

Twinned Data Set Processing with CrysAlis^{Pro}



Indexing the diffraction pattern in the Lattice Wizard

1. Open the Lattice Wizard from its icon



at the top of the Power Tools panels on the left.



2. Like for a single component data set, run (1) Peak hunting, (2) Unit cell finding and (3) Refine.



**Lattice wizard**



LATTICE
Current cell (CSD: install)
7.0930 7.0930 7.0930 90.0 90.0 90.0 356.8534
Lattice reduction
No reduced unit cell present!

PEAK TABLE
User-loaded table
0 obs out of 0 (total:0,skipped:0)

INSTRUMENT MODEL
Goniometer
beam: 0.00000 alpha: 90.00000 beta: 0.00000
om zero: 90.00000 th zero: 0.00000 ka zero: 0.00000

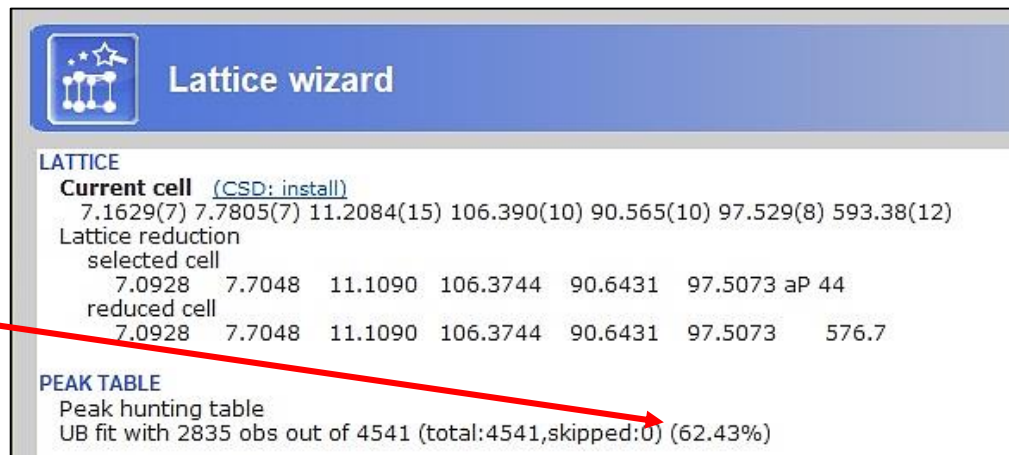
 Peak hunting  Unit cell finding

 Ewald explorer - reciprocal space  Reindexation with current cell

 Refine instrument model  Lattice transformation

Indexing the diffraction pattern in the Lattice Wizard

3. The Lattice Wizard shows:
- The unit cell parameters.
 - The number and % reflections indexed. Only 62.43% reflections are indexed in this example.



Lattice wizard

LATTICE

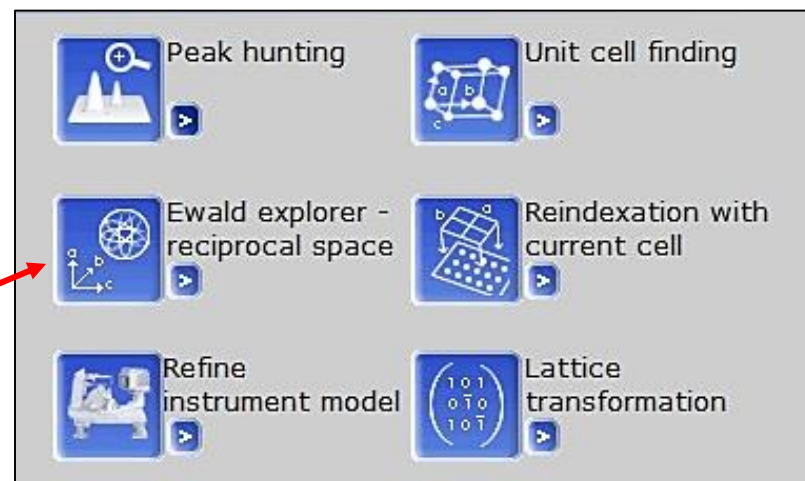
Current cell ([CSD: install](#))
7.1629(7) 7.7805(7) 11.2084(15) 106.390(10) 90.565(10) 97.529(8) 593.38(12)

Lattice reduction
selected cell
7.0928 7.7048 11.1090 106.3744 90.6431 97.5073 aP 44
reduced cell
7.0928 7.7048 11.1090 106.3744 90.6431 97.5073 576.7

PEAK TABLE
Peak hunting table
UB fit with 2835 obs out of 4541 (total:4541,skipped:0) (62.43%)

A red arrow points from the text "Only 62.43% reflections are indexed in this example." to the "(62.43%)" value in the Peak TABLE section.

4. If % reflections indexed < 90%, there may be a significant, ordered 2nd component present in the data as a twin component. To check for the presence of a twin, open 'Ewald explorer – reciprocal space' from the Lattice Wizard.



The interface displays a grid of icons for various functions:

