

Reciprocal Workshop: Rigaku Oxford Diffraction

Handling twins

Some notes on corrections

les macles

The twins

双晶



Overview

- Twin/Multi-crystal
- New twin processing for better accuracy
- Step-by-step processing on easy twin and lattice faker
- Twin example on Crystalline Sponge
(Vacant [cyclohexane x 5] but twined, guideline for the worst condition before soaking)
 $R_{int} < 3.0\%$, $R_1 < 7.0\%$ @ redundancy > 2.5
- Time permitting: Some notes on corrections:
Empirical and numeric corrections

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

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

Question:

Twin vs. multi-crystal – what is what?

*non-merohedric

Screening tool for quickly judging sample quality

SM Screening

Screening

Mount Screening >

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

QUALITY

Resolution(A)	N	I/sig	I/sig ₀
inf - 1.23	91	24.0	26.5
1.28- 1.23(last)	10	12.9	14.2

Well diffracting sample

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

Experiment - Complete data for publication

Name: exp_209

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

Exposure time: 1.0 s

Start Pre-Exp. (5 min) Edit

Goniometer

Omega	Theta	Kappa	Phi	Distance
20.0	-35.0	0.0	0.0	52.0

Generator

kV	mA
50.0	0.80

SM Screening

Screening

Mount Screening >

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

QUALITY

Resolution(A)	N	I/sig	I/sig ₀
inf - 0.91	749	0.7	10.8
1.05- 0.91(last)	83	0.5	7.1

Weakly diffracting sample

NOTE: Quality estimation may be unreliable - you can:

- Increase image binning to 4x4
- Increase exposure time
- Run pre-experiment with suggested exposure time
- Change sample

Experiment - Complete data for publication

Name: exp_101

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

Exposure time: 60 s

Start Pre-Exp. (35 min) Edit

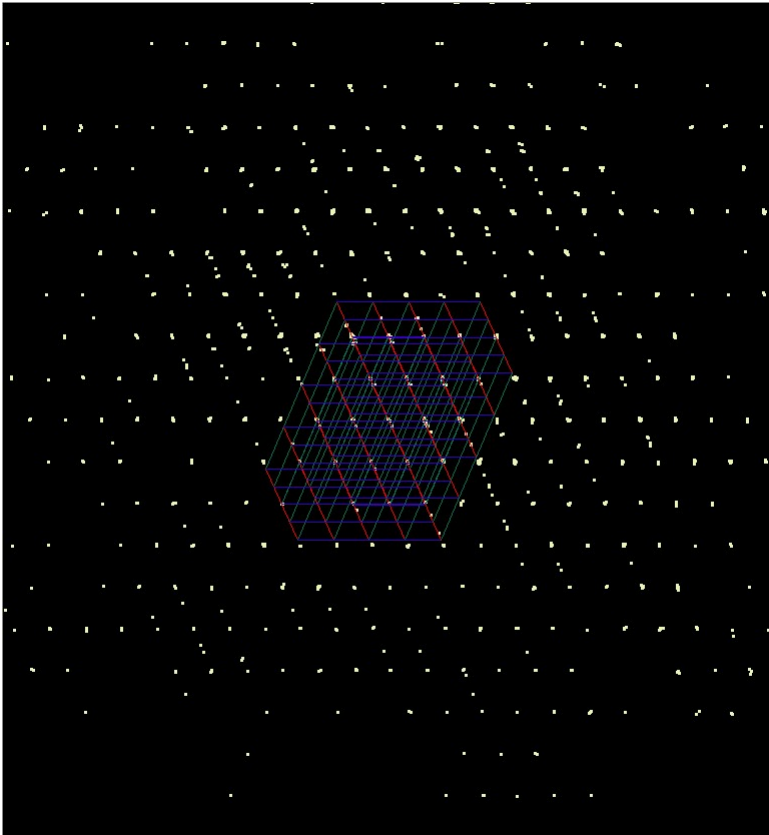
Goniometer

Omega	Theta	Kappa	Phi	Distance
20.0	0.0	0.0	0.0	41.9

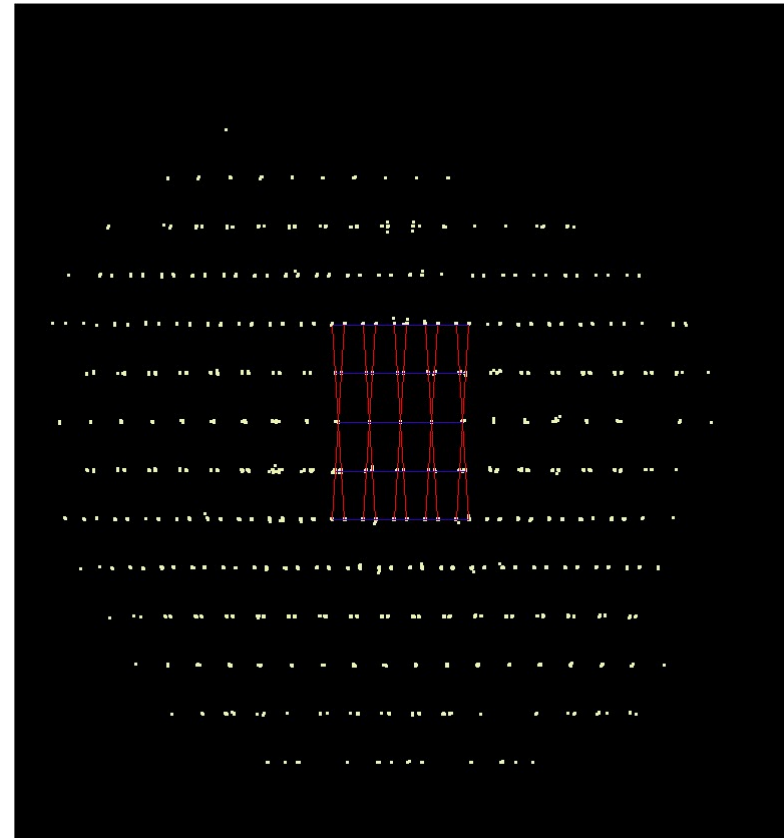
Generator

kV	mA
0.0	0.00

Twin types



- Easy twin

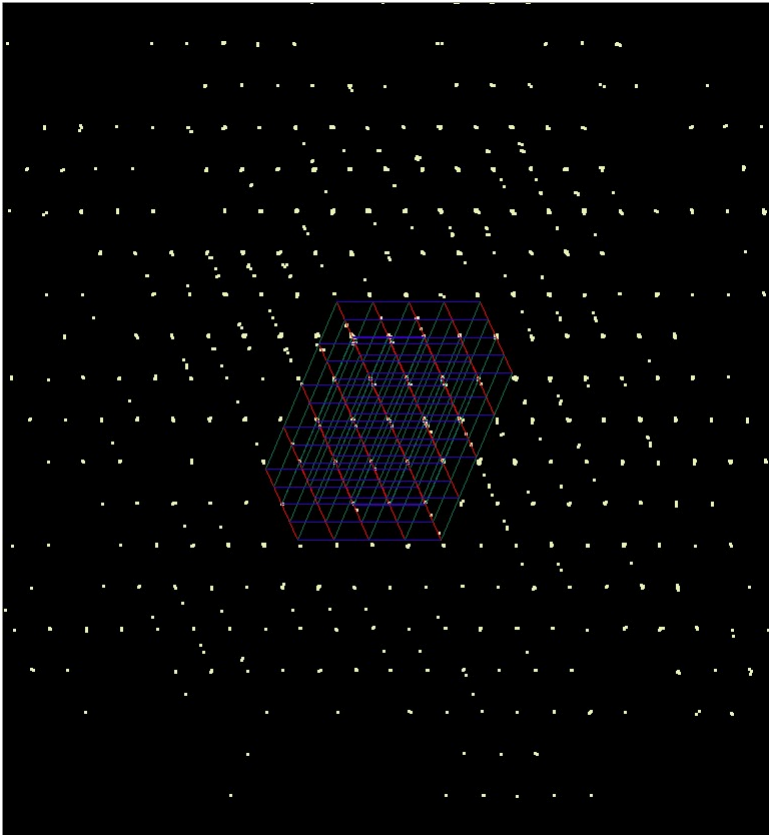


- Super lattice "faker"

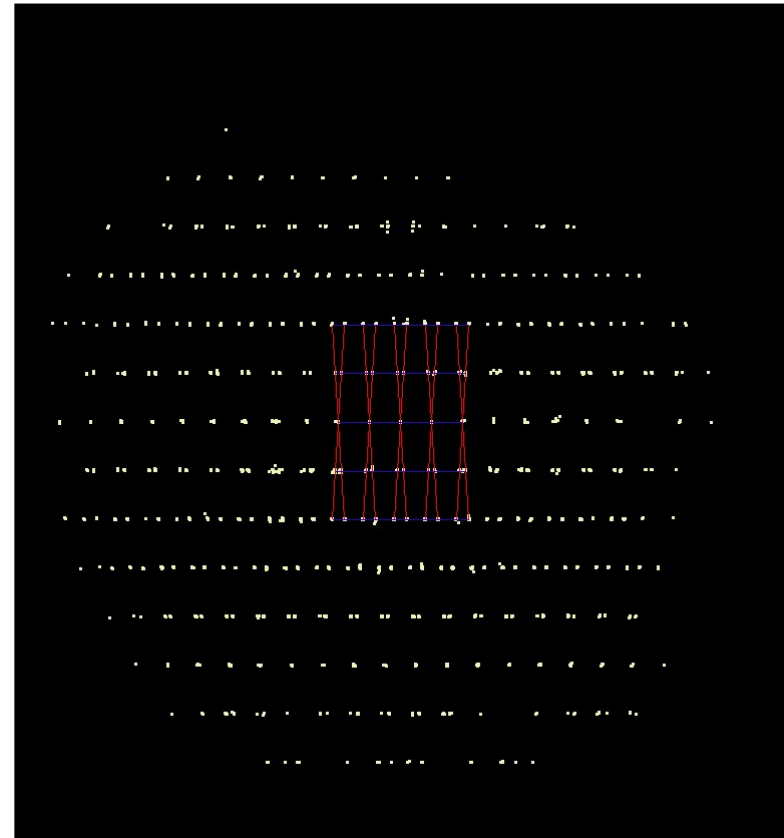
Data sets for these examples

- Pol411, pol426
- CCD data; kindly provided by Prof. Maria Gdaniec (Poznan, Poland)
- You can try to apply the concepts presented here to these data sets.

Twin types



- Easy twin (pol426)



- Super lattice “faker” (pol411)

Easy twin

Lattice wizard (1.0.32)

Lattice wizard

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

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

INSTRUMENT MODEL
Goniometer
beam: 0.04194 alpha: 50.04288 beta: -0.01668
om zero: -0.73625 th zero: -0.00479 ka zero: -0.90939
Detector
x-rot: 0.46124 y-rot: -0.20948
x-zen: 523.89276 y-zen: 507.49376 distance: 60.50539
Wavelength Mo (Ang): A1 0.70930 A2 0.71359 B1 0.63229

Peak hunting
Ewald explorer - reciprocal space
Refine instrument model
Twinning - multi-crystals

