Twinned Data Set Processing with CrysAlis^{Pro}

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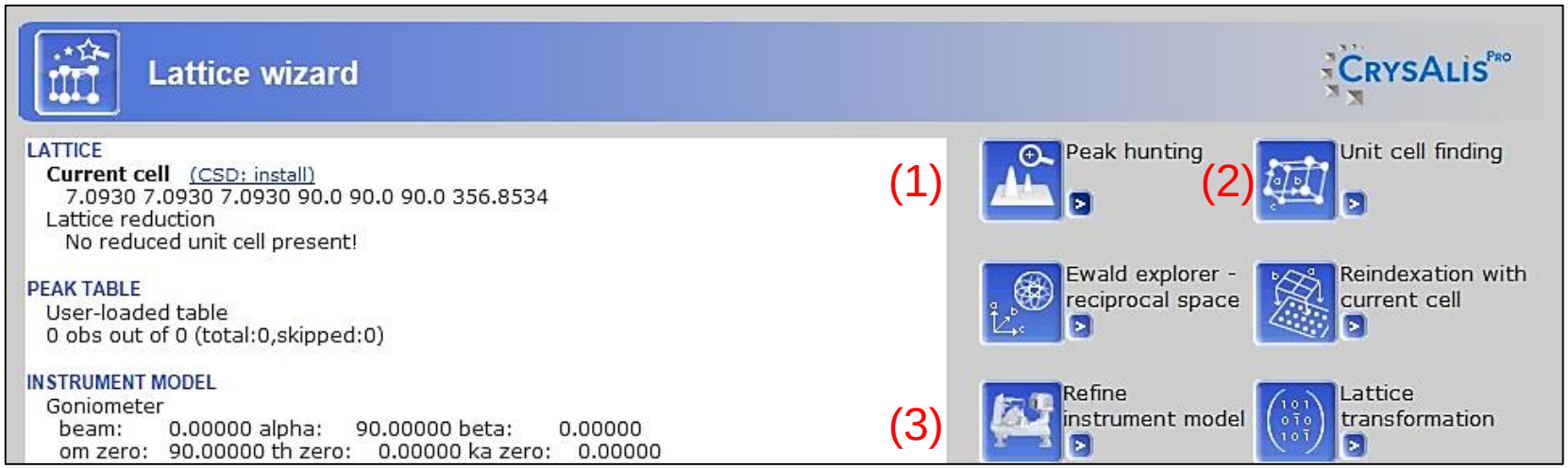
Indexing the diffraction pattern in the Lattice Wizard

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.



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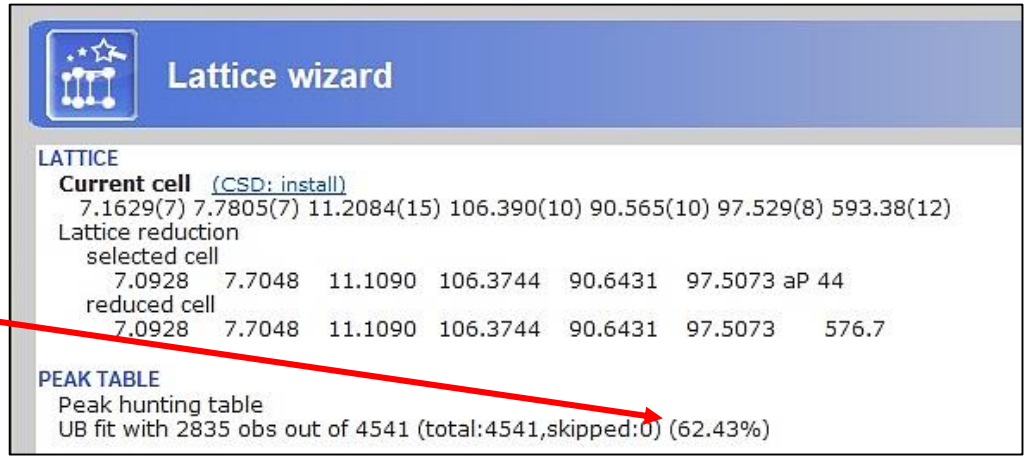
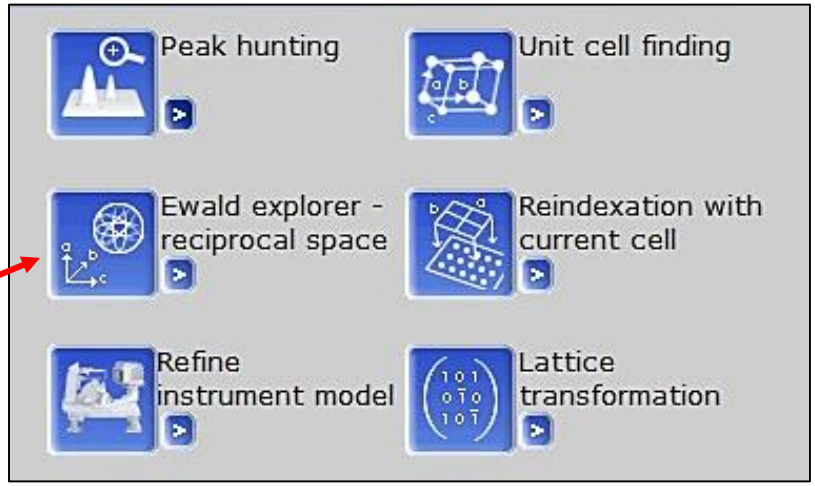
3. The Lattice Wizard shows:
   - The unit cell parameters.
   - The number and % reflections indexed. Only 62.43% reflections are indexed in this example.



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.