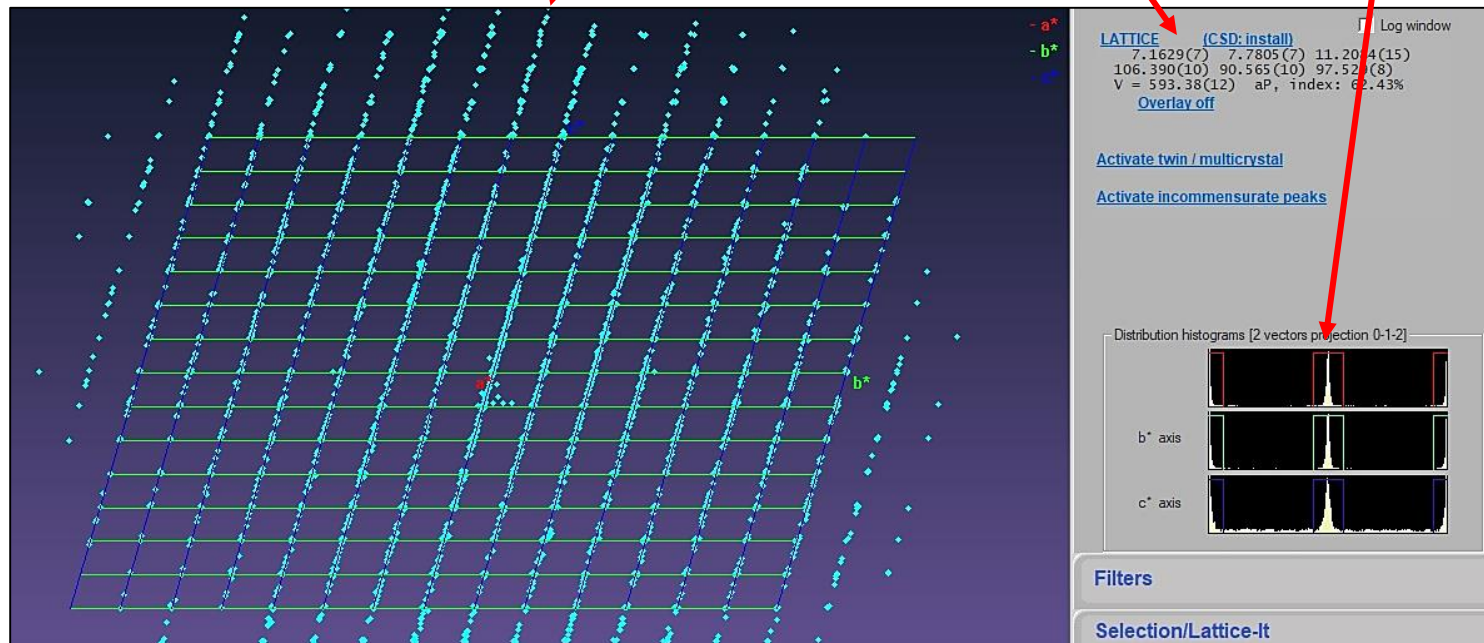
- Peak hunting
- Unit cell finding
- Ewald explorer - reciprocal space
- Reindexation with current cell
- Refine instrument model
- Lattice transformation

A red arrow points from the text "open 'Ewald explorer – reciprocal space' from the Lattice Wizard." to the 'Ewald explorer - reciprocal space' icon.

Indexing the diffraction pattern in the Lattice Wizard

5. The reciprocal space interface shows:

- The unit cell parameters and % reflections indexed.
- The predictions (red, green and blue boxes) and reflections for the current component match each other along a^* , b^* and c^* , indicating the unit cell parameters are correct.
- Lots of reflections are present in between lattice points along c^* and are thus unindexed.



Indexing the diffraction pattern in the Lattice Wizard

6. To activate twin indexing in 'Ewald explorer', click 'Activate twin / multicrystal' and select 'Automatic twin finding'.
'Automatic twin finding' should handle most twin situations.

The screenshot displays the Lattice Wizard software interface. The main window shows a 2D diffraction pattern with a grid of peaks. A red arrow points from the text 'Automatic twin finding' to the corresponding menu item in the 'Activate twin / multicrystal' dropdown. Another red arrow points from the text 'Activate twin / multicrystal' to the corresponding button in the Lattice Wizard panel.

Lattice Wizard Panel:

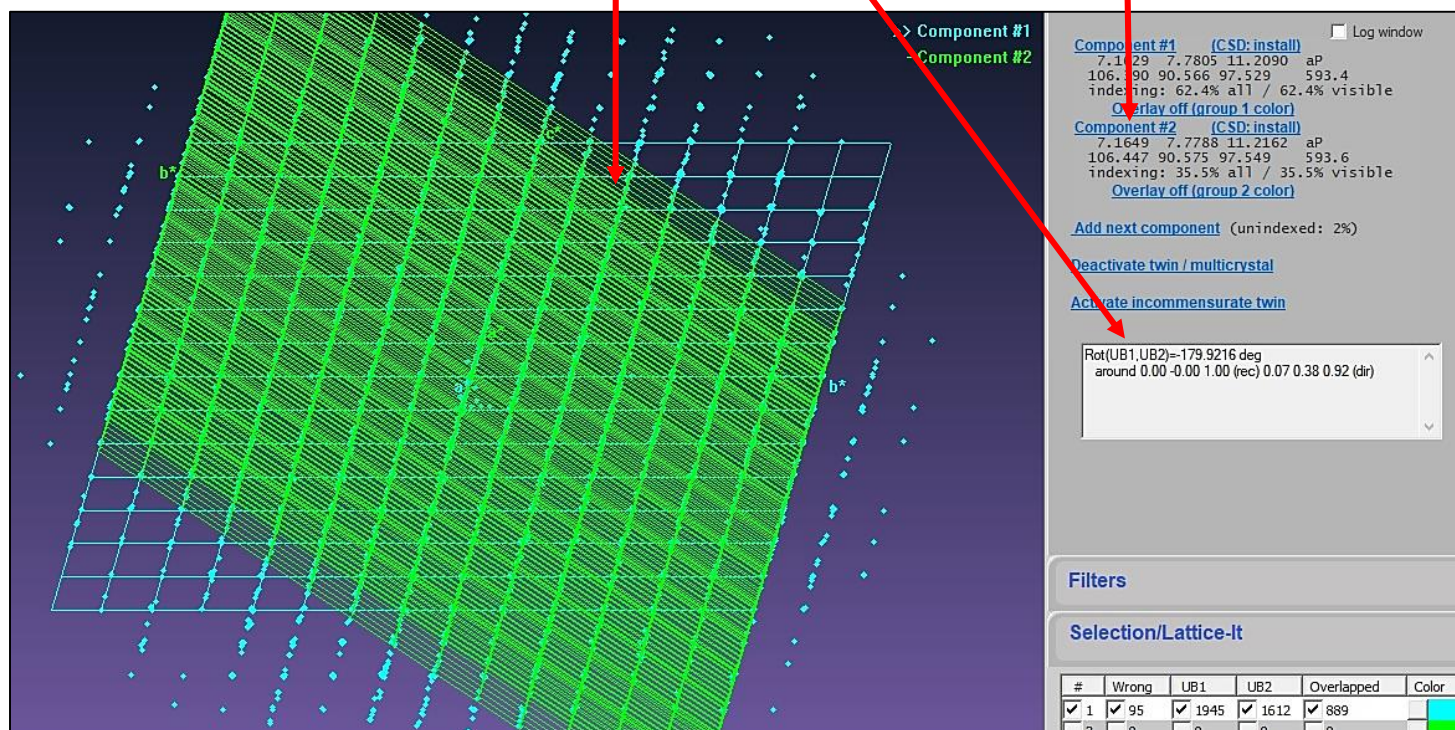
- LATTICE (CSD: install)**
7.1625(7) 7.7805(7) 11.2084(15)
106.390(10) 90.565(10) 97.529(8)
V = 593.38(12) aP, index: 62.43%
[Overlay off](#)
- [Activate twin / multicrystal](#)
- [Activate incommensurate peaks](#)
- Distribution histograms [2 vectors projection 0-1-2]**
 - a* axis
 - b* axis
 - c* axis
- Filters**
- Selection/Lattice-It**

#	Indexed	Wrong	Color
✓ 1	✓ 2835	✓ 1706	
2	0	0	

Indexing the diffraction pattern in the Lattice Wizard

7. The reciprocal space interface now shows:

- The unit cell parameters and % reflections indexed for the 2nd component.
- The twin law (rotation angle and rotation axis) between the two components.
- The lattice for the 2nd component in green.