Unit cell
Reindex current
Lattice transform
Incommensurate

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

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

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

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

Z: 1.00

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

C80 H40 F24

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

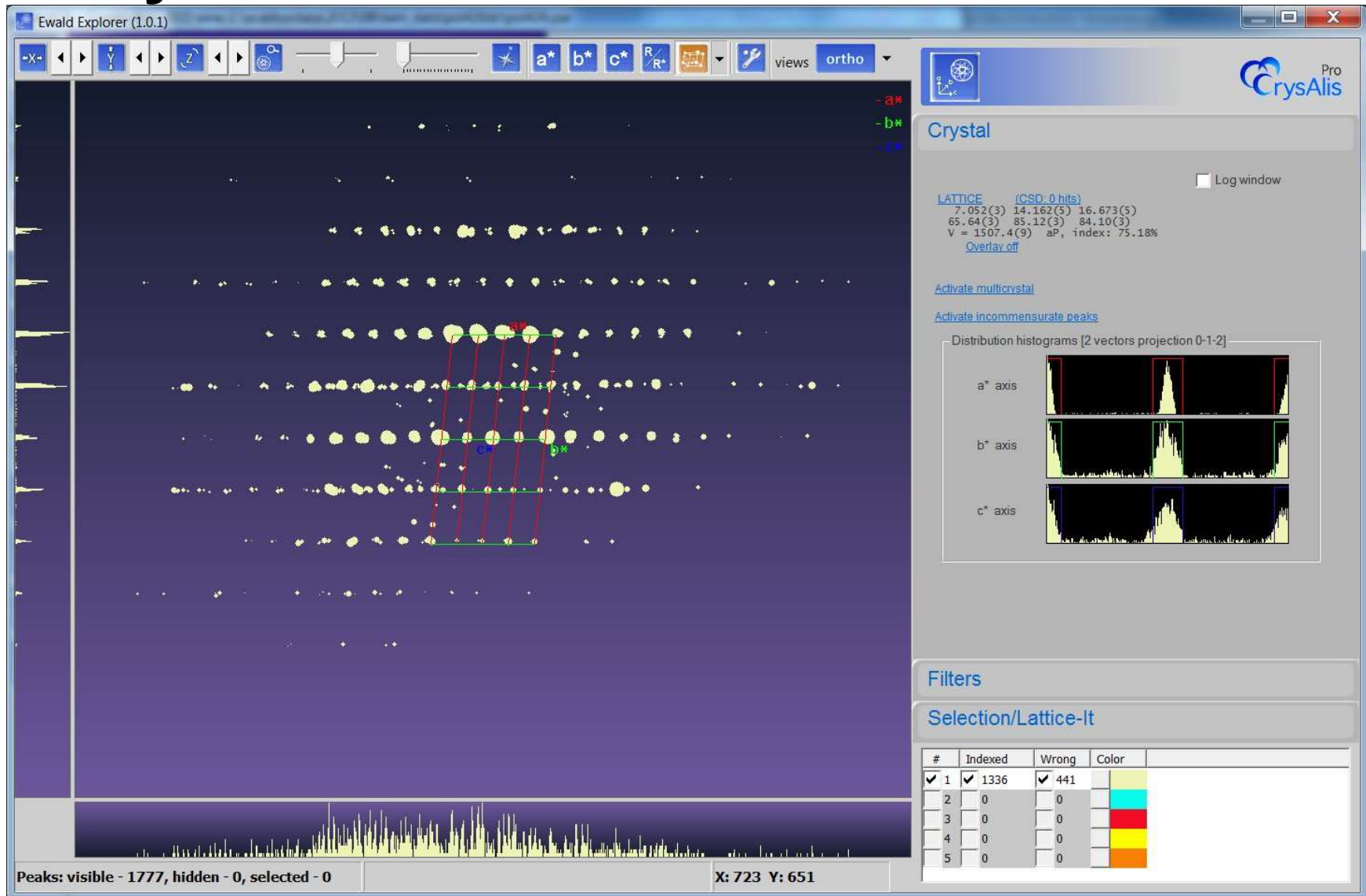
mu (mm-1) 0.14490 Edit mu

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

We do peak hunting and auto-indexing gives:

- Indexation <90%
- Chemical content consistent

Easy twin in Ewald^{Pro}



Easy twin in Ewald^{Pro}

Crystal

LATTICE (CSD: 0 hits)
 7.052(3) 14.162(5) 16.673(5)
 65.64(3) 85.12(3) 84.10(3)
 V = 1507.4(9) aP, index: 75.18%
[Overlay off](#)

[Activate multicrystal](#)

[Activate incommensurate peaks](#)

Distribution histograms [2 vectors projection 0-1-2]

a* axis

b* axis

c* axis

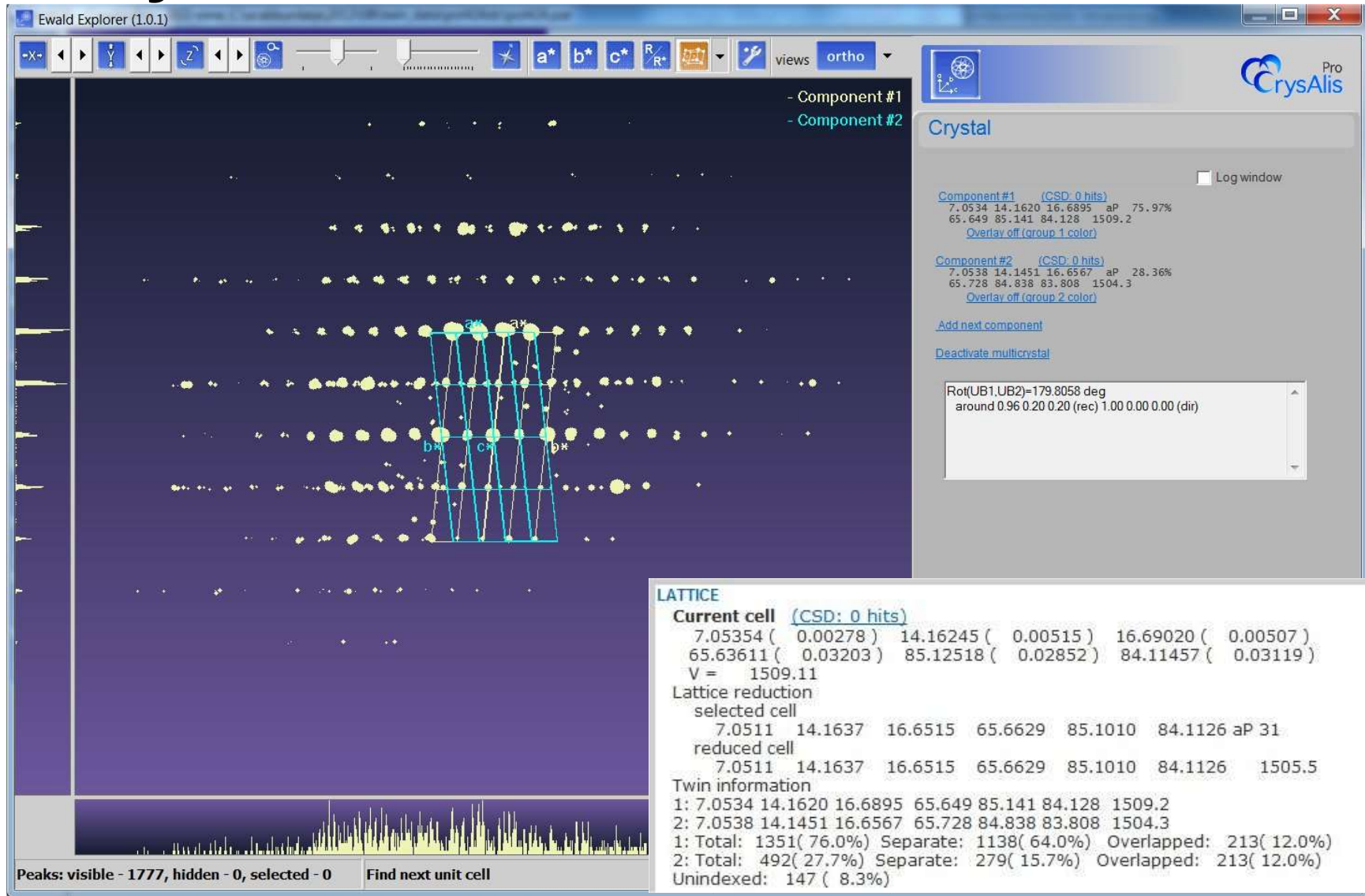
Filters

Selection/Lattice-It

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

X: 958 Y: 813

Easy twin in Ewald^{Pro}



Super lattice faker

Lattice wizard (1.0.32)

Lattice wizard

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

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

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

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

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

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

C14H12N3

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

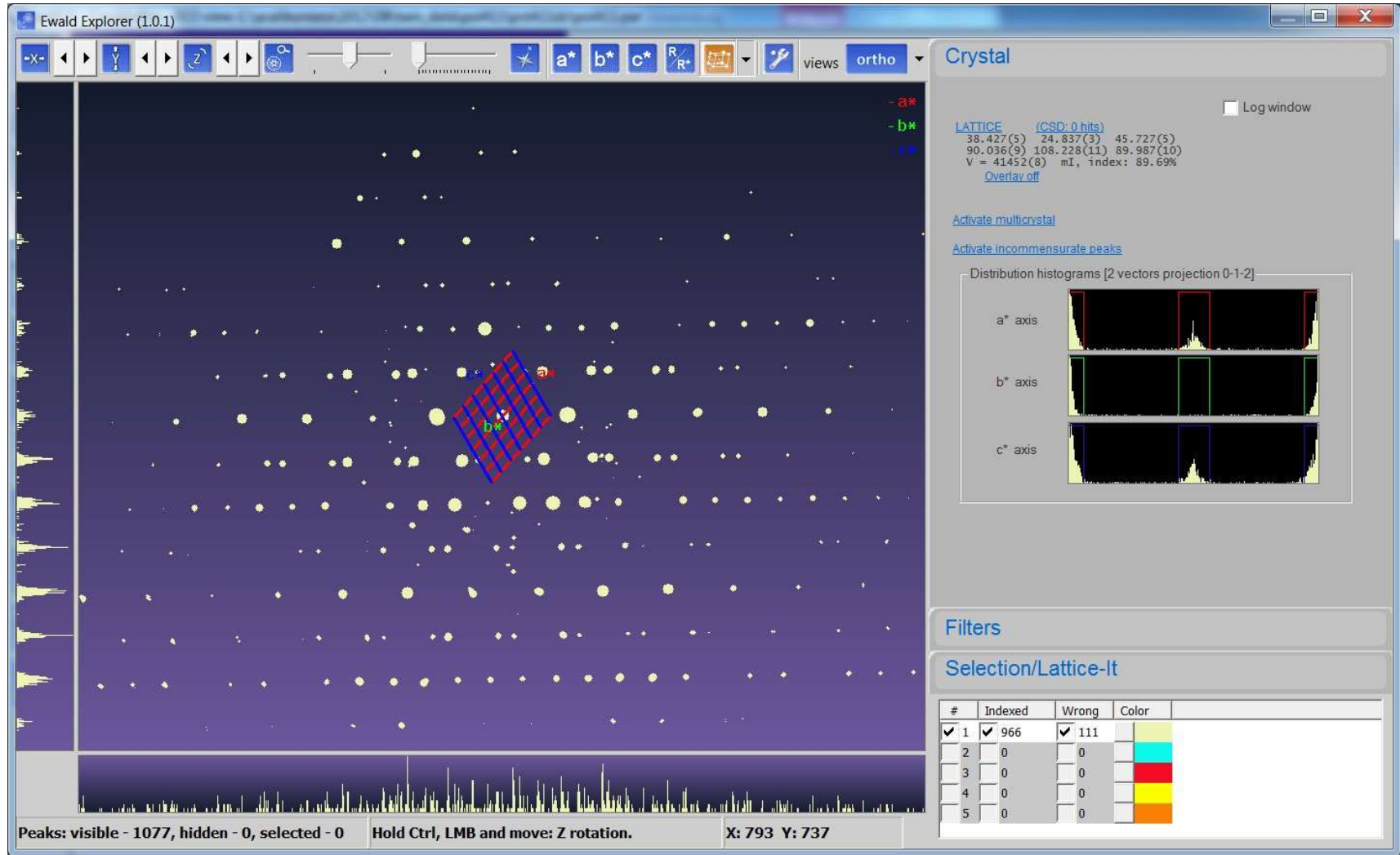
mu (mm-1) 0.07370 Edit mu

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

Log window

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

Super lattice faker in Ewald^{Pro}



Super lattice faker in Ewald^{Pro}

Crystal

Filters

Selection/Lattice-It

Select all Invert selection Deselect all

Selection tools

None Rubber band

Lattice-It find period, refine angle Hints

#	angle	period	width

New Remove

Angle: Period: Width:

Operations

Move selected to: Group2

Lattice-It usage:
 To modify period by 1/6: scroll mouse wheel or hit period scroll buttons.
 To adjust period by smaller factor (zoom related) hold SHIFT button and hit period scroll buttons.

