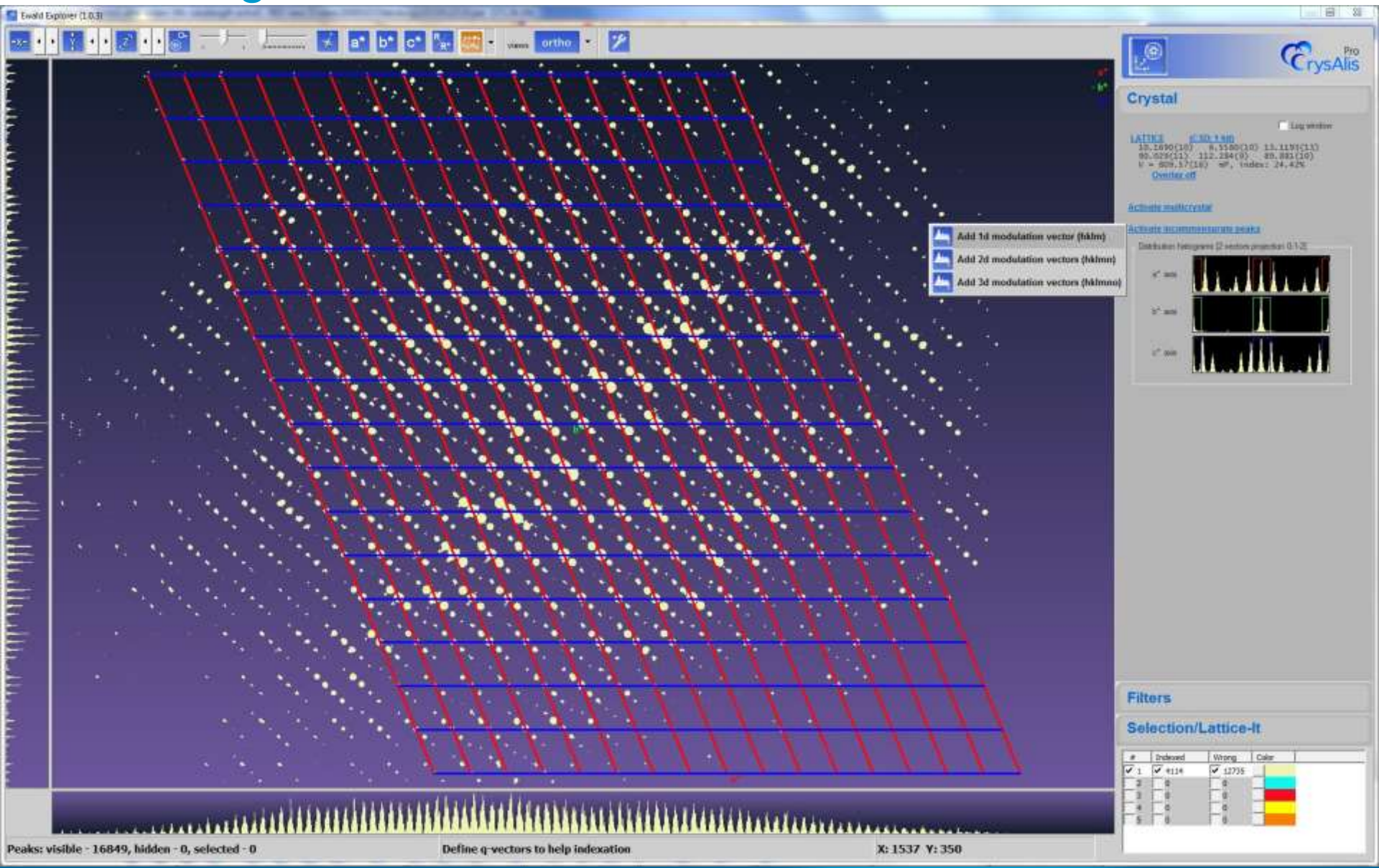
Oldenburg:



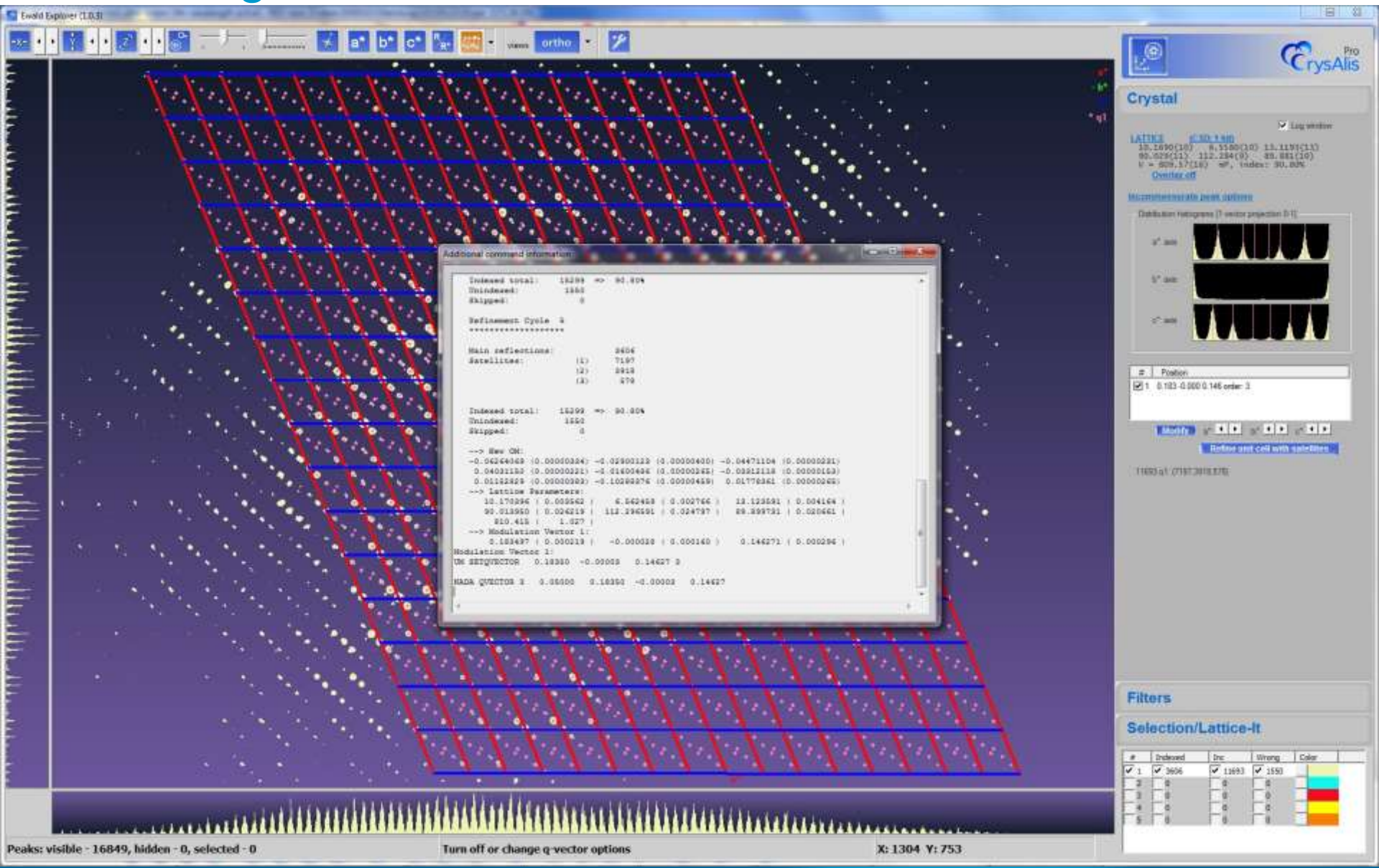
Agilent Technologies

Oldenburg:



Agilent Technologies

Oldenburg:



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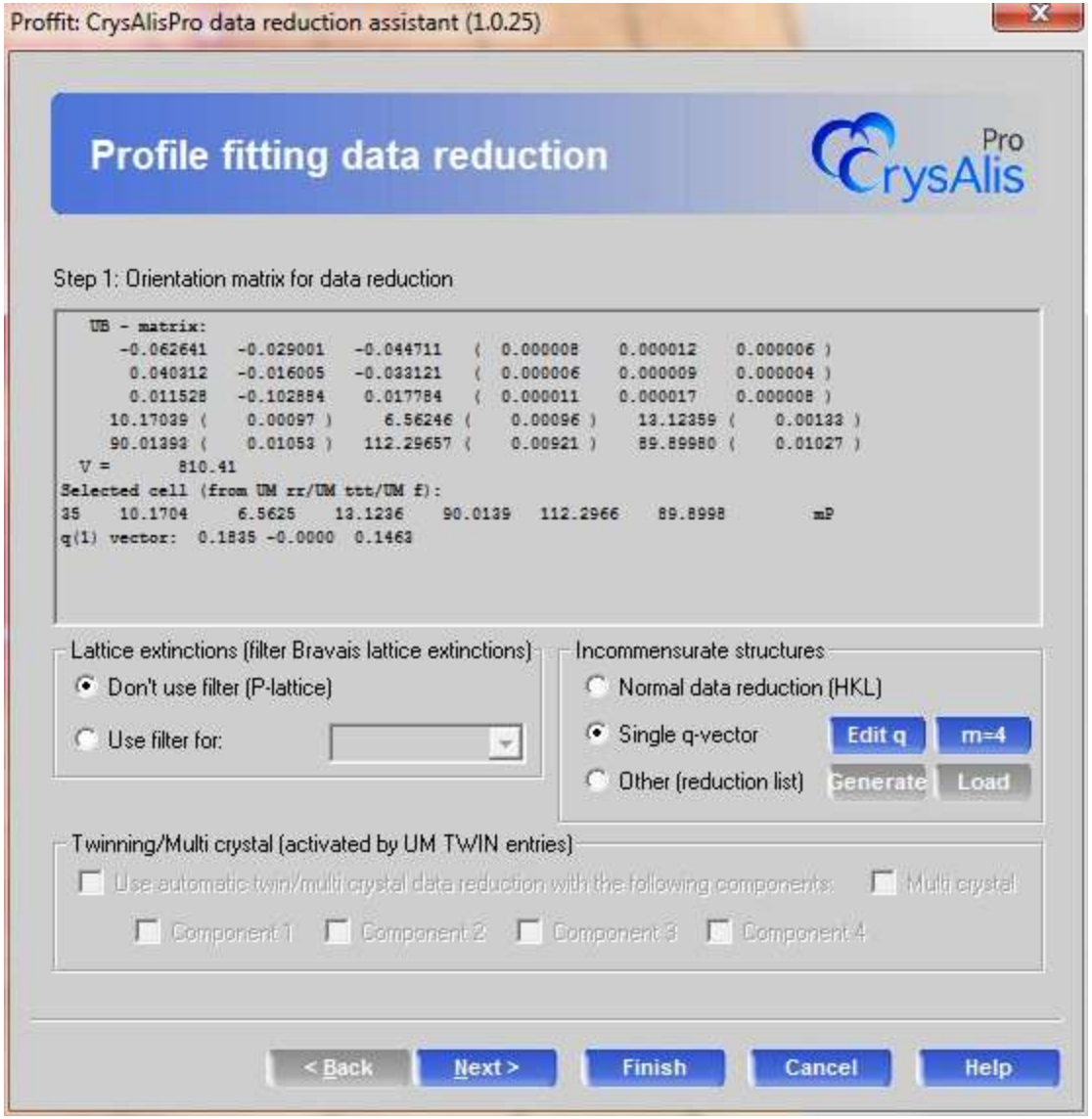
Oldenburg:


Lattice wizard (1.0.32)
Lattice wizard
Pro CrysAlis
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
Load information
SAVE
Save information
Unwarping - Precession images
Log window
Close

Oldenburg:


Proffit: CrysAlisPro data reduction assistant (1.0.25)
Profile fitting data reduction
Pro CrysAlis
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)
10.17039 (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 rr/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
Edit q
m=4
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
< Back
Next >
Finish
Cancel
Help