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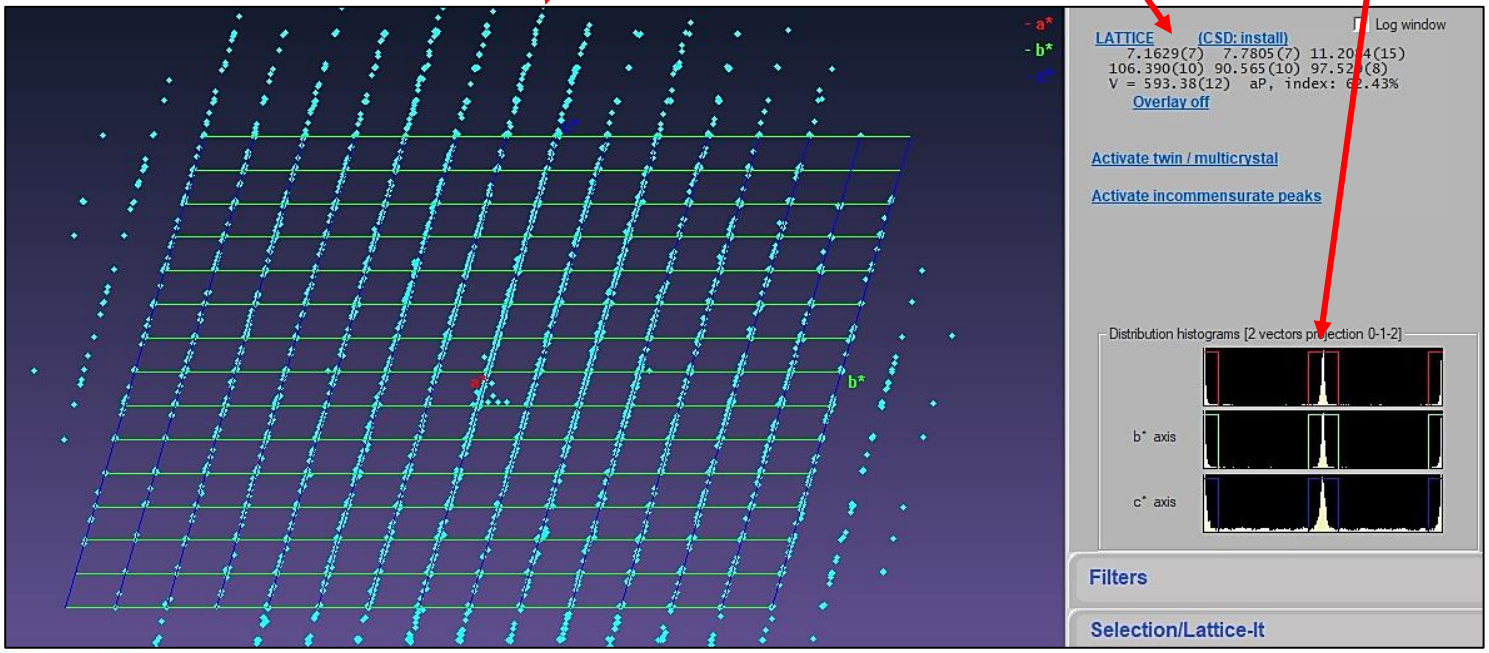
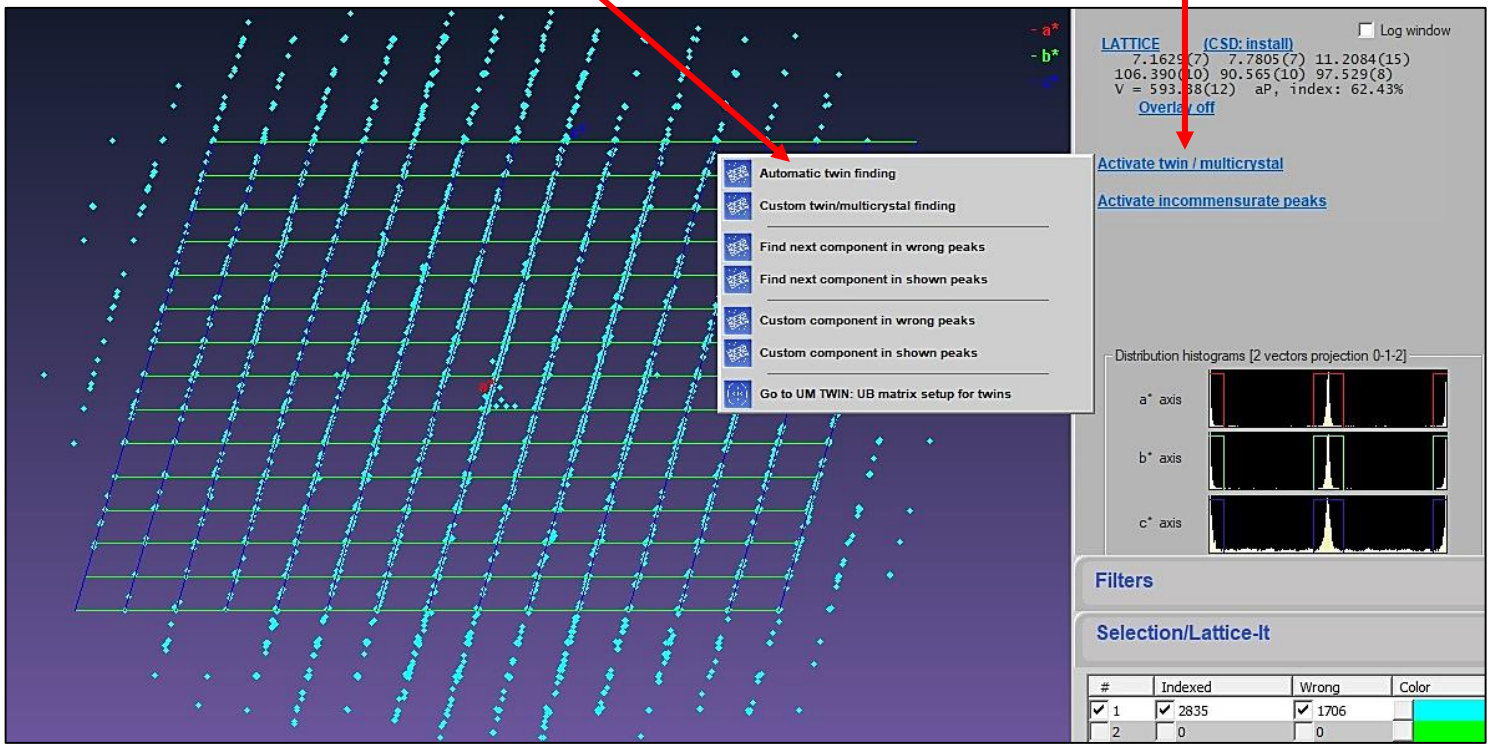
5. The reciprocal space interface shows:
   - The unit cell parameters and % reflections indexed.
   - The predictions (red, green and blue boxes) and reflections for the current component match each other along a*, b* and c*, indicating the unit cell parameters are correct.
   - Lots of reflections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



Indexing the diffraction pattern in the Lattice Wizard

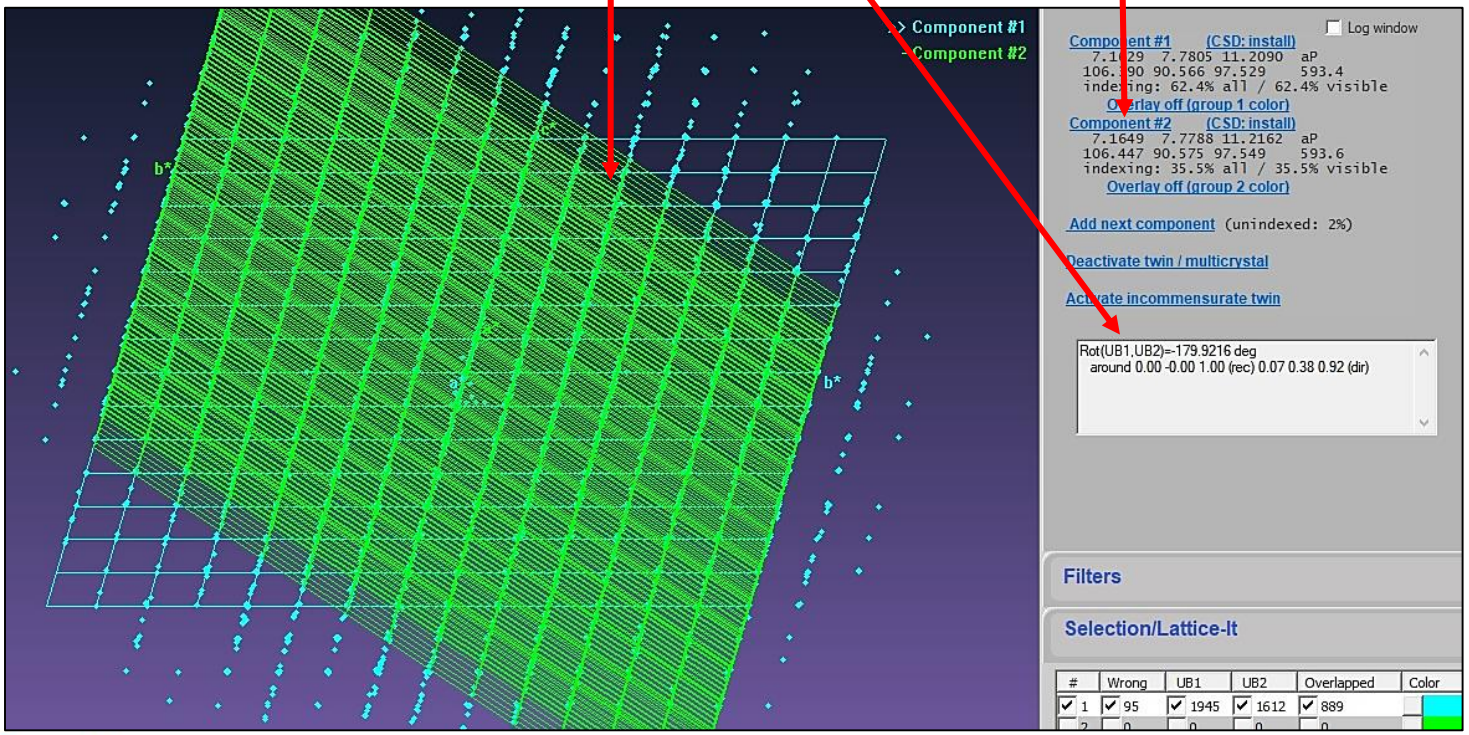
6. To activate twin indexing in ‘Ewald explorer’, click ‘Activate twin / multicrystal’ and select ‘Automatic twin finding’.