#	Indexed	Wrong	Color
<input checked="" type="checkbox"/> 1	<input checked="" type="checkbox"/> 966	<input checked="" type="checkbox"/> 111	
<input type="checkbox"/> 2	<input type="checkbox"/> 0	<input type="checkbox"/> 0	
<input type="checkbox"/> 3	<input type="checkbox"/> 0	<input type="checkbox"/> 0	
<input type="checkbox"/> 4	<input type="checkbox"/> 0	<input type="checkbox"/> 0	
<input type="checkbox"/> 5	<input type="checkbox"/> 0	<input type="checkbox"/> 0	

Peaks: visible - 1077, hidden - 0, selected - 0 Hold Ctrl, LMB and move: Z rotation. X: 937 Y: 635

Super lattice faker in Ewald^{Pro}

The screenshot shows the Ewald Explorer (1.0.1) interface. The main window displays a diffraction pattern with overlaid lattice lines. The control panel on the right includes the following sections:

- Crystal**
- Filters**
- Selection/Lattice-It**
 - Buttons: Select all, Invert selection, Deselect all
 - Selection tools:
 - None
 - Rubber band
 - Lattice-It (find period, refine angle) Hints
 - Table:

#	angle	period	width
<input checked="" type="checkbox"/> 1	120.0	9.57 Ang.	12.5 %
 - Buttons: New, Remove
 - Angle: Period: Width:
- Operations**
 - Move selected to: Group2
- Lattice-It usage:**
 - To modify period by 1/6: scroll mouse wheel or hit period scroll buttons.
 - To adjust period by smaller factor (zoom related) hold SHIFT button and hit period scroll buttons.
- Table:**

#	Indexed	Wrong	Color
<input checked="" type="checkbox"/> 1	<input checked="" type="checkbox"/> 966	<input checked="" type="checkbox"/> 111	
<input type="checkbox"/> 2	<input type="checkbox"/> 0	<input type="checkbox"/> 0	
<input type="checkbox"/> 3	<input type="checkbox"/> 0	<input type="checkbox"/> 0	
<input type="checkbox"/> 4	<input type="checkbox"/> 0	<input type="checkbox"/> 0	
<input type="checkbox"/> 5	<input type="checkbox"/> 0	<input type="checkbox"/> 0	

Peaks: visible - 1077, hidden - 0, selected - 441 MWheel: Change period for active lattice. X: 555 Y: 361

Super lattice faker in Ewald^{Pro}

The screenshot shows the Ewald Explorer (1.0.1) software interface. The main window displays an Ewald sphere plot with a grid of peaks. A context menu is open over the plot, listing several options:

- Auto unit cell finding in shown peaks
- Refine unit cell
- Custom unit cell finding in shown peaks
- Lattice improvement with tolerance
- Modify lattice type
- Modify lattice type with user matrix

The right-hand panel shows the Crystal information section, including a table of lattice parameters:

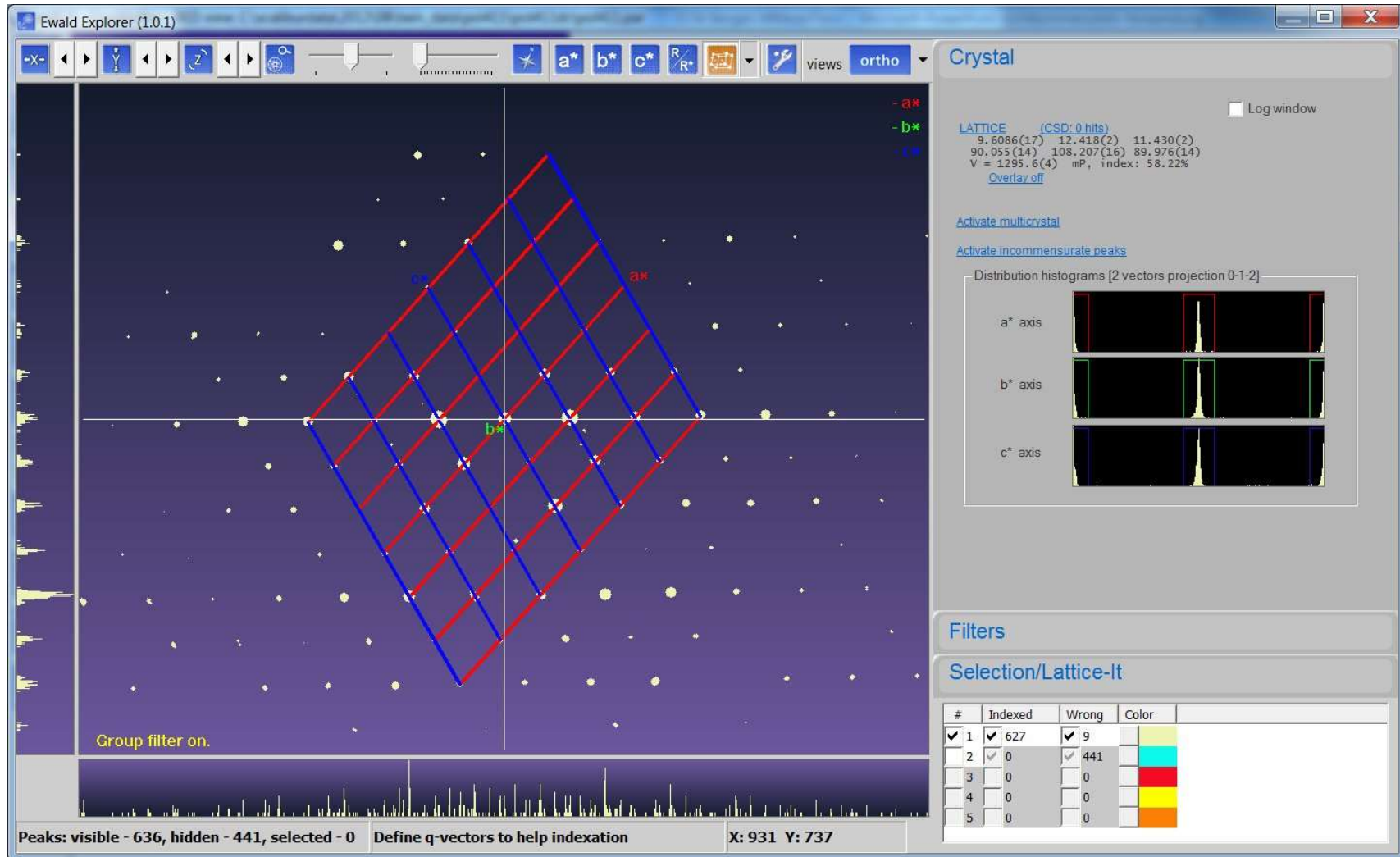
LATTICE	(CSD: 0 hits)
24.843(3)	49.670(6) 34.169(4)
89.985(9)	100.524(10) 89.973(9)
V = 41453(8)	mC, index: 88.77%

Below the Crystal information, there are sections for "Filters" and "Selection/Lattice-It". The "Selection/Lattice-It" section contains a table with the following data:

#	Indexed	Wrong	Color
1	582	54	Yellow
2	374	67	Cyan
3	0	0	Red
4	0	0	Yellow
5	0	0	Orange

At the bottom of the interface, there is a status bar with the following information: "Peaks: visible - 636, hidden - 441, selected - 0", "Find or change unit cell", and "X: 939 Y: 701".

Super lattice faker in Ewald^{Pro}



Super lattice faker in Ewald^{Pro}

Crystal

LATTICE (CSD: 0 hits)
 9.6086(17) 12.418(2) 11.430(2)
 90.055(14) 108.207(16) 89.976(14)
 V = 1295.6(4) mP, index: 58.22%
[Overlay off](#)

[Activate multocrystal](#)
[Activate incommensurate peaks](#)

Distribution histograms [2 vectors projection 0-1-2]

a* axis
 b* axis
 c* axis

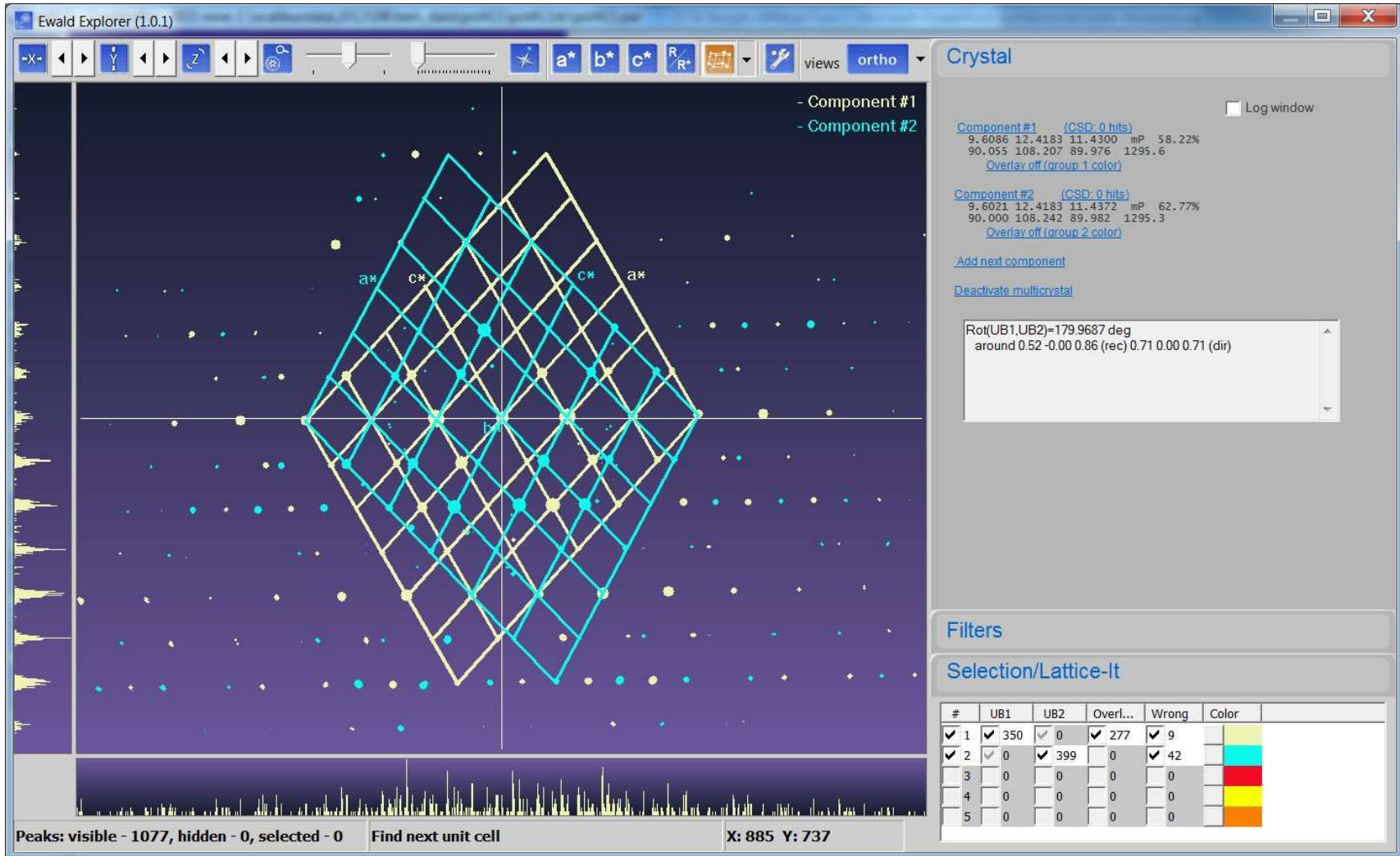
Filters

Selection/Lattice-It

#	Indexed	Wrong	Color
✓ 1	✓ 627	✓ 9	Yellow
2	0	✓ 441	Cyan
3	0	0	Red
4	0	0	Yellow
5	0	0	Orange

Peaks: visible - 636, hidden - 441, selected - 0 Find next unit cell X: 932 Y: 414

Super lattice faker in Ewald^{Pro}



Super lattice faker in Ewald^{Pro}

Cell and wavelength
 9.60859 12.41834 11.43003 90.055 108.207 89.976 1295.581
 Mo-radiation
 Z: 4.00

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

C14H12N3

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

mu (mm-1) 0.07039 Edit mu

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

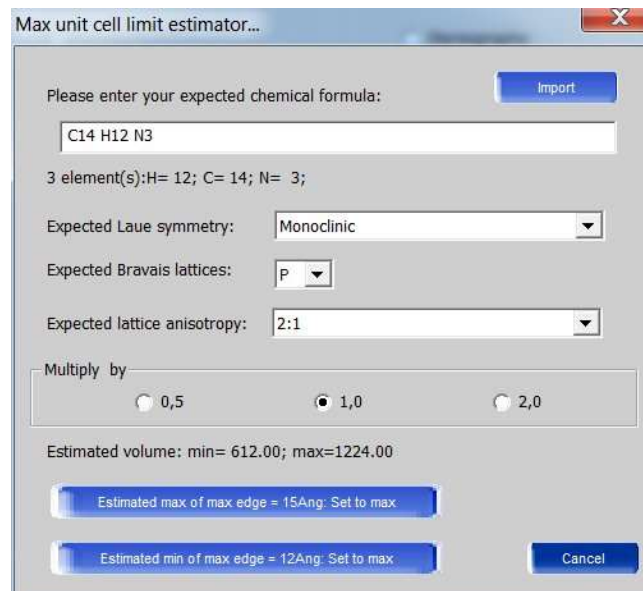
Set mu and formula Cancel

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%)

Peaks: visible - 1077, hidden - 0, selected - 0 Find next unit cell X: 8

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

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



Max unit cell limit estimator...