Oldenburg: Strength of satellites

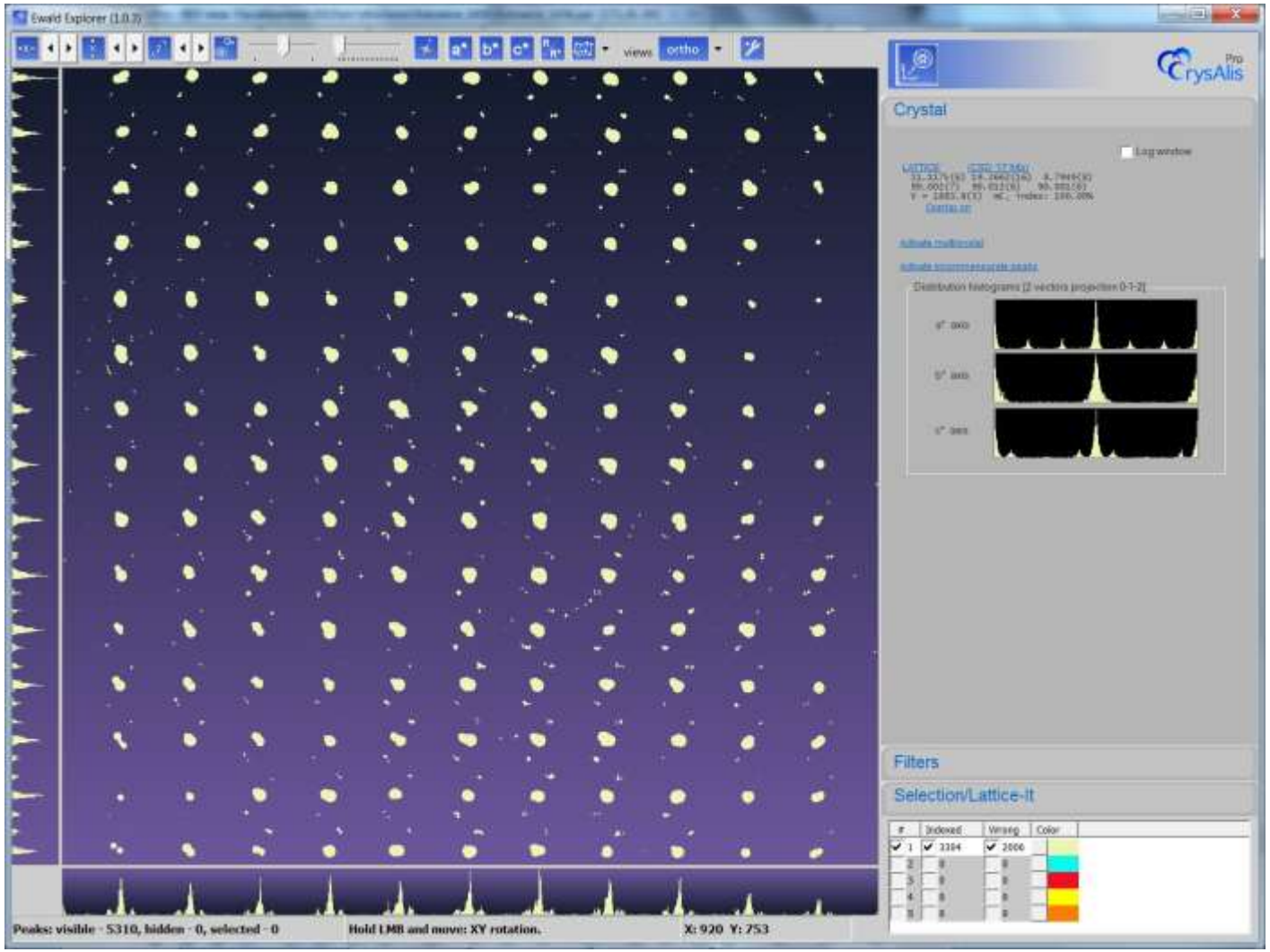
Main reflections

resolution(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

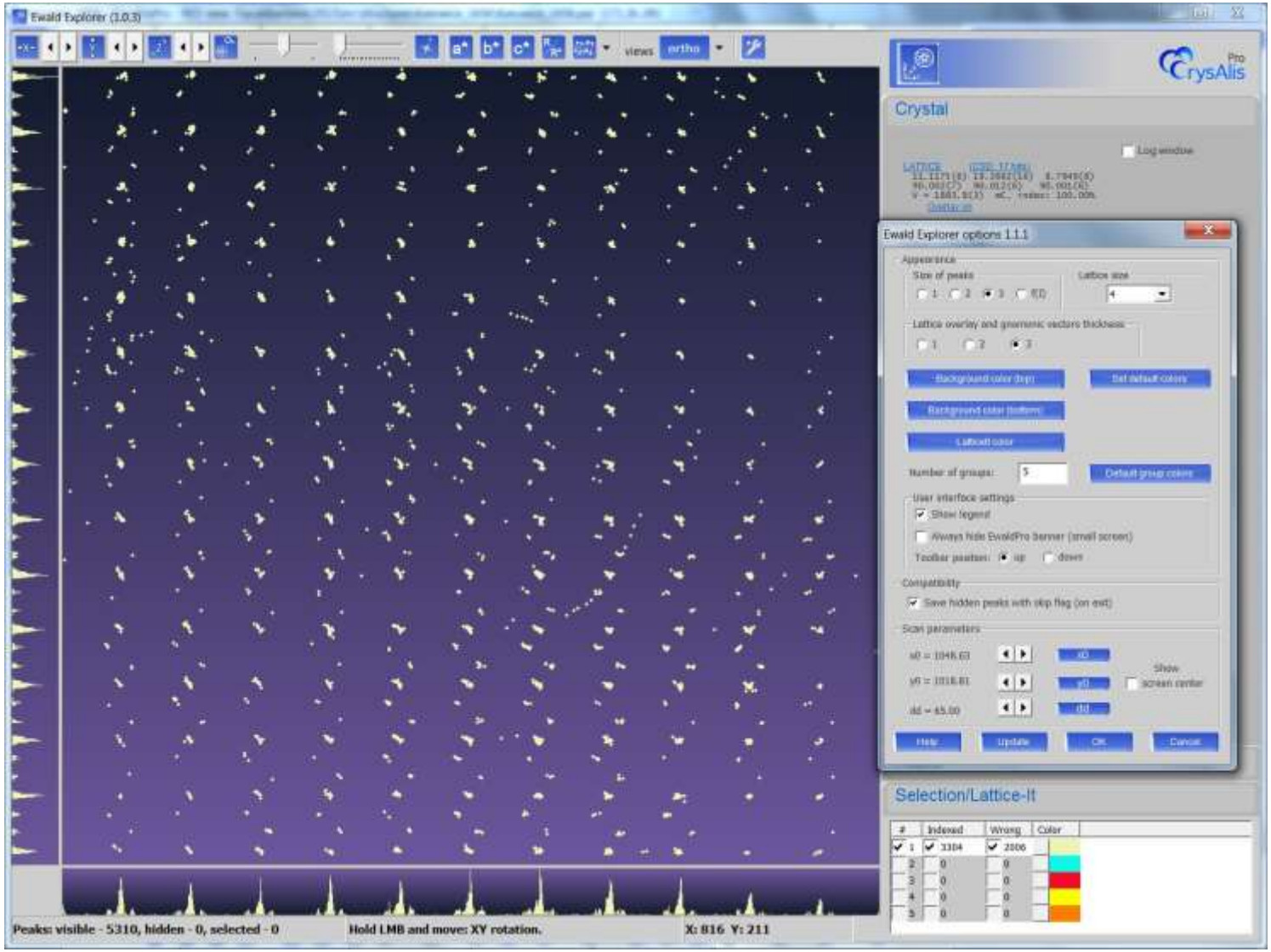
Recognition of incommensurate structures in CrysAlis

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

Katowice_165K:



Katowice_165K:



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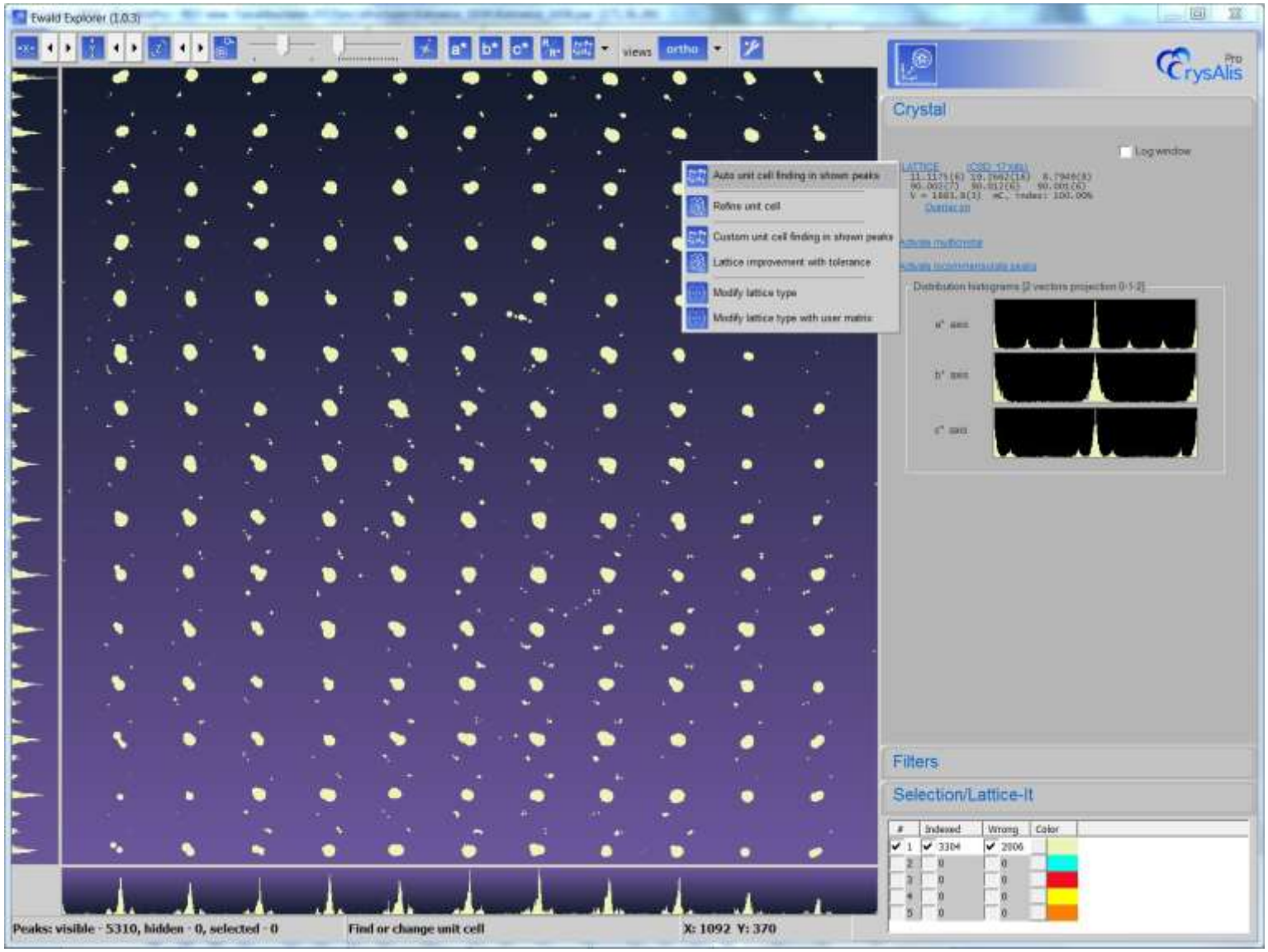
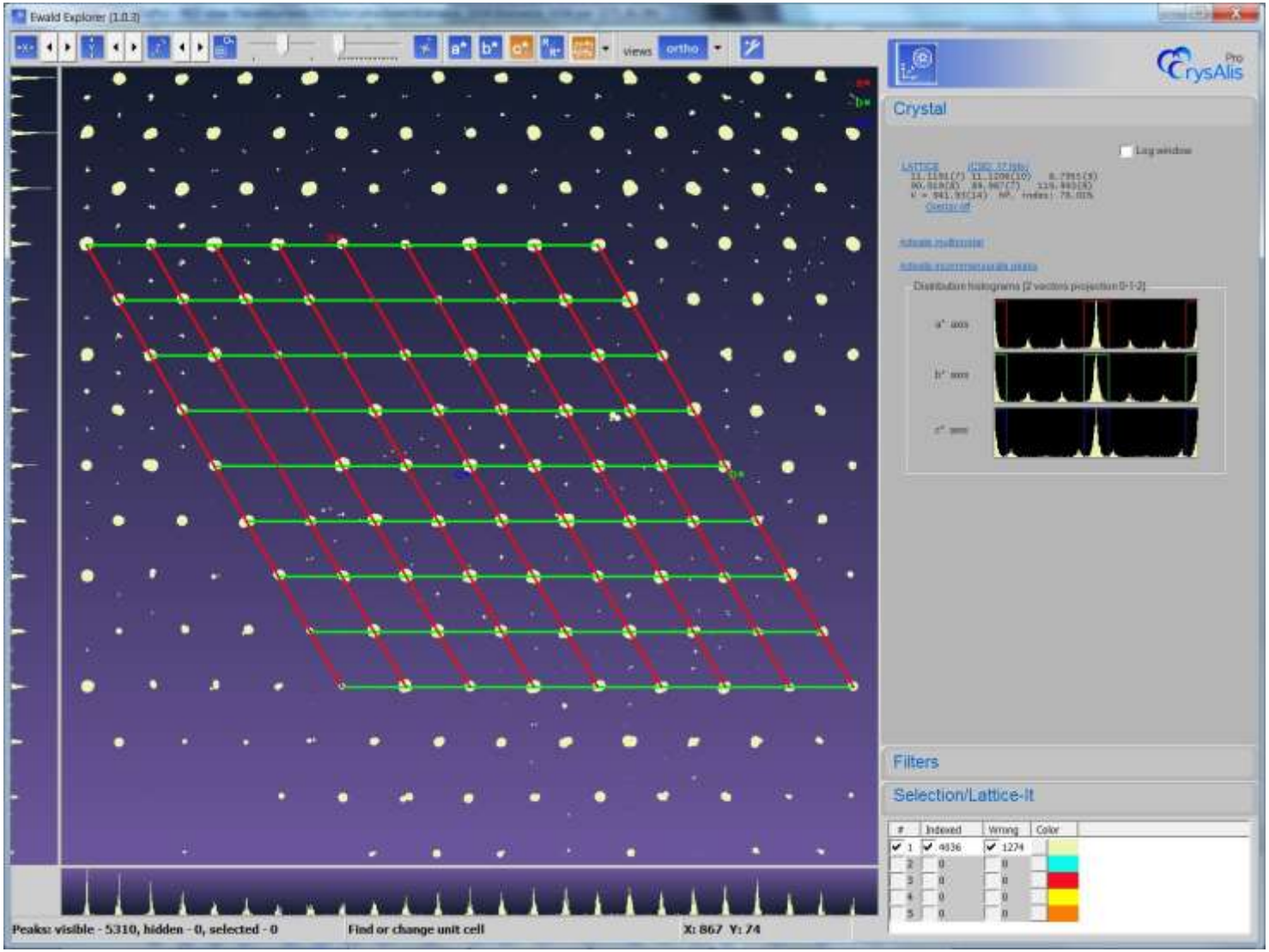
Katowice_165K:

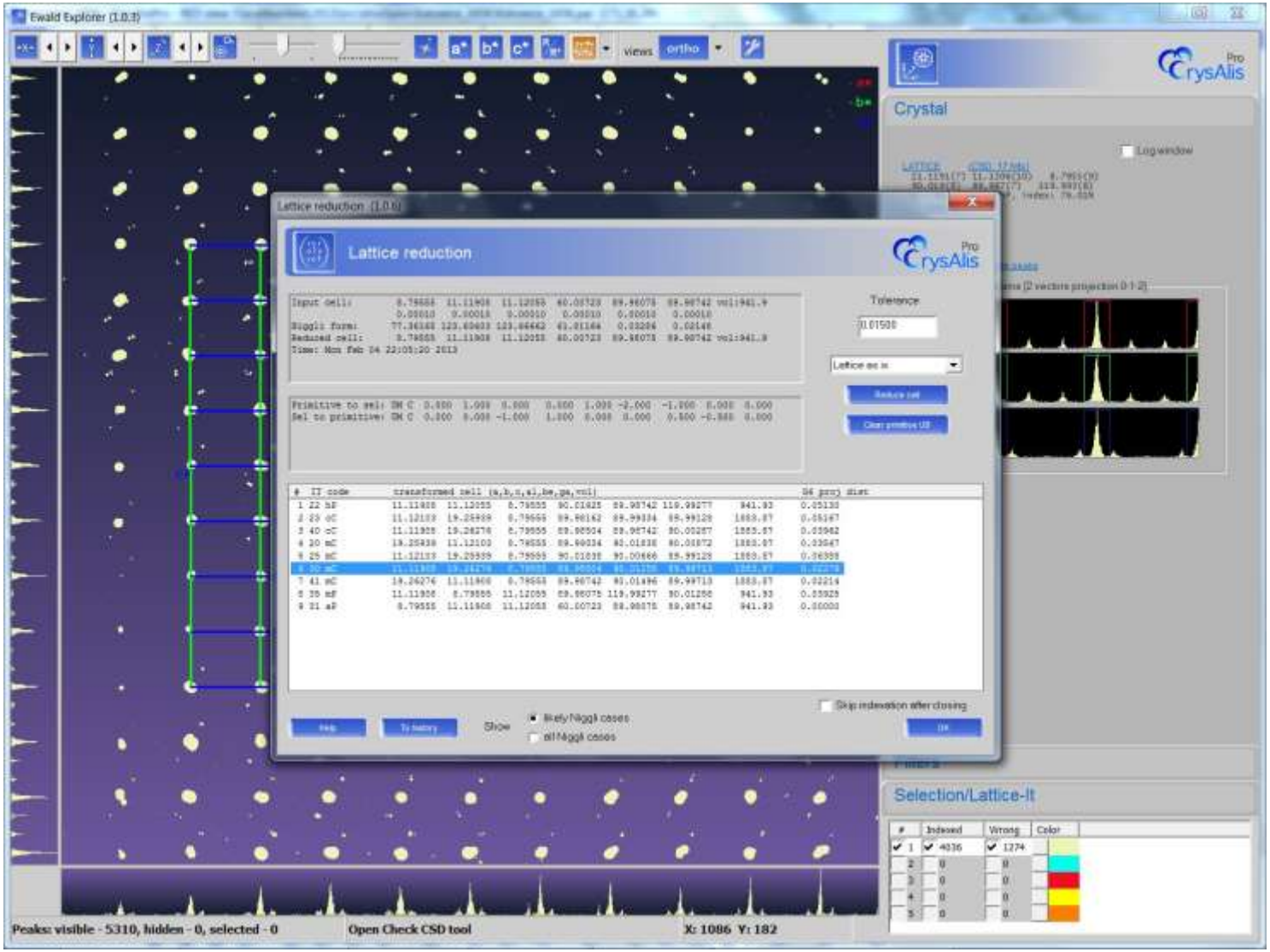


Agilent Technologies

Katowice_165K:

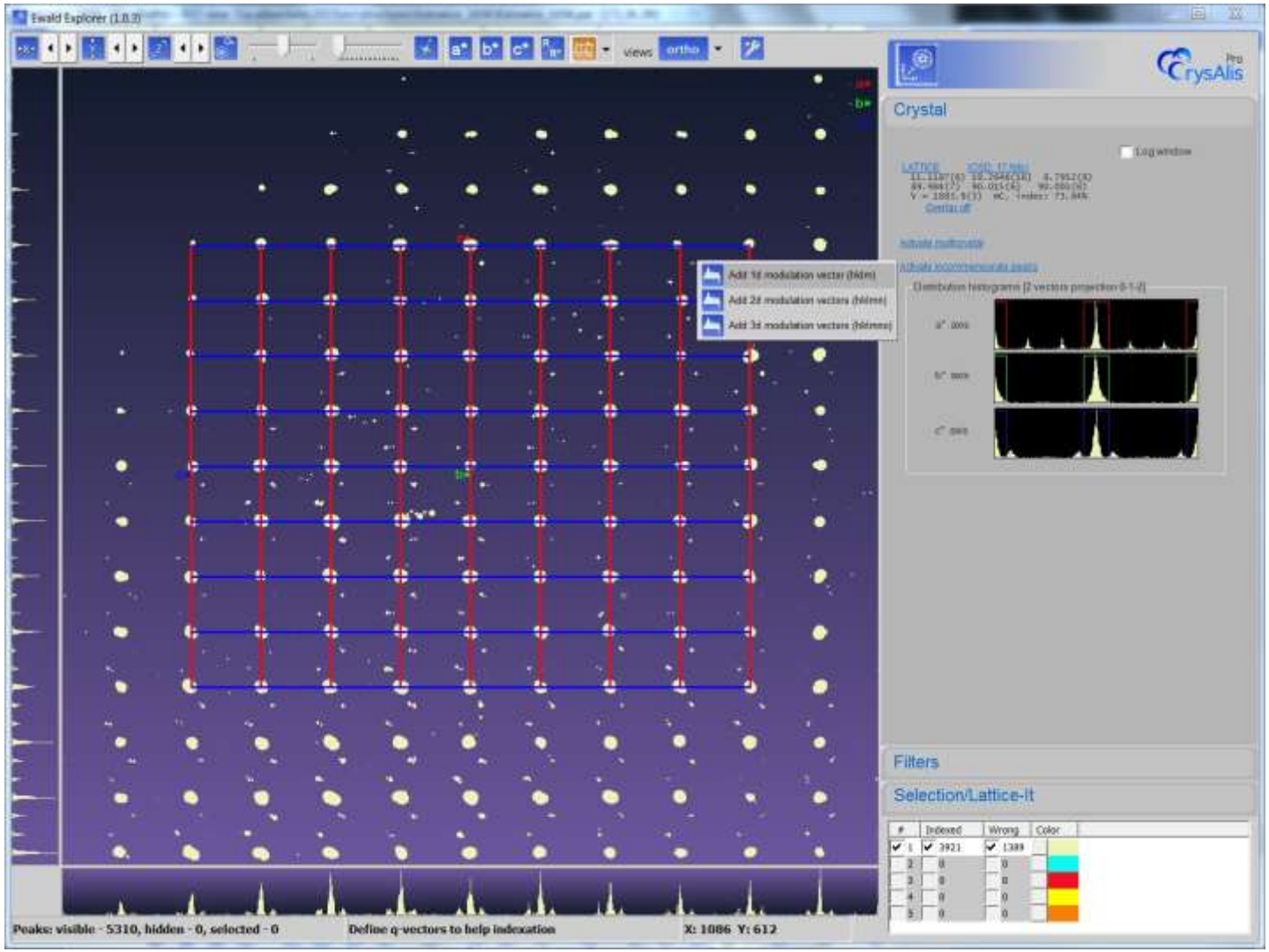


Katowice_165K:



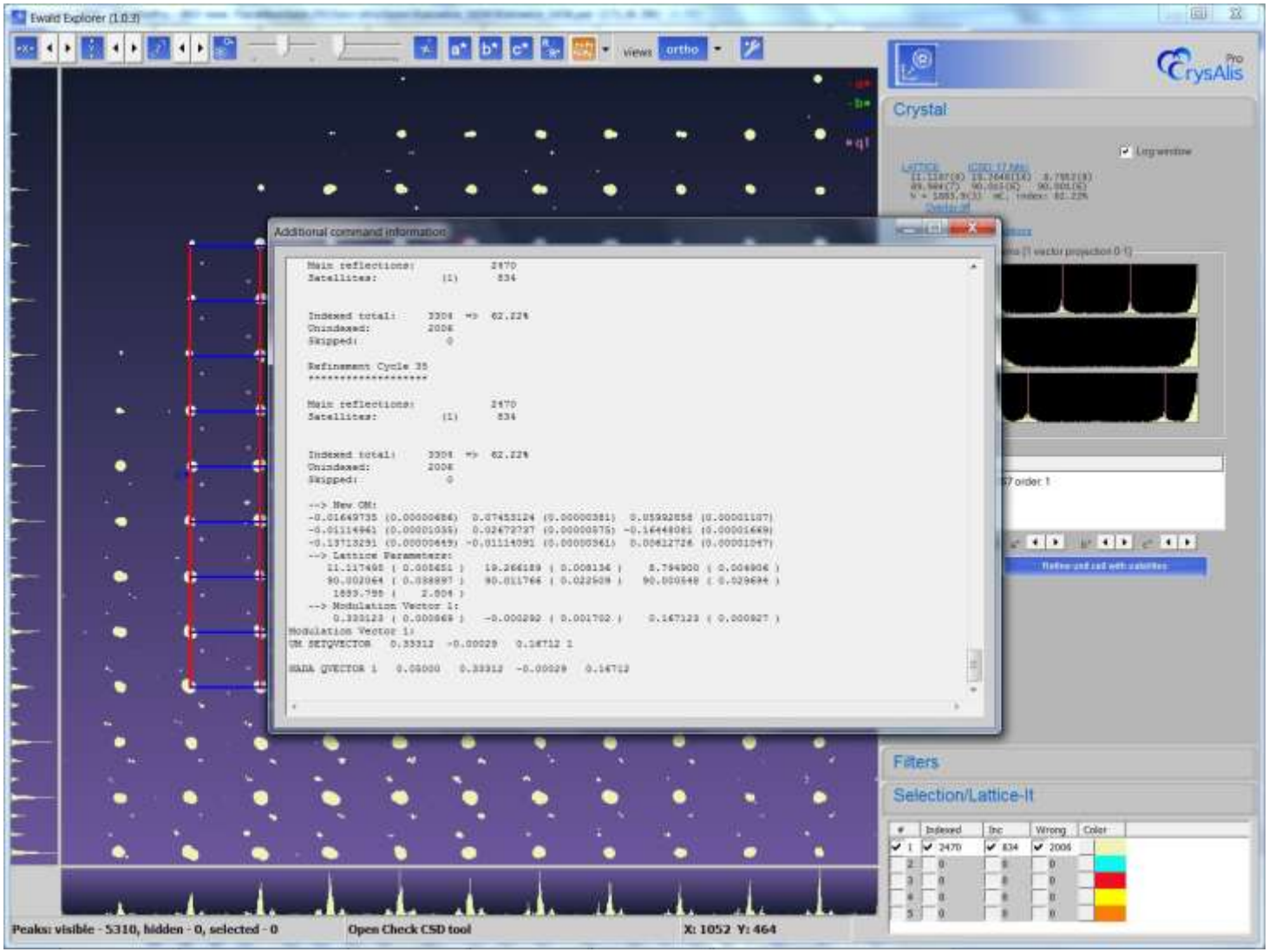
Agilent Technologies

Katowice_165K:



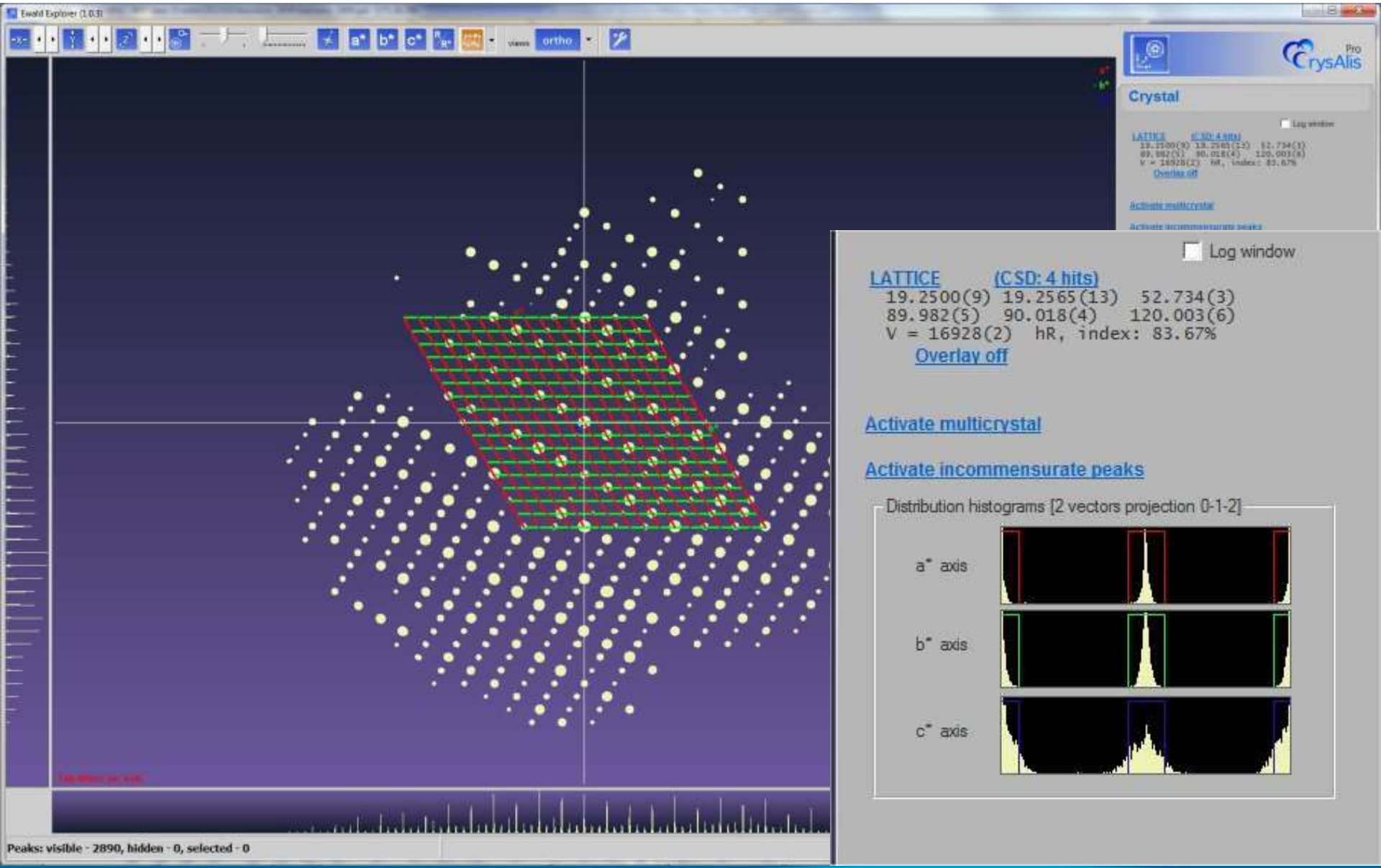
Agilent Technologies

Katowice_165K:



Agilent Technologies

Katowice_165K:



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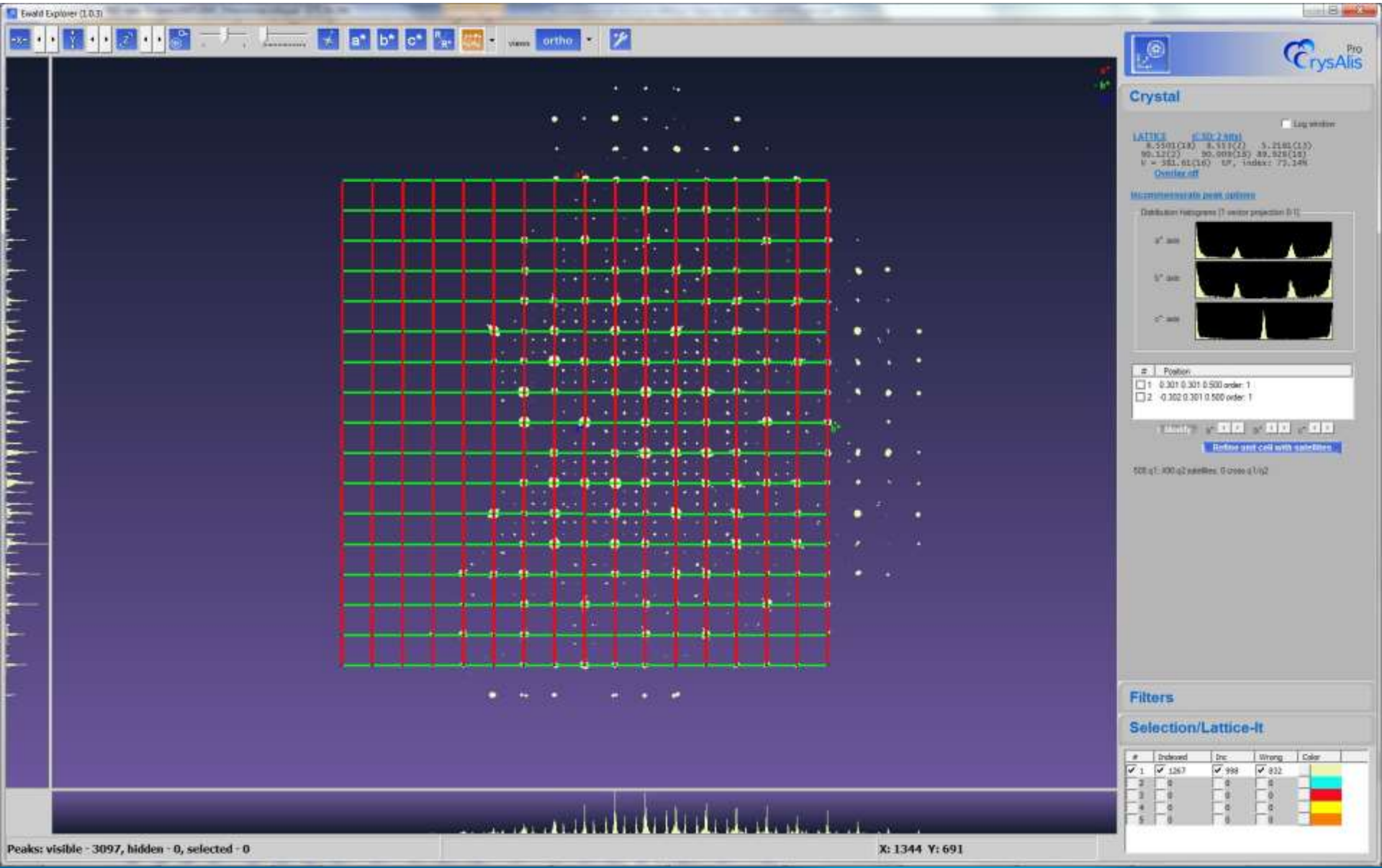
Recognition of incommensurate structures in CrysAlis

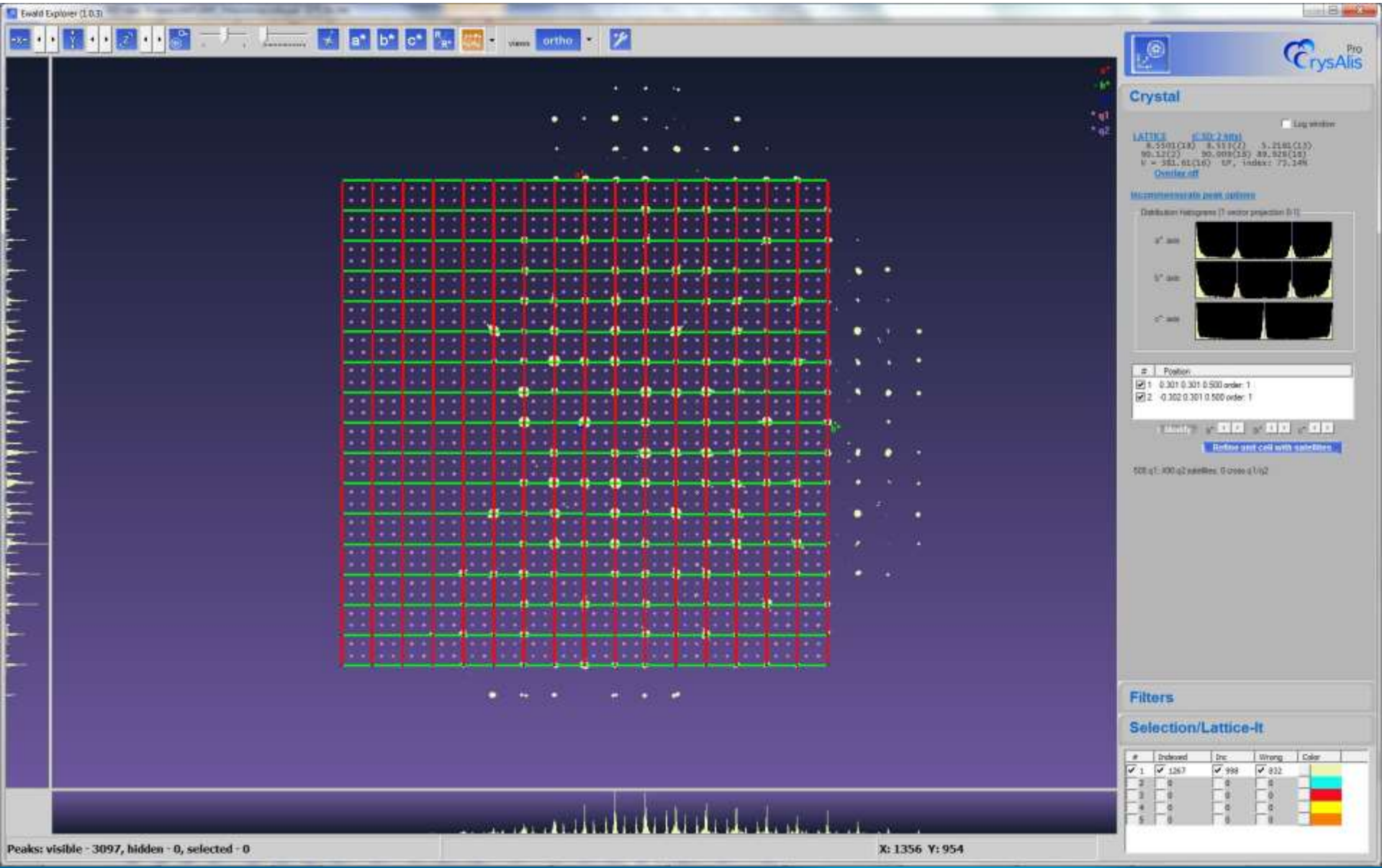
- Case fresno/raccolta
- Tetragonal sample with 2(!) q-vectors!
- Symmetry dictates the orientations

Fresno:



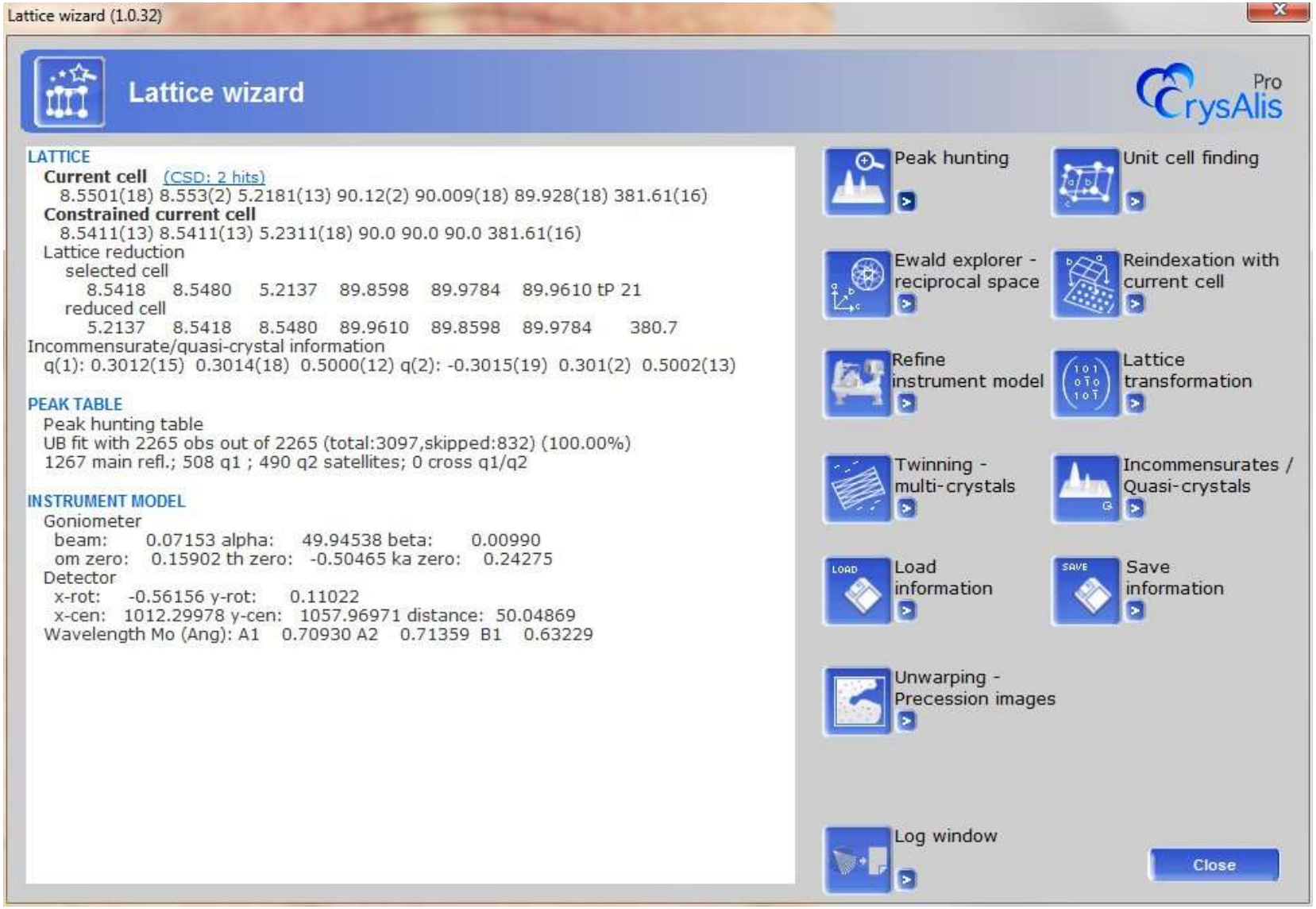
Agilent Technologies

Fresno:

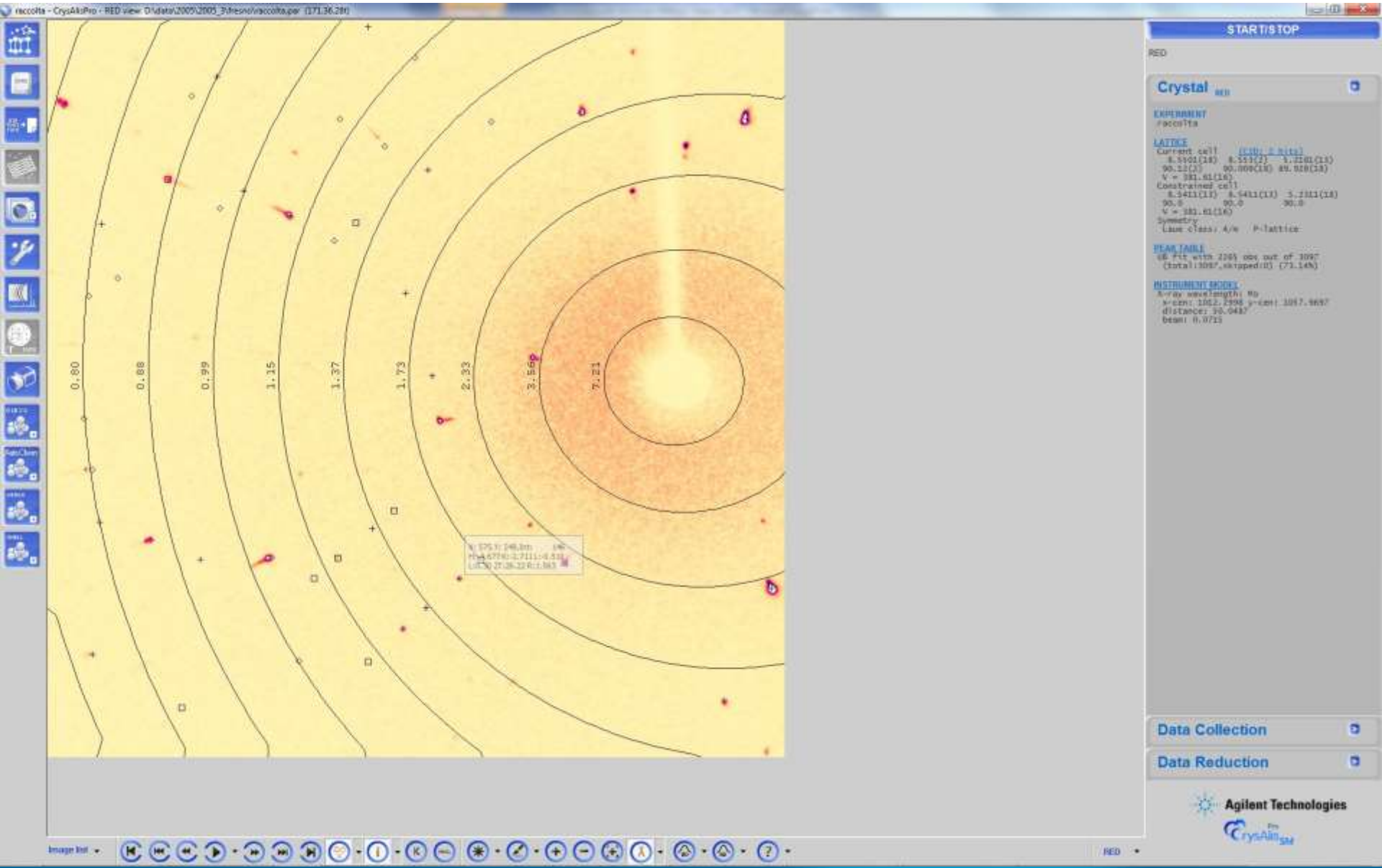


Agilent Technologies

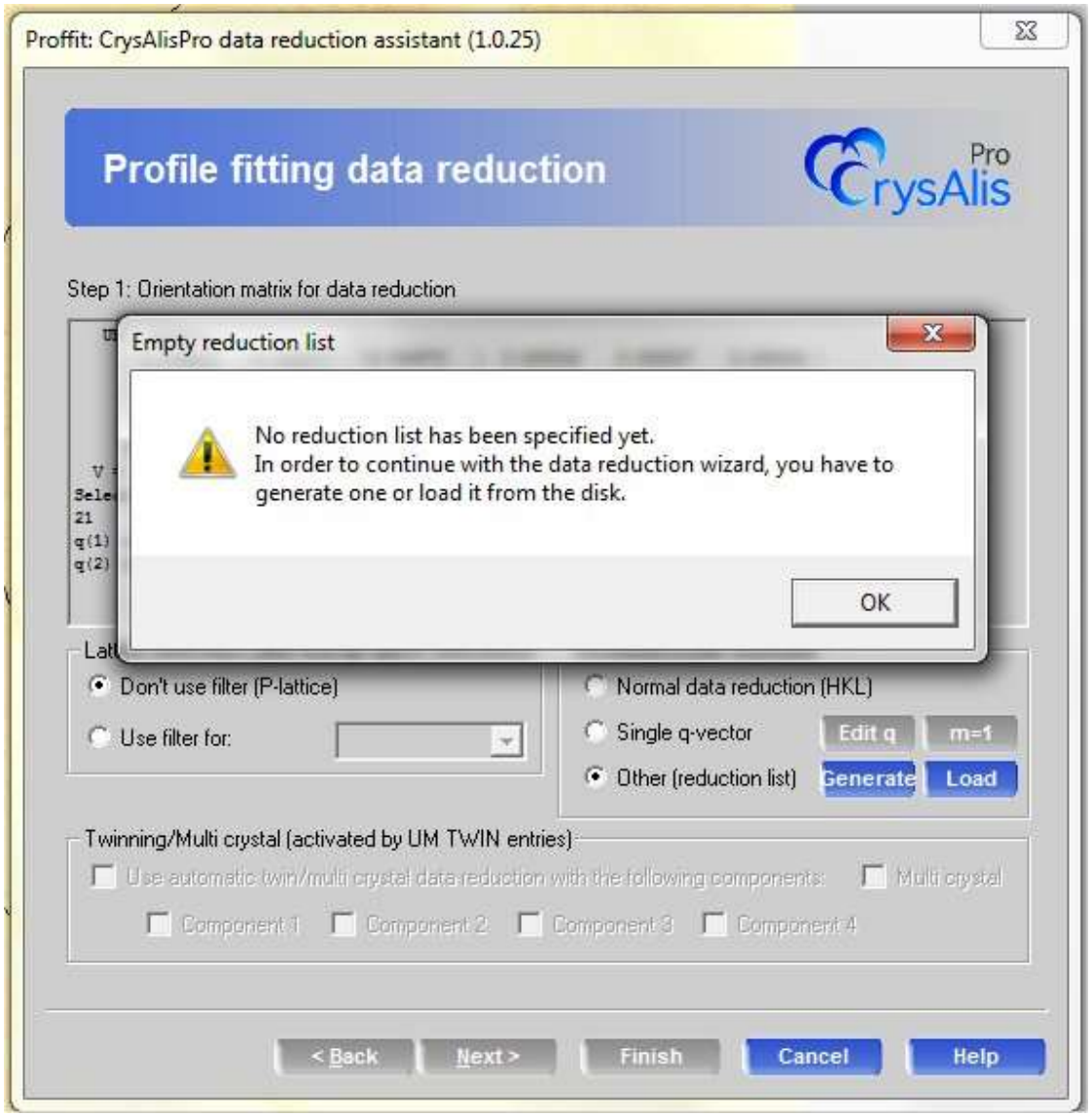
Fresno:


Lattice wizard (1.0.32)
Lattice wizard
Pro CrysAlis
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
Load information
SAVE
Save information
Unwarping - Precession images
Log window
Close

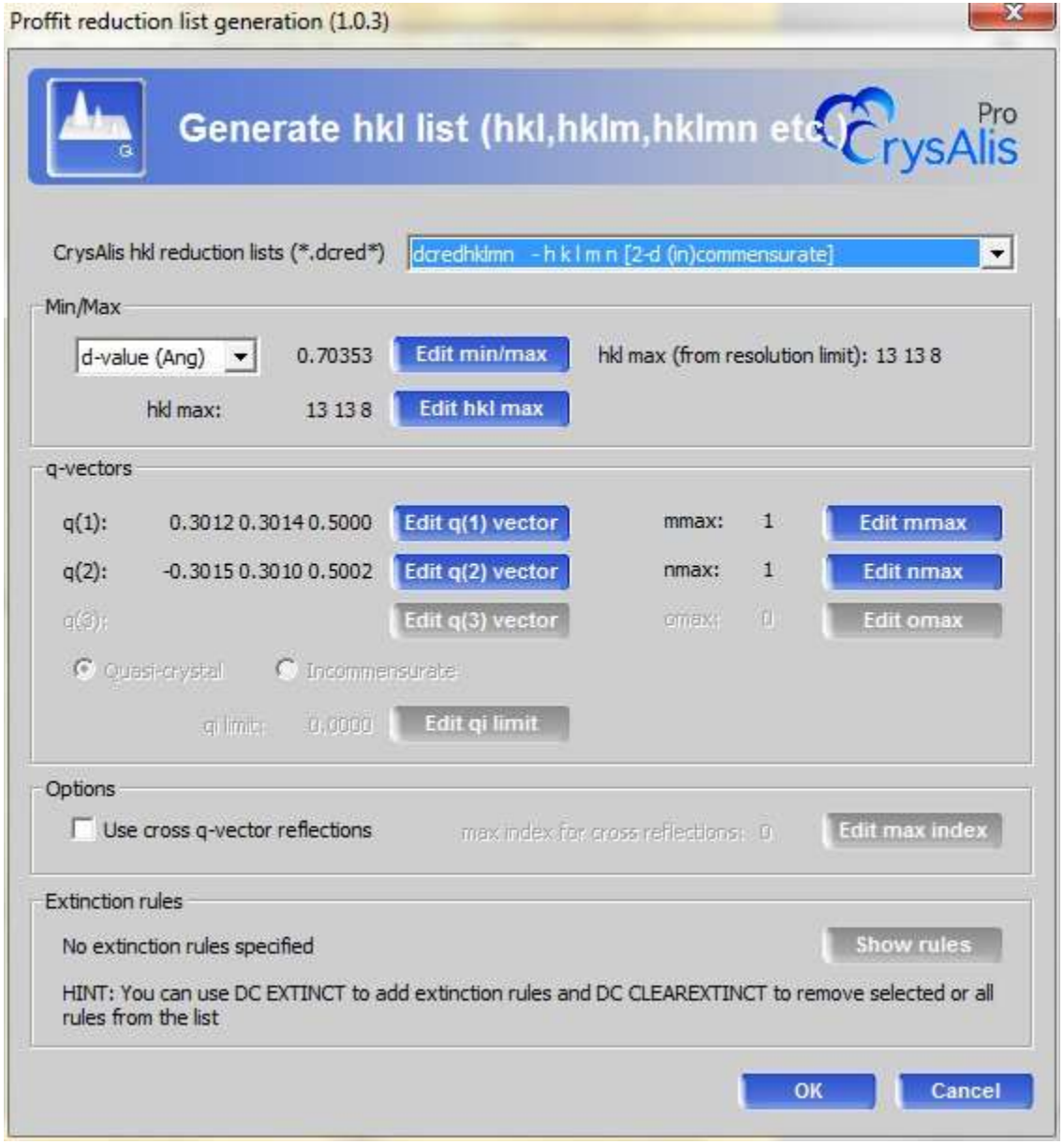
Fresno:


0.80
0.88
0.99
1.15
1.37
1.73
2.33
3.56
7.21
START/STOP
Crystal
Data Collection
Data Reduction
Agilent Technologies

Fresno:


Proffit: CrysAlisPro data reduction assistant (1.0.25)
Profile fitting data reduction
Pro
CrysAlis
Step 1: Orientation matrix for data reduction
Empty reduction list
No reduction list has been specified yet.
In order to continue with the data reduction wizard, you have to generate one or load it from the disk.
OK
Don't use filter (P-lattice)
Use filter for:
Normal data reduction (HKL)
Single q-vector
Edit q
m=1
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
< Back
Next >
Finish
Cancel
Help

Fresno:


Proffit reduction list generation (1.0.3)
Generate hkl list (hkl,hklm,hklmn etc)
CrysAlis Pro
CrysAlis hkl reduction lists (*.dcred*)
dcredhklmn - h k l m n [2-d (in)commensurate]
Min/Max
d-value (Ang)
0.70353
Edit min/max
hkl max (from resolution limit): 13 13 8
hkl max:
13 13 8
Edit hkl max
q-vectors
q(1):
0.3012 0.3014 0.5000
Edit q(1) vector
mmax:
1
Edit mmax
q(2):
-0.3015 0.3010 0.5002
Edit q(2) vector
nmax:
1
Edit nmax
q(3):
Edit q(3) vector
omax:
0
Edit omax
Quasi-crystal
Incommensurate
qi limit:
0.0000
Edit qi limit
Options
Use cross q-vector reflections
max index for cross reflections: 0
Edit max index
Extinction rules
No extinction rules specified
Show rules
HINT: You can use DC EXTINCT to add extinction rules and DC CLEAREXTINCT to remove selected or all rules from the list
OK
Cancel