   ‘Automatic twin finding’ should handle most twin situations.



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.



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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 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.

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Data Processing

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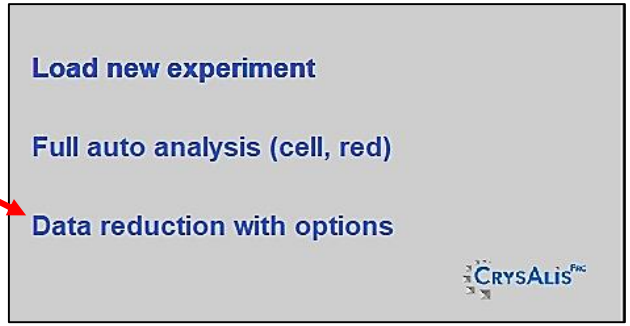
Data Reduction

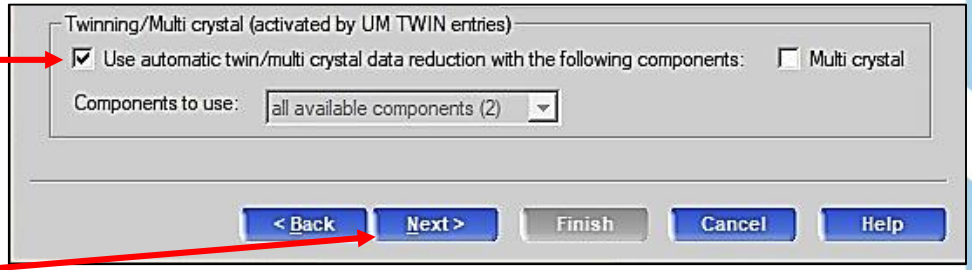
1. Click on START/STOP

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’.



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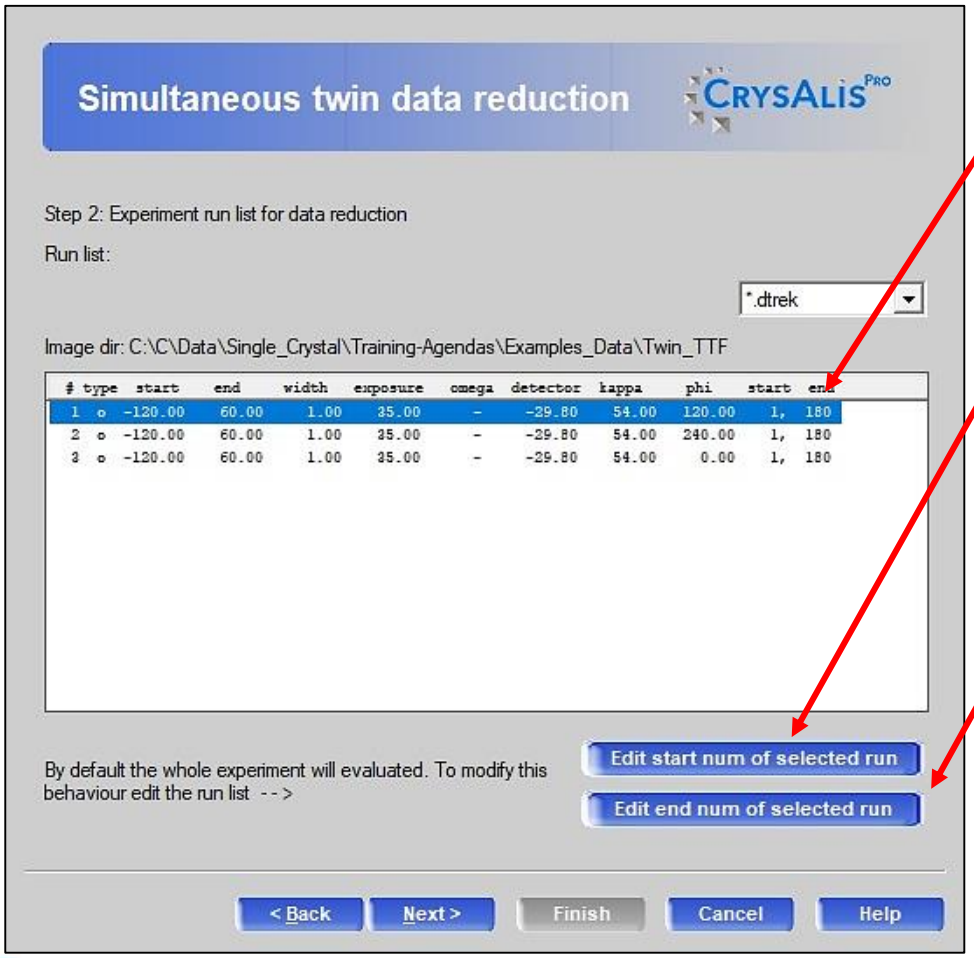
Step 2. Image range selection

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



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’.