Please enter your expected chemical formula:

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

Expected Laue symmetry:

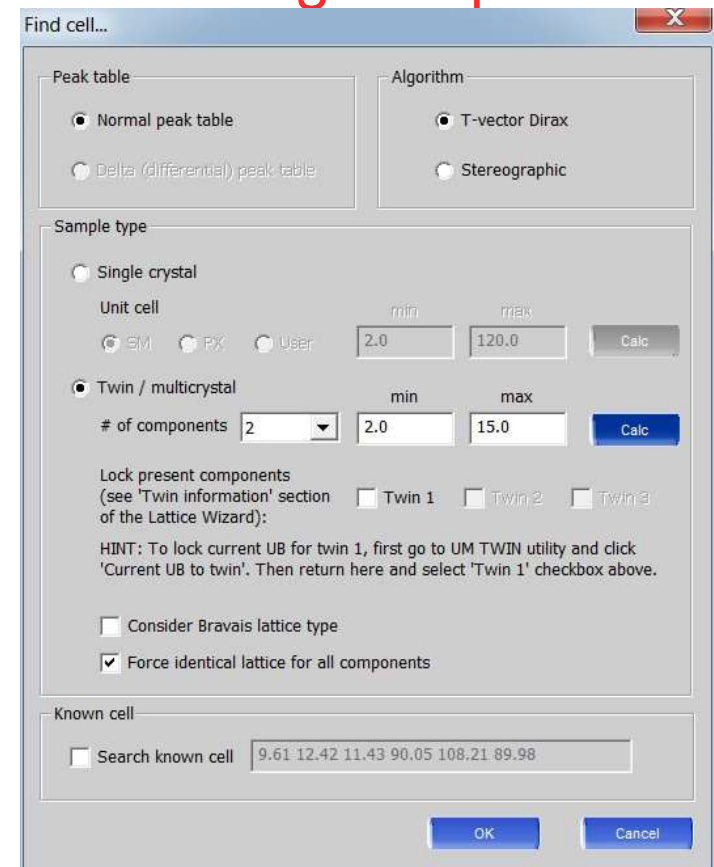
Expected Bravais lattices:

Expected lattice anisotropy:

Multiply by

0,5 1,0 2,0

Estimated volume: min= 612.00; max=1224.00



Find cell...

Peak table

Normal peak table

Delta (differential) peak table

Algorithm

T-vector Dirax

Stereographic

Sample type

Single crystal

Unit cell

BM PX User

min max

Twin / multycrystal

of components min max

Lock present components
(see 'Twin information' section of the Lattice Wizard):

Twin 1 Twin 2 Twin 3

HINT: To lock current UB for twin 1, first go to UM TWIN utility and click 'Current UB to twin'. Then return here and select 'Twin 1' checkbox above.

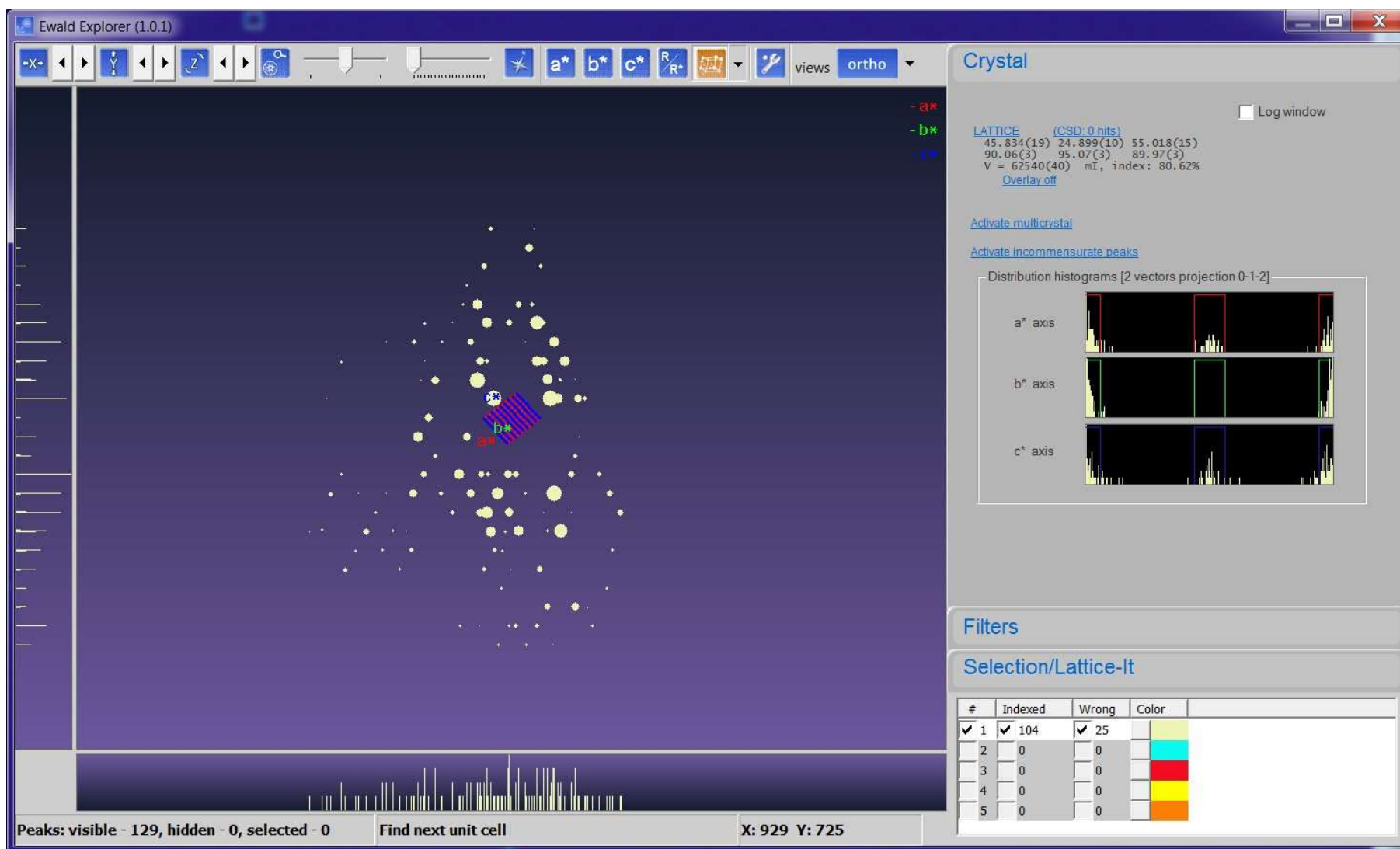
Consider Bravais lattice type

Force identical lattice for all components

Known cell

Search known cell

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



Use the custom unit cell tool...

Find cell...

Peak table
 Normal peak table
 Delta (differential) peak table

Algorithm
 T-vector Dirax
 Stereographic

Sample type
 Single crystal
 Unit cell: min 2.0 max 120.0 [Calc]
 Twin / multocrystal
 # of components: 2 min 2.0 max 15.0 [Calc]
 Lock present components (see 'Twin information' section of the Lattice Wizard):
 Twin 1 Twin 2 Twin 3
 HINT: To lock current UB for twin 1, first go to UM TWIN utility and click 'Current UB to twin'. Then return here and select 'Twin 1' checkbox above.
 Consider Bravais lattice type
 Force identical lattice for all components

Known cell
 Search known cell [9.61 12.42 11.43 90.05 108.21 89.98]

OK Cancel

Crystal

LATTICE (CSD: 0 hits)
 11.457(6) 12.434(6) 9.631(5)
 90.02(4) 108.30(5) 89.96(4)
 V = 1303(1) mC, index: 66.67%
[Overlay off](#)

[Activate multocrystal](#)
[Activate incommensurate peaks](#)

Distribution histograms [2 vectors projection 0-1-2]

a* axis
 b* axis
 c* axis

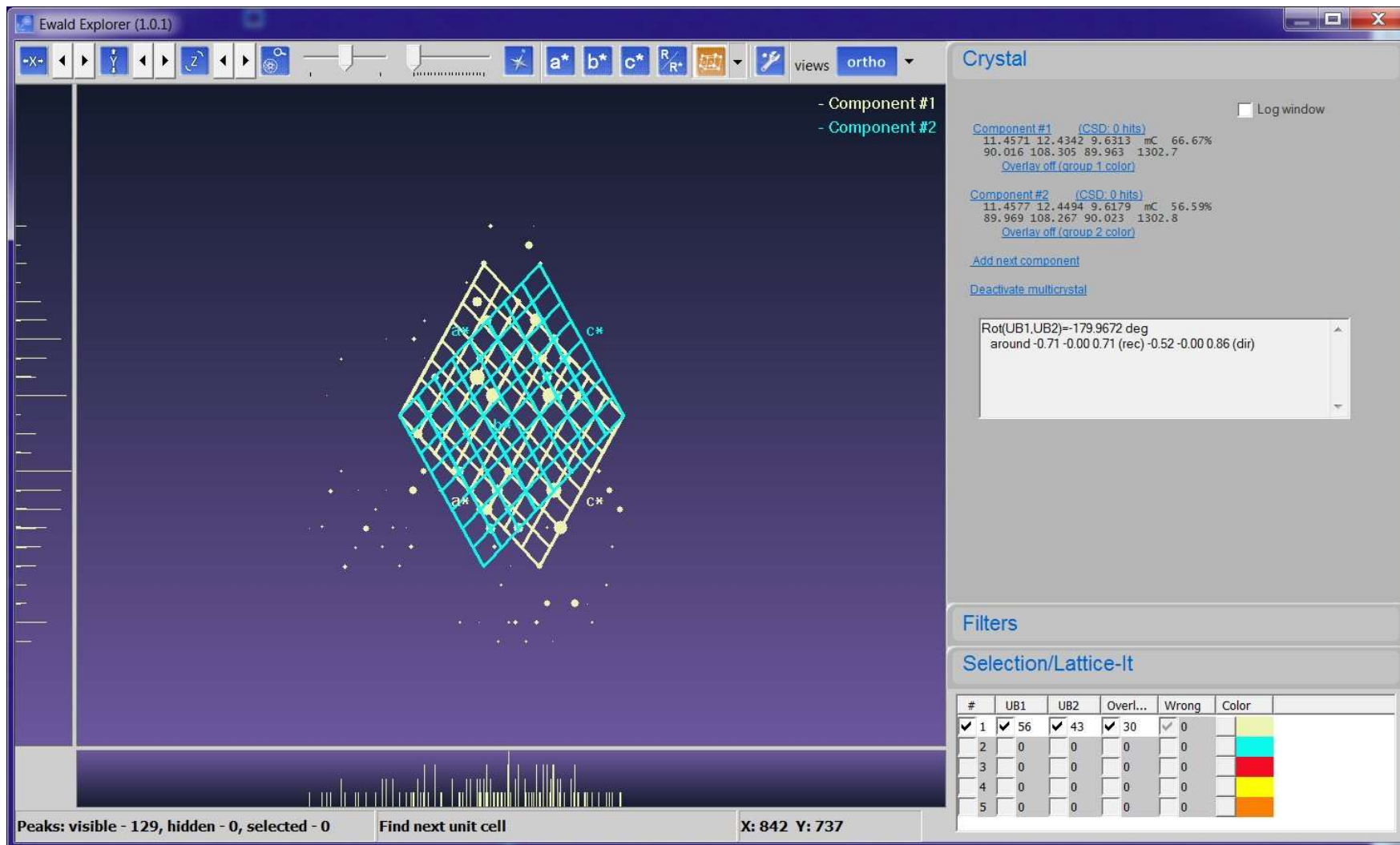
Filters

Selection/Lattice-It

#	Indexed	Wrong	Color
<input checked="" type="checkbox"/> 1	<input checked="" type="checkbox"/> 86	<input checked="" type="checkbox"/> 43	
<input type="checkbox"/> 2	<input type="checkbox"/> 0	<input type="checkbox"/> 0	
<input type="checkbox"/> 3	<input type="checkbox"/> 0	<input type="checkbox"/> 0	
<input type="checkbox"/> 4	<input type="checkbox"/> 0	<input type="checkbox"/> 0	
<input type="checkbox"/> 5	<input type="checkbox"/> 0	<input type="checkbox"/> 0	

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

Find the twin from the wrong peaks...