Indexing the diffraction pattern in the Lattice Wizard

8. A successful twin indexing should show:

- 10 % or more reflections indexed in the 2nd unit cell.

If less than 10 % reflections are indexed, the crystal may still be twinned but better processing statistics may be obtained by ignoring the small contribution from the 2nd component. To check this, processing should be performed twice, once by including the 2nd component and once by ignoring it. Then check for best R_{int} and $\langle I/\sigma(I) \rangle$.

- A twin law that makes sense:
 - The rotation angle is related to a known symmetry, such as 180° for an inversion center or 90° for a 4-fold rotation
 - Usually, the rotation axis coincides with a well-defined direction, such as (0 0 1).
 - If these two sets of parameters show random numbers, the crystal is likely to be cracked and the 2nd piece of crystal has a random orientation relative to the main component.

9. Close 'Ewald explorer' by pushing on the cross at the top of the interface

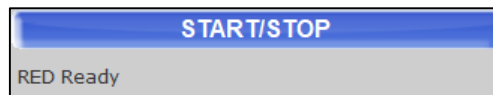


Then close the Lattice Wizard.

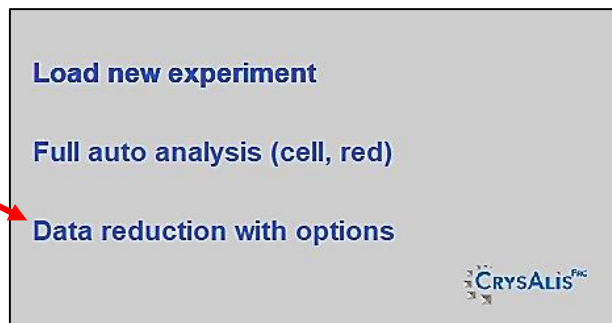
Data Processing

Data Reduction

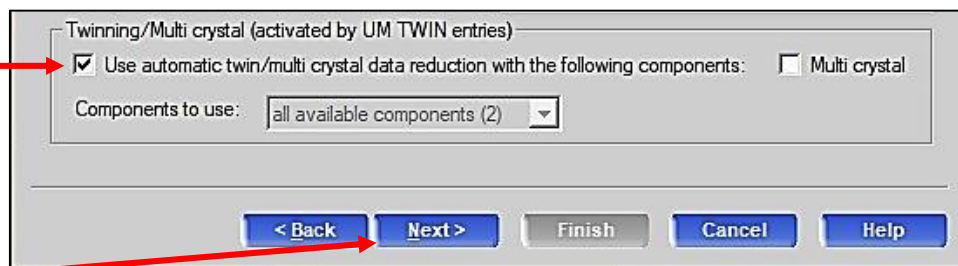
1. Click on START/STOP



2. Select 'Data reduction with options'.



3. In 'Step 1', the box enabling data reduction for twins should be automatically checked.



4. Click on 'Next'.

Step 2. Image range selection

Bad images (icing, scattering from crystal holder, ...) at the beginning or the end of a scan may be removed from reduction using this feature.

Simultaneous twin data reduction

CRYSLIS^{PRO}

Step 2: Experiment run list for data reduction

Run list: *.dtrek

Image dir: C:\C\Data\Single_Crystal\Training-Agendas\Examples_Data\Twin_TTF

#	type	start	end	width	exposure	omega	detector	kappa	phi	start	end
1	o	-120.00	60.00	1.00	35.00	-	-29.80	54.00	120.00	1	180
2	o	-120.00	60.00	1.00	35.00	-	-29.80	54.00	240.00	1	180
3	o	-120.00	60.00	1.00	35.00	-	-29.80	54.00	0.00	1	180

By default the whole experiment will be evaluated. To modify this behaviour edit the run list -->

Edit start num of selected run

Edit end num of selected run

< Back Next > Finish Cancel Help

1. Select the desired scan.
2. If bad frames at the beginning of the scan, click here and enter the number of the 1st good frame to integrate.
3. If bad frames at the end of the scan, click here and enter the number of the last good frame to integrate.
Note: entering 'zero' in 2. or 3. above will remove the entire scan from reduction.
4. Click 'Next'.

Step 3. Special Parameters

Simultaneous twin data reduction

CRYSA LIS^{Pro}

Step 3: Basic algorithm parameters

Reflection position prediction ☐ Skip model refinement ☐

☐ Auto select optimal prediction approach on run basis

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

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

☐ Follow sudden (discontinuous) changes of sample orientation

Orientation search range (max 10 deg) 2.00 Search steps/deg (max 10) 4

Edit special pars

Data from previous run of 'do profit'
3d profile information and/or integration results on
the disk

Clear data from previous run

Scratch all tmp data of previous profit run
Empty tmp folder from all analysis files including
background

Clear all data from tmp

< Back Next > Finish Cancel Help

1. Click here to modify special parameters (next slide).

2. Click on these to delete all info from previous processing runs and start afresh.

3. Click Next

Special Parameters Options

1. To enter a resolution cutoff, check this box and select 'Edit limits'

2. To reject the worst shaped reflections or discard ice rings, check this box.

The screenshot shows a software dialog box titled 'Special Parameters Options' with several sections and checkboxes:

- 3D intensity integration**
 - ☐ 2D profile fitting (recommended only for very strong diffraction data)
 - ☒ 3D profile fitting (improves weaker data, default option)
- Reflection positioning and integration**
 - ☐ Single wavelength only (recommended exclusively for data up to 1.5 Ang, i.e. large molecules)
 - ☐ HKL check in 3D peak analysis (recommended when reflections are very close to each other)
- Extra corrections**
 - ☐ Apply inverse float correction (f.ex. undo flood field correction)
 - ☐ Apply float correction (f.ex. additional flood field correction)
 - ☐ Apply pixelwise absorption correction (prepared by DC ABSTORJUN)
 - ☐ Apply monitor renormalization ☐ Use file for monitor values
- Skip filters**
 - Lorentz min = 0.0500
 - ☐ HP cell opening reject 0.00
 - ☐ Use resolution limits
 - ☐ Reject reflections with bad profiles (e.g. for HP data)
 - d-value (Ang): Inf- 0.68
 - 2theta (deg): 0.00- 68.24
 - &
- Extinction rules**
 - No extinction rules specified
 - HINT: You can use DC EXTINGT to add extinction rules and DC CLEAREXTINGT to remove selected or all rules from the list
- DC JETSHADOW (to visualize beforehand use 'beamstop mask')**
 - ☐ Use JetShadow
 - alpha: 30.00, beta: 0.00, jet_width: 13.00, jet_distance: 6.00
- Profile fitting**
 - Override integration mask size (generally not recommended, but smaller mask can be useful for strongly overlapping reflections e.g. twins) ☐ 1.00 of original size
 - ☐ Follow profile size changes with incidence angle
 - ☐ Adjust masks according to prediction uncertainty (for high angle data)
 - ☐ Print average profiles to history window
 - ☐ Consider reflection spot drift along diffraction vector (e.g. due to high mosaicity and/or beam divergence)

At the bottom right are and buttons.

Red arrows from the text blocks point to the following elements in the dialog:

- Arrow 1 points to the '3D profile fitting' radio button.
- Arrow 2 points to the 'Reject reflections with bad profiles' checkbox.
- Arrow 3 points to the 'Use JetShadow' checkbox.
- Arrow 4 points to the 'OK' button.

3. Check this box so the detector area covered by the cryo-nozzle shadow is removed from reduction. This must be done if Cu radiation was used for data collection

4. Click OK.

Step 4. Background Subtraction

Simultaneous twin data reduction

CRYSA LIS^{PRO}

Step 4: Background evaluation

Background for 3D centroids

For an accurate evaluation of integrated intensities a good background determination is essential. Two parameters control this evaluation: The evaluation range R_e and the repeat frequency F_r .

$R_e = 25$ [Edit \$R_e\$](#) $F_r = 25$ [Edit \$F_r\$](#)

Binning may reduce the memory requirements for the background evaluation. Default is 1. You may use 2 or 4 in case of lack of physical memory on your machine (risk of swapping)!

☒ 1 ☐ 2 ☐ 4 ☒ Reduce background accumulation to SHORT type (saves memory)

Required disk/memory space for background evaluation: 53.8/12.5 Mb

Background for 3D integration

☐ Average background from 3D centroid evaluation (good for stable & low background, fast)

☒ Smart background (combination of local and average background computation, good for weaker data with high background and locally varying features, e.g. protein data, slower)

Frame range = 1 [Edit range](#) Memory usage: 5.3 Mb

< Back Next > Finish Cancel Help


1. Select 'Average background' if the X-ray background is low throughout the data set.

Select 'Smart background' if the X-ray background is high and/or irregular throughout the data set.

If in doubt, run processing twice using one and then the other background option. Then check for best R_{int} and $\langle I/\sigma(I) \rangle$.

2. Click 'Next'.

Step 5. Outlier Rejection

Simultaneous twin data reduction 

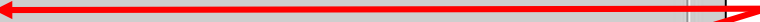
Step 5: Outlier rejection


CCD data sets usually contain more than the unique data required for the structure determination. This redundant data can be used to check for measurement outliers.

The rejection is based on R. Blessing (1997), J. Appl. Cryst. and additional CCD specific criteria.

Outlier rejection

☐ Don't use outlier rejection

☒ Use outlier rejection: 

 1

aP 7.16293 7.78051 11.20846 106.39120 90.56476 97.52915

☒ Use Friedel mates as equivalent

< Back Next > Finish Cancel Help

1. Ensure this box is checked and that the Laue group and unit cell parameters are all correct.

2. Click 'Next'.

Step 6. Output

1. You must change the name of the final reflection file or else the reflection file created previously by CrysAlis^{Pro} upon automatic processing will be **overwritten**. It is recommended to keep the reflection file from automatic processing as a backup. Click here and input a filename of your liking for the reflection file in the next dialog.

Simultaneous twin data reduction

Step 6: Output

Tip: You may change the output name and directory to keep results of data reductions under different parameter sets (UB, supercells...)

Output file name:
C:\CData\Single_Crystal\Training-Agendas\Examples_Data\Twin_TTF\ET_TCNB_RT_02272014_new

Change output name

Finalization options

☒ Space group determination ☒ Automatic ☐ Manual

☐ Automatic structure solution (AutoChem)
Chemical formula not available

☐ Completeness computation

☐ Make unwarp pictures

Max order (one for h, k, l): Resolution:

AutoChem options
Edit formula

< Back **Next >** **Finish** **Cancel** **Help**

2. Enter chemical formula and Z, if not already done.

3. Click 'Finish' to exit the dialog and start processing. CrysAlis^{Pro} will reduce the data, determine the space group and scale the reduced data at once.

Data Processing Results

At the end of the data reduction run, a summary of the data processing statistics is shown in the 'Data Reduction' tab on the right.

1. Mosaicity along a, b and c.

3. Parameters used by CrysAlis^{Pro} for scaling:

- Absorption harmonics (evens and odds)
- # images per scale factor.