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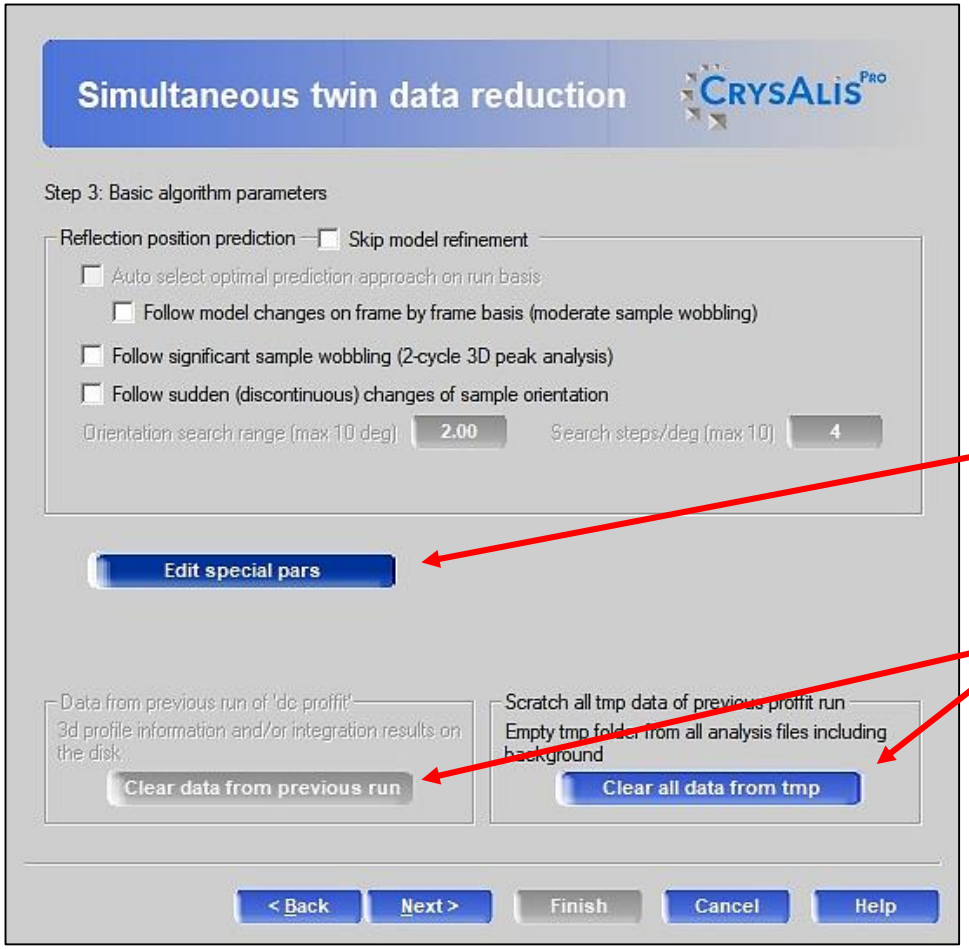
Step 3. Special Parameters



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

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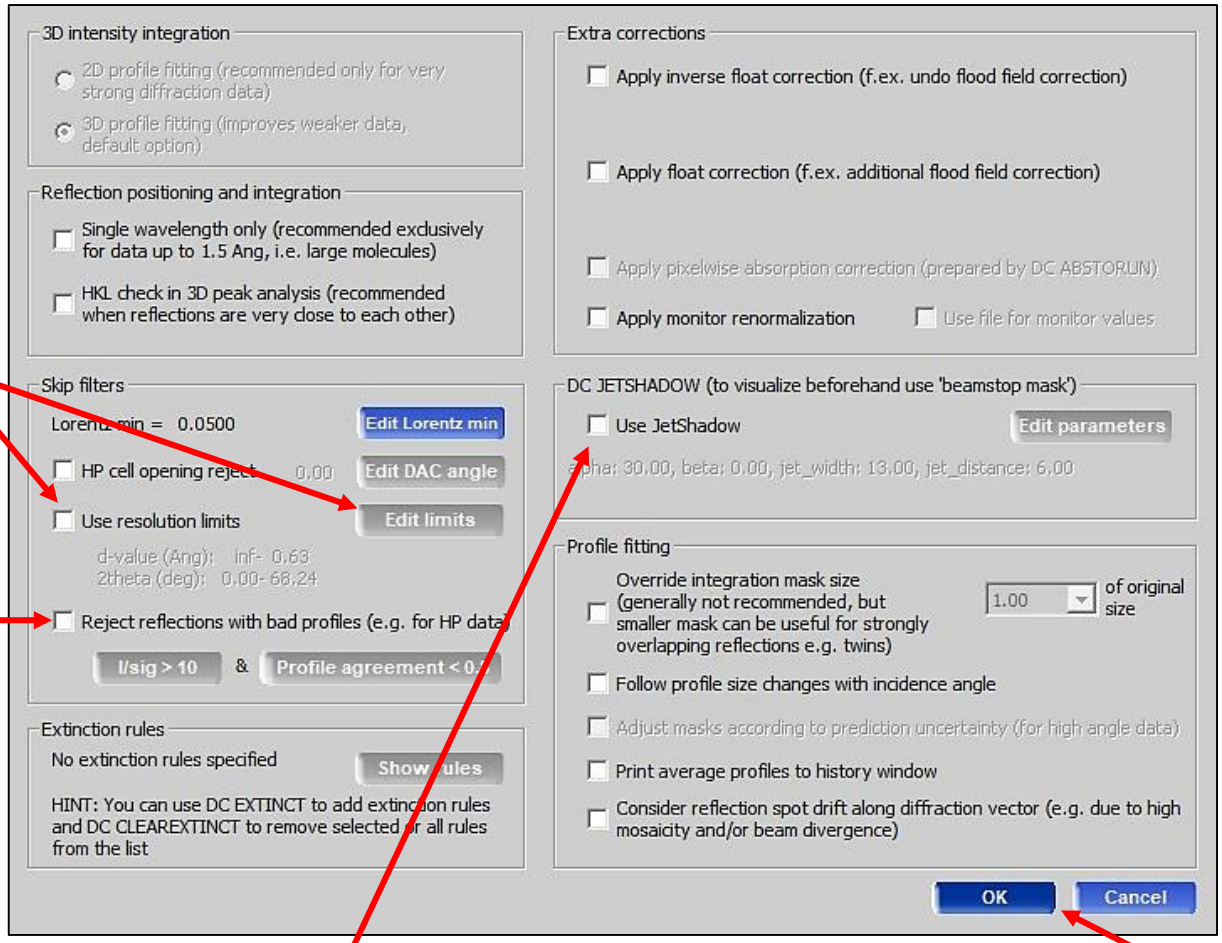
Special Parameters Options

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.



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.

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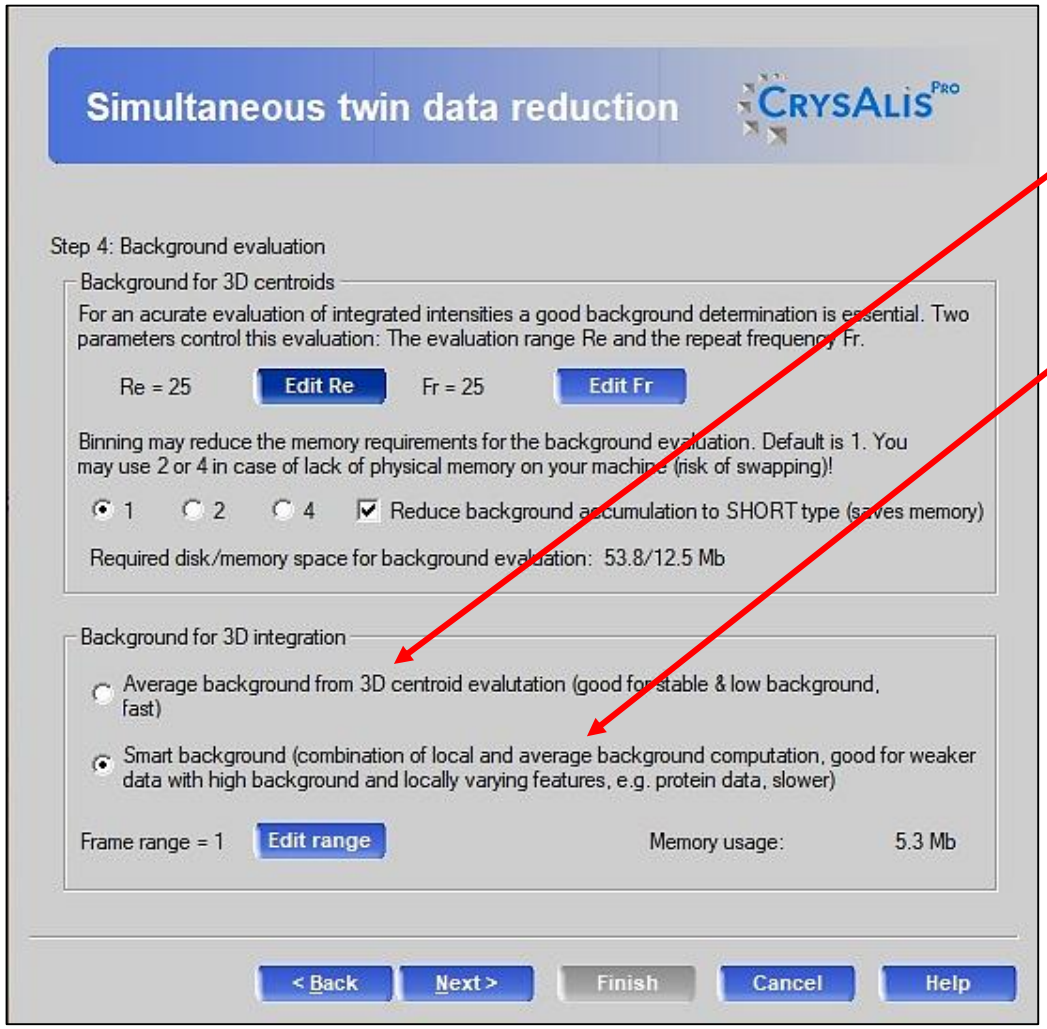
Step 4. Background Subtraction