Strategy with known twin law

Experiment Strategy (1.1.8)

Experiment Strategy

Unit cell for Strategy Calculation (CSD: 0 hits)
 Cell: 11.430(2) 12.418(2) 9.6086(17) 90.024(14) 108.207(16) 89.945(14) 1295.6(4) mC C-lattice 58.22% (627 of 1077 reflections) Lattice Wizard

Strategy parameters
 Resolution Theta 2Theta 0.800
 Laue group Other 2/m (b-unique)
 Friedel mates are equivalent (uncheck for high quality absolute configuration data)
 Detector Distance 60.14 Advanced

Time prediction
 The same time for all theta positions
 Different time for each theta positions
 default time: 10.00
 your time: 10.00 [25.88, 27.05]
 Scan width: 1.00

Settings/Options

Strategy mode
 Complete data for twins
 limit 100.0 IUCr limit Max 99.74 %
 Generates runs that reach completeness limit for all twin components

Calculate New Strategy
 Aug 06 07:21:55 2012 Update Completeness Manually Edit Run List

iteness/Coverage tables
 s in 2/m (b-unique)

Help Save experiment Cancel

Data reduction

CrysAlisPro: Data reduction (1.13) ✕

Proffit: CrysAlisPro data reduction assistant (1.0.26) ✕

Load new experiment

Full auto analysis (cell, red)

