CrysAlis^{Pro}

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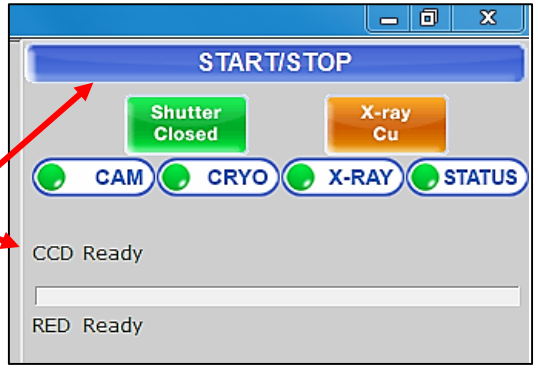
Getting Started

1. Launch the online version of CrysAlis^{Pro} by pushing the blue CrysAlis^{Pro} icon.

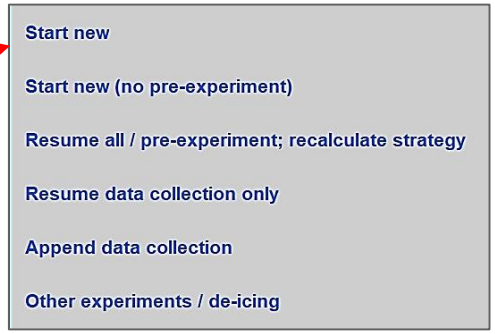
2. Wait for the instrument to initialize and show ‘Ready’.



3. You should be in Small Molecule mode.
   Click once on this icon if it shows ‘PX’.

4. Click on ‘START/STOP’.

5. Click on ‘Start new’.



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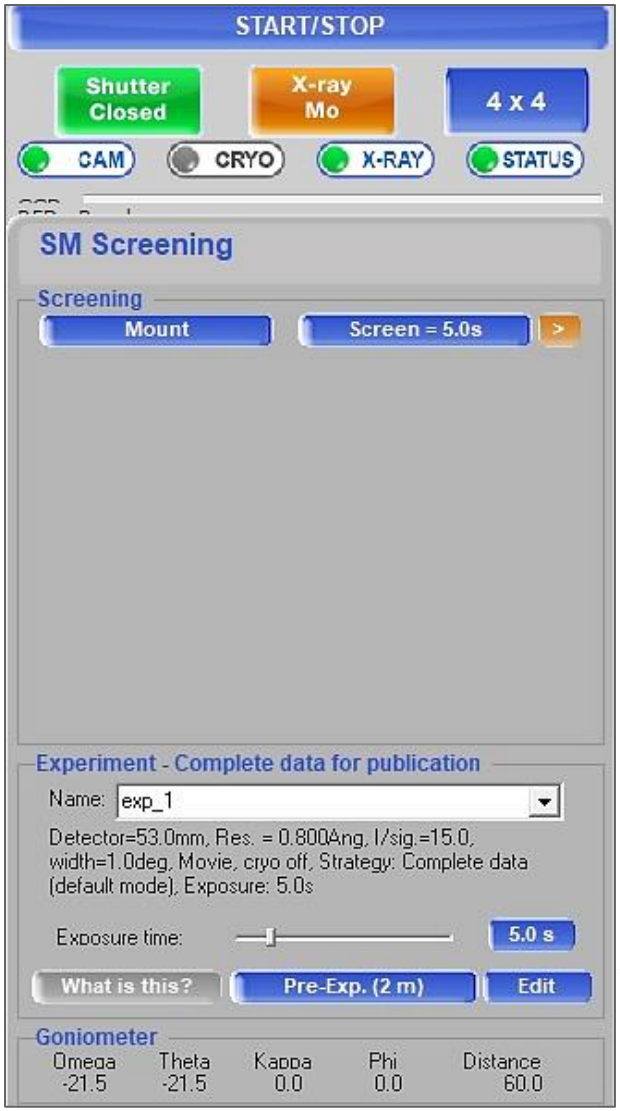
The Crystal Screening Panel Opens . . .



Click on Edit

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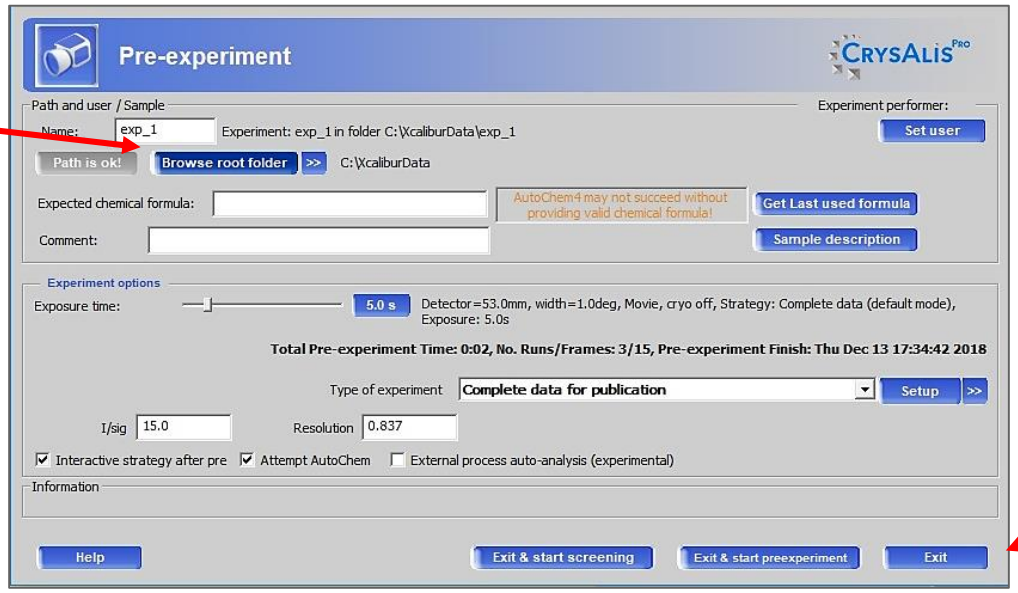
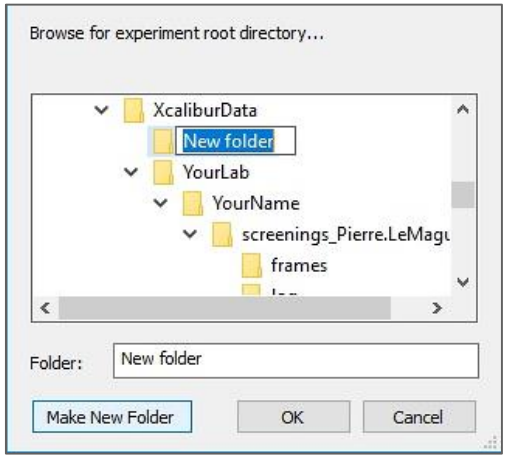
First, Setup User Info ! !

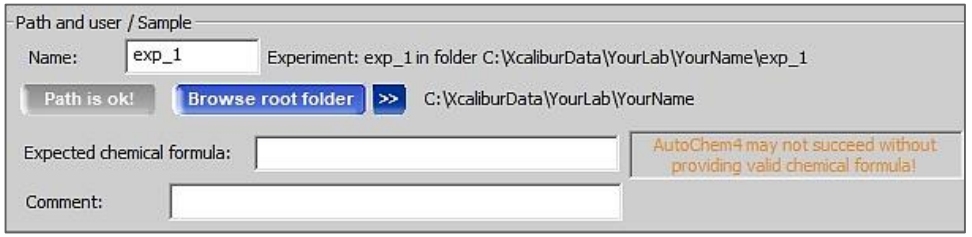
1. Open File Explorer



3. Click Exit

2. Browse to an existing folder or create new folders to something as shown below.





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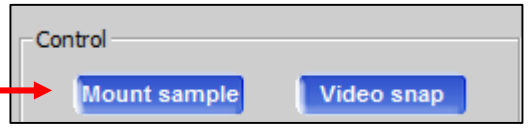
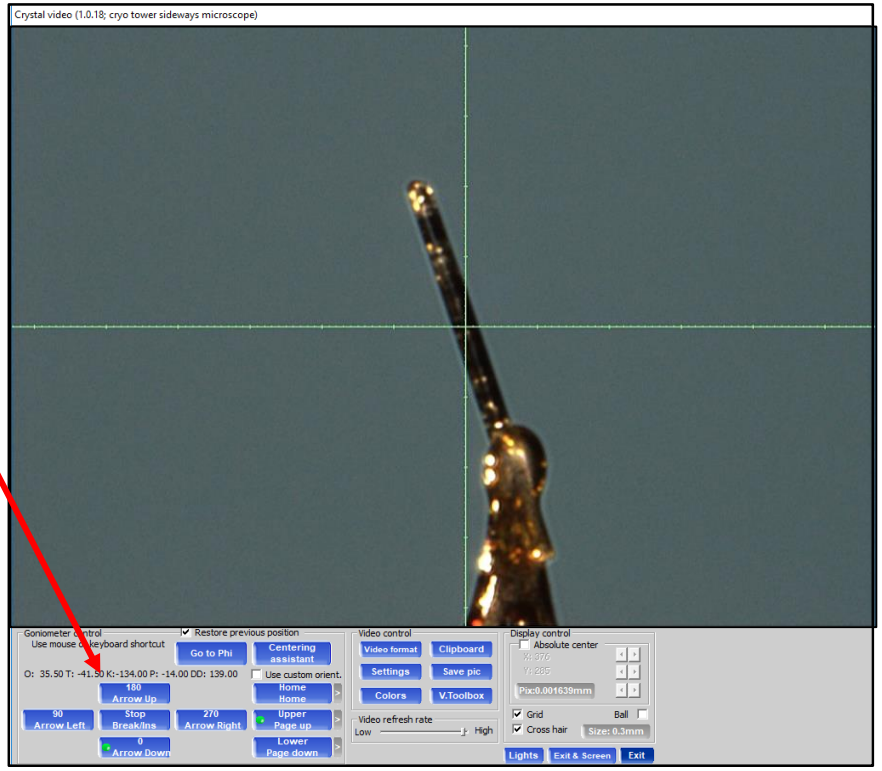
Second, Mount & Center a Crystal

1. Click ‘Mount sample’ to open the mounting & centering window.



2. Mount the crystal & center to the crosshairs in several Phi orientations. Click on these buttons or use arrow keys shortcuts to switch between 0, 90, 180 and 270° Phi orientations.
   The crystal must be centered as well as possible. This picture shows an example of a needle that is centered exactly to the crosshair.



3. Click ‘Exit’ or press ‘ESC” on the keyboard.

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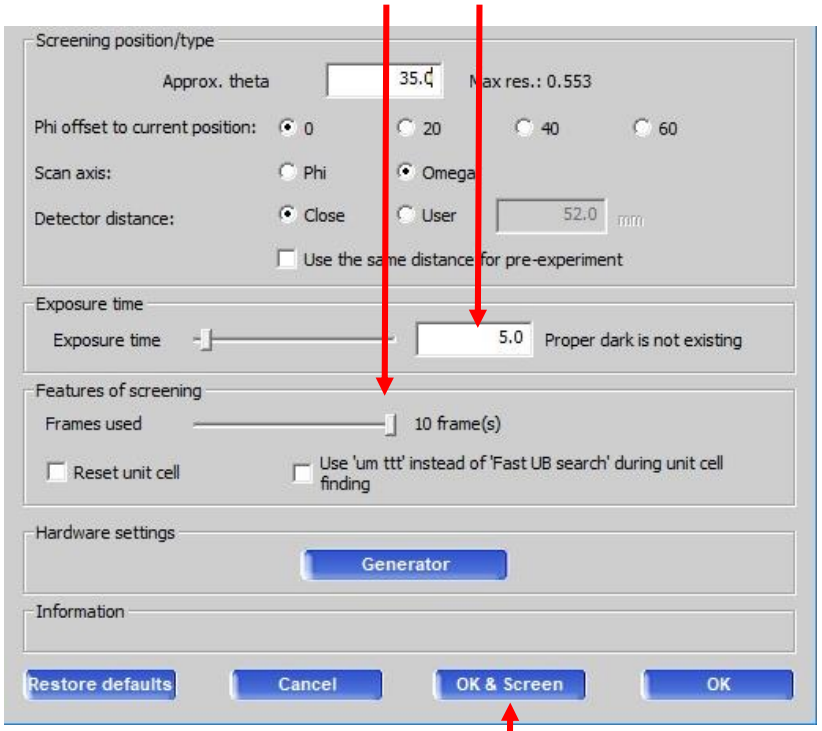
Third, Enter Your Screening Parameters

1. If you click directly on ‘Screen’, 10 frames will be collected in a single crystal orientation and using the displayed exposure time.
2. To change the parameters, click here. Then change the exposure time and # frames in the next dialog.