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’.

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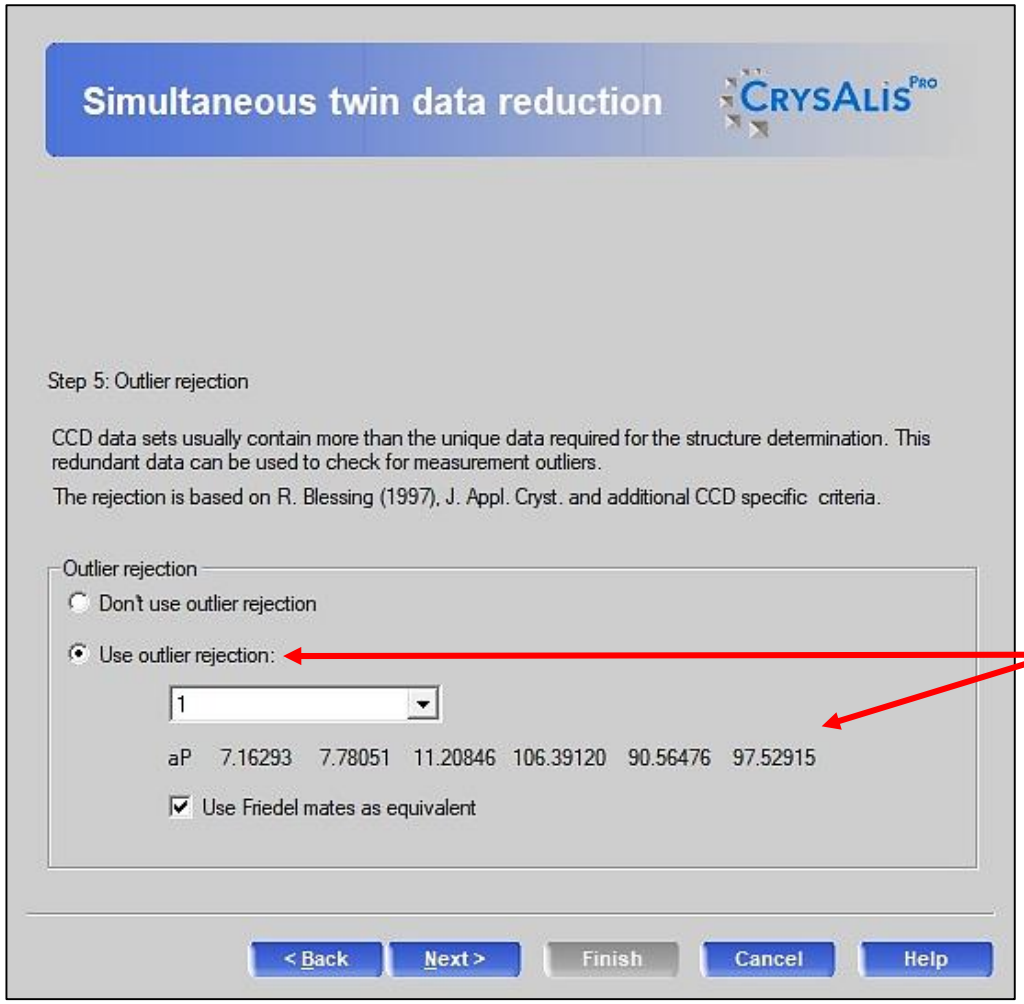
Step 5. Outlier Rejection



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

2. Click 'Next'.

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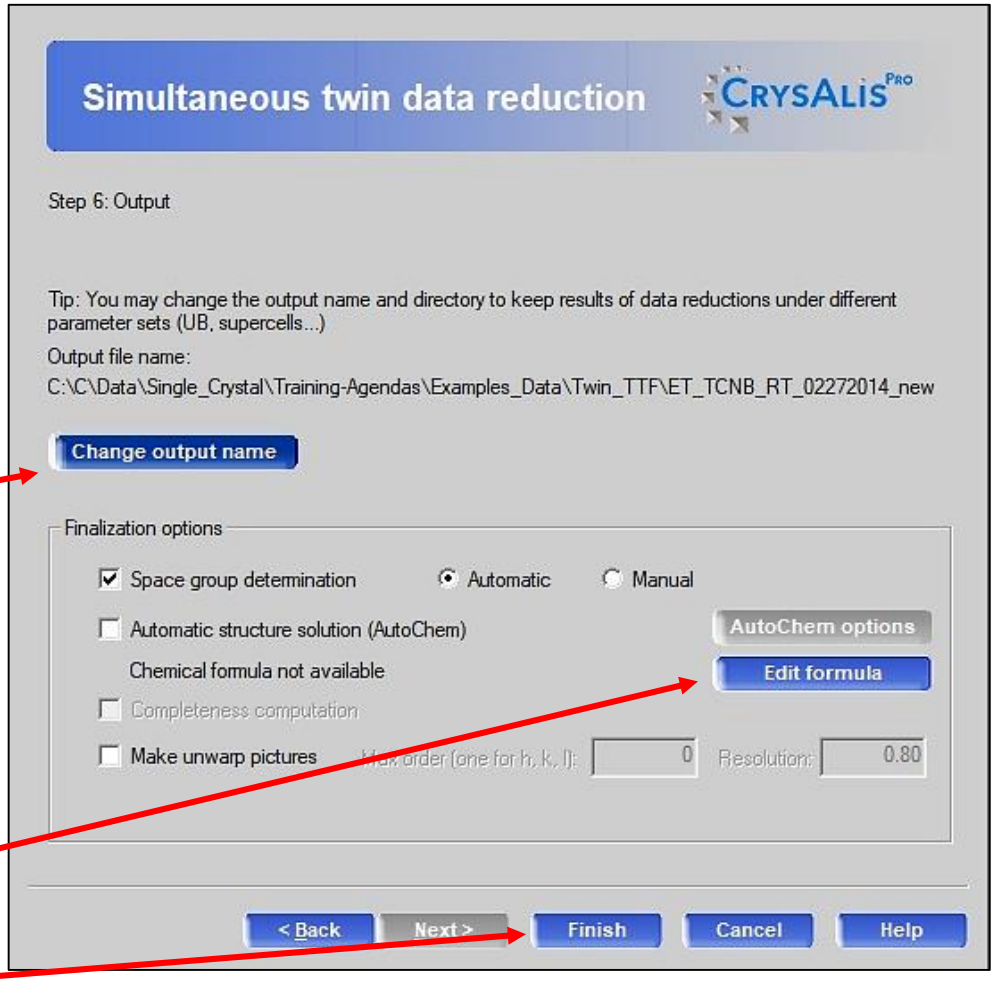
Step 6. Output

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.