6. Space group found by CrysAlis^{Pro}.

```
Data Reduction

FRAMES / RUNS
In run list: 532/4, used: 532/4
3D PROFILE ANALYSIS (twin component #2)
Frames done: 532
Reflections tested: 4501, used: 973
Avg mosaicity (in degrees) - 4 run(s)
e1=0.79, e2=0.73, e3=0.73

3D INTEGRATION & FITTING
Frames done: 532
Fitted: 9115, overflow: 0, hidden: 17
Outliers rejected: 0
FINALIZATION INPUT FILE
Filename: pol411_Pierre
FINALIZATION OUTPUT HKL FILE
Filename: pol411_Pierre_twin1_hklf4
SCALING / NUMERICAL ABSORPTION (SYM:2/m (b-unique))
Empirical abs (e=2 o=0): min=0.96,max=1.03
Frame scales (2/scale): min=0.73,max=1.46
TWIN ANALYSIS
Component Ratio Isolated Overlapped
1 0.51 3848 595
2 0.49 3837 595
DECOMPOSED TWIN DATA STATISTICS (<0.40 overlap)
Component Redundancy F2/sig(F2) Rint
1 2.7 7.6 0.050
2 2.7 7.0 0.048
Overlap limit for HKLF4 export: 0.40
TWIN HKLF5 STATISTICS
Components Redundancy F2/sig(F2) Rint
1,2 2.7 7.7 0.050
SPACE GROUP DESCRIPTOR
C2/c Group #: 15 (2 SG found)
DATA REDUCTION OPTIONS
3D profile fitting used
```

2. # frames processed and # peaks fitted

4. # isolated and overlapped reflections for each component.

5. Overall redundancy, $\langle I/\sigma(I) \rangle$ & R_{int} for each twin component and for the merged data.

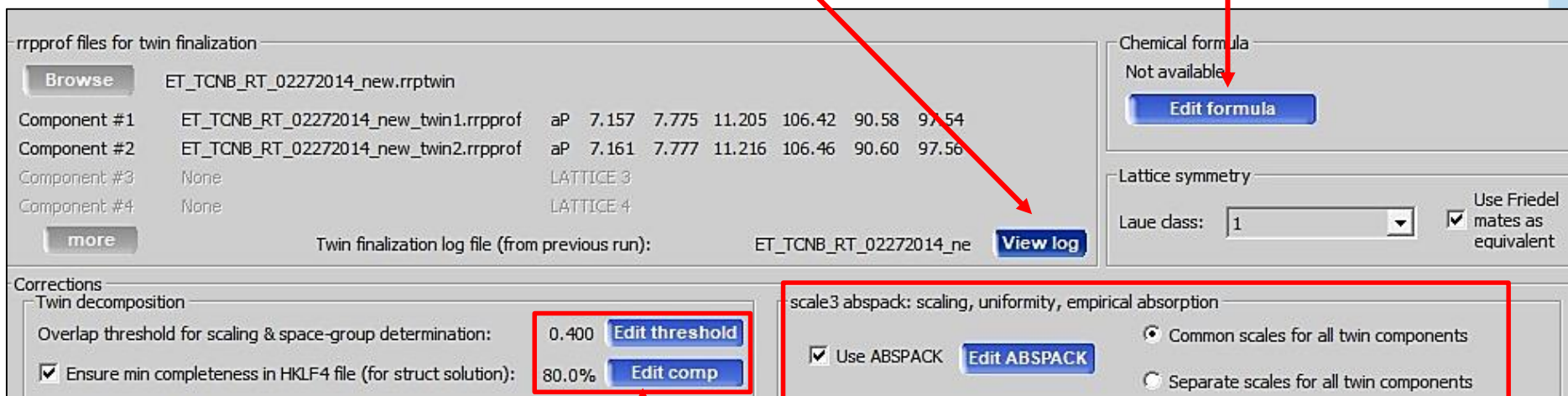
Data Refinalization (re-scaling) and Absorption Correction

Twin Data Finalization_1

1. Click  in the Power Tools panel on the left to open the twin data finalization module.

2. Review log file from latest scaling.

3. Enter chemical formula and Z, if not already done.



rrpprof files for twin finalization

Browse ET_TCNB_RT_02272014_new.rrptwin

Component #	File	Scale	aP	7.157	7.775	11.205	106.42	90.58	97.54
Component #1	ET_TCNB_RT_02272014_new_twin1.rrpprof	aP	7.157	7.775	11.205	106.42	90.58	97.54	
Component #2	ET_TCNB_RT_02272014_new_twin2.rrpprof	aP	7.161	7.777	11.216	106.46	90.60	97.56	
Component #3	None	LATTICE 3							
Component #4	None	LATTICE 4							

more

Twin finalization log file (from previous run): ET_TCNB_RT_02272014_ne **View log**

Chemical formula
Not available **Edit formula**

Lattice symmetry
Laue class: 1 ☒ Use Friedel mates as equivalent

Corrections
Twin decomposition

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

☒ Ensure min completeness in HKLF4 file (for struct solution): 80.0% **Edit comp**

scale3 abspack: scaling, uniformity, empirical absorption

☒ Use ABSPACK **Edit ABSPACK**

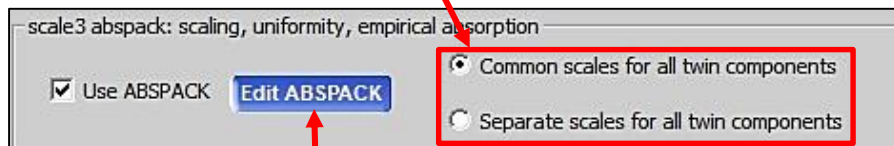
☒ Common scales for all twin components
☐ Separate scales for all twin components

4. The target completeness for the reflection file for each twin component (hkl4 files) is 80%. CrysAlis^{Pro} grabs all isolated reflections first, and then progressively adds reflections overlapping with their neighbors in incremental amounts. If two reflections overlap by more than 40%, they are not included.

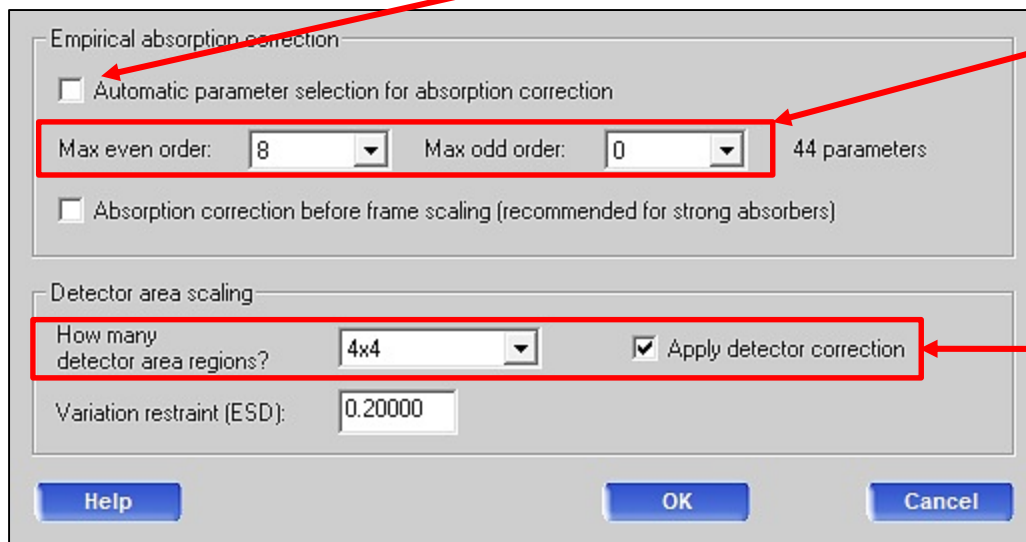
5. Next slide.