3. Click here to start screening.

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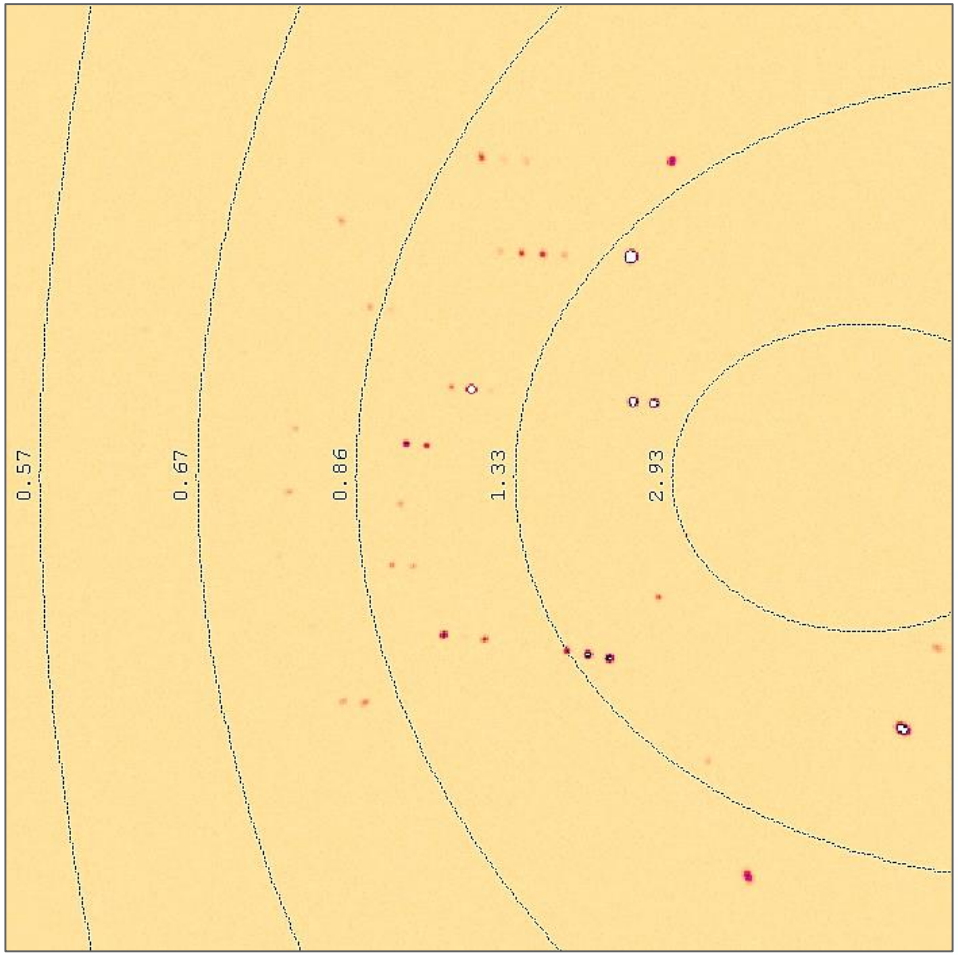
Examine the frames

Criteria for a good diffraction pattern:

1. Well-defined, single spots. If some spots appear split, you may have a cracked or twinned crystal.
2. Ordered, non-intertwined array of spots. Rows of spots crossing each other (intertwined) are indicative of multiple diffraction patterns. You may have mounted more than 1 crystal.
3. No rings of diffraction. If there are, then you have either mounted a polycrystalline sample or there is ice on the crystal.
4. Strong enough diffraction. Spots should be visible to 0.8-0.9 Å.



5. If 1.-3. are not satisfied, click ‘Start new’ again and screen another crystal. If 4. is not satisfied, increase the exposure time.

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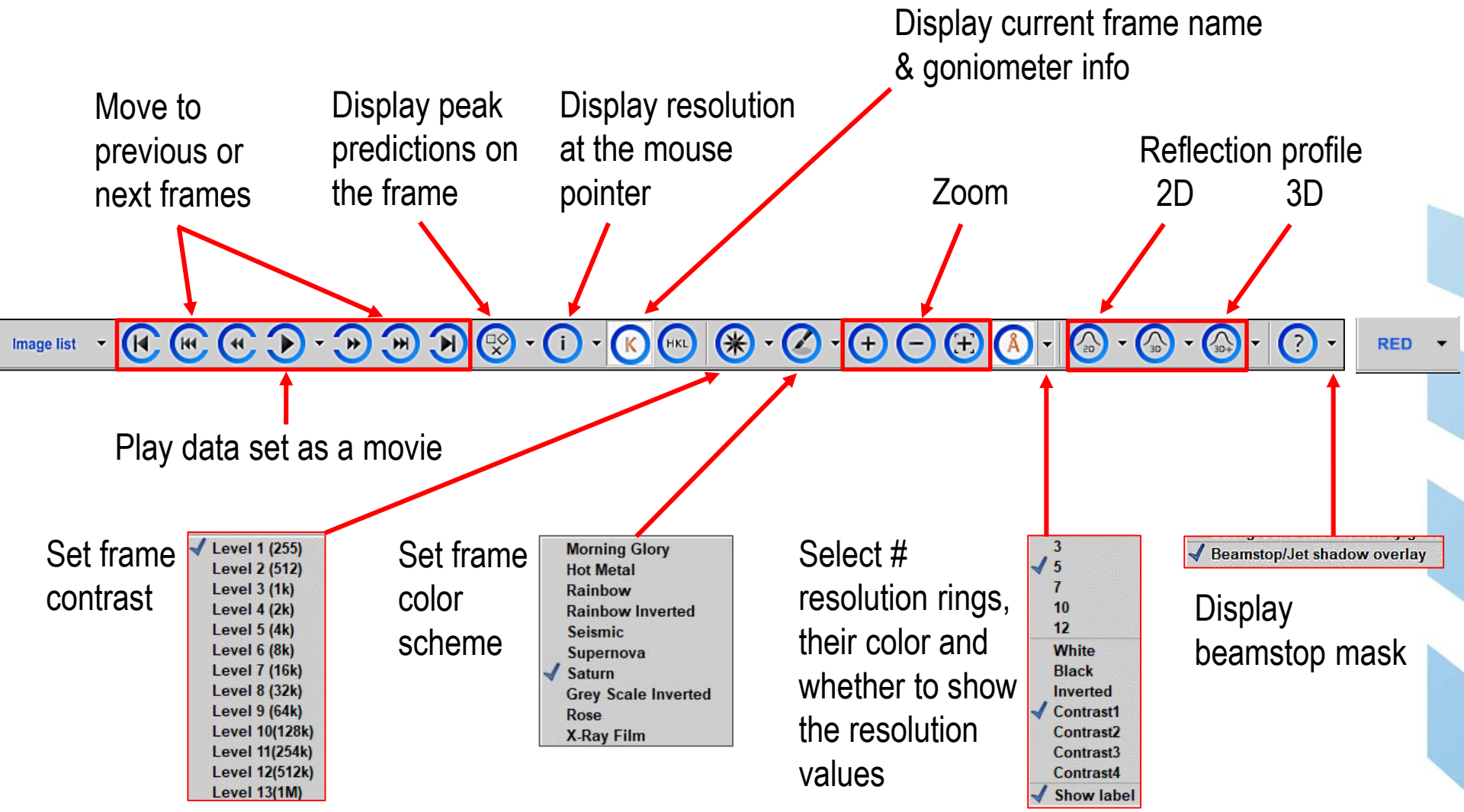
Use the Toolbar Functions



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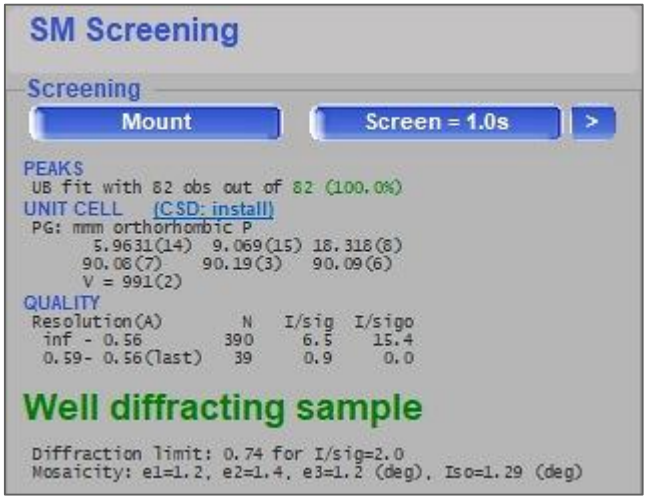
Check the Auto-Indexing Results

% and # reflections indexed

Unit cell & Bravais lattice

<I/σ(I)> estimate

Mosaicity in 3 directions



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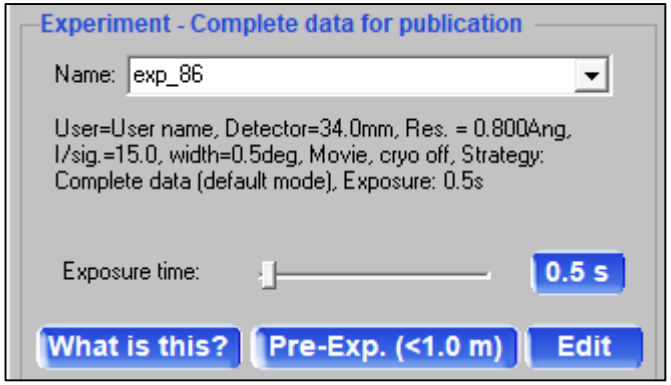
Run ‘What is this?‘ experiment or ‘Pre-experiment‘



Choose **What is this?** if the aim is to obtain a quick structure (quality control, filtering out starting materials …). This features will collect a partial data set (60-70% completeness) with a target <I/σ(I)> of 5 to 1 Å.

Choose **Pre-experiment** if you plan to collect a full data set to IUCr standards. For this option, go to Page 12.

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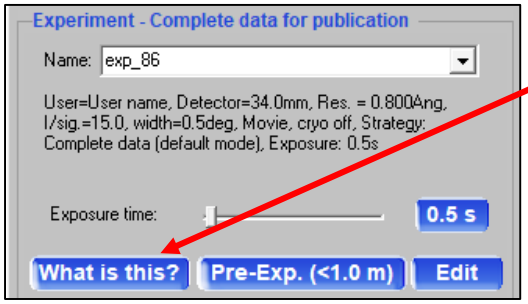
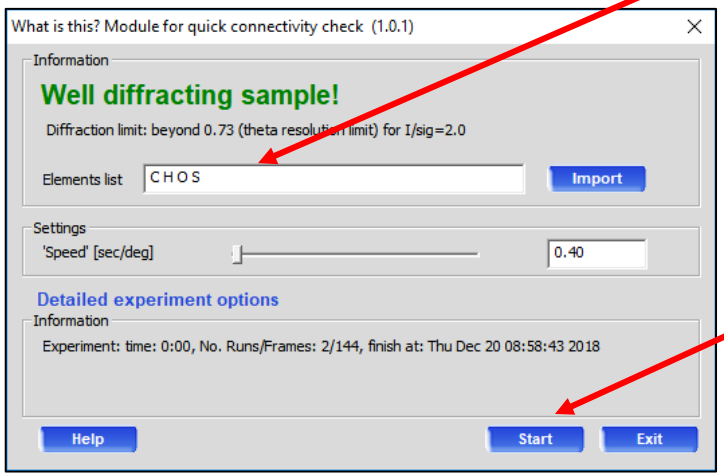
Setting up a ‘ What is this‘ experiment?



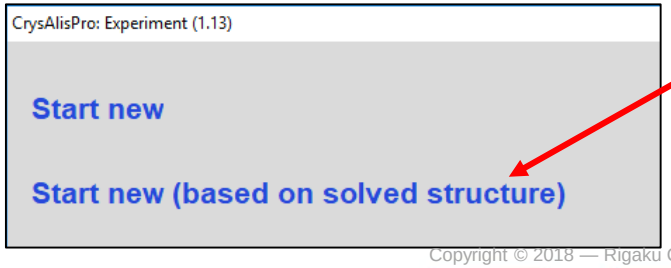
1. Click ‘What is this?’