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.

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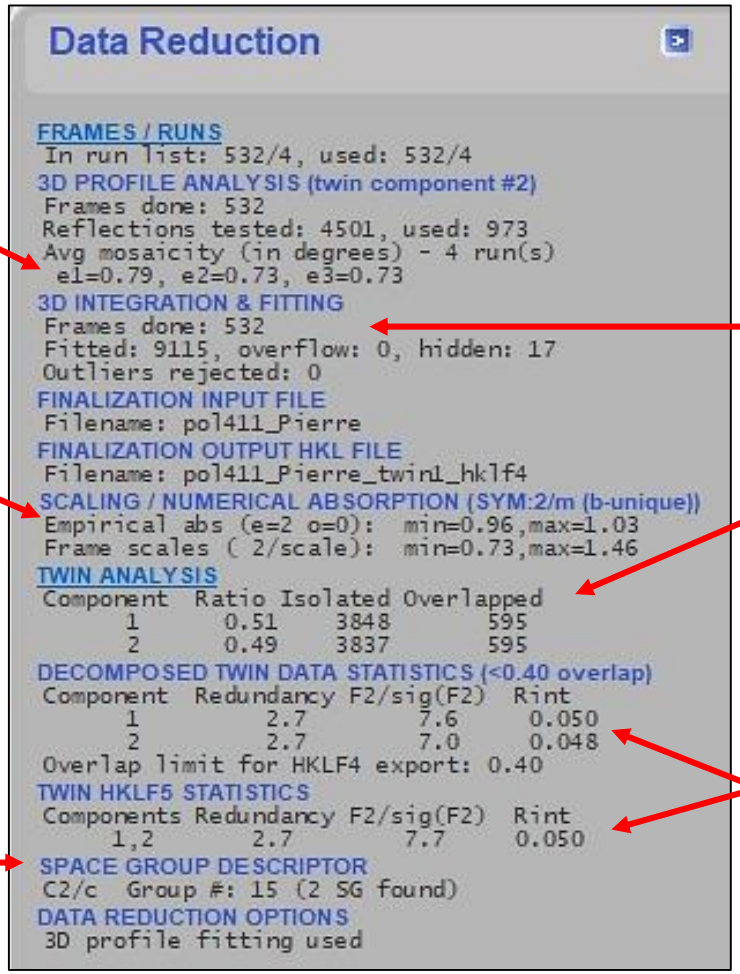
Data Processing Results

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}.



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.

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Data Refinalization (re-scaling) and Absorption Correction

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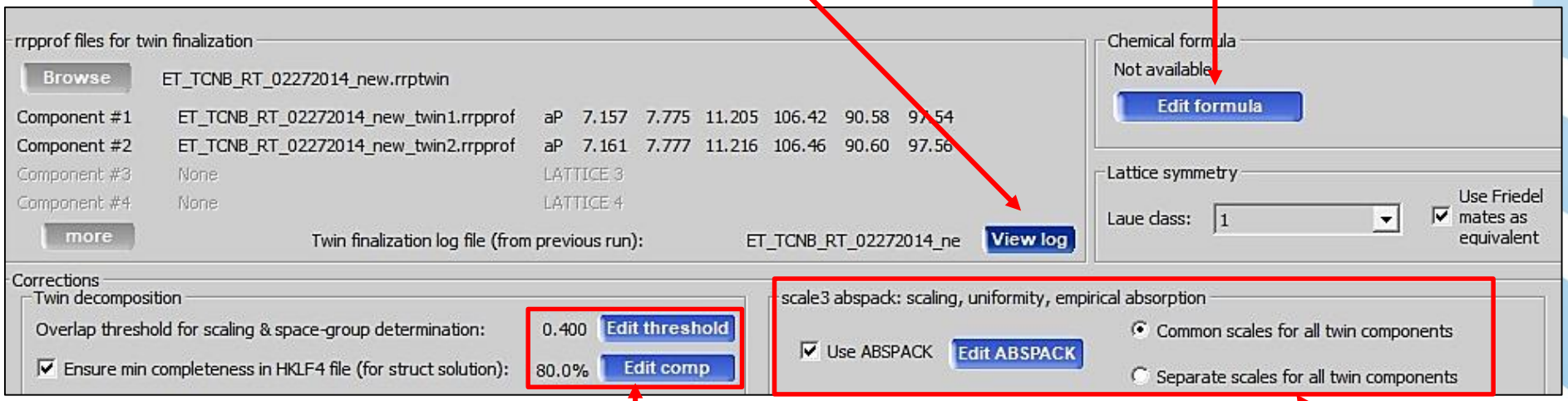
Twin Data Finalization_1

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.



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.

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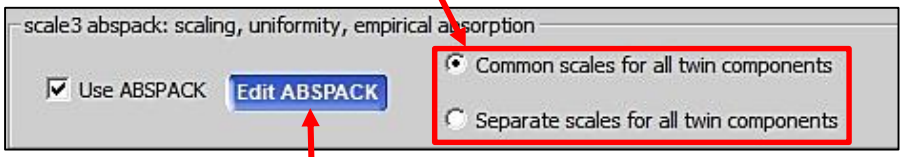
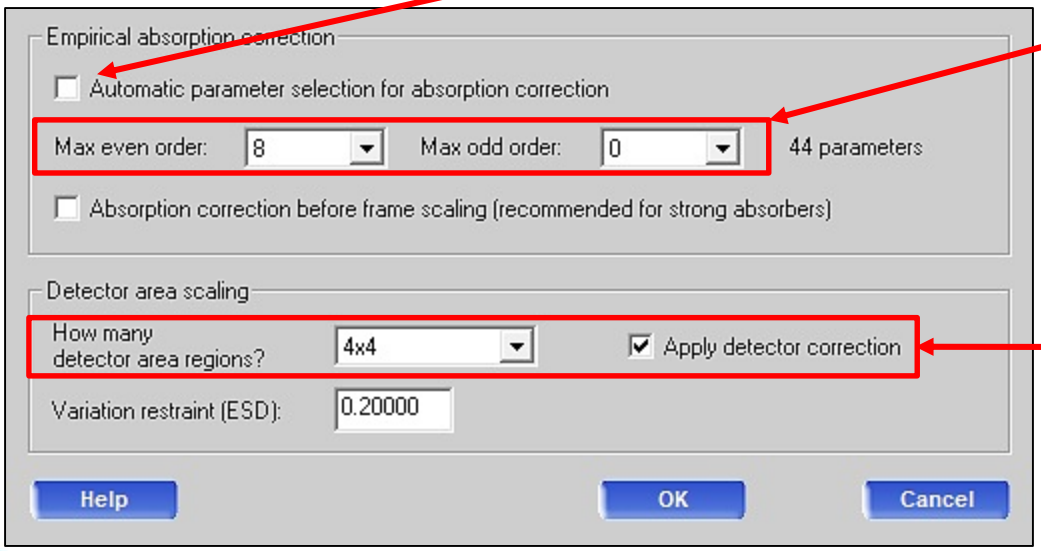
Twin Data Finalization_2

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 $\langle I/\sigma(I)\rangle$.