Twin Data Finalization_2

6. Select one of these options to refinalize the data with the same or different scale factor for each twin component. Run Refinalize by clicking 'OK' at the bottom of the dialog and check for best R_{int} and $\langle I/\sigma(I) \rangle$.



7. Click here to open the dialog shown below and access additional scaling parameters.

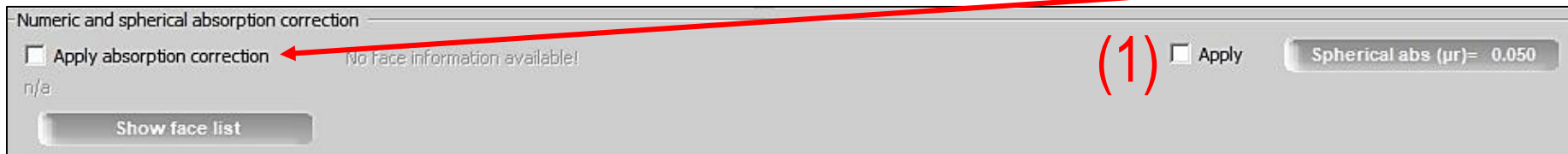


8. Optional for mediocre data sets: uncheck this box and incrementally increase the coefficients for the even & odd orders of the spherical harmonics. Check for best R_{int} and $\langle I/\sigma(I) \rangle$.

9. Optional for mediocre data sets: check this box and vary the array dividing up the detector into smaller areas for local scaling. Check for best R_{int} and $\langle I/\sigma(I) \rangle$.

Twin Data Finalization_3

10. Empirical absorption correction is applied by default. If you want to also apply a numerical absorption correction, you must first create a 3D model of the crystal via face indexing, provided crystal pictures were taken by the collection program. If so, select this option and go to pages 22-25.

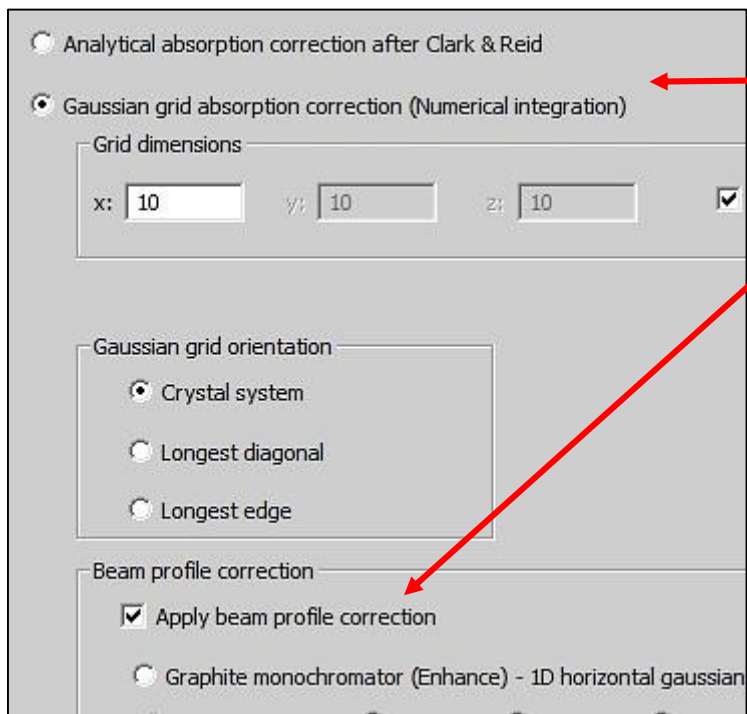


Numeric and spherical absorption correction

☐ Apply absorption correction No face information available! (1) ☐ Apply Spherical abs (μr) = 0.050

n/a

Show face list



☐ Analytical absorption correction after Clark & Reid

☒ Gaussian grid absorption correction (Numerical integration)

Grid dimensions

x: 10 y: 10 z: 10 ☒

Gaussian grid orientation

☒ Crystal system

☐ Longest diagonal

☐ Longest edge

Beam profile correction

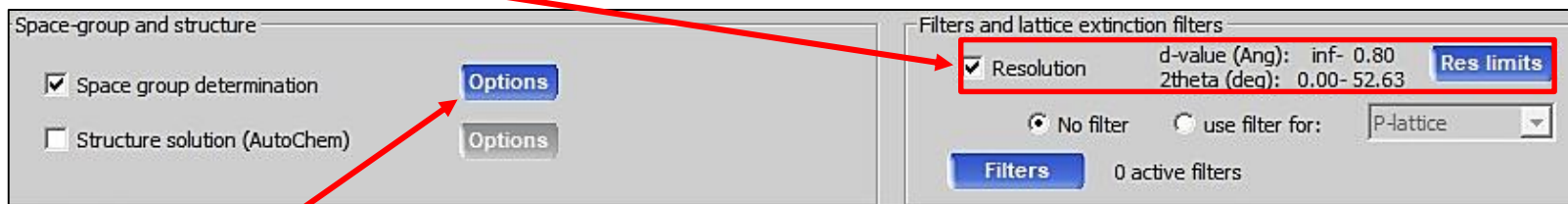
☒ Apply beam profile correction

☐ Graphite monochromator (Enhance) - 1D horizontal gaussian

11. You may run an Analytical or a Gaussian absorption correction. Check for best R_{int} and $\langle I/\sigma(I) \rangle$. For Gaussian, also check this box. For anisotropic crystals with at least one dimension larger than the X-ray beam, CrysAlis^{Pro} will take into account that only part of the crystal is bathed in the beam at once.
12. You may also run a spherical absorption correction (1 on the picture at the top). Check for best R_{int} and $\langle I/\sigma(I) \rangle$ to determine the best absorption correction procedure.