2. Enter the expected chemical formula or at least the chemical elements.

3. Click ‘Start’. If a good structure with R_1 < 15 % is obtained, then you can use it to plan for the full experiment, should you decide to do so at this point (see below).

4. For a full experiment, click START/STOP



5. Select this option to calculate a data collection strategy from the structure results.

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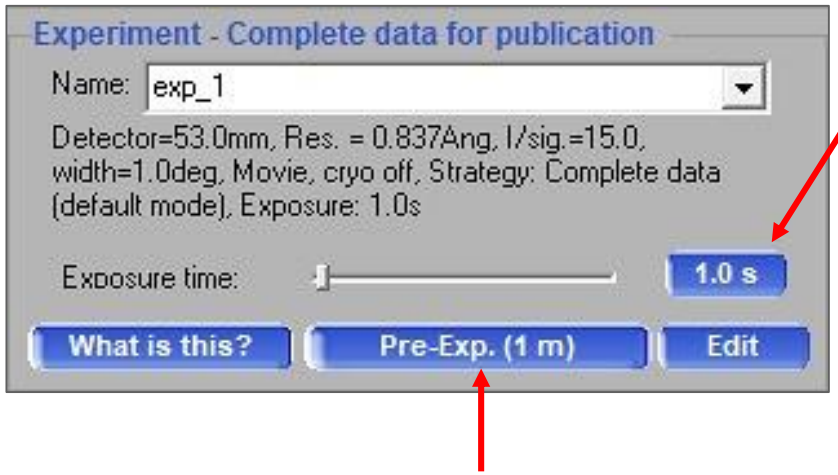
Pre_experiment

The ‘Screening’ step is really a way to have a quick X-ray snapshot of the crystal diffraction (quality, intensity, preliminary unit cell).

A more sophisticated screening is done via ‘Pre_experiment’, whereby a few orthogonal scans, each of 10 frames, are collected. This will yield a better indexing, orientation matrix and <I/σ(I)> estimate, leading to a more accurate data collection strategy.

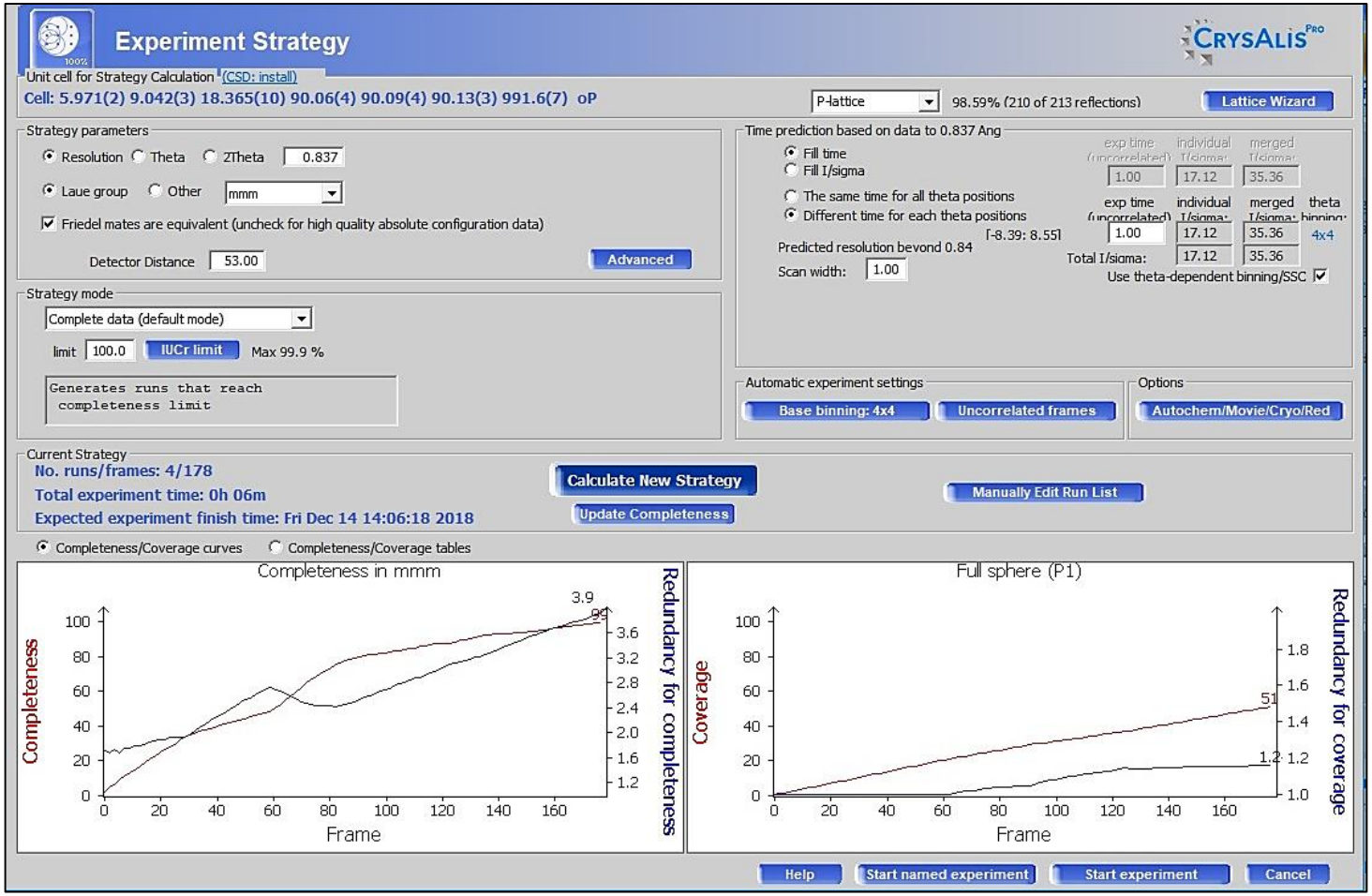


1. This exposure time is selected by CrysAlis^{Pro} using the <I/σ(I)> estimated upon screening. Click on the button to change the exposure time.
   Note that this number is a rate per degree. Since the frame width is 0.5°, an exposure time of 0.5 second per frame would actually be used.

2. Click ‘Pre_Exp’. In parenthesis, the estimated time for the pre_experiment is shown. Concurrent indexing takes place during the pre_experiment. Check the indexing results in the right hand side panel, as described on Page 9.

The Strategy Module Opens . . .

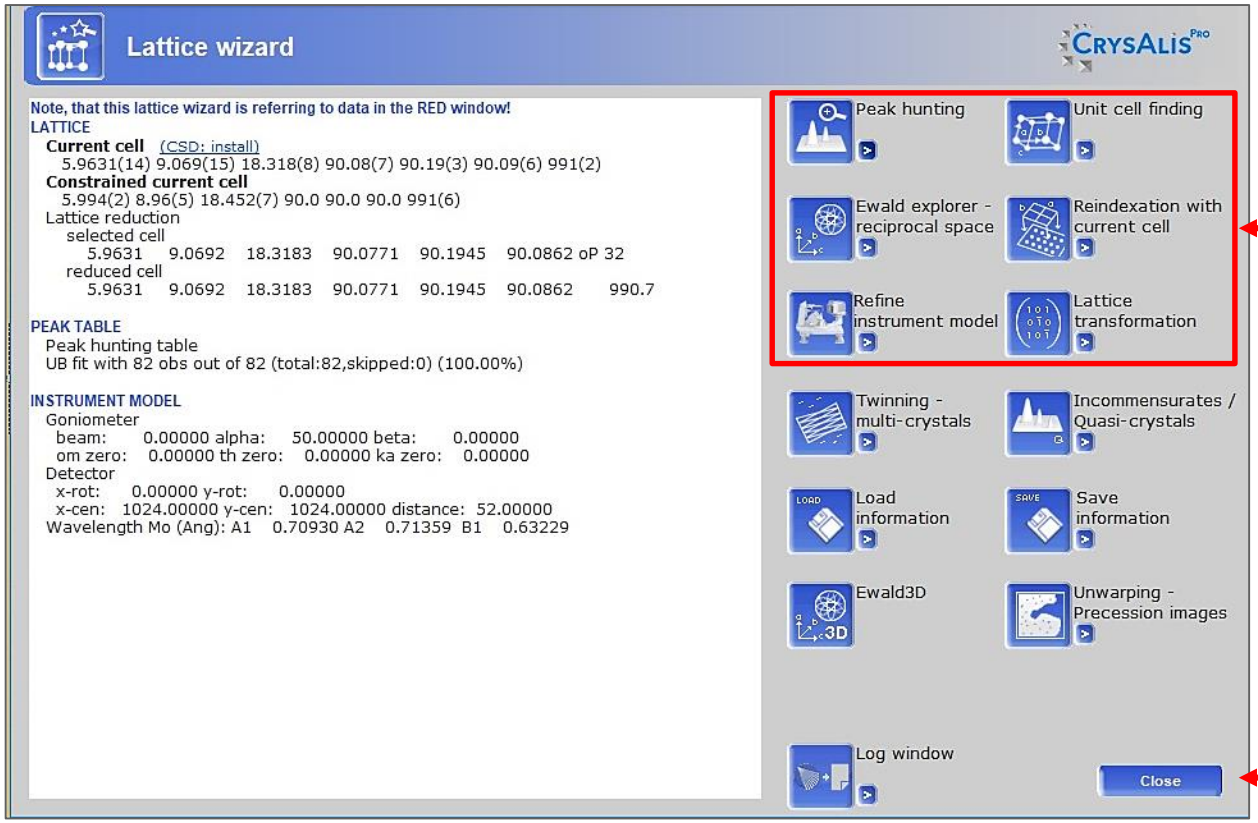
And a strategy is automatically calculated, but you may want to change some of the parameters …



Open the Lattice Wizard to Check/Improve Indexing

Unless 90-100% reflections are already indexed, it is recommended to re-index the diffraction pattern manually. Open the Lattice Wizard from the strategy interface by clicking on 

at the top right corner of the interface.



Use these functions (next page)

Click on ‘Close’ when finished.

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Peak Search with User Settings

- **Traditional peak hunting**: Each frame pixel is scanned and compared to the 'Threshold'. If above the 7x7 average is checked to be higher. If yes, some further 2D peak shape tests are made and the pixel is accepted if passed. These are the 2D peak locations. In a further algorithm step, these 2D locations are assembled to 3D peaks. This method works well when no background is present (for high background. use the background subtraction). Its benefit is speed, but the 3D assembled peaks are not as accurate as the 3D profiles.

- **Automatic background and threshold detection**: This is an extension of the 'Traditional peak hunting' in that it finds automatically the approximate background and according to the findings adjusts the thresholds. For speed, some shortcuts are made.

- **Smart peak hunting**: This method was first developed for proteins and is useful for weaker data. It uses local background to cope with the situation of small peaks on strongly varying background. Slower than traditional and automatic peak hunting.

- **3D peak extraction**: This method is based on the dc proffit 3D profile learning routines. It locates potential peaks and then extracts 3D profiles from it to compute an accurate 3D centroid. The slowest method, but excellent profile position information and better at extracting reflections from weakest data.