Fresno: Strength of satellites

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

Main reflections

Statistics vs resolution - point group symmetry: P4/m

resolution(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

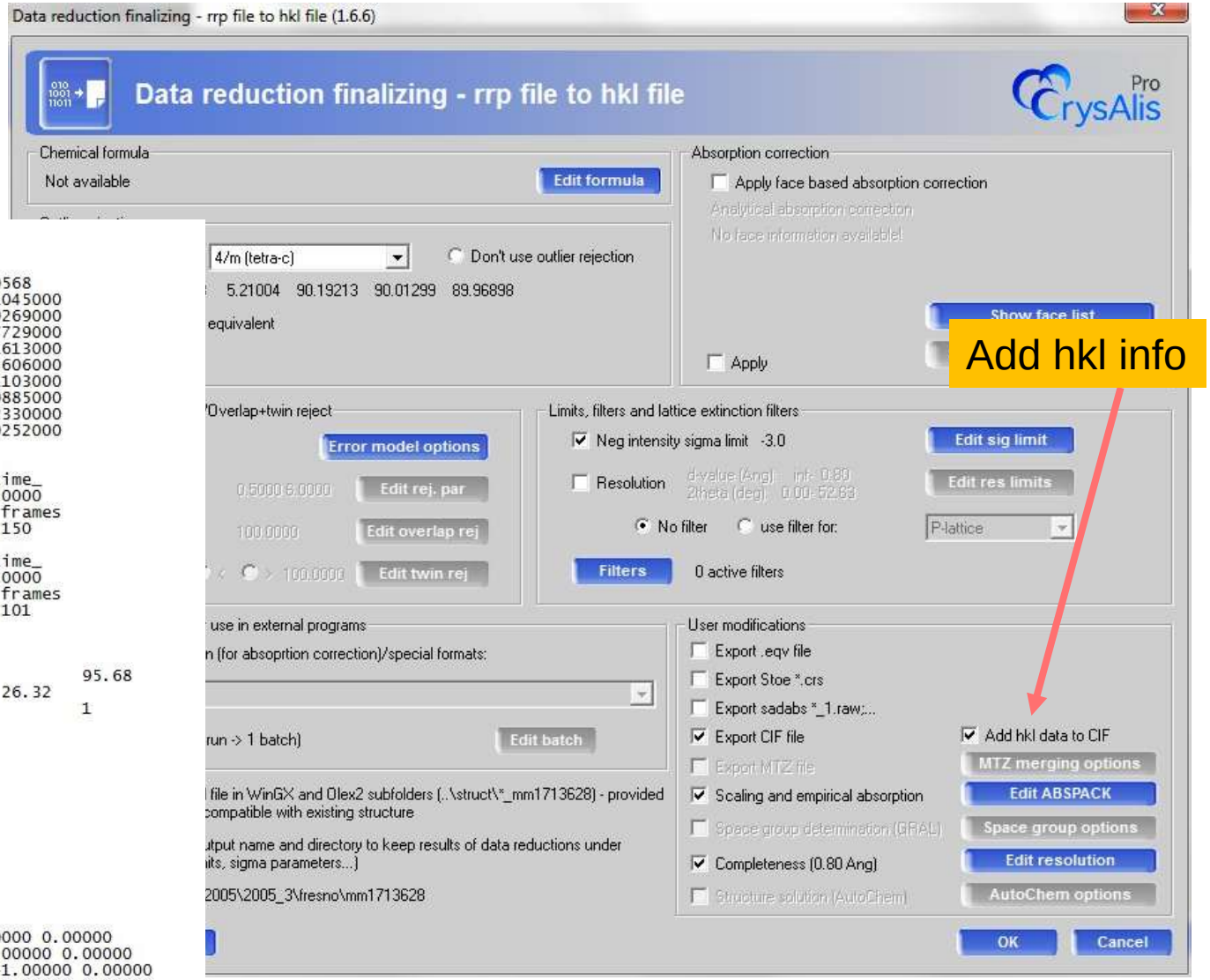
Satellite reflections

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

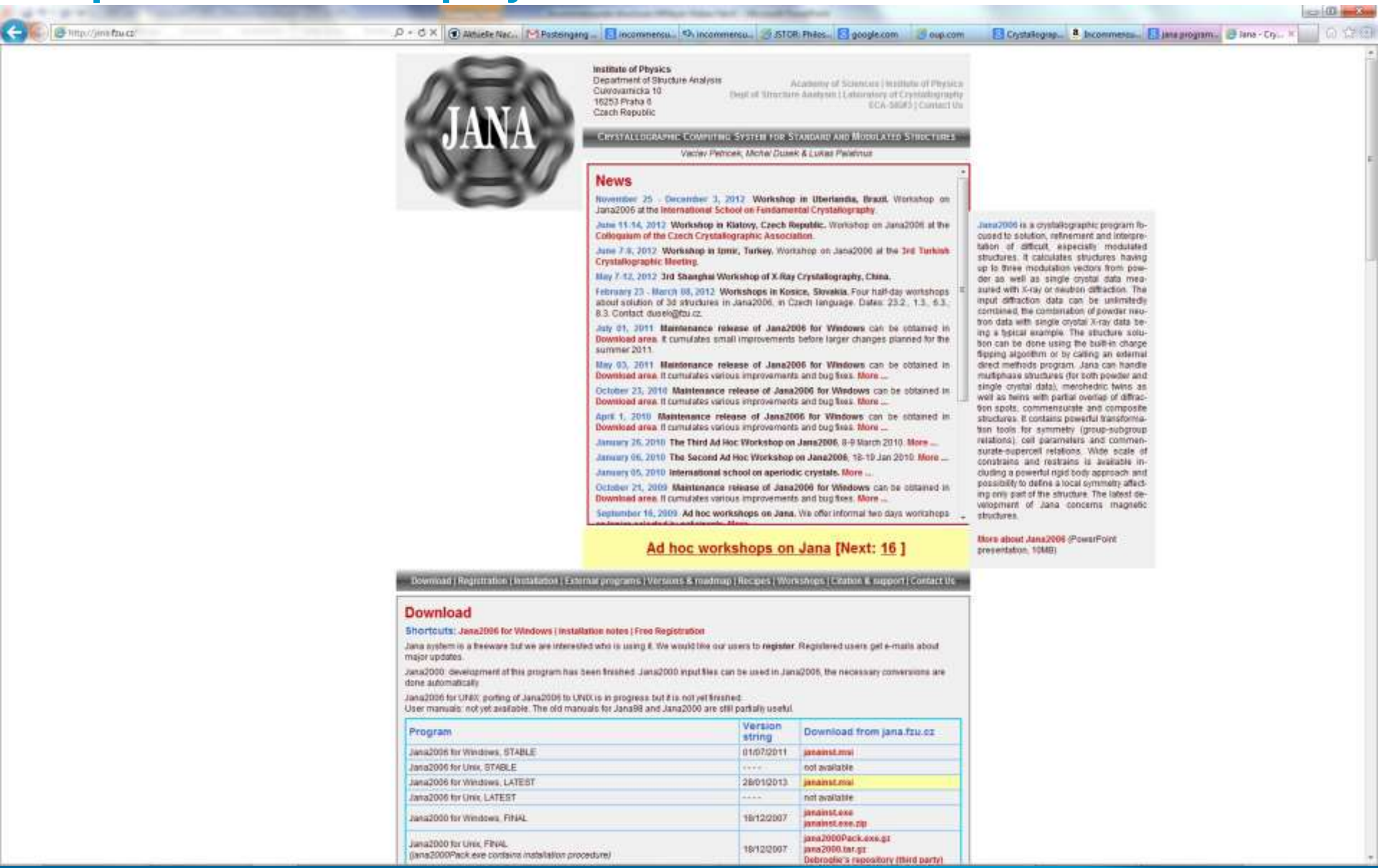
Export to Jana

- Via hkl or CIF

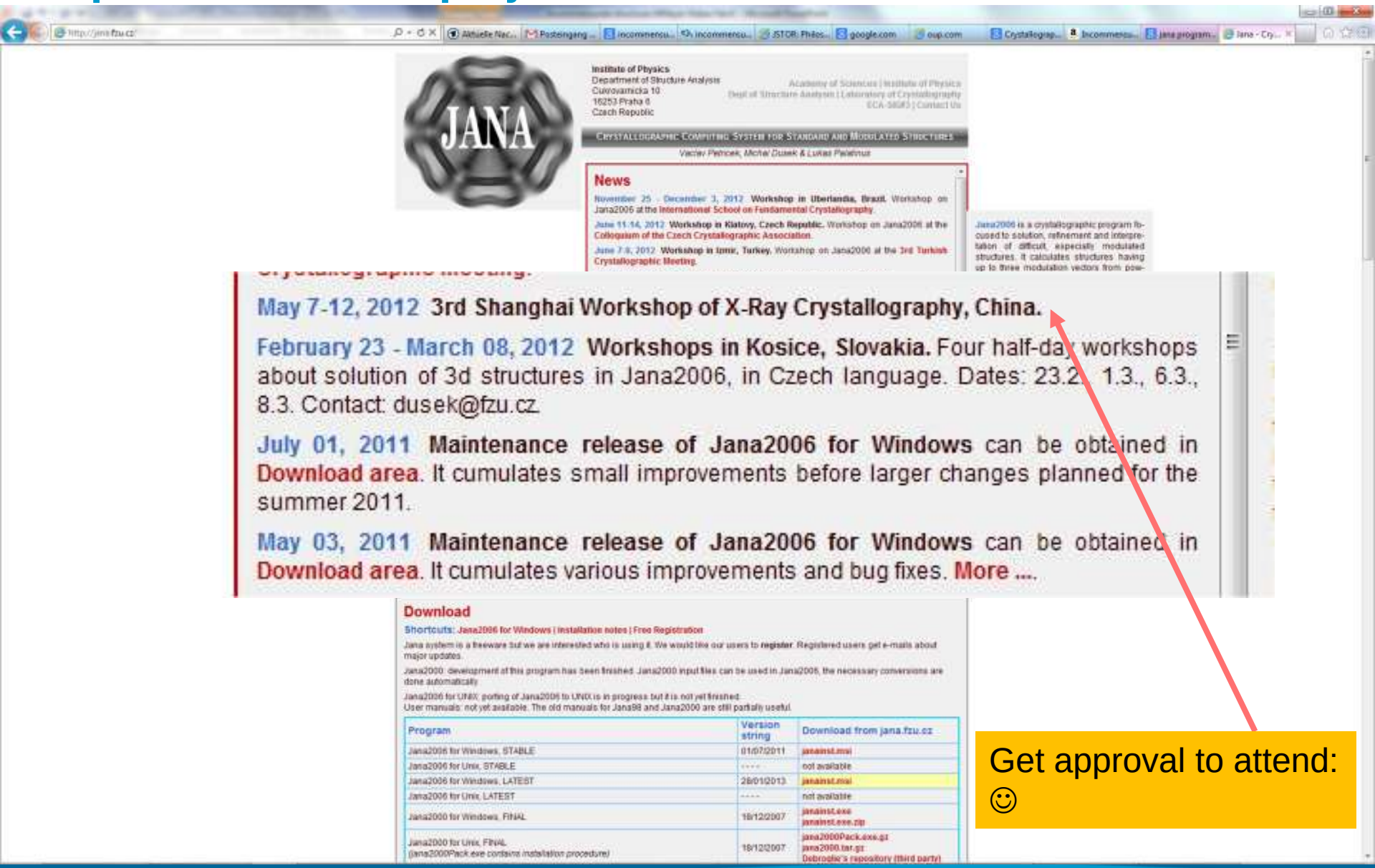
```
_diffrn_refln_index_l_max 0
_diffrn_reflns_theta_min 4.5503
_diffrn_reflns_theta_max 29.4106
_diffrn_reflns_theta_full 26.3154
_diffrn_measured_fraction_theta_full 0.9568
_diffrn_orient_matrix_UB_11       0.0761045000
_diffrn_orient_matrix_UB_12      -0.0229269000
_diffrn_orient_matrix_UB_13      -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
```



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



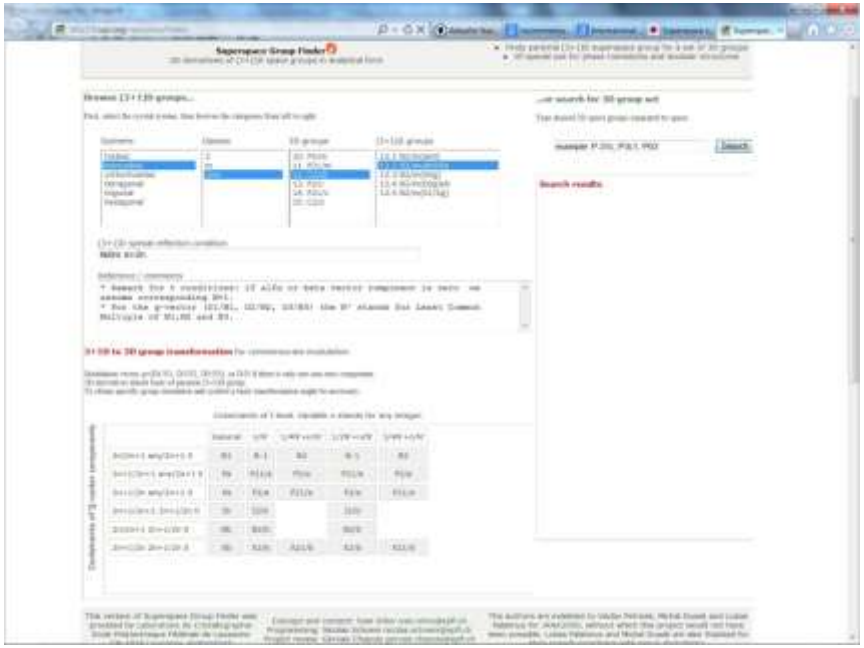
Export to Jana: http://jana.fzu.cz/



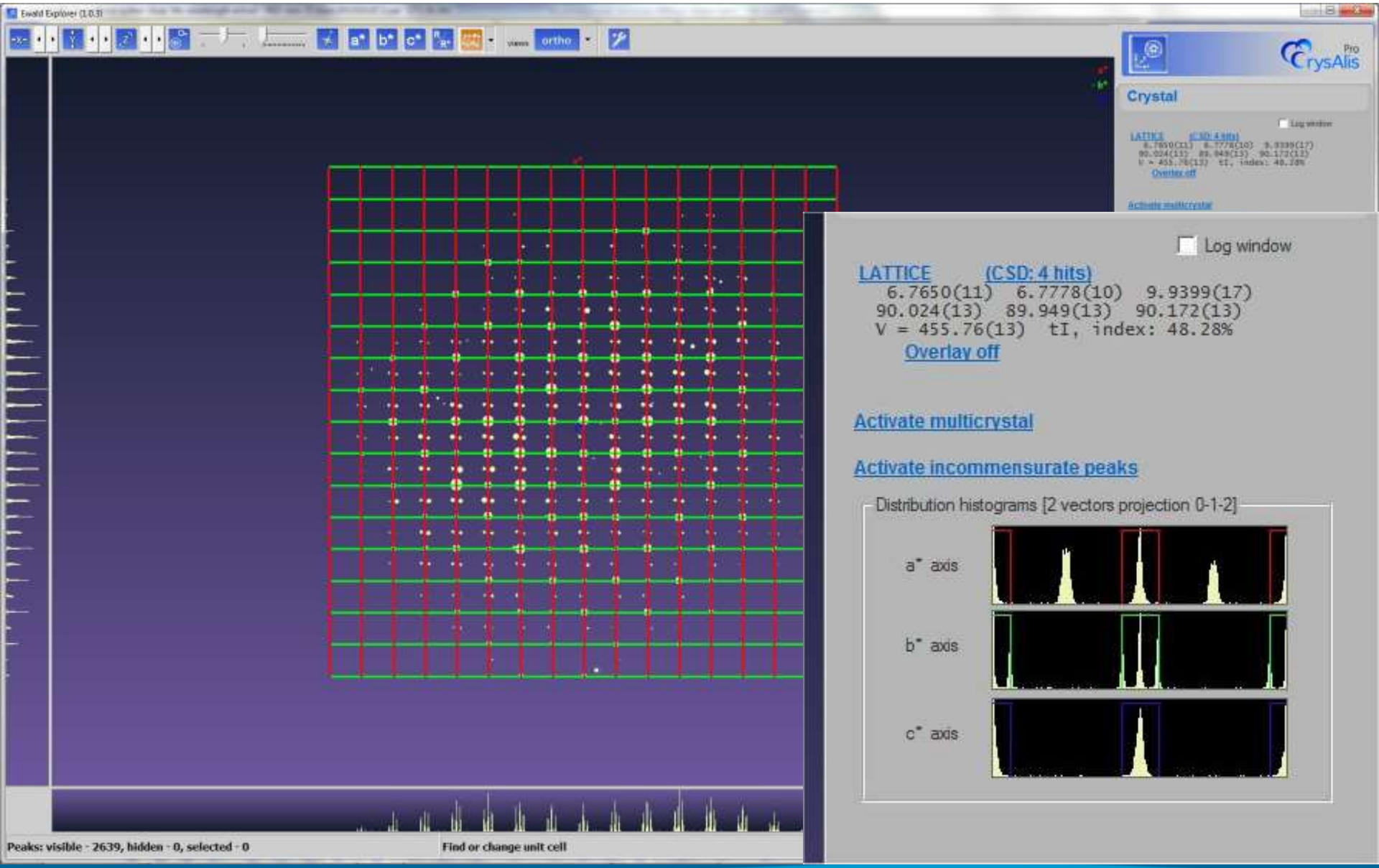
Get approval to attend: ☺

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/)



IC:



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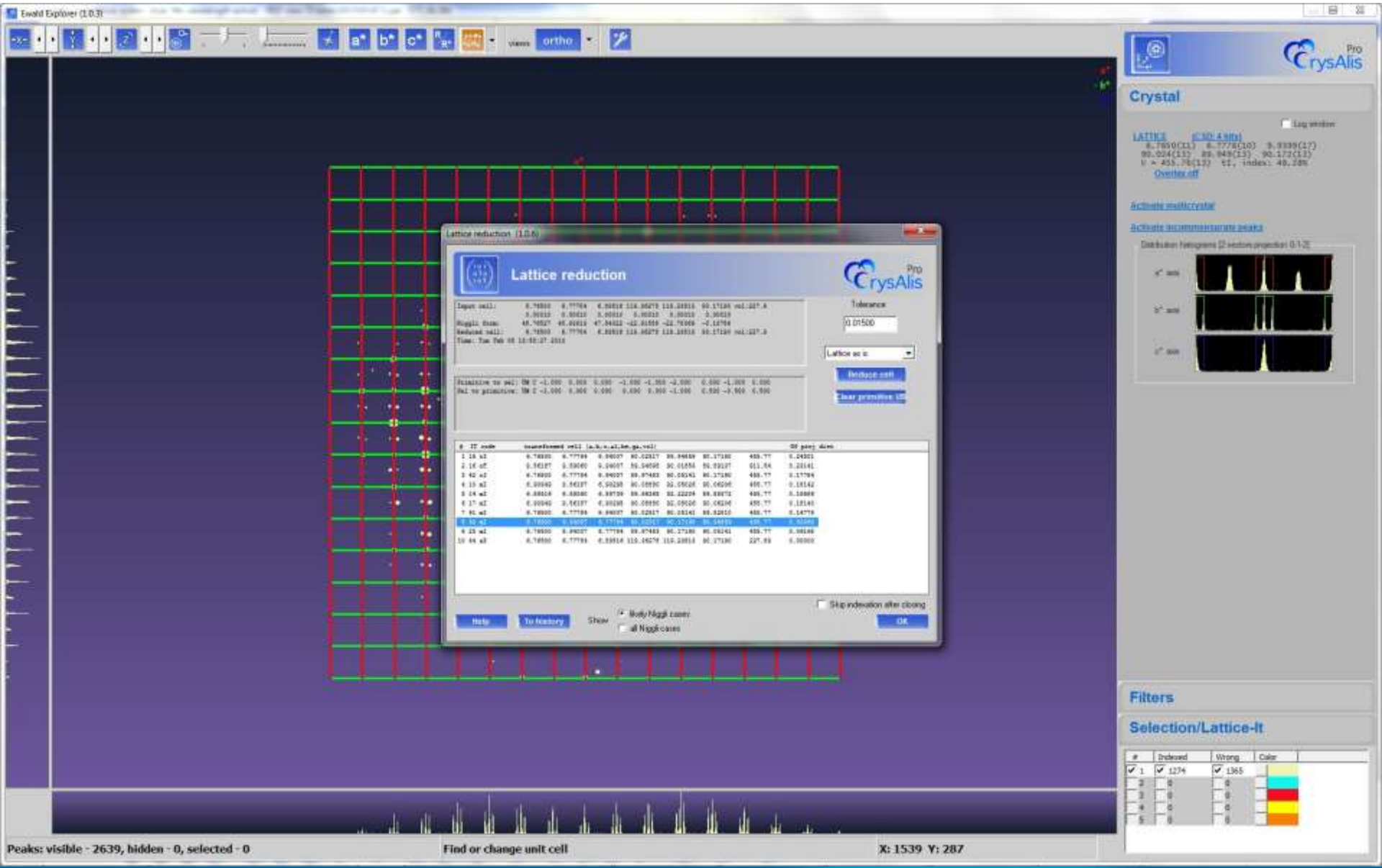
IC:



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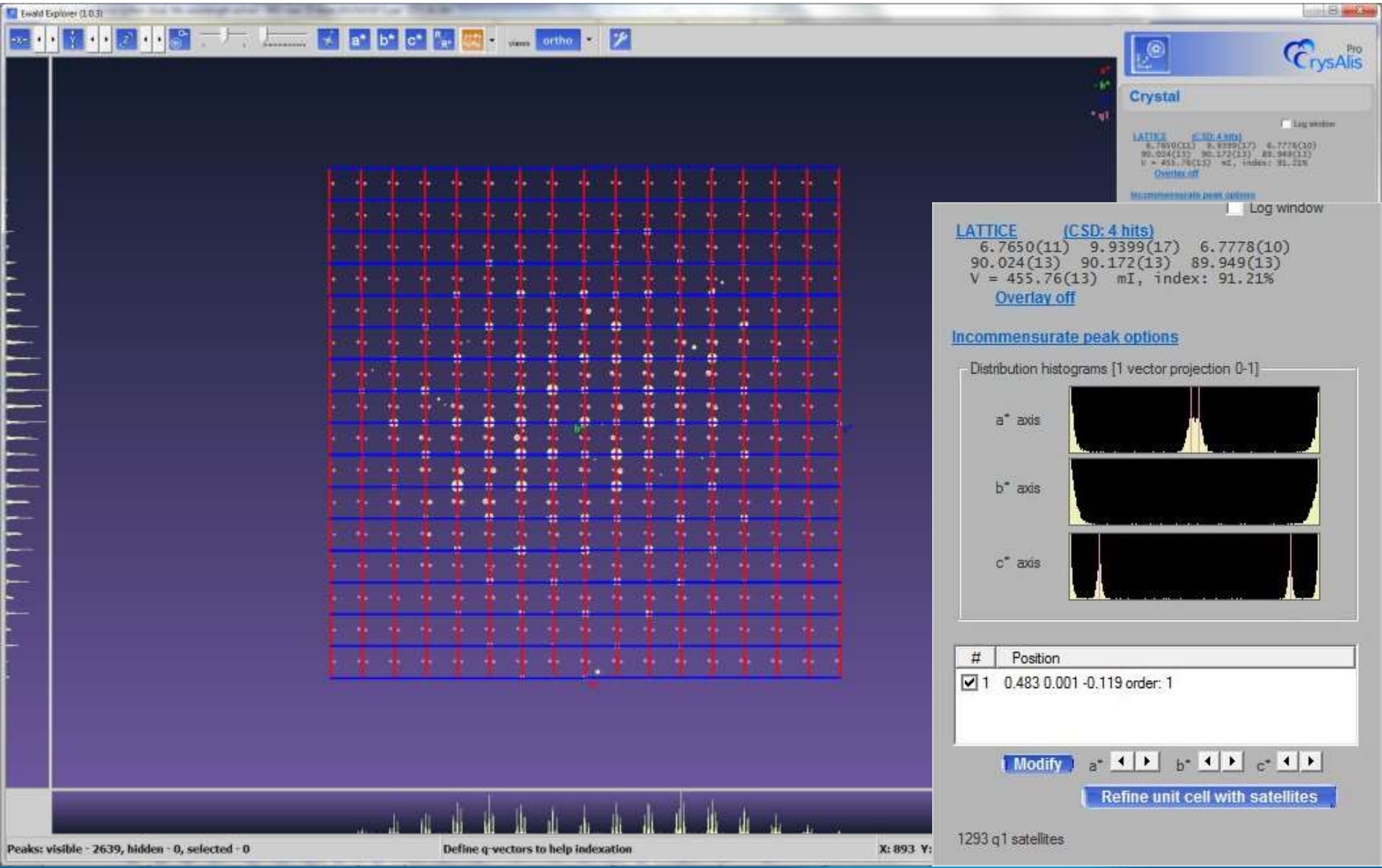
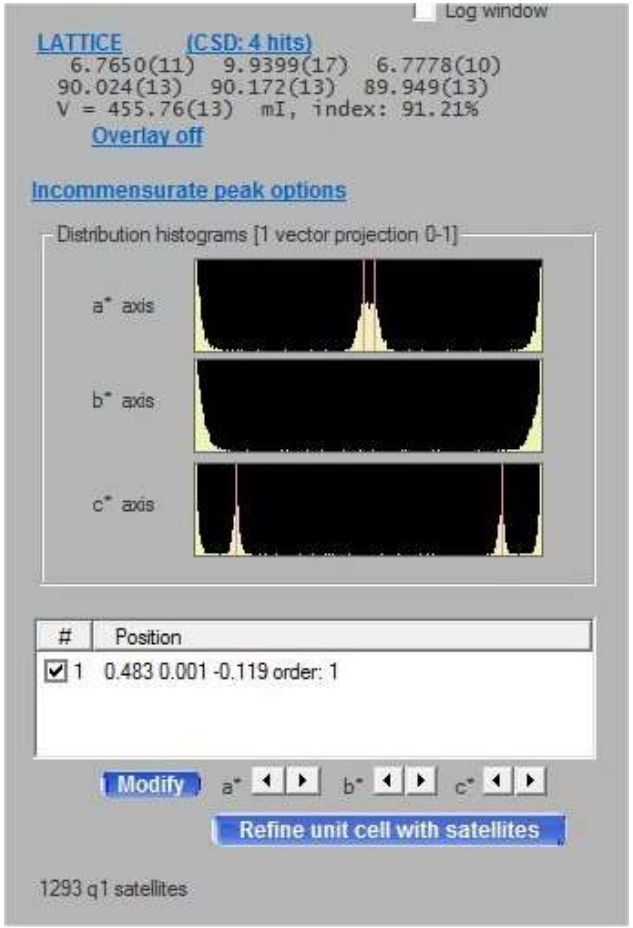
IC:

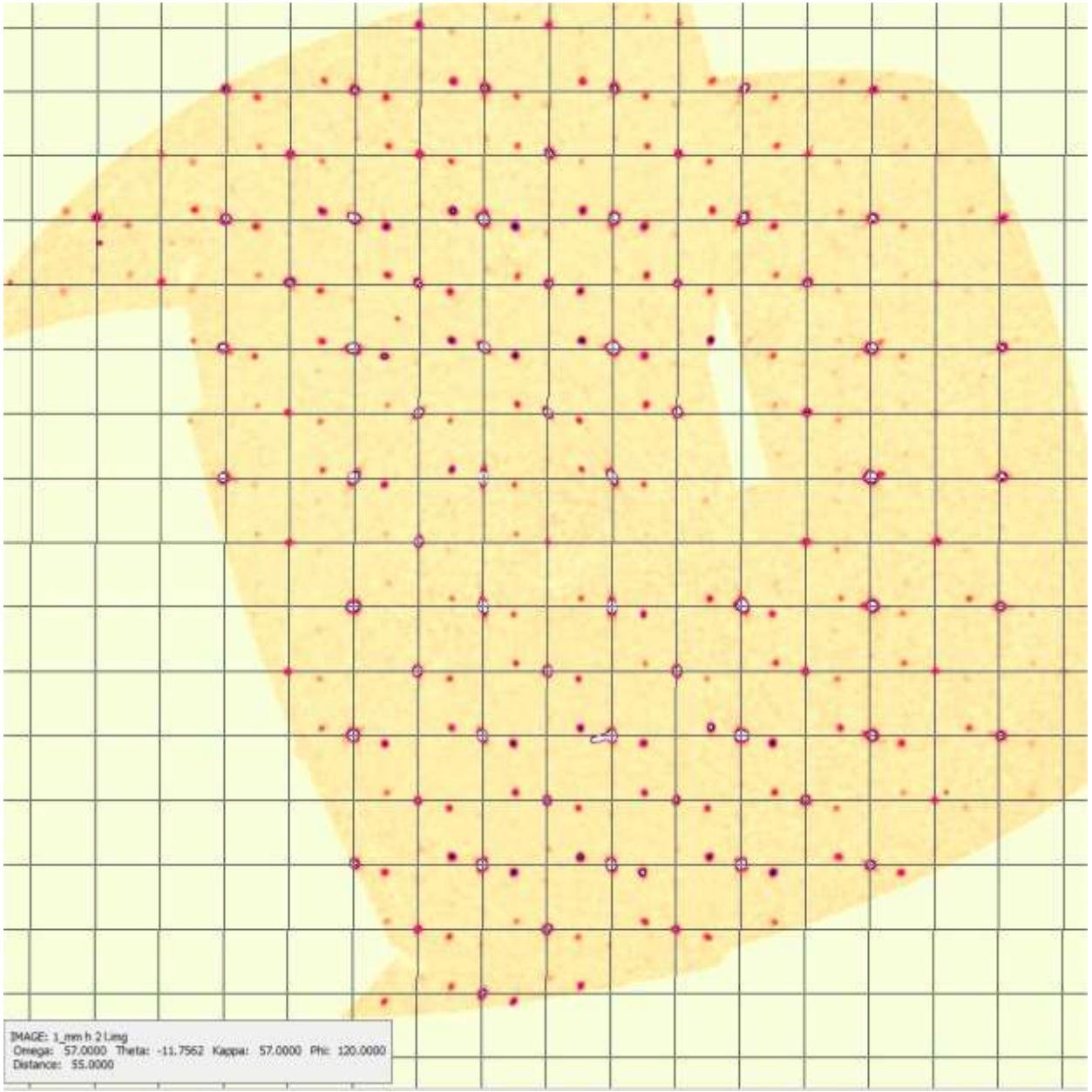

Ewald Explorer (1.0.3)
ortho
CrysAlis Pro
Crystal
Log window
LATTICE (CSD: 4 hits)
6.7650(11) 9.9399(17) 6.7778(10)
90.024(13) 90.172(13) 89.949(13)
V = 455.76(13) mI, index: 91.21%
Overlay off
Incommensurate peak options
Distribution histograms [1 vector projection 0-1]
a* axis
b* axis
c* axis
Position
1 0.483 0.001 -0.119 order: 1
Modify
a*
b*
c*
Refine unit cell with satellites
1293 q1 satellites
Peaks: visible - 2639, hidden - 0, selected - 0
Define q-vectors to help indexation
X: 893 Y:

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)

resolution(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*scintillator thickness. Eos has a front pixel size of: 1x1 = 33microns; 2x2 = 66microns; 4x4 = 132microns. Atlas has a front pixel size of: 1x1 = 50microns; 2x2 = 100microns; 4x4 = 200microns. So 2x2 and 4x4 binning are reasonable with respect to resolution. Your camera's 1x1 pixel size you find on the 'instrument model I' tab.
- The source divergence is of the order of (5-10mRad = 0.3-0.6deg). 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

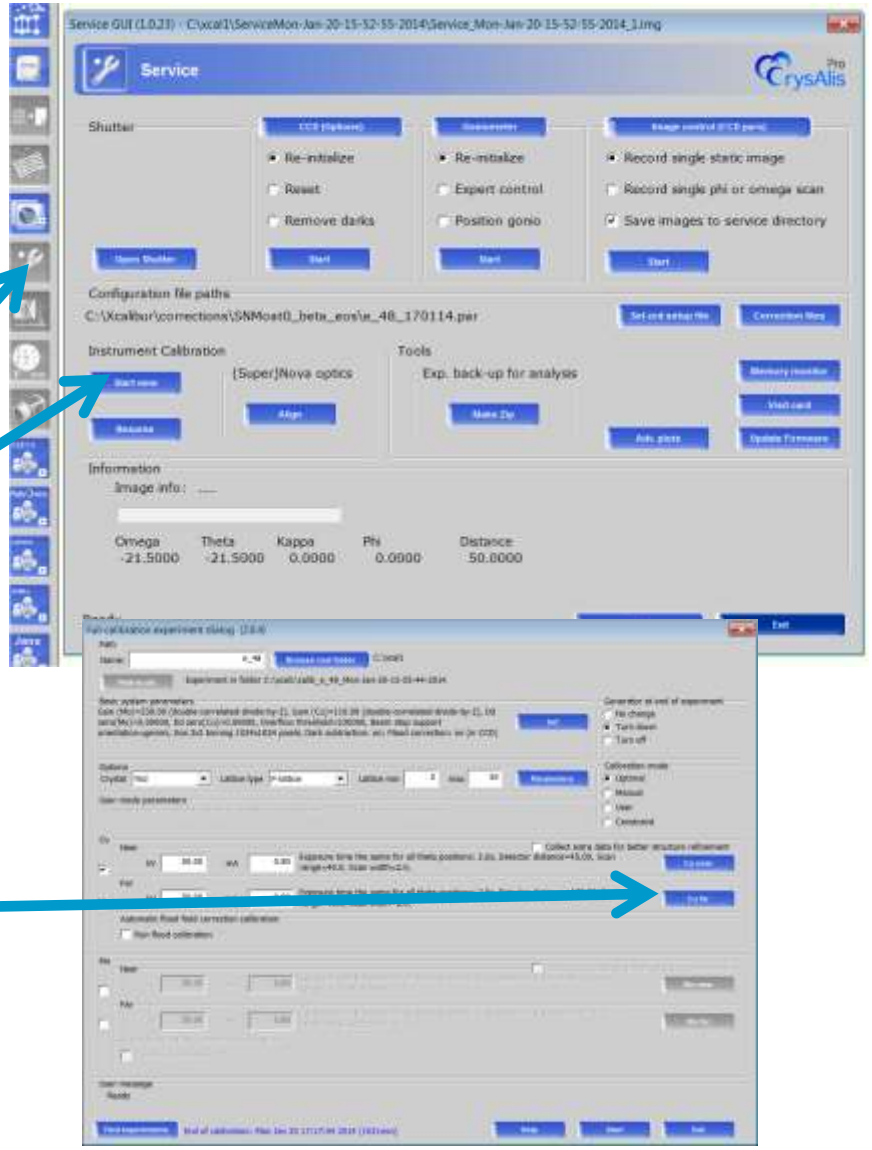
- As was said: The instrument is calibrated for the single crystal application. The calibration is only exact at the ‘near’ calibration point. Experiments at distances other than the near point rely on the model interpolation in conjunction with ‘refine model’. This means that the instrument model might 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…