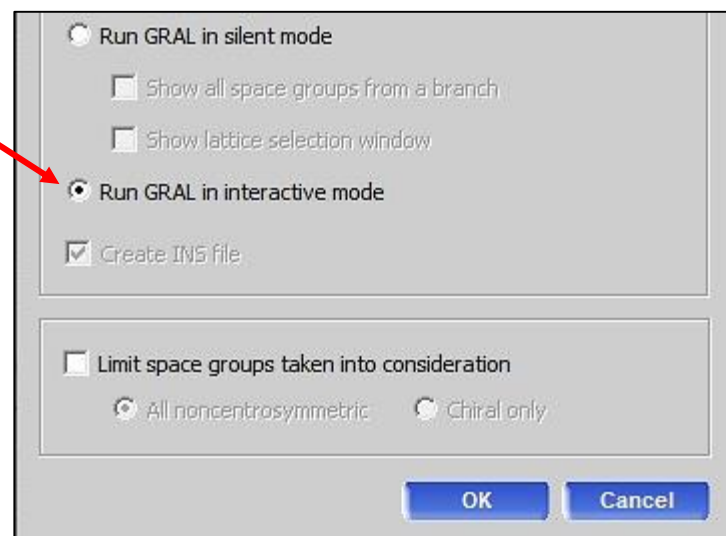
Twin Data Finalization_4

13. Check this box and then click 'Res limits' if a resolution cutoff needs to be used.



14. If you want to manually select the space group, click here to open the space group search dialog shown below.

15. Click here to switch from running GRAL (space group search algorithm) in silent mode (automatic) to interactive mode (manual).




16. Click 'OK' and follow the instructions in the next dialog.

Note: GRAL is the equivalent of XPREP.

Face Indexing_1

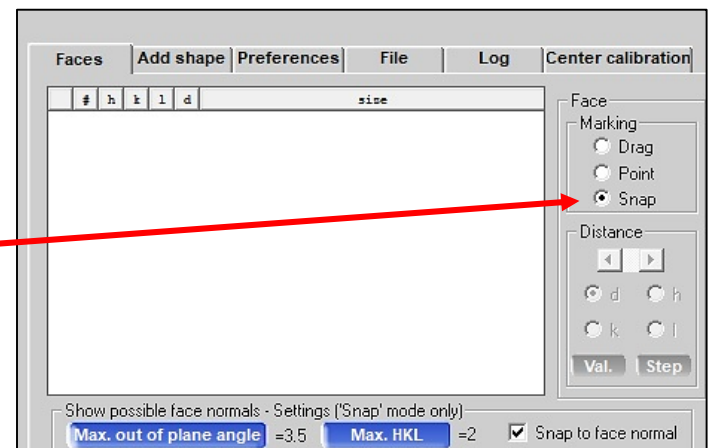
1. This can be done only if the collection program has taken visible pictures of the crystal. Note that none were taken for this data set. The following pages are taken from a different example.

Click  in the Power Tools panel on the left to open the Data Inspection Module

2. At the bottom of the Data Inspection module, click on 'Crystal movie'.

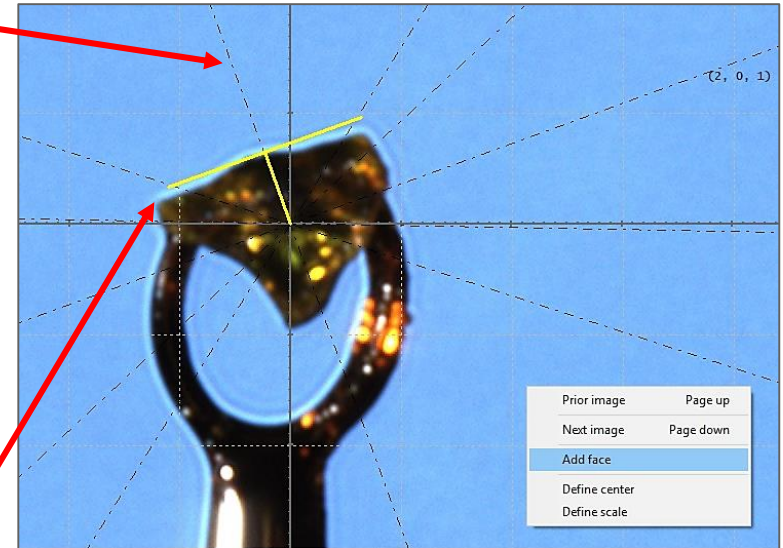


3. In the 'Crystal shape' window, ensure 'Snap' is selected.



Face Indexing_2

4. Select a crystal view that shows normal directions to crystal faces (dotted lines). Different crystal orientations may be displayed using the mouse's wheel or the Prev/Next buttons underneath the image. The crystal view can also be rotated by $\pm 90^\circ$ or 180° at once using the corresponding buttons on the right of the image.
5. Left-click anywhere and drag along a dotted line until the mark (in yellow on this picture) coincides with a crystal face.
6. Right-click on the picture and select 'Add face'.
7. In the new dialog, ensure this option is selected and click 'Add face' to validate the face.