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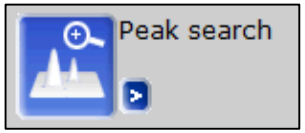
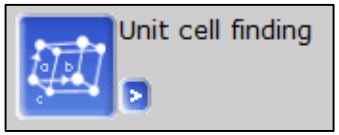
Lattice Wizard Manual Functions

(Big button = auto function, small arrow button = options)

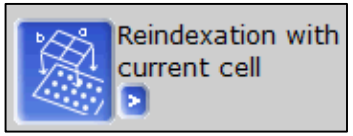


Peak search



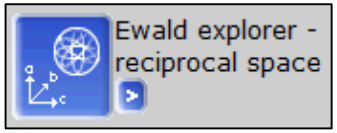
Find the best unit cell compatible with current peak table

Refine parameters and instrument model to improve the fit



Index with current cell to improve the fit

Potential fits to other Bravais lattices from the same primitive unit cell



Access to a reciprocal space viewer with some additional features

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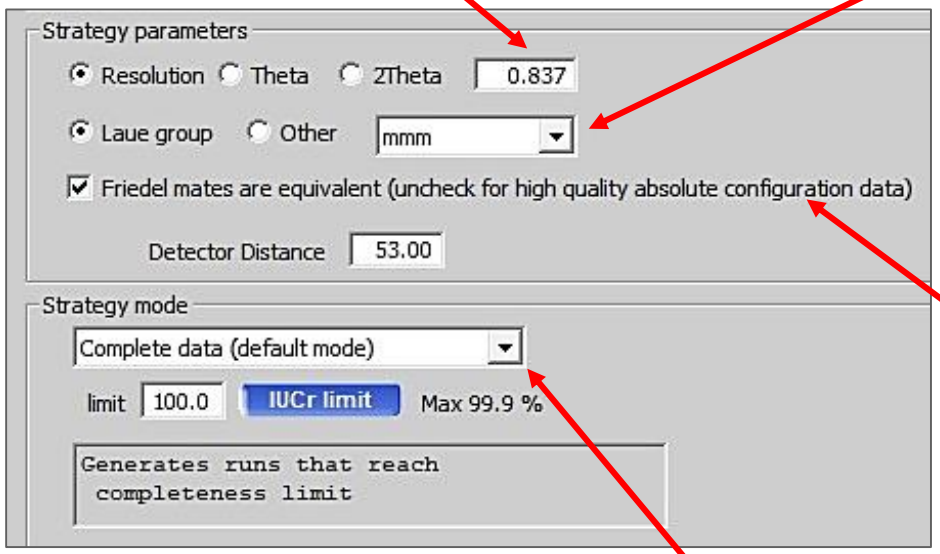
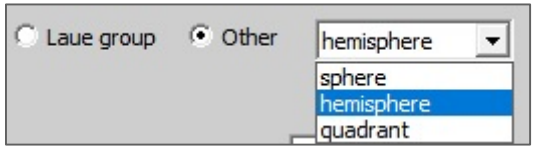
Adjust the Strategy

1. When closing the Lattice Wizard, you are asked whether an updated strategy should be calculated, Click on YES.

2. Optional: adjust the resolution

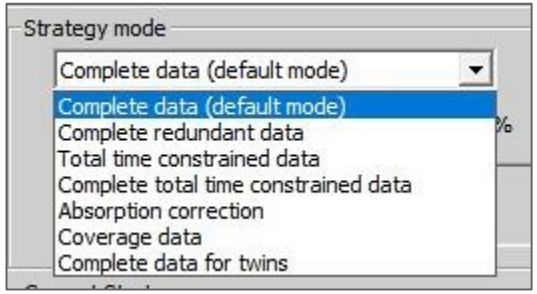


3. Optional: select the desired Laue group if different from CrysAlis^{Pro} solution. You can also select a sphere, hemisphere or quadrant from the 'Other' pull-down menu in the option.



4. Check/uncheck to make the Friedel mates equivalent/merged.

5. Optional: change the Strategy mode according to the type of experiment planned. An explanation of the strategy type selected is given in the panel underneath.



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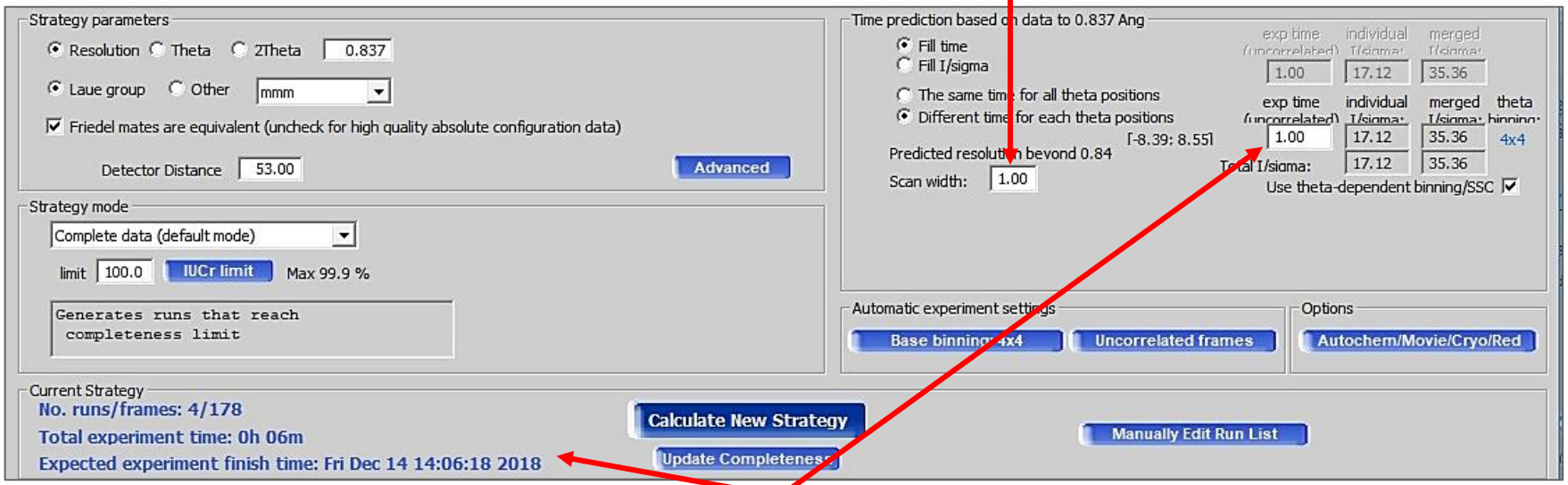
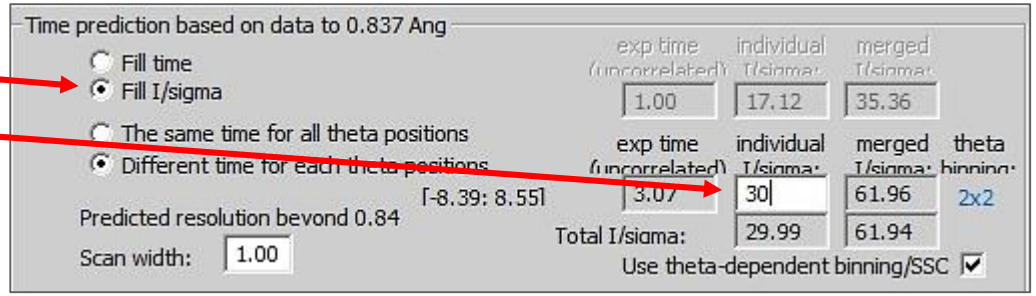
Adjust the Strategy (continued)

6. Optional: change the rotation width if you want finer or wider slicing. Don't forget to change the exposure time accordingly to keep the same rate per degree.



7. Optional: the exposure time was calculated by CrysAlis^{Pro} based upon a user-input target <I/σ()> (15 is a good target for medium to well-diffracting crystals). You can change it at will, and the data collection time will be updated automatically.

8. Optional: click on 'Fill I/Sigma' and enter a new <I/σ(I)> target. The exposure time in the box on the left will automatically be updated.



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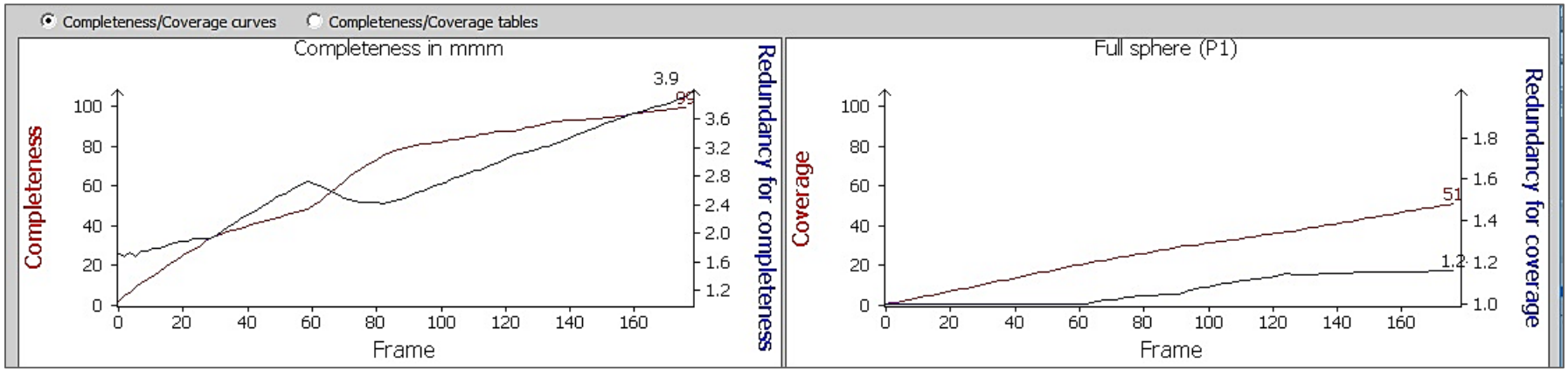
View the Predicted Completeness/Redundancy

View as a graph



View as a table

Completeness/Coverage curves Completeness/Coverage tables

Res		Completeness (under Laue symmetry) #Data	#Theory	%	Redundancy	Coverage (under P1) #Total	#Data	#Theory	%	Redundancy	#Total
18.36-	1.94	100	107	93.46%	4.1	406	424	706	60.06%	1.2	516
1.94-	1.49	107	107	100.00%	4.5	482	444	706	62.89%	1.2	523
1.49-	1.30	107	107	100.00%	5.3	571	505	706	71.53%	1.2	610
1.29-	1.17	107	107	100.00%	5.2	552	439	706	62.18%	1.2	507
1.16-	1.08	107	107	100.00%	4.7	498	389	706	55.10%	1.2	474
1.08-	1.00	107	107	100.00%	3.9	420	339	706	48.02%	1.2	398
1.00-	0.96	107	107	100.00%	3.7	394	313	706	44.33%	1.2	362
0.95-	0.91	107	107	100.00%	2.8	296	260	706	36.83%	1.1	276
0.91-	0.87	107	107	100.00%	2.6	276	252	706	35.69%	1.0	260
0.87-	0.84	113	113	100.00%	2.6	291	240	706	33.99%	1.1	260
18.36-	0.84	1069	1076	99.35%	3.9	4186	3605	7060	51.06%	1.2	4186

theta settings: 1, abs minimum theta: 8.39, abs maximum theta: 8.39, max resolution: 0.743

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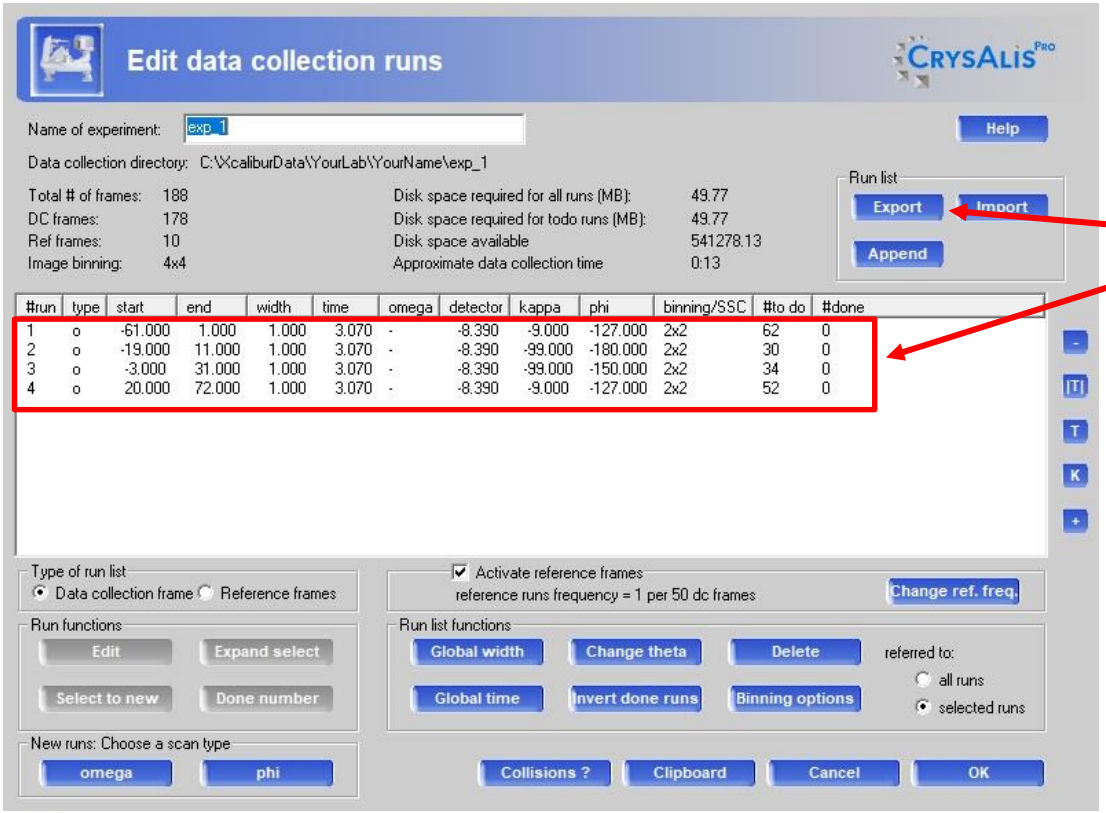
Calculate a New Strategy and review the Scans

1. Once you have adjusted all the parameters, click ‘Calculate New Strategy’.

2. Click ‘Manually Edit Run List’ if you wish to see the calculated scans.



All scans are listed and you can export the strategy.