Data reduction with options

Pro CrysAlis

Simultaneous twin data reduction

Orientation matrix for data reduction

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

V = 1302.30
Selected cell (from UM rr/UM ttt/UM f):
14  11.4572  12.4428  9.6208  89.9433  108.2831  89.9651  mC
Auto analyse found P-lattice in peak hunting data!
Twin 1: 11.45724 12.44279 9.62081 89.9433 108.2831 89.9651 1302.30
Twin 2: 11.45006 12.44351 9.62704 90.0025 108.2504 90.1021 1302.65
```

Lattice extinctions (filter Bravais lattice extinctions)

Don't use filter (P-lattice)

Use filter for: C-lattice

Incommensurate structures

Normal data reduction (HKL)

Single q-vector Edit q m=0

Other (reduction list) Generate Load

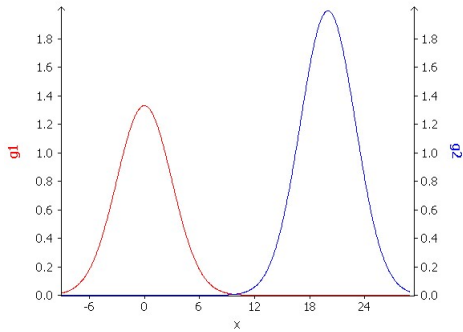
Twining/Multi crystal (activated by UM TWIN entries)

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

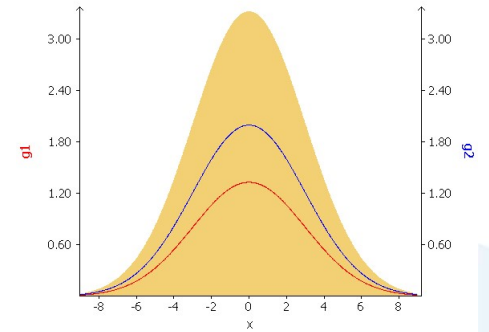
Component 1 Component 2 Component 3 Component 4

< Zurück Weiter > Fertig stellen Abbrechen Hilfe

Twin profile data

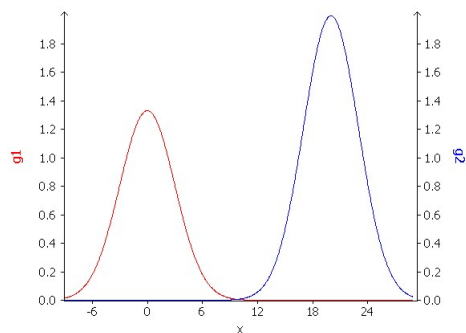


Fully separated

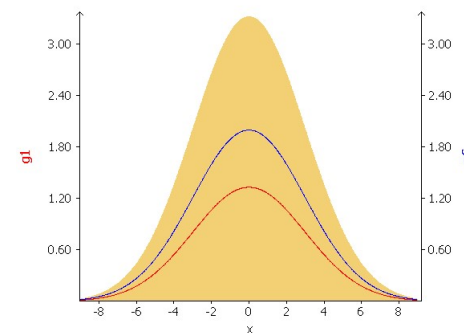


80% - 100%
Overlap data

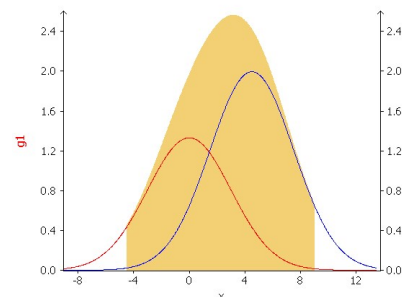
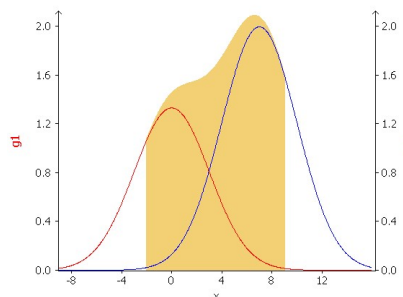
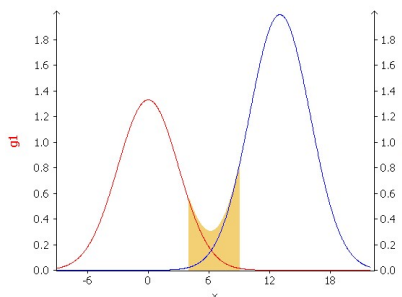
Twin profile data



Fully separated



80% - 100%
Overlap data



0%

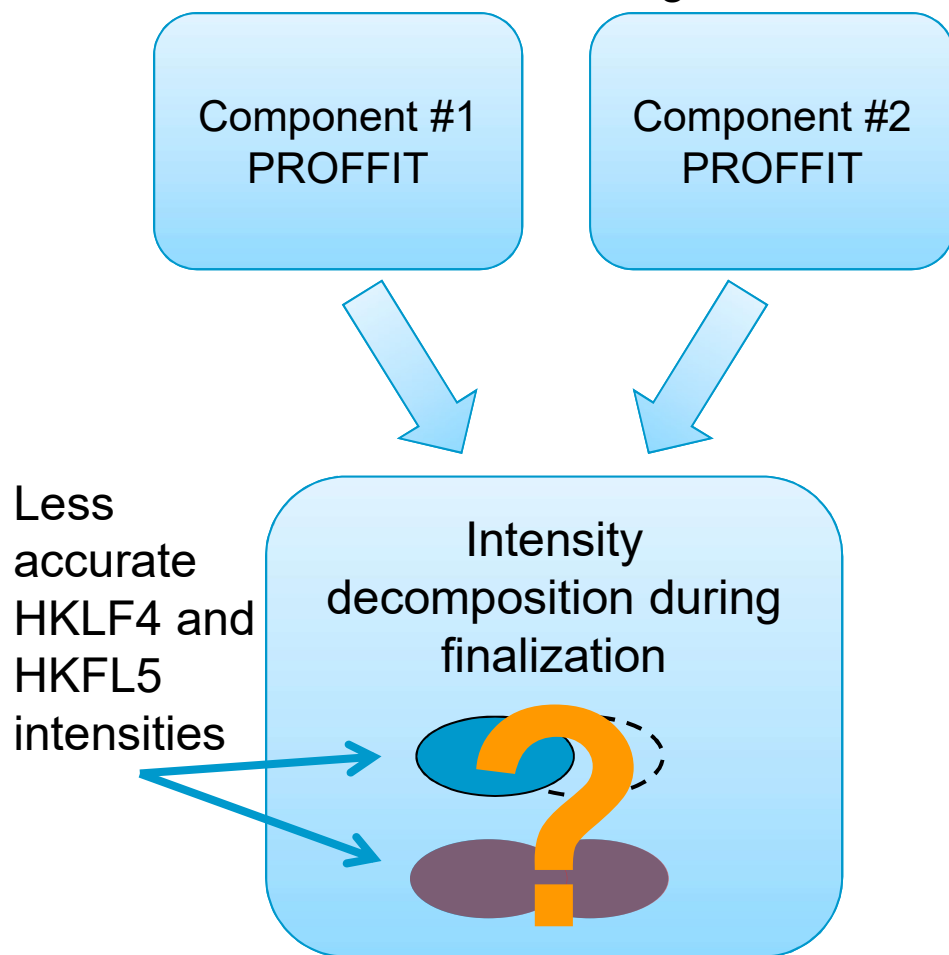
Deconvolution
possible

80%

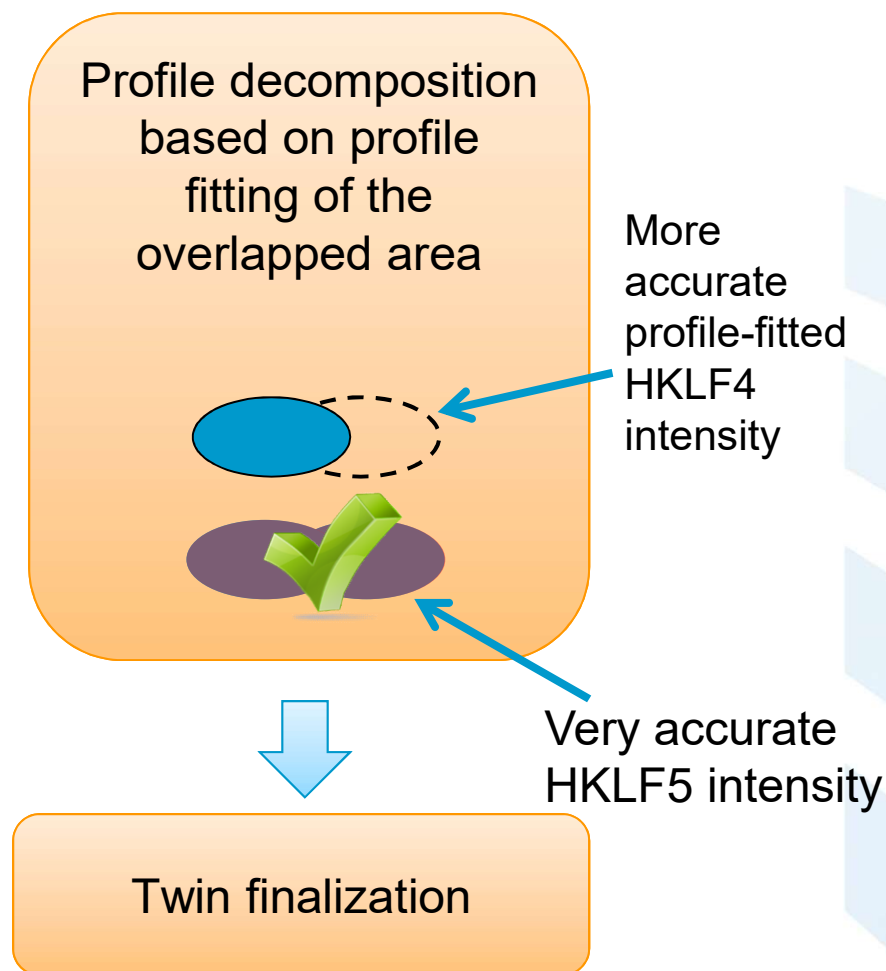
Pre-37 vs. new 37 approach



Pre-37 twin integration

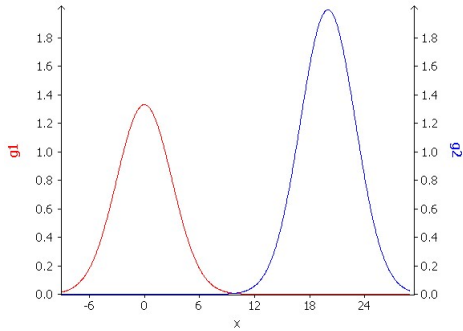


New simultaneous integration

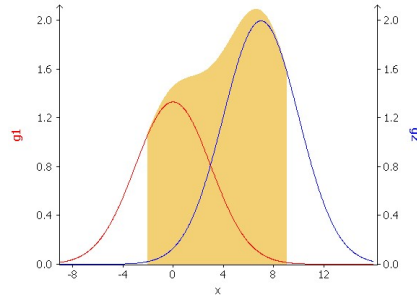


HKLF4 and HKLF5 play modes

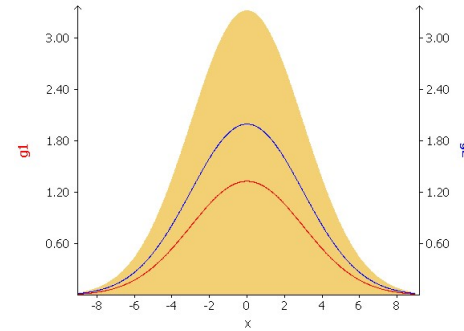
Fully separated



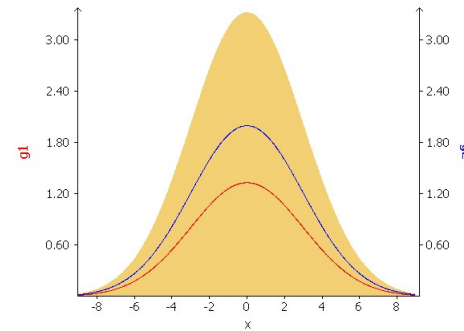
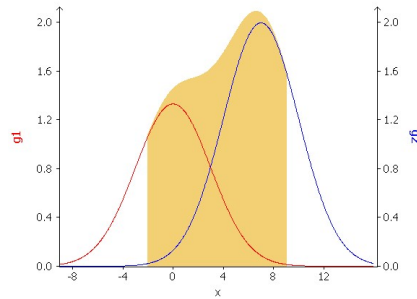
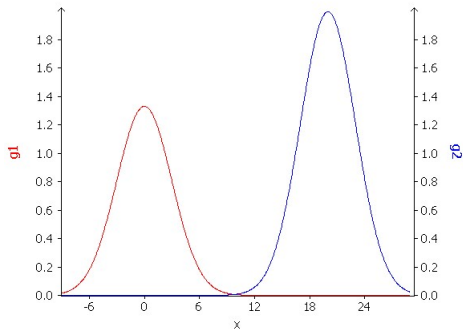
Partially overlapped



'Full' overlap



Component 1



Component 2

...

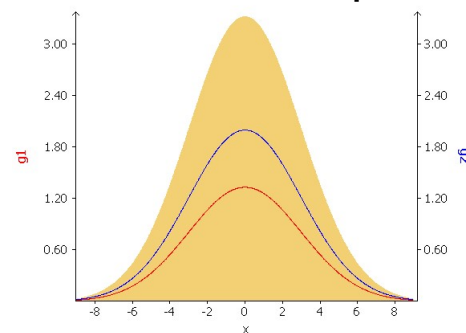
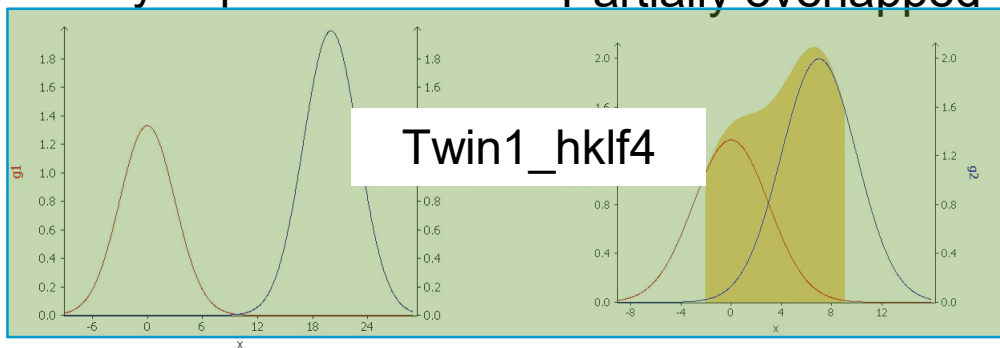
Component n

HKLF4 and HKLF5 play modes: 1st trial

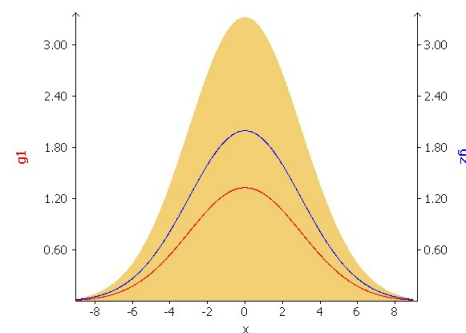
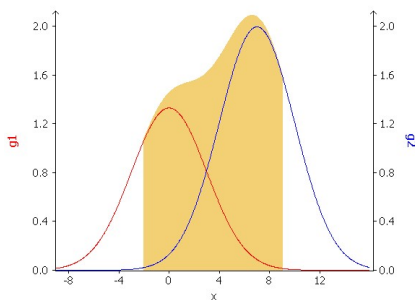
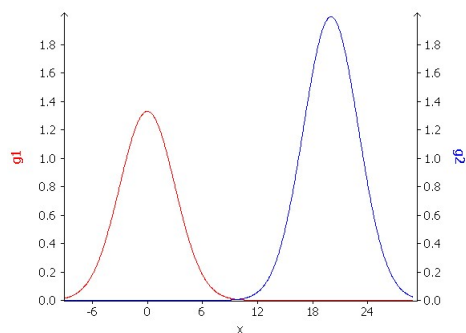
Fully separated

Partially overlapped

'Full' overlap



Component 1



Component 2

...

Component n

Structure solve and refine using AutoChem^{2.0}

The screenshot displays the Olex2 software interface. The central part of the window shows a 3D ball-and-stick model of a complex organic molecule with a central core and several side chains. The atoms are color-coded: carbon (grey), hydrogen (white), nitrogen (blue), and sulfur (yellow). The background is a dark blue gradient.