Index selection

☐ Real hkl: -0.222 -11.098 -5.097

☐ Integer hkl: 0 -11 -5

☐ Integer hkl small: 0 -5 -2

☐ Custom hkl: 0 -2 -1

☒ Integer hkl small (Snap mode): 0 -2 -1

Distance selection

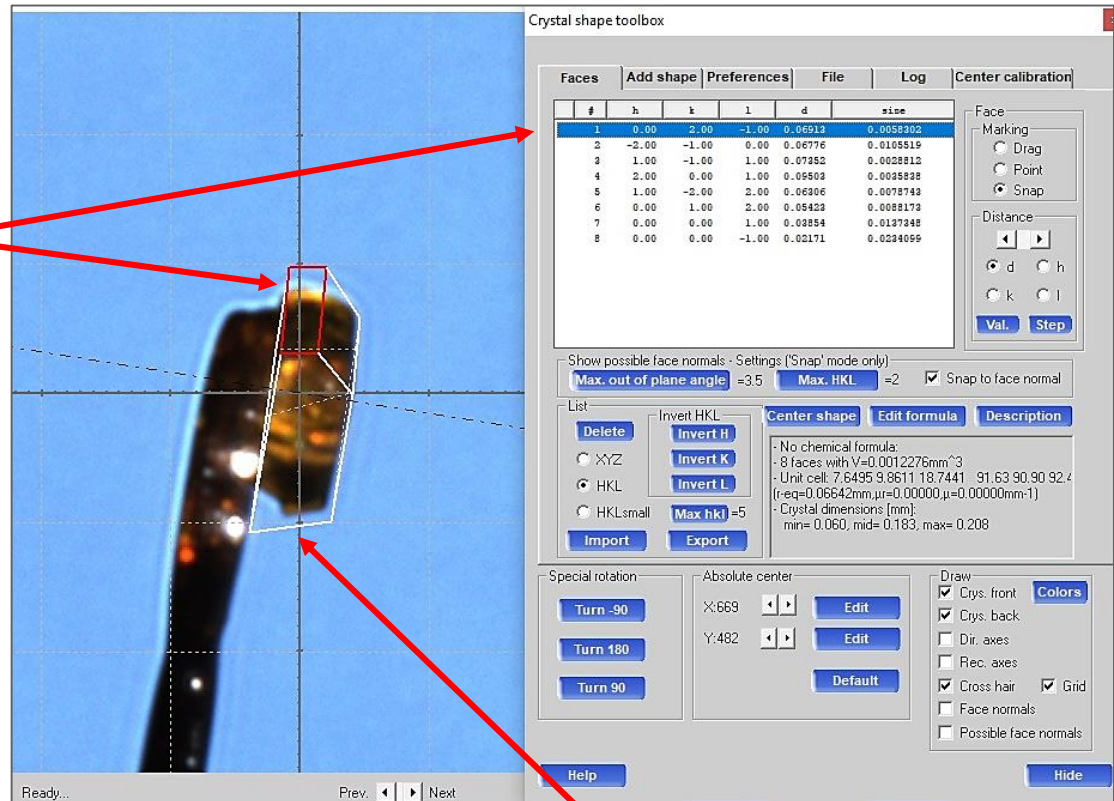
☒ Measured distance 0.06476

☐ Custom distance: 0.06476

Face Indexing_3

8. The new face is added to the list in the right hand side panel, along with its miller indices. A face highlighted in blue in the list is shown in red on the crystal picture.

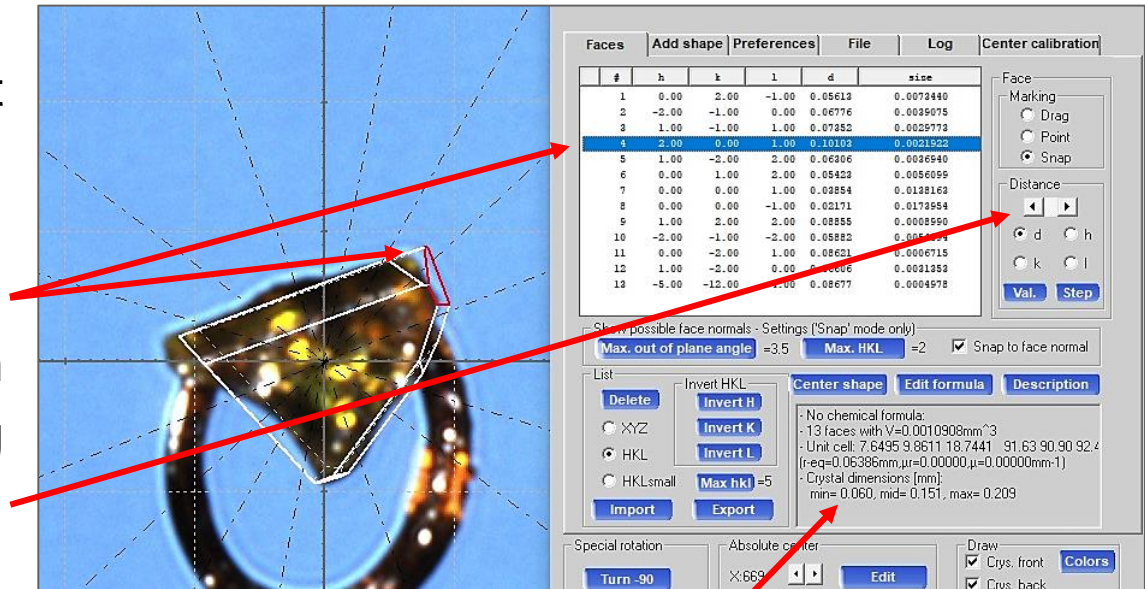
9. A good practice is to select a face view of the crystal first and create as many faces as possible. Then rotate by 90° and add more faces.



10. Once a few faces have been created, CrysAlis^{Pro} will come up with an idealized shape. However, it is likely to be imperfect at this point.

Face Indexing_4

11. Add faces from intermediate crystal orientations to carve out the shape so it fits best to the crystal. Also, you can move an existing face towards or away from the crystal to make it flush with a crystal edge by selecting it in the list and using the 'Distance' arrows.

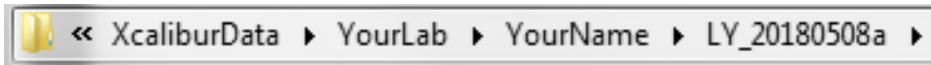



Note: A * symbol preceding a face in the list means this face has become too small and should be deleted.

12. As the shape is being adjusted, the 3 dimensions of the crystal are automatically calculated by CrysAlis^{Pro}. If the correct chemical formula and Z have been previously entered, the crystal density and absorption coefficient μ are also automatically calculated by CrysAlis^{Pro}.

Where Are My Files?

Go to the experiment data directory:



- The images are in the 'frames' directory.
- The crystal pictures are in the 'movie' directory.
- All log files for the current experiment are in the 'log' directory.
- The reflections file for each twin component are named '*twin1_hkl4.hkl', '*twin2_hkl4.hkl', ...
The reflections file for the merged data is called '*twin1_hkl5.hkl'.
- The input file for structure solution is named '*twin1_hkl4.ins'. To open it in Olex2, click  At the bottom of the Power Tools panel on the left and select '*twin4.ins' in the next dialog. Upon opening this file for the 1st time, Olex2:
 - creates a 'struct' directory in the experiment data directory (alongside 'frames', 'movies' and 'log'), followed by a sub-folder name 'Olex2_ExperimentName_hkl4'.
 - transfers the files '*twin1_hkl4.hkl', '*twin1_hkl5.hkl' and named '*twin1_hkl4.ins' in the Olex2 sub-folder.

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