3. Click OK when finished.

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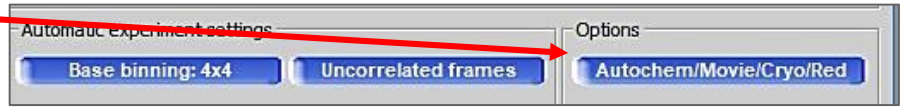
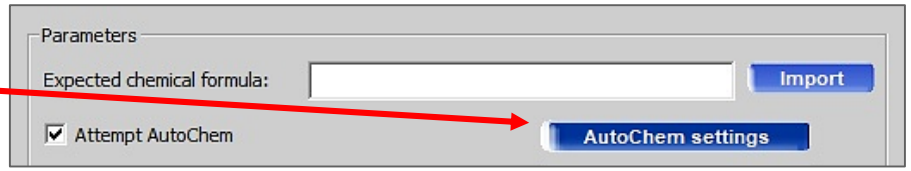
Parameters for Automatic Structure Solution

1. Open the ‘Options’ dialog.

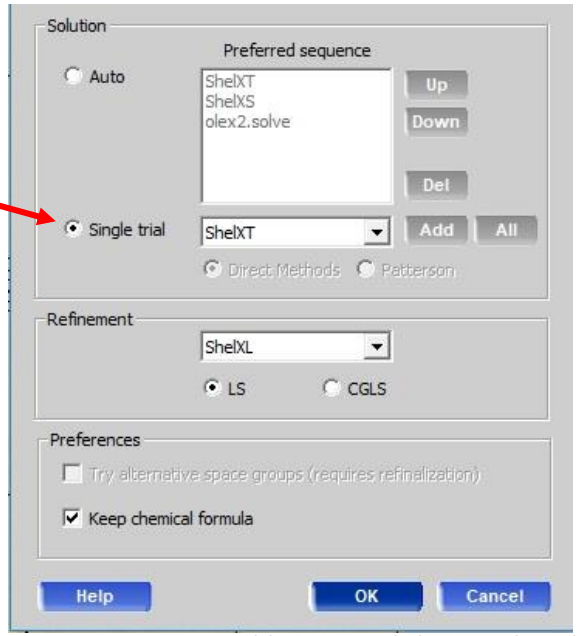


2. Open the AutoChem settings dialog.



3. Select ‘Single trial’ and ShelXT from the pull-down menu.
   AutoChem will only run ShelxT when attempting structure solution during data collection.

4. Click ‘OK’.



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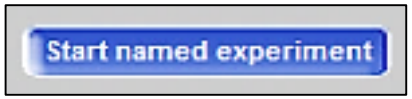
Start Data Collection

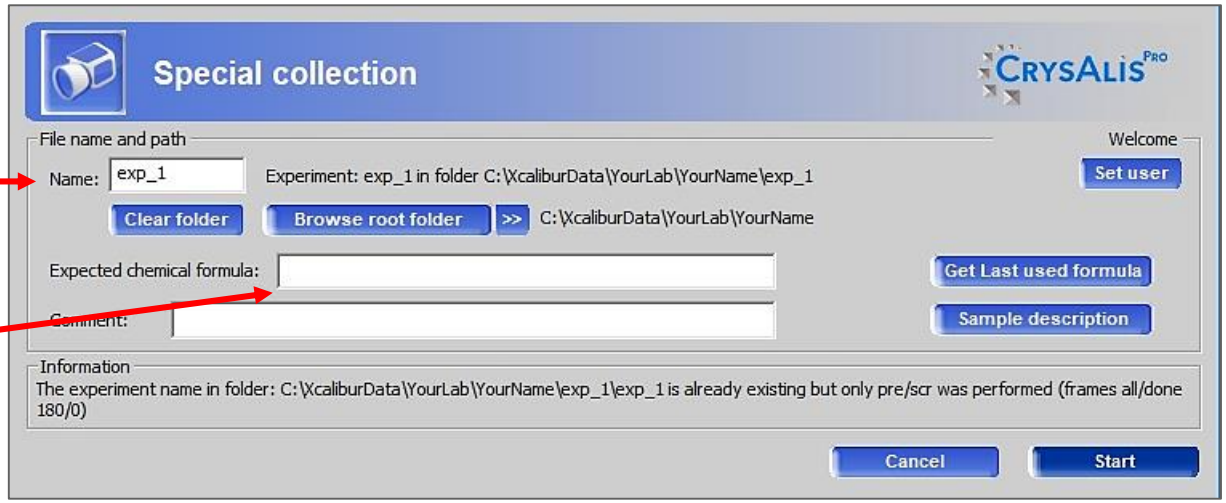
5. Check the predicted finish time for the experiment.

6. Click on 'Start Named Experiment to access the 'Special collection' dialog.



7. Enter a name for the experiment.

8. Enter the chemical formula or chemical content, if known.



9. Click 'Start'. 60 pictures, one every 6º along phi, will first be taken of the crystal. Then, concurrent data reduction and structure solution will take place during data collection.

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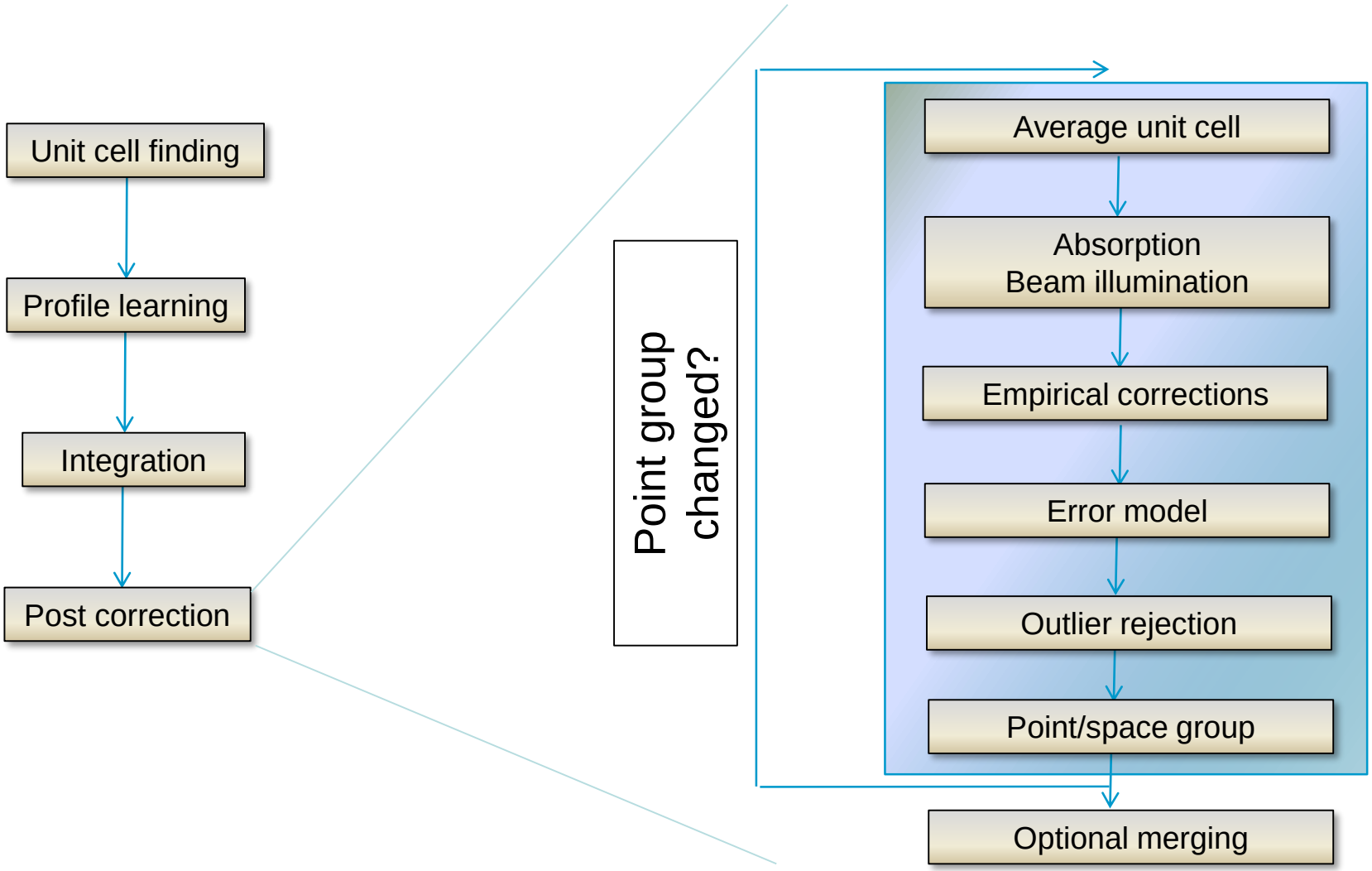
Data Processing Flow Chart



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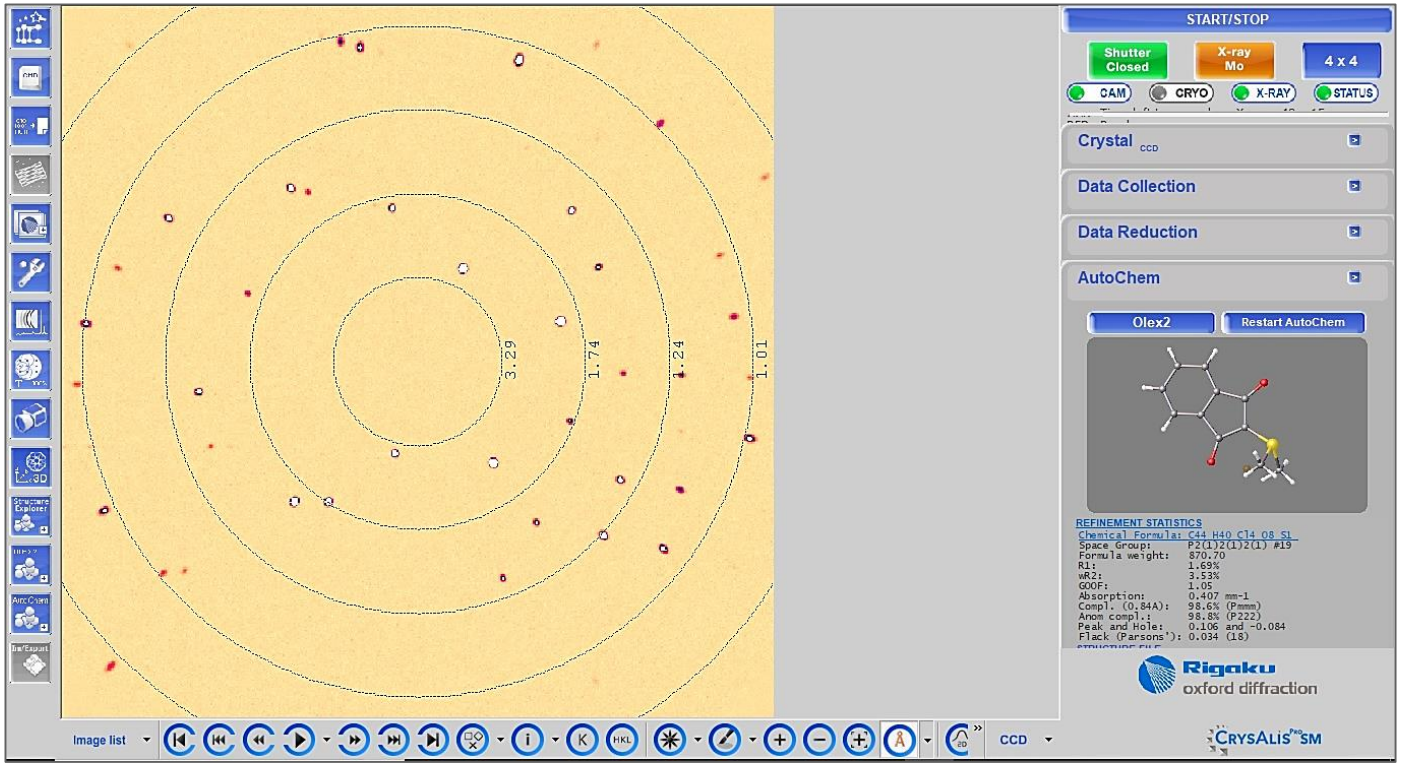
End of Data Collection



At the end of data collection, the interface should look like the above:

- The last diffraction frame is displayed in the main panel
- The current structure along with the current refinement statistics are displayed in the right hand side panel.
- A summary of data reduction statistics can be viewed by clicking on the 'Data Reduction' tab (see Page 26).