At the top right, the software title bar reads "Olex2". Below it, the menu bar includes "File", "View", "Structure", "Mode", "Tools", "Edit", "Model", "Select", and "Help".

On the right side, there is a panel titled "ania1" showing the following information:

- File path: C:/Data/2009/katowice/Oct2009/katowice/ania1/struct/olex2_ania1/ania1.res
- Chemical formula: $C_{26}H_{32}CuHgN_{12}S_4$
- Unit cell parameters: $a=9.2673(3)$; $b=25.0701(8)$; $c=14.7153(5)$ Å
- Angles: $\alpha=90^\circ$; $\beta=100.663(3)^\circ$; $\gamma=90^\circ$
- Statistics: Mean I/σ: 23.8; Rint: 4.08%; Completeness: 86.89%

Below this, there are tabs for "Work", "View", "Tools", and "Info". The "Autochem2" panel is active, showing the following details:

- Buttons: Run AutoChem, Revert, Refine Only, Solve Only, Report Only, STOP, Edit USER PHIL
- Solution Program: Auto; Solution Method: Auto
- Refinement Program: Auto; Refinement Method: Auto
- Log: ++++++ Starting AC2 ++++++ ania1
- SOLUTION STAGE: --> ShelXS, Patterson Method (0.64 s); VSS = 72; VATA = 36;
- REFINEMENT STAGE:
 - > Iteration 0 R1 = 22.33%; ATA = 14
 - > Iteration 1 R1 = 8.65%; ATA = 27
 - > Iteration 2 R1 = 7.15%; ATA = 50
 - > Iteration 3 R1 = 6.61%; ATA = 52
 - > Iteration 4 R1 = 6.33%; ATA = 52
- Continue? const(0.0) + rint(4.1)*3 + ios(23.8)*0 ==> 18.2 (6.3): Yes
- SECONDARY REFINEMENT STAGE:
 - Anisotropic Sensible? R1 = 4.84%; 0.95 YES
 - > Iteration f1 R1 = 4.51%; ATA = 59
 - > Iteration f2 R1 = 4.50%; ATA = 59
 - > Iteration f3 R1 = 4.01%;
- POLISHING STRUCTURE:
 - > Iteration f1 R1 = 4.01%;
 - Finished with ATA = 59 and R1 = 4.01 **** (21.2 s)

At the bottom left, there is a text box with the following information:

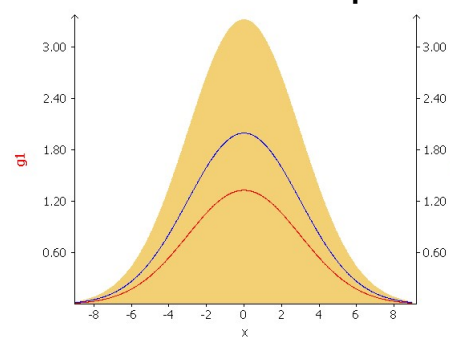
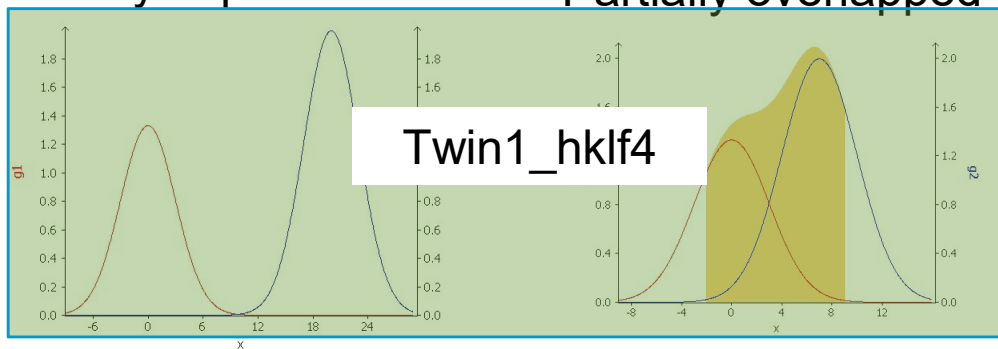
```
R1 = 0.0459 for 6358 unique reflections after merging for Fourt
Highest peak 2.75 at 0.4141 0.1483 0.7021 [ 0.98 Å from F1 ]
Deepest hole -1.33 at 0.3676 0.0921 0.7434 [ 0.88 Å from G1 ]
+++++
+ ania1 finished at 15:59:41 Total CPU time: 2.0 secs +
+++++
22.33 8.65 7.15 6.61 6.33 4.84 4.51 4.5 4.01 4.01 Refinement CIF file has been merged with the meta-data cif file
>>
```

Get extra data in to solve the structure...

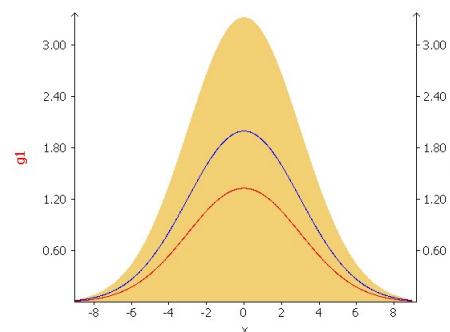
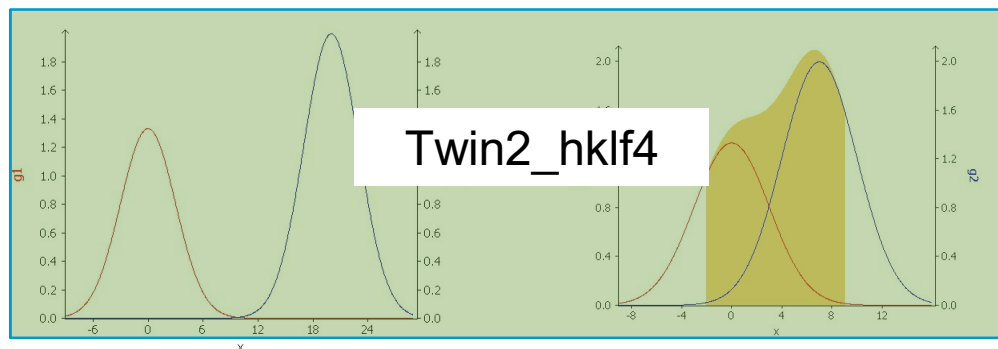
Fully separated

Partially overlapped

'Full' overlap



Component 1



Component 2

...

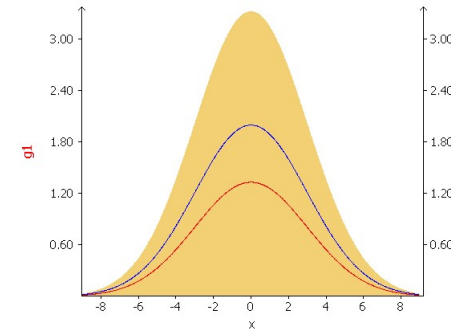
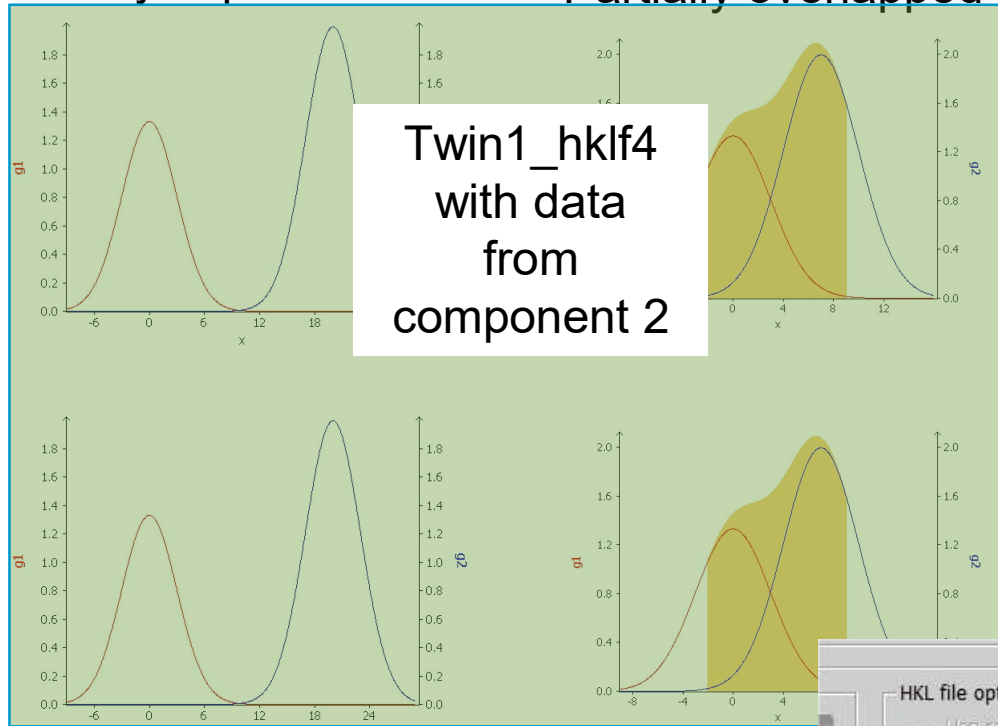
Component n

Get extra data in to solve the structure...

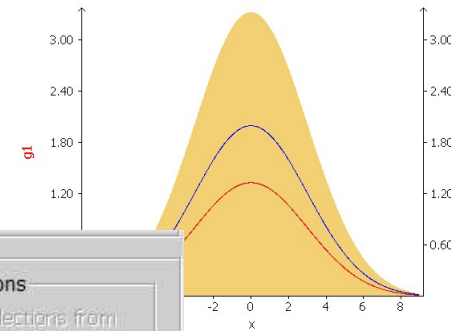
Fully separated

Partially overlapped