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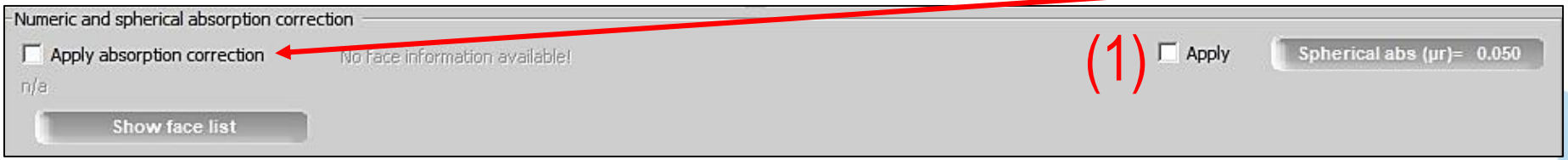
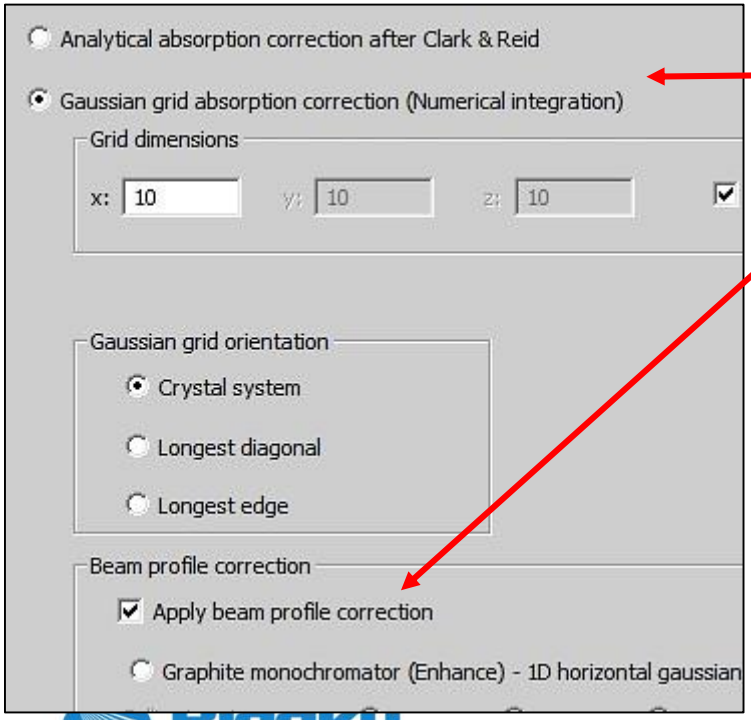
Twin Data Finalization_3

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.



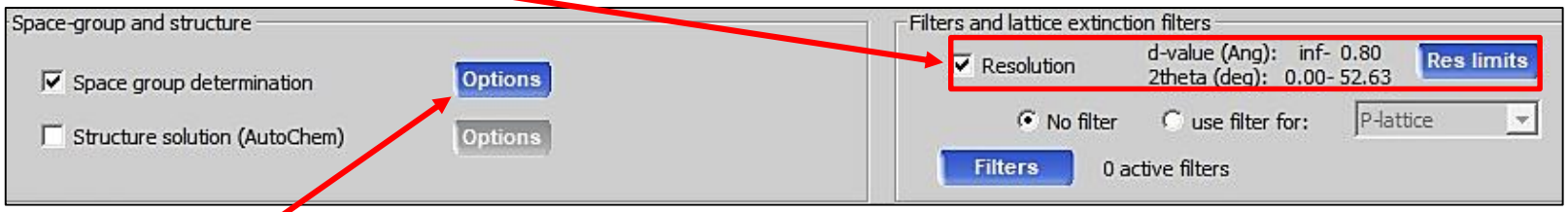


11. You may run an Analytical or a Gaussian absorption correction. Check for best R_{int} and $\langle I/\sigma(I)\rangle$.
For Gaussian, also check this box. For anisotropic crystals with at least one dimension larger than the X-ray beam, CrysAlis^{Pro} will take into account that only part of the crystal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

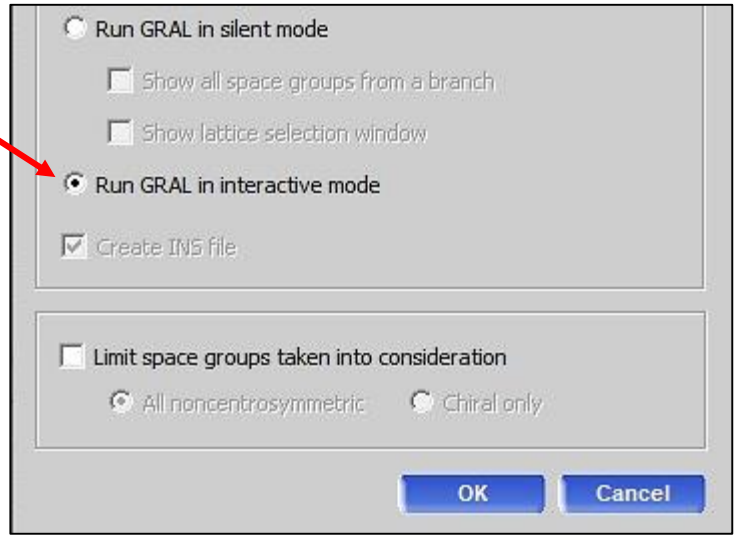
13. Check this box and then click ‘Res limits’ if a resolution cutoff needs to be used.



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.



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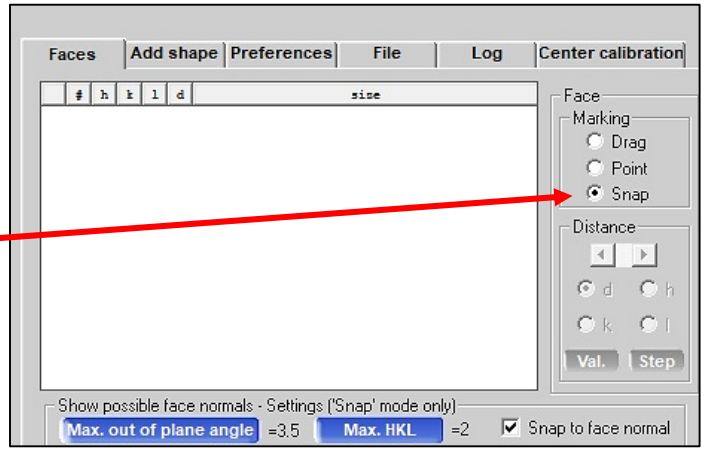
Face Indexing_1

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.