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Relying on the Automatic Processing from CrysAlis^{Pro}

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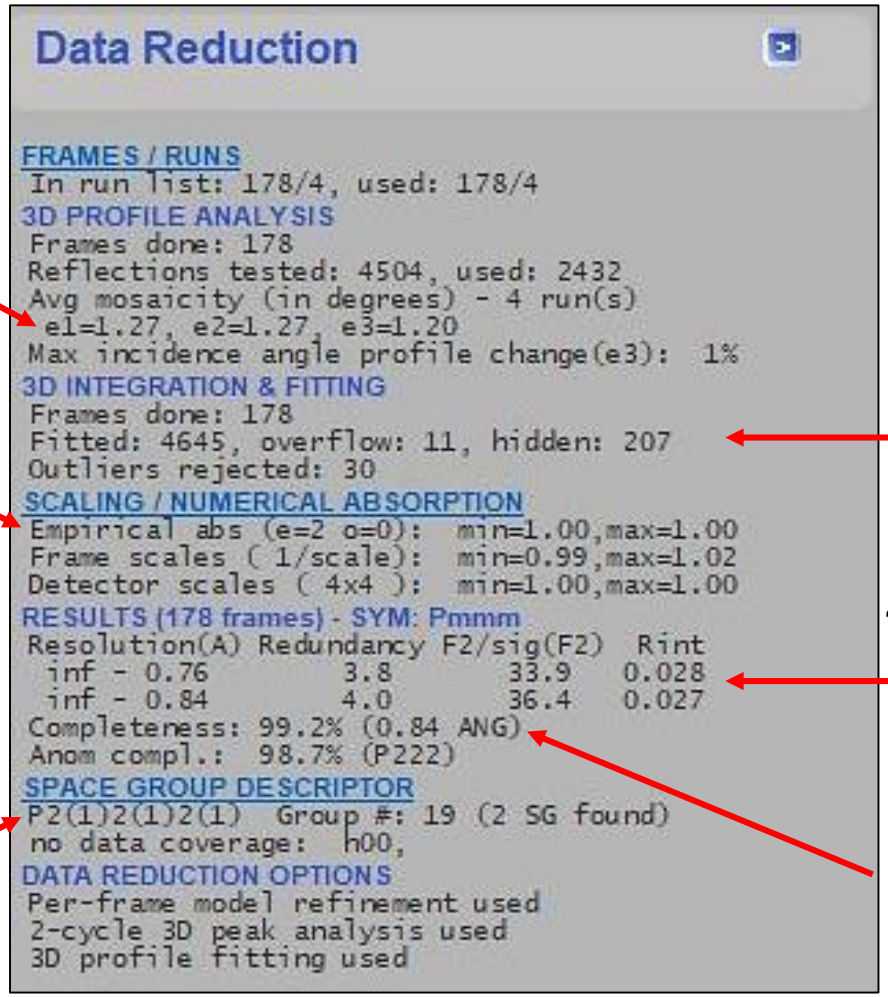
Data Processing Results

1. Mosaicity along a, b and c.

3. Parameters used by CrysAlis^{Pro} for scaling:
   - Absorption harmonics (evens and odds)
   - # frames per scale factor.

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



2. # frames processed and # peaks fitted

4. Overall redundancy, $\langle I/\sigma(I) \rangle$ and R_{int} at 0.837 Å and overall.

5. Overall completeness.

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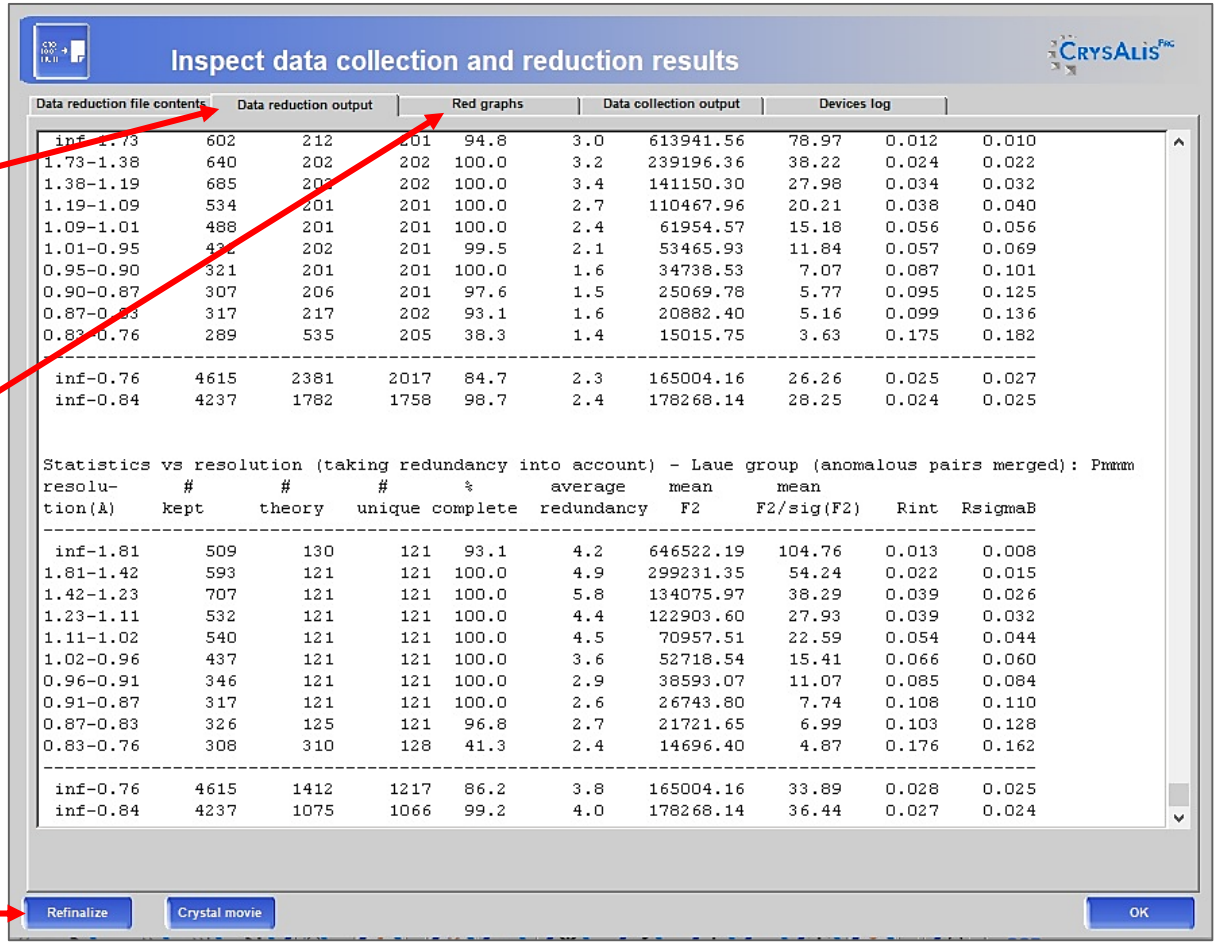
Data Inspection Module

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

2. Check statistics tables

3. Check statistics graphs (see next 2 pages)

4. Click Refinalize to open the scaling options dialog and apply different types of absorption correction.



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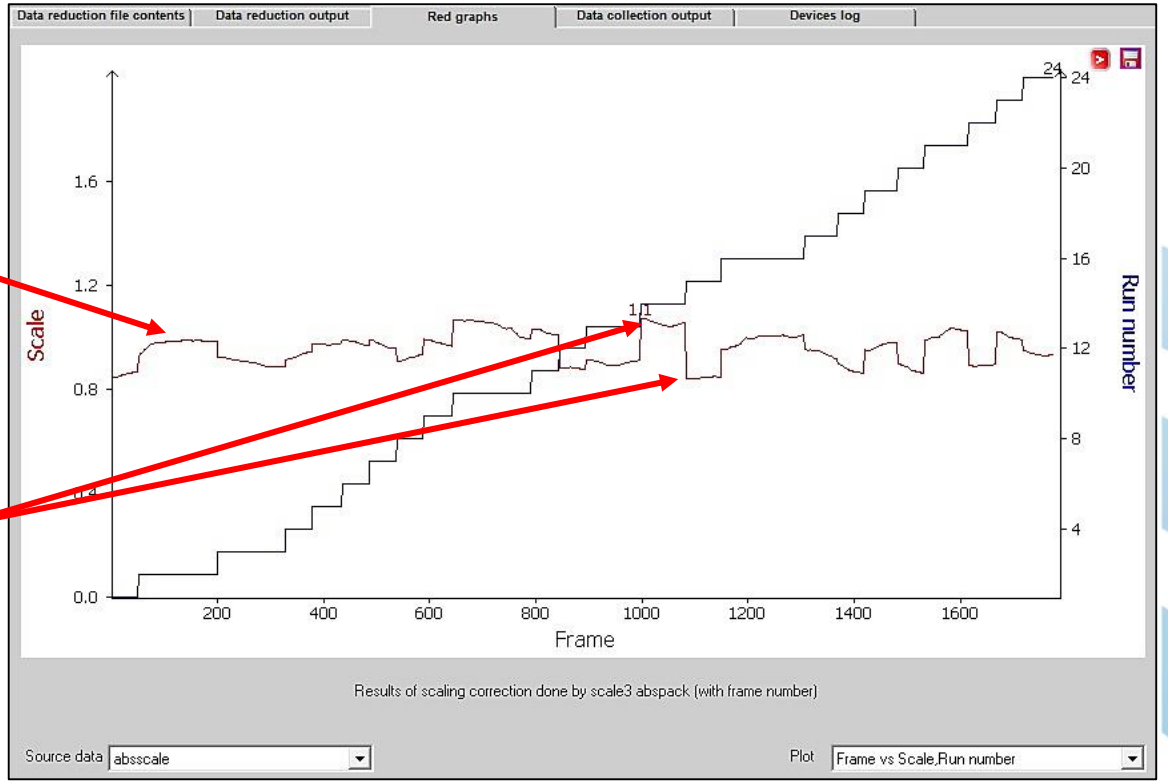
Data Inspection Module – Scale Factors per Frame

1. The 1st useful plot to look at is that of the scale factors per frame. It should always be smooth, i.e. not jittery as scale factors (dependent on the average diffraction intensity per frame) should remain almost identical for consecutive frames.

2. The values are likely to change for different runs, as different orientations of the crystal will vary the average intensity per frame and thus the scale factors.



3. Scale factors between runs will vary more for anisotropic crystals (needles, rods, plates) than for isotropic crystals (blocs, cubes, rounder crystals), as different amounts of crystalline matter are in the beam for different crystal orientations. For the same reason, for anisotropic crystals, scale factors may slowly vary over wide ranges of rotation … but remember, this variation must remain smooth.