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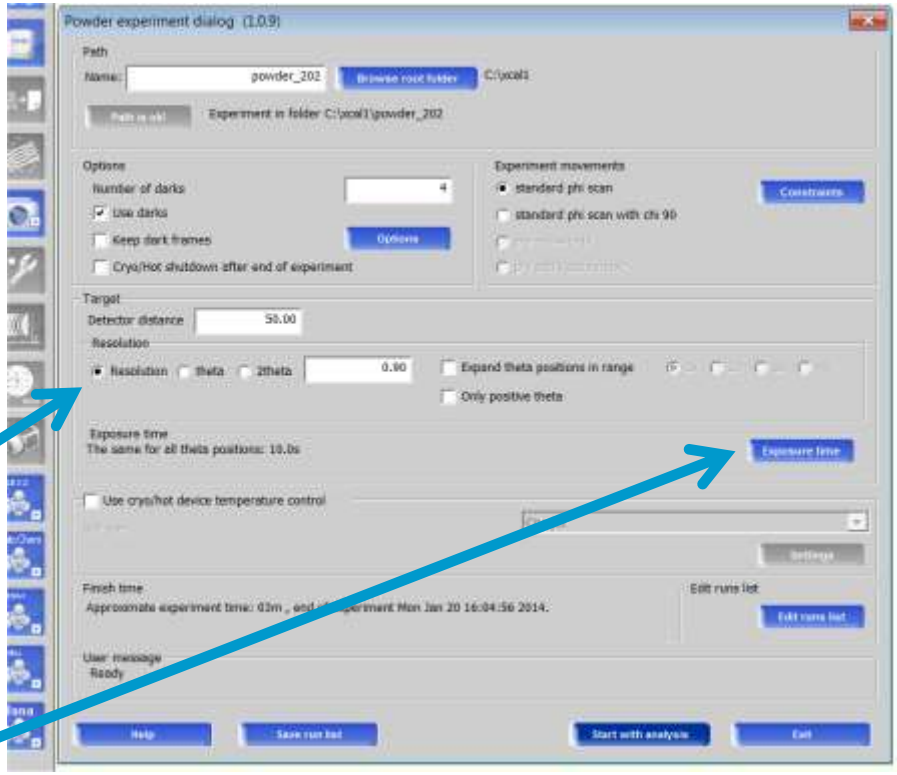
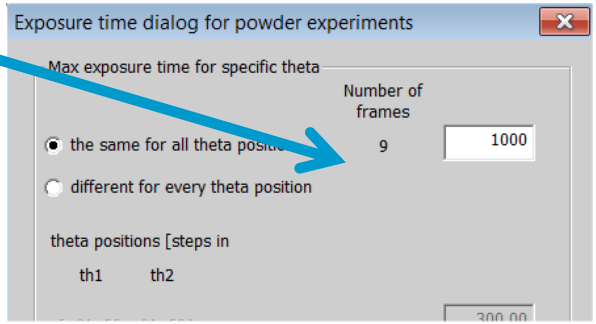
Powder experiments

- After a successful calibration you can use the instrument for powder experiments at the near calibration point.
- Mount you 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





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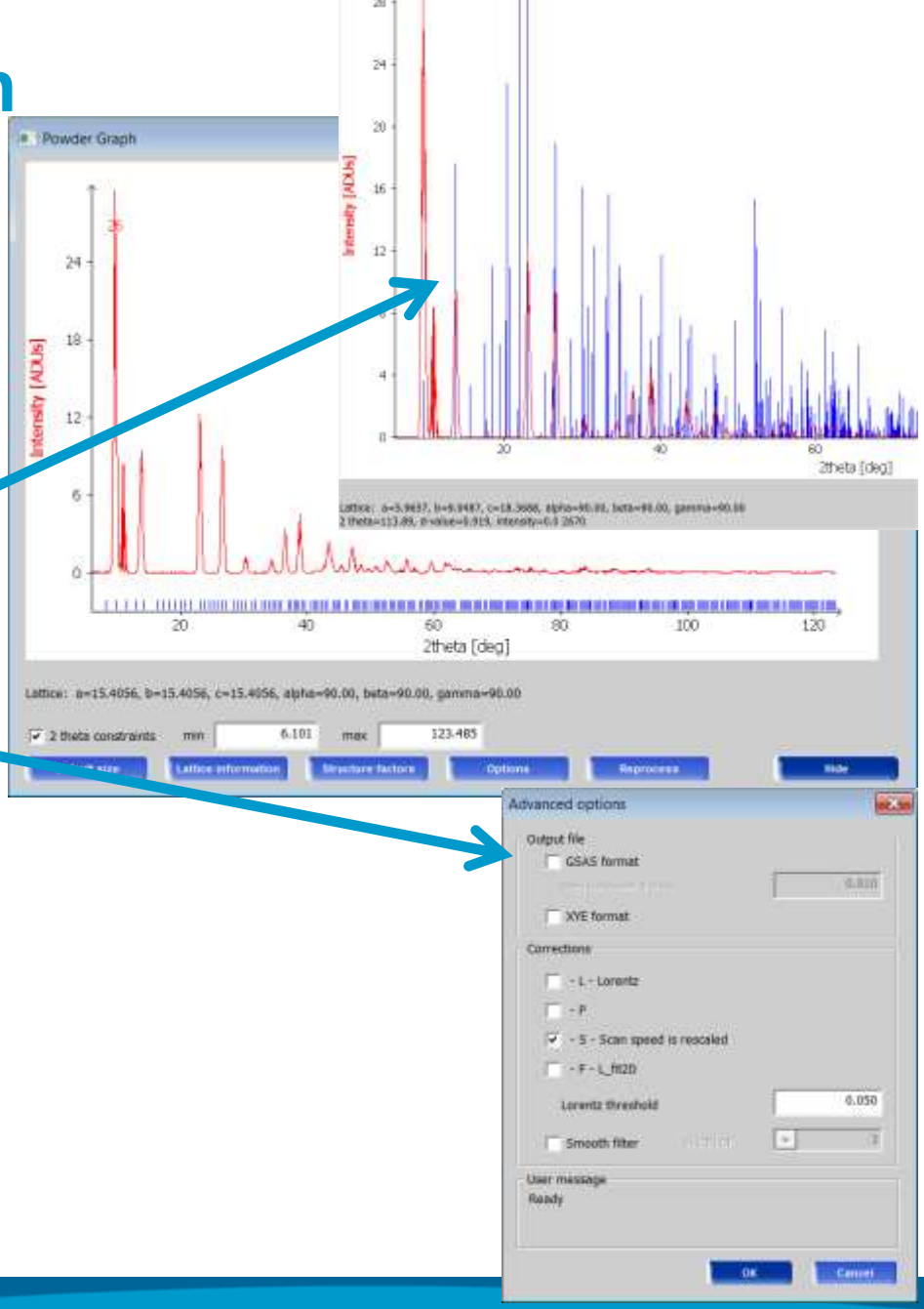
Powder pattern extraction

- 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:\xcal1\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:\xcal1\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:\xcal1\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:\xcal1\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:\xcal1\calib_e_48_Mon-Jan-13-19-00-06-2014\Mo\Near45mm\Mo_Near45mm.hkl
```

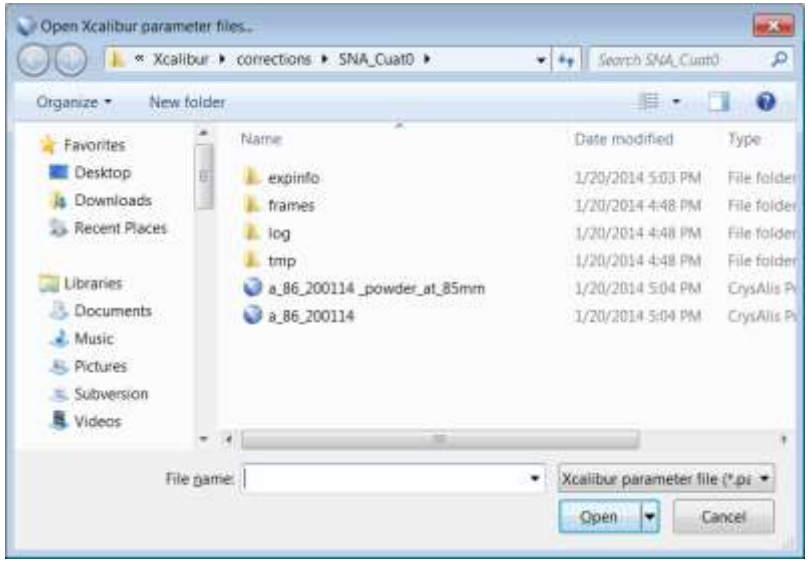
- Note that the extracted pattern will land in a file.

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

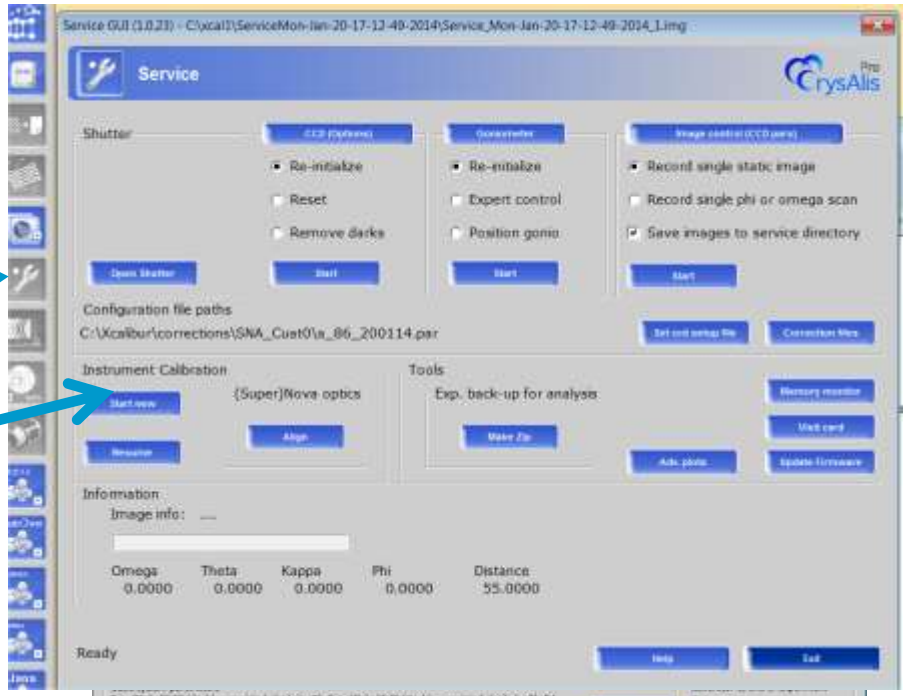
PEAAD 1

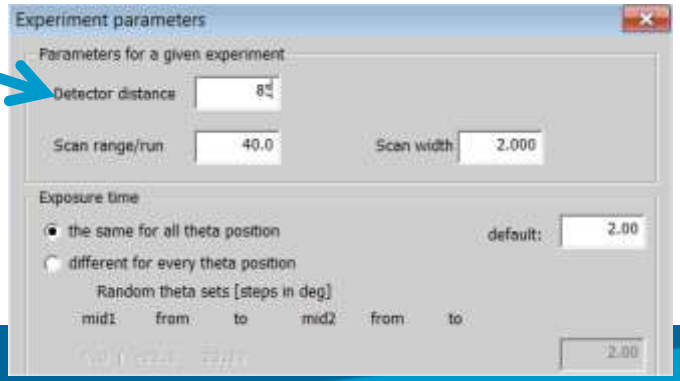
- Make a copy of your current par file: Mine is ‘a_86_200114.par’. The powder one I will call ‘a_86_200114_powder_at_85mm.par’.
- The new par is selected as the new ccd configuration file (under ‘Configuration file paths’ -> ‘Set ccd setup file’).
- The software prompts to restart the software, which 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.

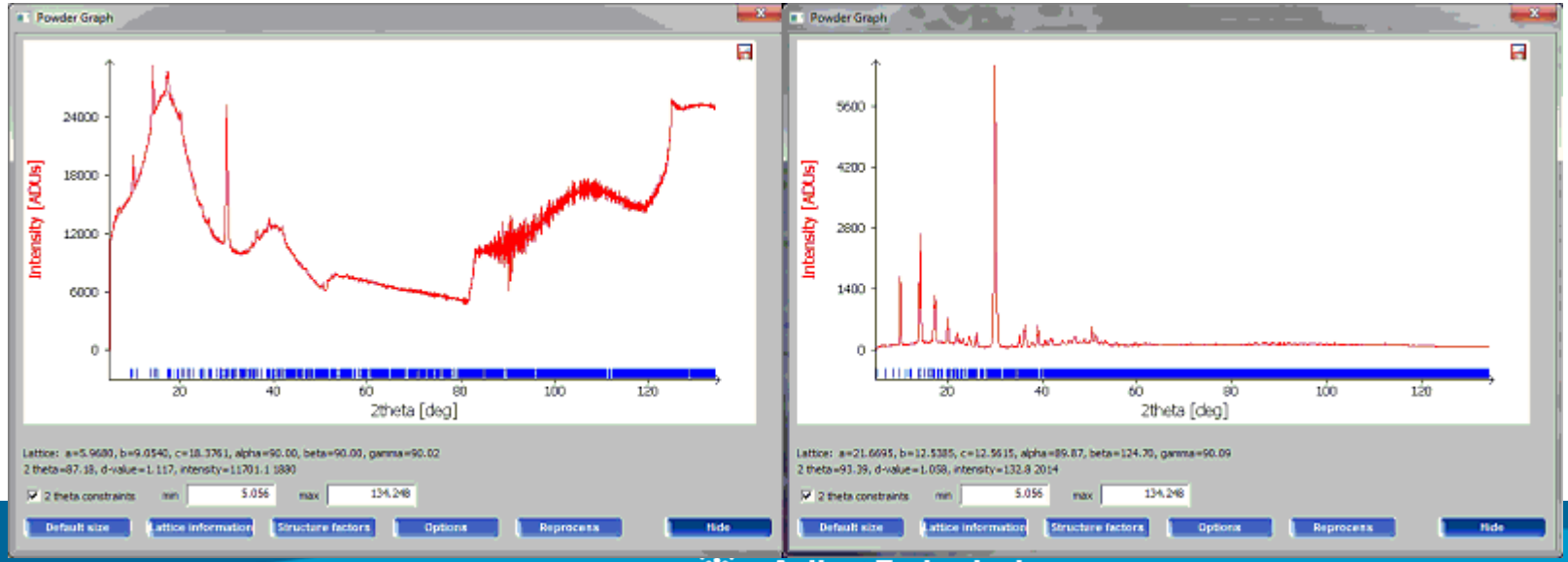
Powder: common errors

- 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 calbration 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 $X_{raw,i}$ as X-ray counts (ADUs/system gain). In parallel we accumulate the number of hits C_i and the corrected counts $X_{corr,i}$ (optional L, P and scan time rescale).
- The final pattern is outputted in the following way: The $\sigma(X_{raw,i}) = sqrt(X_{raw,i})$ and $\sigma(X_{corr,i}) = \sigma(X_{raw,i})\ X_{corr,i} / X_{raw,i}$. We output as I_i -> $X_{corr,i} / C_i$ and $\sigma(I_i)$ -> $\sigma(X_{corr,i})/ C_i$. For proper plotting an additional rescale is applied on the max(I_i; (i 1 to N)) is <10.

Thank you for listening!

Find out more at

www.agilent.com/chem/xrd