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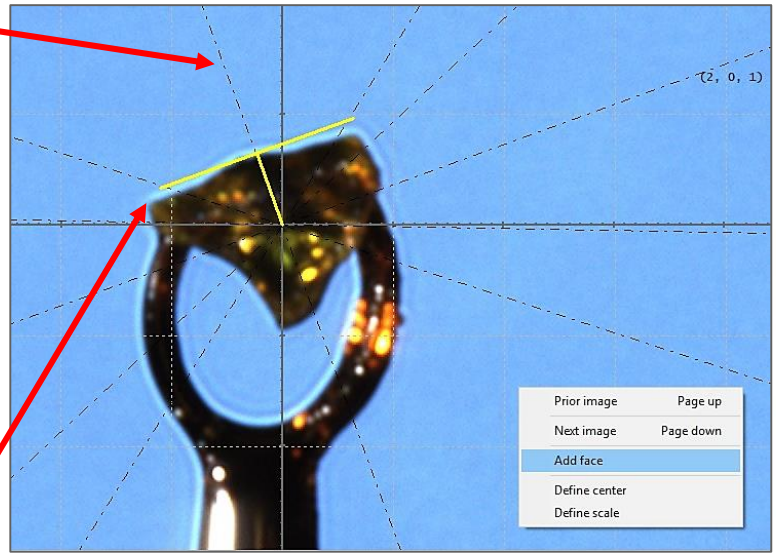
Face Indexing_2

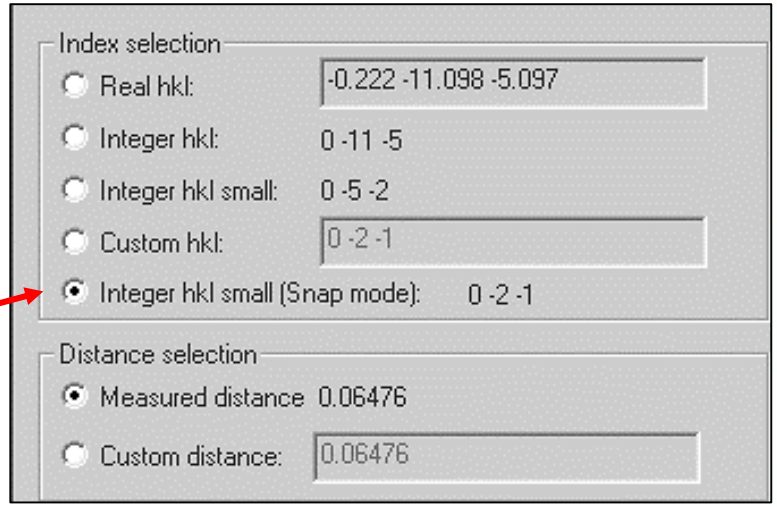
4. Select a crystal view that shows normal directions to crystal faces (doted lines). Different crystal orientations may be displayed using the mouse's wheel or the Prev/Next buttons underneath the image. The crystal view can also be rotated by +/- 90° or 180° at once using the corresponding buttons 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.



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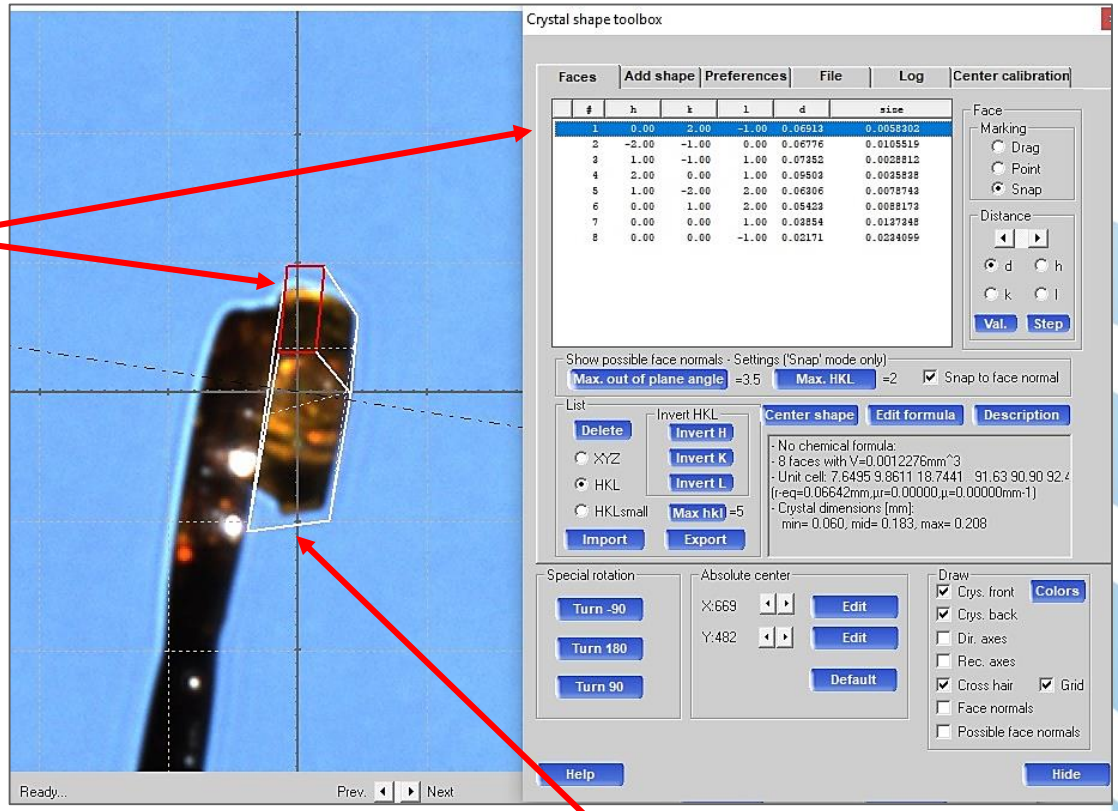
Face Indexing_3

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.

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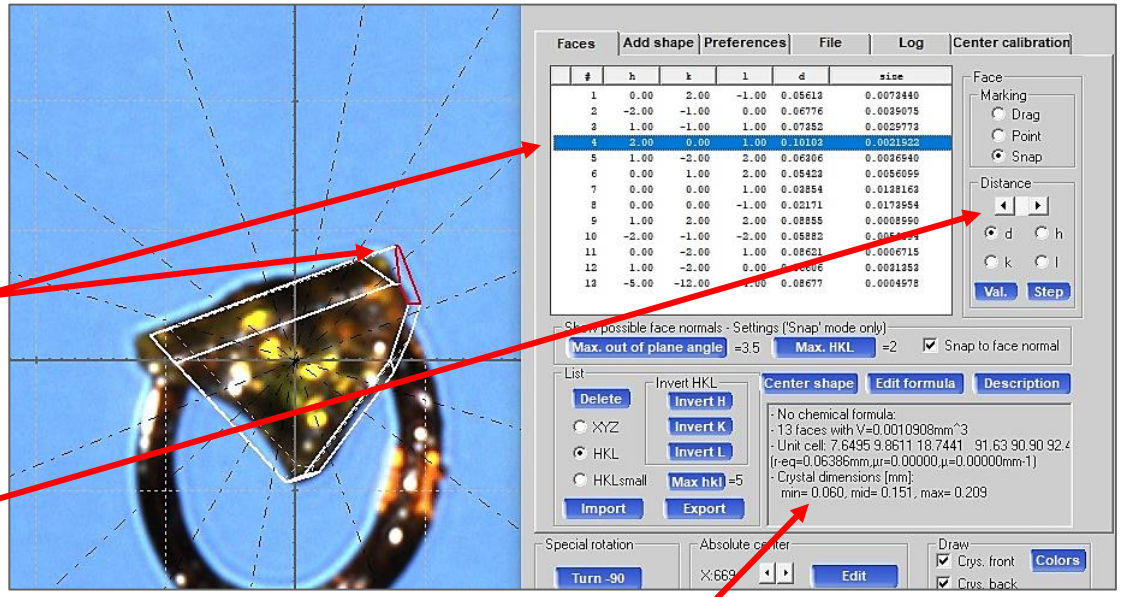
Face Indexing_4

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}.

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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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