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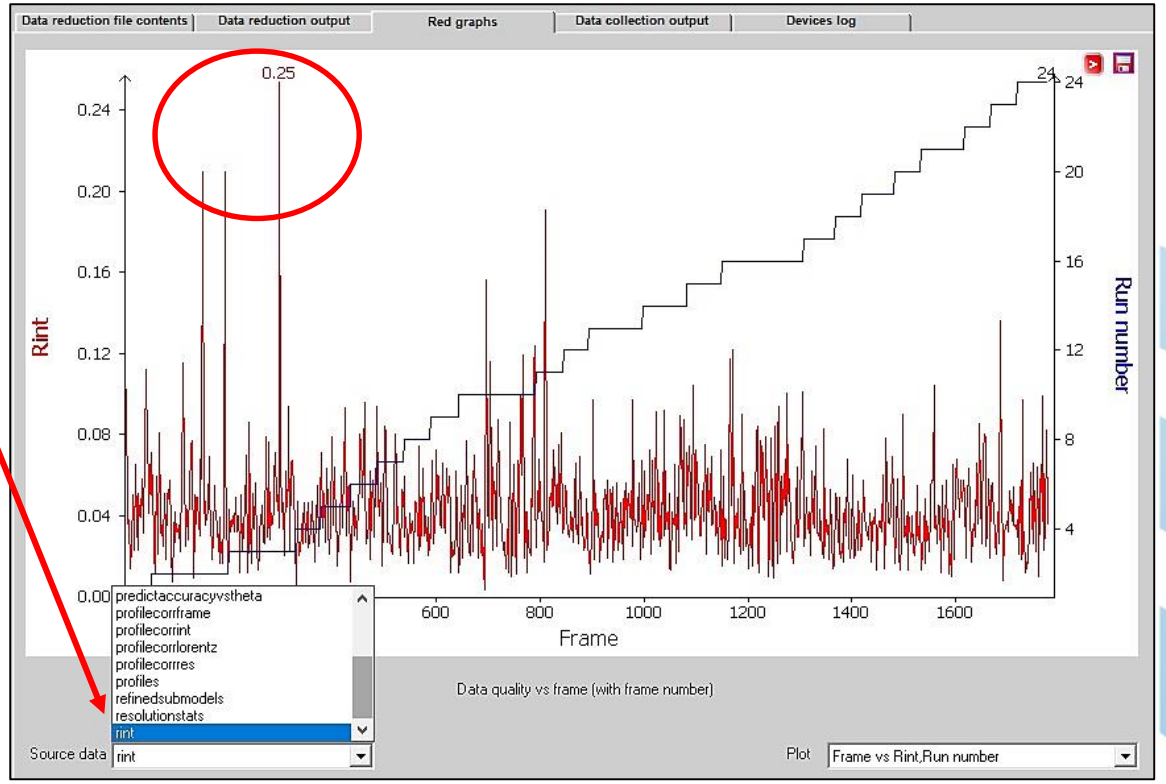
Data Inspection Module – R_{int} per Frame

1. The 2nd plot to look at is that of the R_{int} per frame. Open the pull-down menu at the bottom of the interface, scroll down and select 'Rint'.

2. This plot is always jittery, as shown on the picture. You only want to identify obvious outliers, i.e. single frames whose R_{int} will be grossly greater than its neighbors'. In this example, the 3 frames within the red circle may barely be considered outliers.



3. If single frames or parts or full runs are identified as bad according to their scale factor or R_{int} values, you may reject them by filtering them out (see page 31). However, ensure that data completeness does not go below the minimum required by the IUCr for publications as a result.

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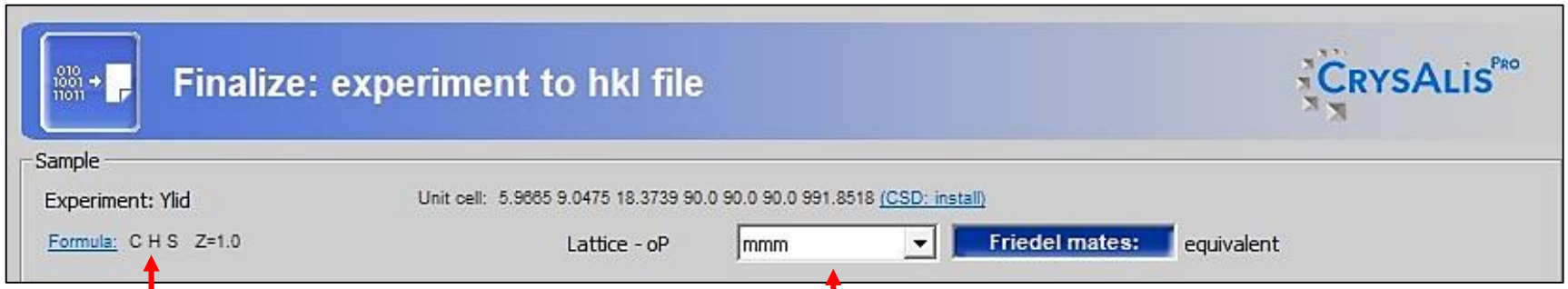
Refinalize Module - Crystal Data



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

2. Ensure the correct Laue group is shown.

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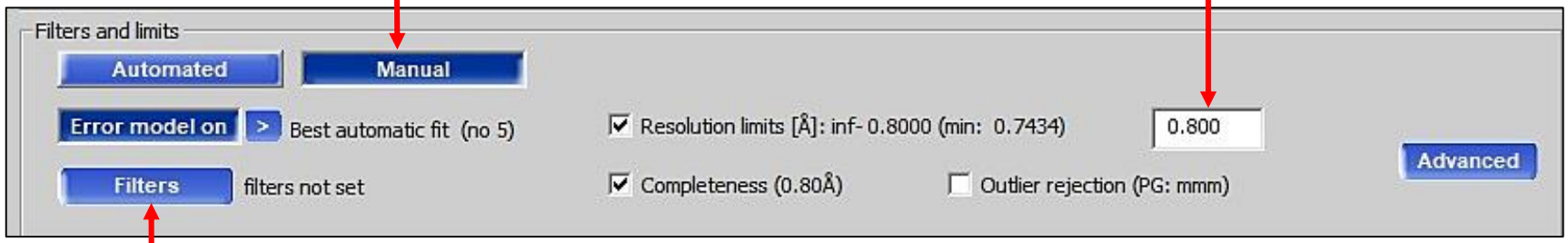
Refinalize Module – Filters and limits

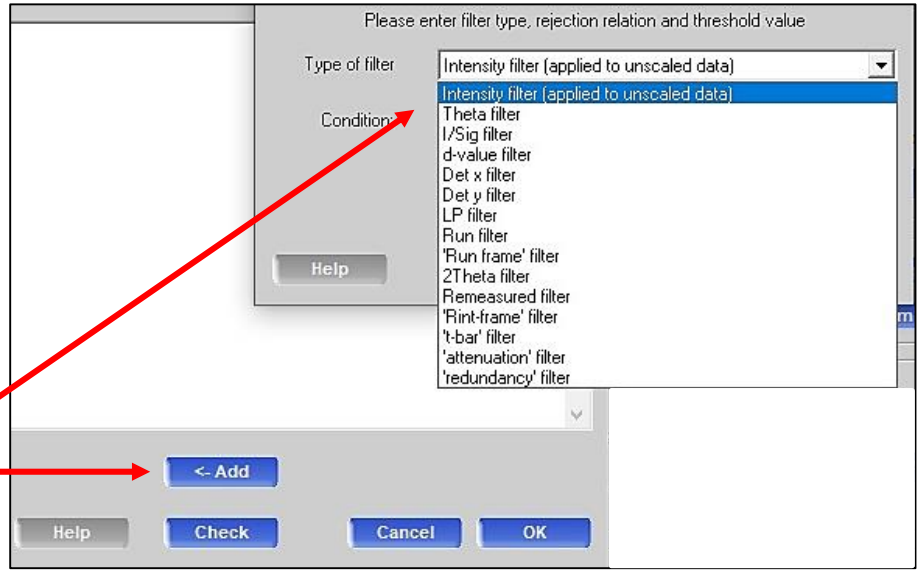
1. Click here to expand the manual filters panel.

2. Check this box and enter a resolution cutoff, if needed.



3. Click here to open the filters dialog and eliminate bad frames.

4. In the filters dialog, click '< Add' and expand the list of available filters.



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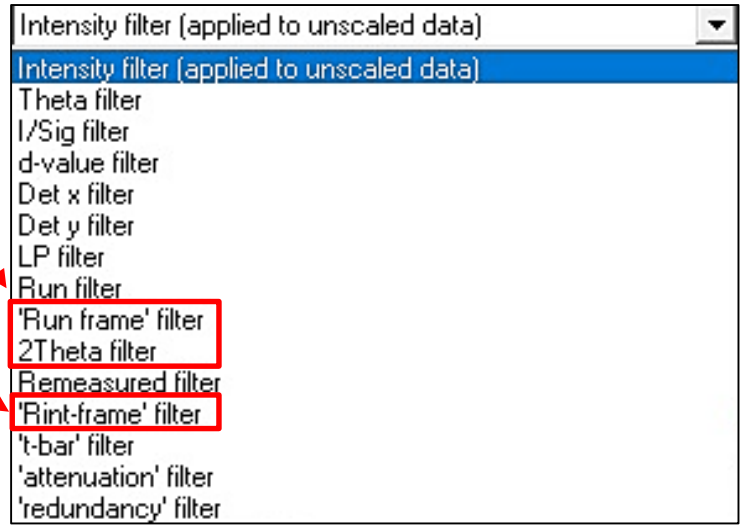
Refinalize Module – Filters and limits (2)

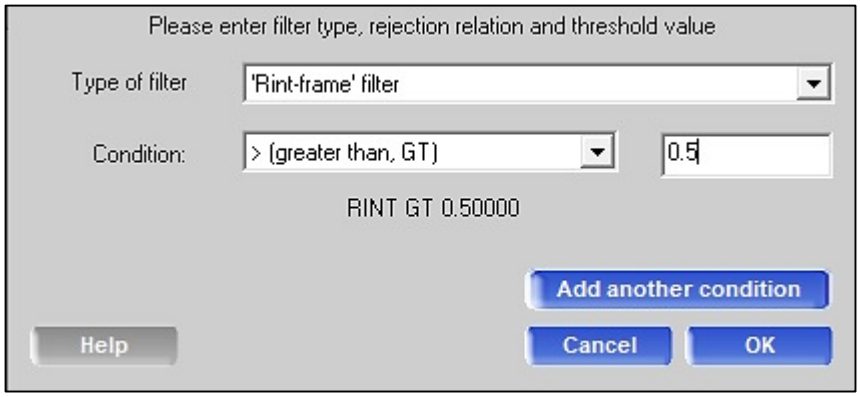
5. The most commonly used filters are:
   - ‘Run filter’: removes entire runs from scaling.
   - ‘Run frame filter’: allows to define a range of frame within a run to be removed from scaling.
   - ‘Rint-frame filter’: single frames with R_{int} > threshold are removed from scaling.



For instance, this picture shows how single frames with R_{int} > 50% would be removed:

- ‘Rint-frame filter’ selected.
- Condition is ‘greater than’
- Threshold is 0.5, i.e. 50%. The threshold must be entered as a fraction.

Upon clicking ‘OK’, that filter is added to the list. Several filters may be used at once.



6. Run Refinalize by clicking ‘OK’ at the bottom of the dialog and check for best R_{int} and $\langle I/\sigma(I)\rangle$.

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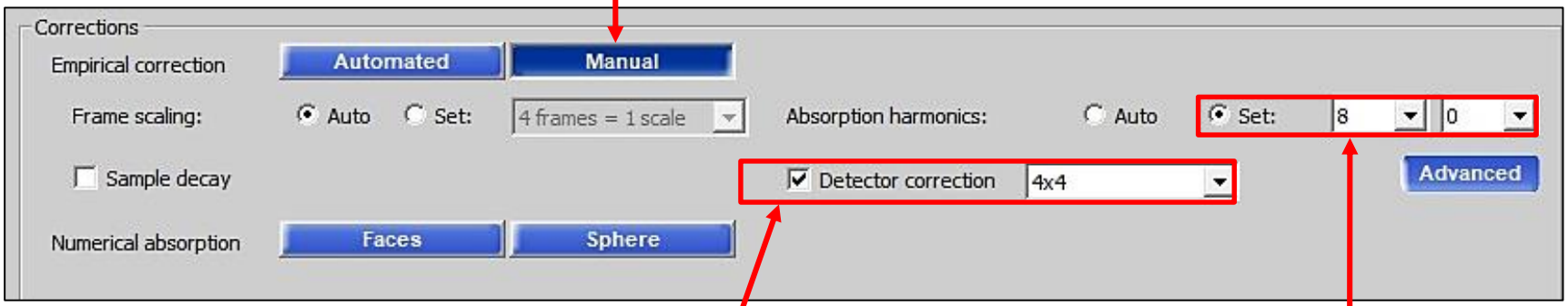
Refinalize Module – Absorption Correction

1. Click here to access the empirical absorption correction options.



2. Optional for mediocre data sets: check this box and vary the array dividing up the detector face into smaller areas for local scaling. Check for best R_{int} and <I/σ(I)>.