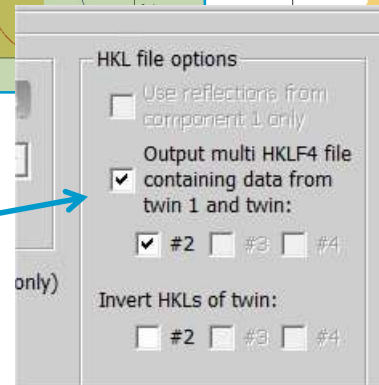
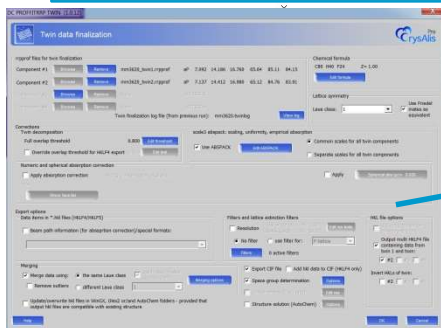
'Full' overlap



Component 1



Component 2



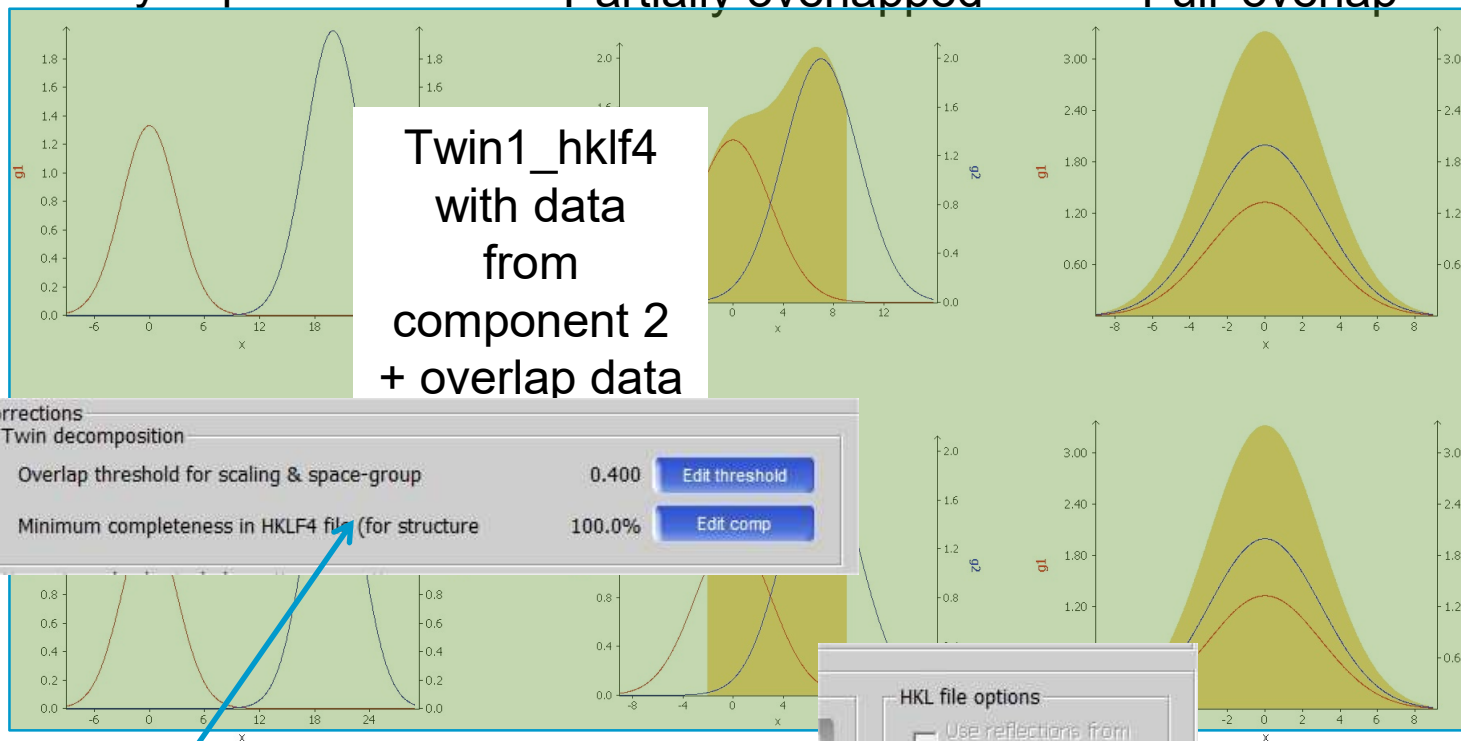
...
Component n

Not enough yet: Add full overlap data...

Fully separated

Partially overlapped

'Full' overlap



Component 1

Component 2

...
Component n

Corrections
Twin decomposition

Overlap threshold for scaling & space-group: 0.400 Edit threshold

Minimum completeness in HKLF4 file (for structure): 100.0% Edit comp

HKLF file options

Use reflections from component 1 only

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

#2 #3 #4

Invert HKLFs of twin:

#2 #3 #4

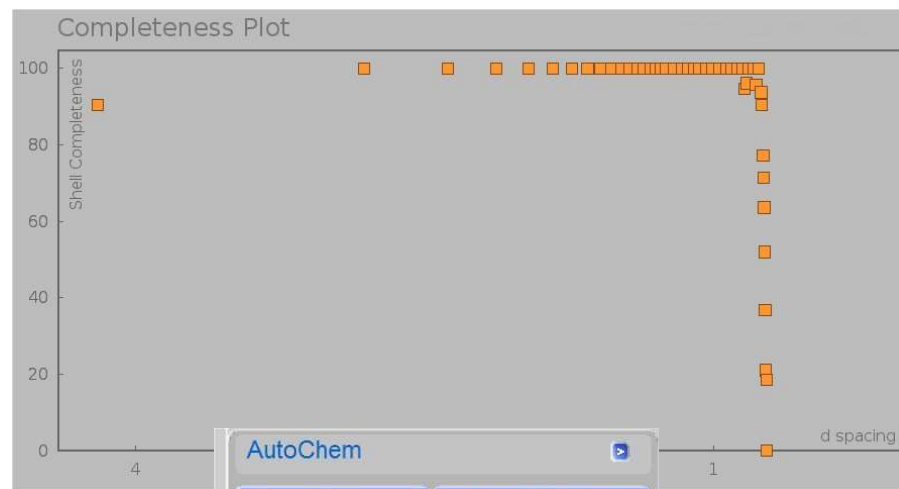
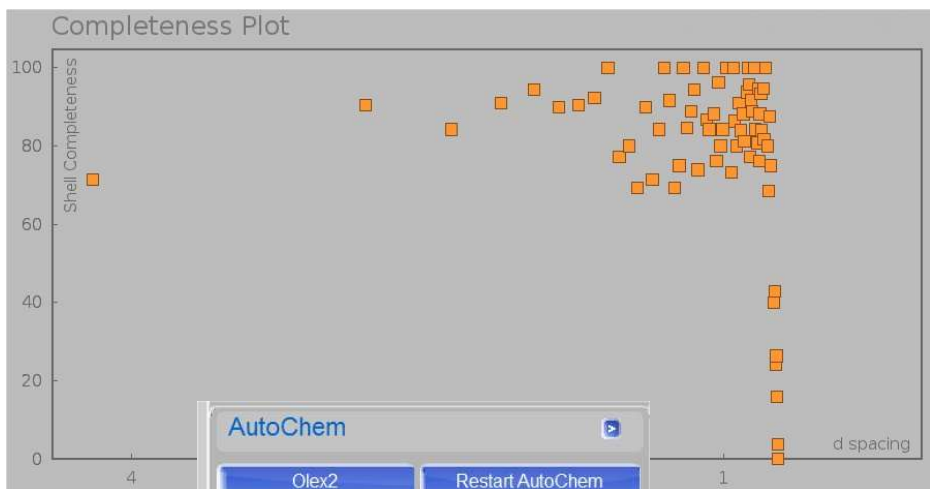
Twin data finalization

Component #1: #1 #2 #3 #4

Component #2: #1 #2 #3 #4

Output multi HKLF4 file containing data from twin 1 and twin: #2 #3 #4

Why?

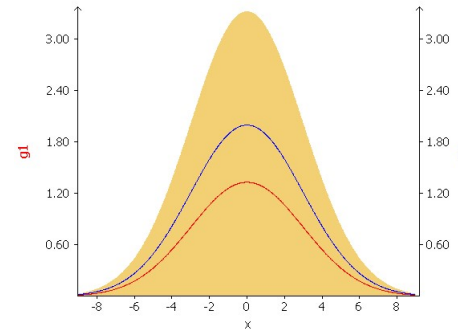
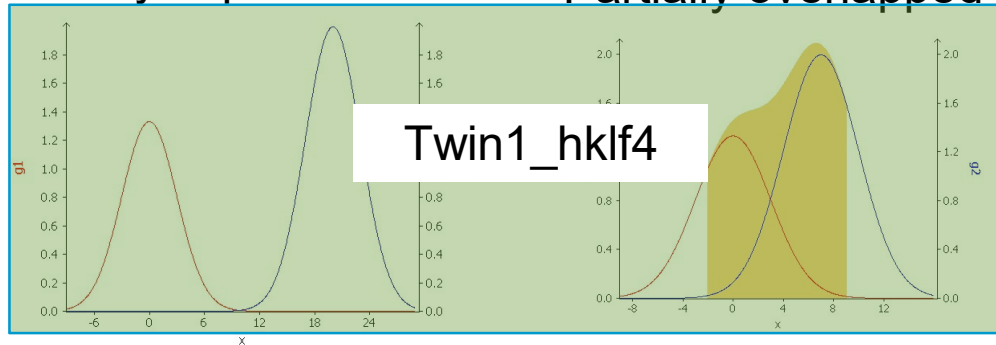


Solve done: Refine on good HKLF4

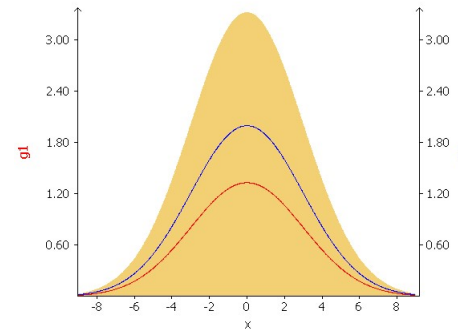
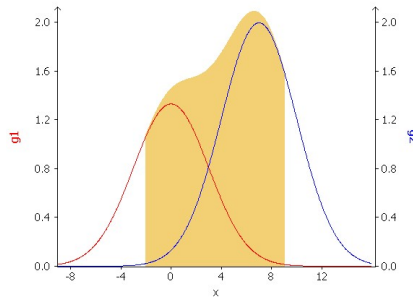
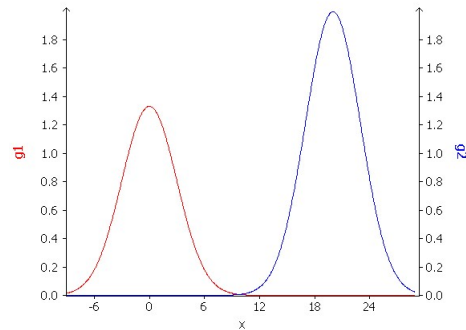
Fully separated

Partially overlapped

'Full' overlap



Component 1



Component 2

...

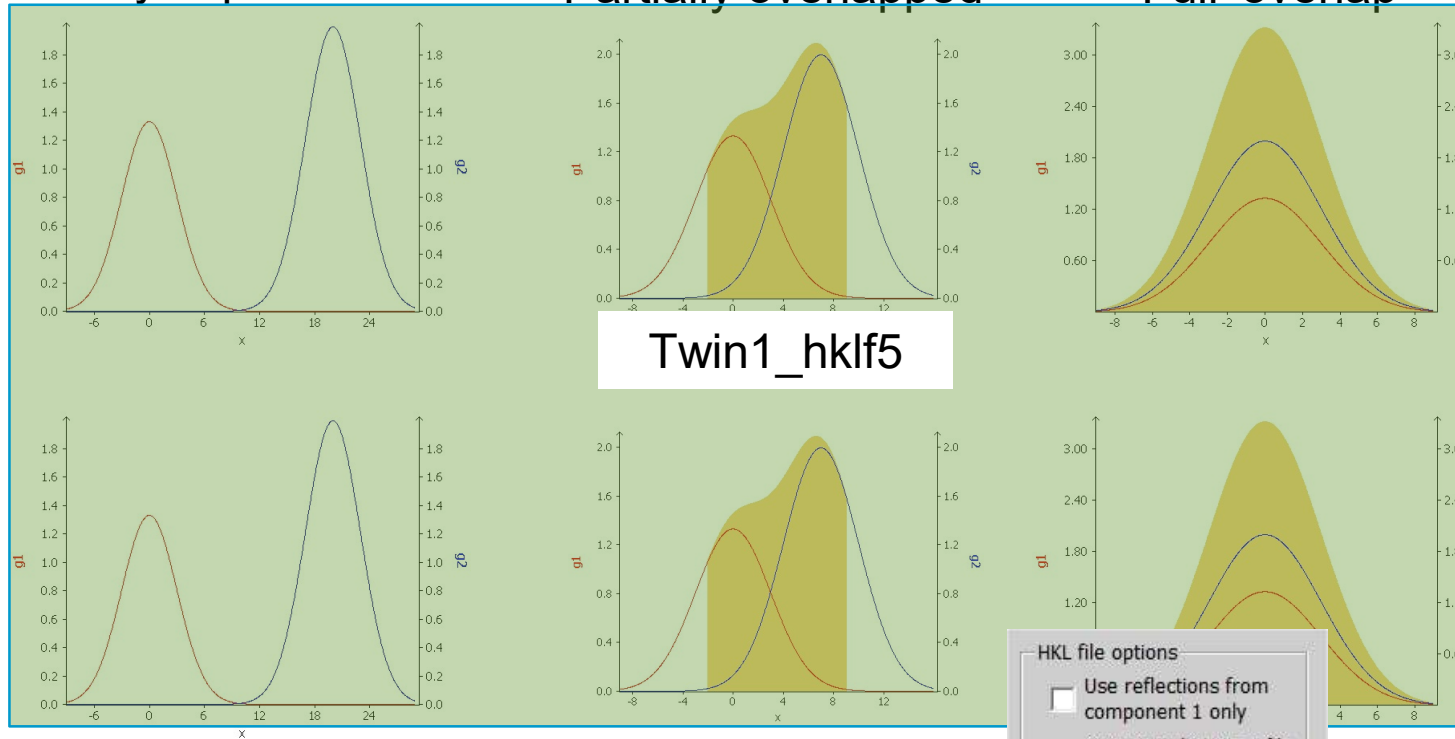
Component n

Solve done: Refine on good HKLF5

Fully separated

Partially overlapped

'Full' overlap



Component 1

Component 2

...
Component n

Merging

Merge data using: the same Laue class Use Friedel mates as equivalent Remove outliers different Laue class

1

Merging options

HKL file options

Use reflections from component 1 only

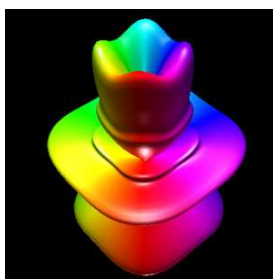
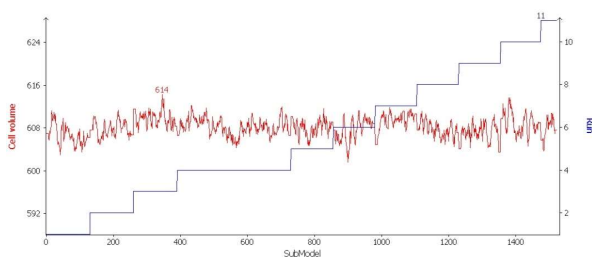
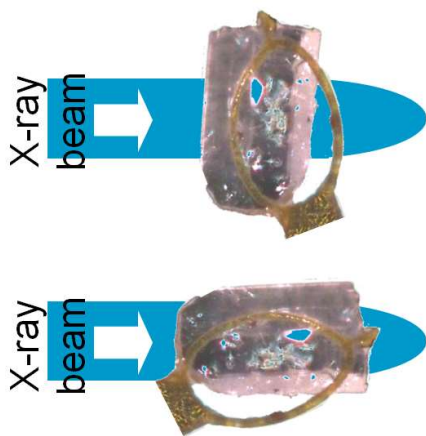
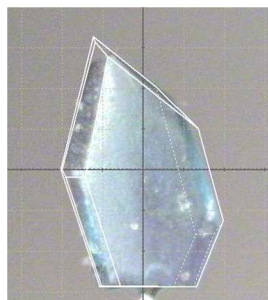
Output multi HKLF4 file containing data from twin 1 and twin:

#2 #3 #4

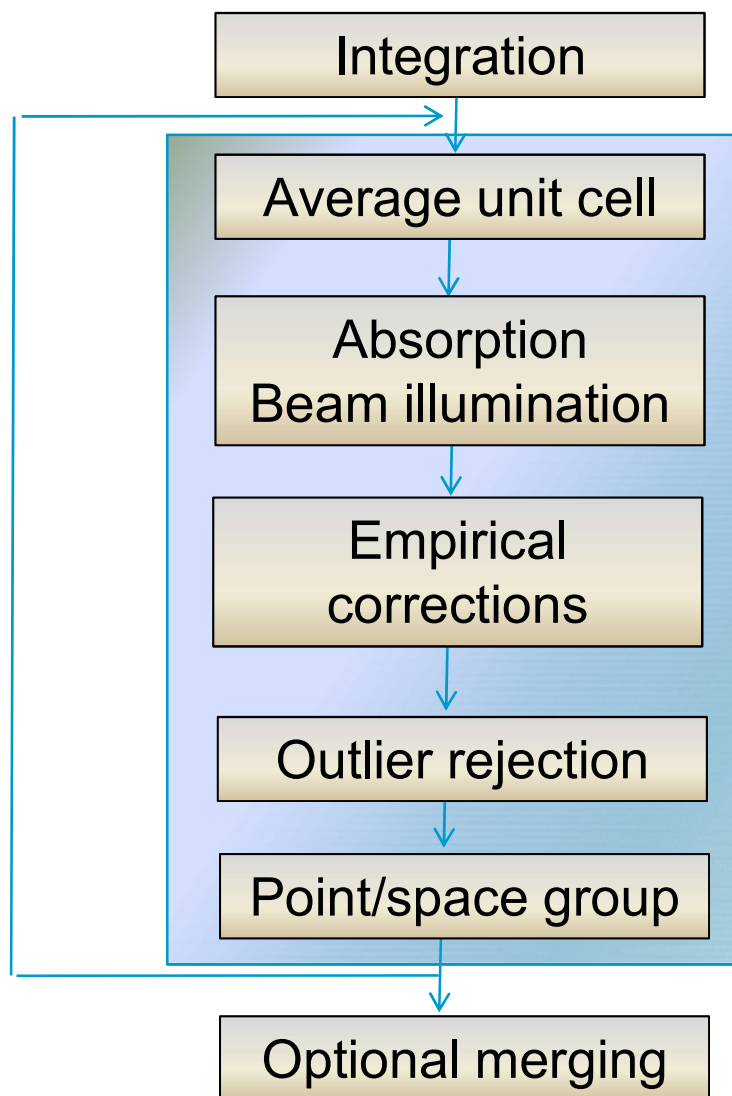
Invert HKLs of twin:

#2 #3 #4

Good data quality through full post corrections



PG changed?



Summary - twin

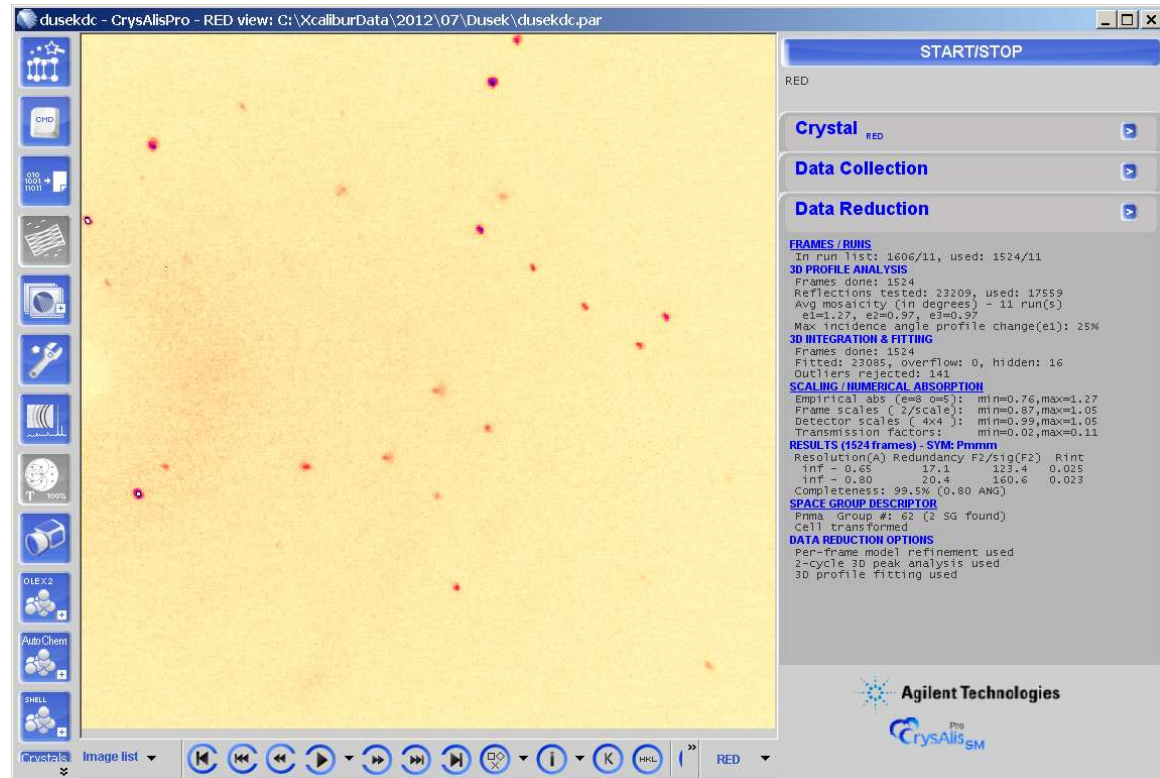
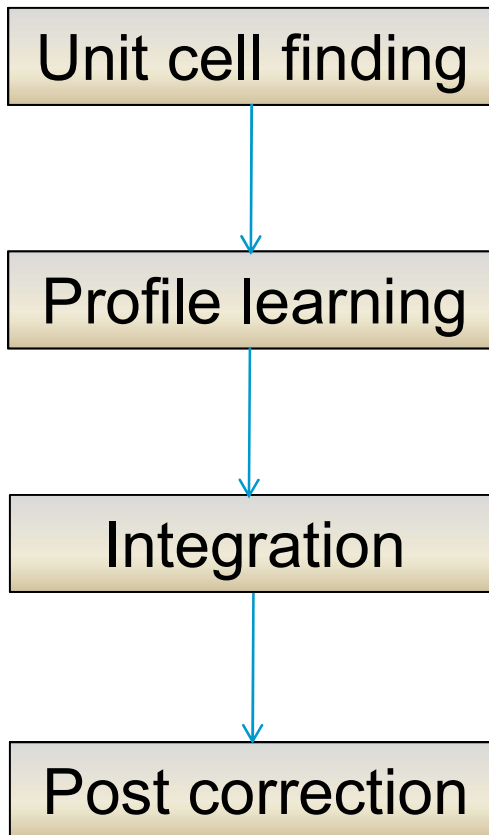
- Twins can be recognized early on during screening/pre-experiment
- With the graphic and computational tools in Ewald^{Pro} twin assignment is easy.
- De-convolution of overlap data gives good HKLF4 files.
- Sometimes solution boot-strapping requires different play modes
- HKLF4 and 5 files can be easily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

Step by step guide on easy twin and super lattice faker

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Typical data reduction sequence



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!