3. Optional for mediocre data sets: select 'Set' and incrementally increase the coefficients for the even & odd orders of the spherical harmonics. Check for best Rint and <I/σ(I)>.

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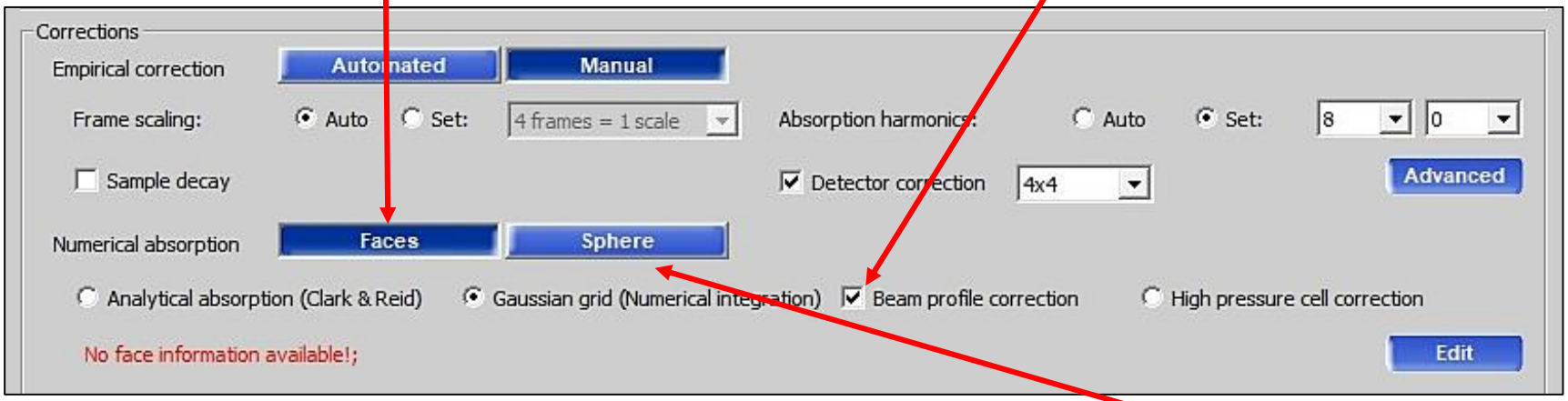
Refinalize Module – Absorption Correction (2)

1. Click here to apply a face-based numerical absorption correction. However, you must first create a 3D model of the crystal via face indexing, provided crystal pictures were taken by the collection program. If so, see next 4 pages.
   If not, skip numerical absorption correction.

2. You may run an Analytical or a Gaussian correction. Check for best R_{int} and $\langle I/\sigma(I)\rangle$. For Gaussian, also check this box. For 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.



3. If a 3D model of the crystal has been created and the crystal is fairly isotropic, click here to run a spherical absorption correction instead of face-based. Check for best R_{int} and $\langle I/\sigma(I)\rangle$ to determine the best absorption correction procedure.

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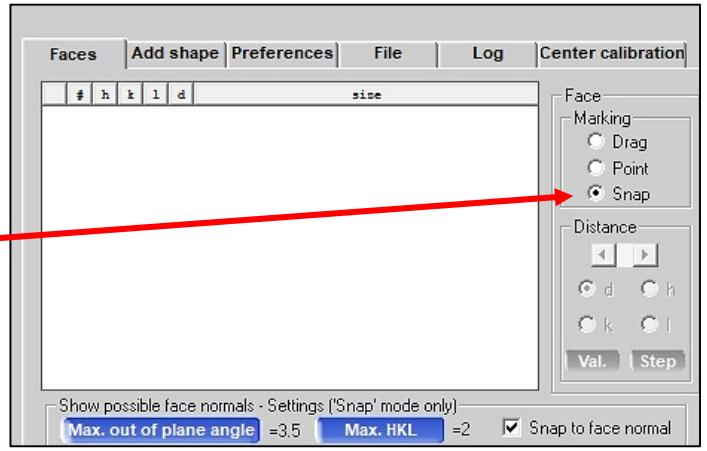
Face Indexing (1)

1. This can be done only if the collection program has taken visible pictures of the crystal. 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 ‘Crystal movie’.

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



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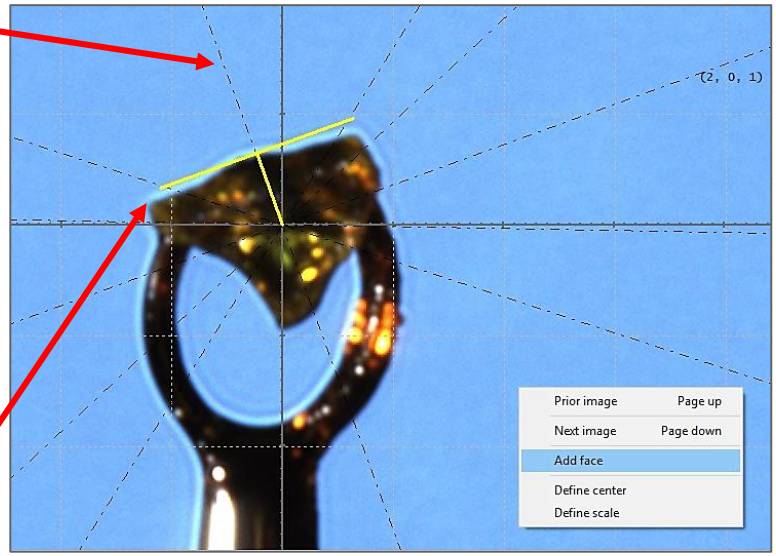
Face Indexing (2)

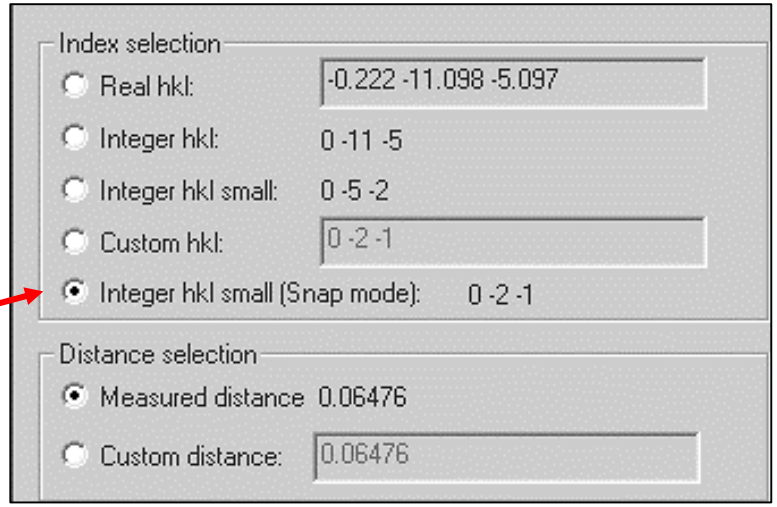
4. Select a crystal view that shows normal directions to crystal faces (doted lines). Different crystal orientations may be displayed using the mouse's wheel or the Prev/Next buttons underneath the frame. The crystal view can also be rotated by +/- 90° or 180° at once using the corresponding buttons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

5. Left-click near the crosshair 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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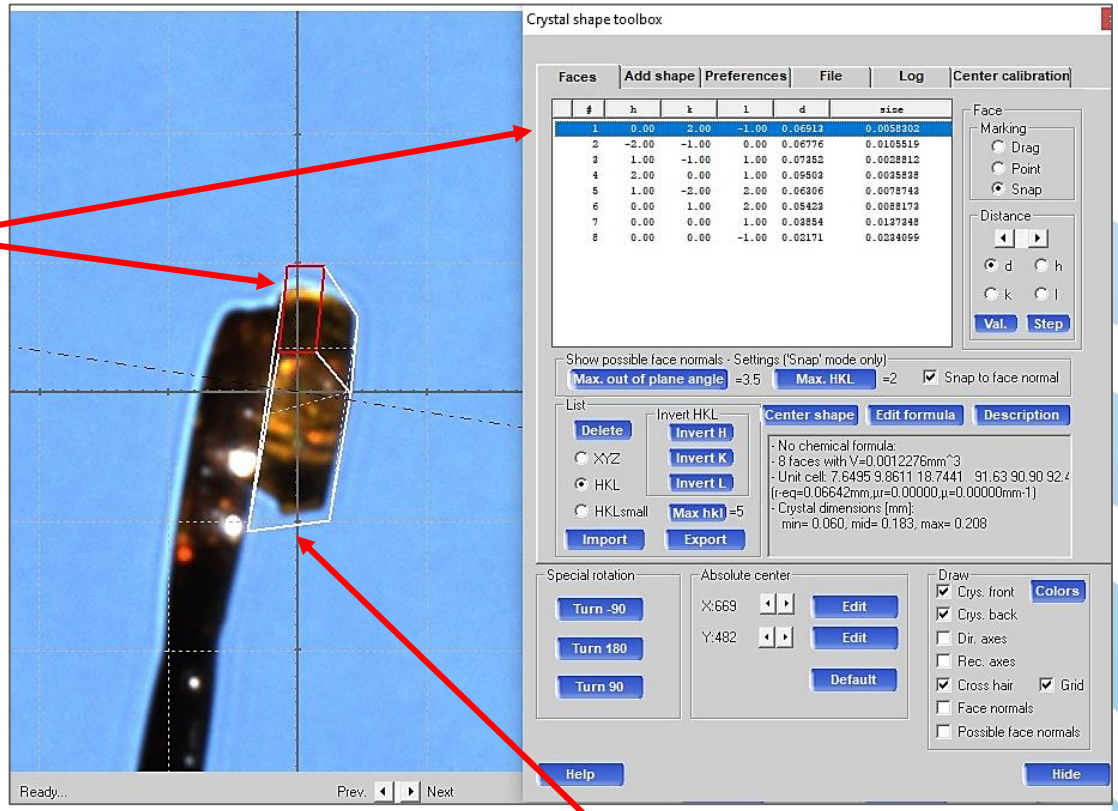
Face Indexing (3)

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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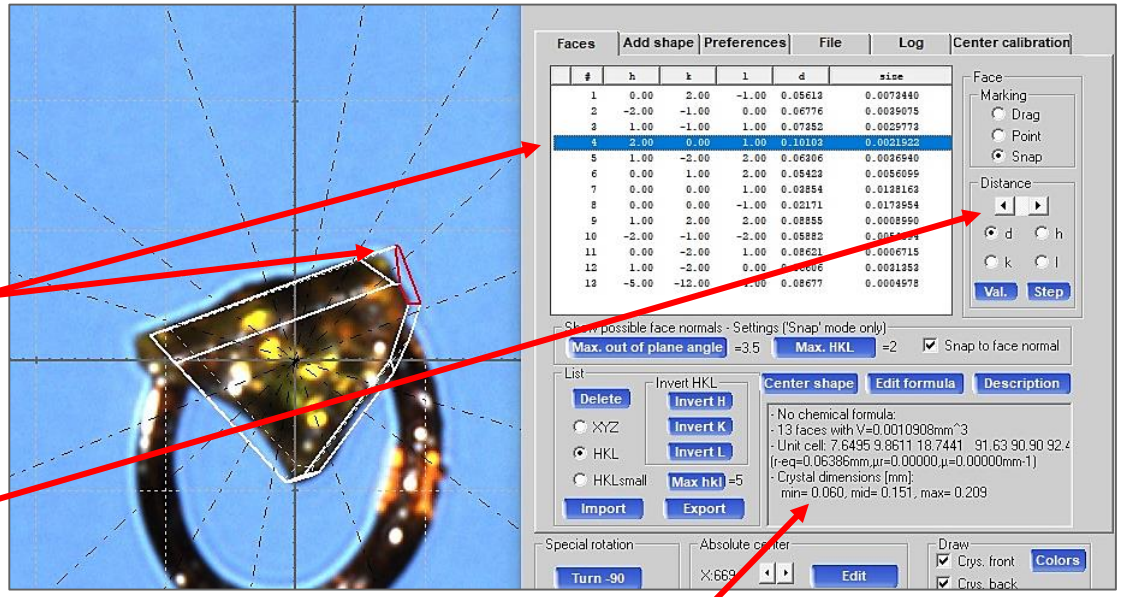
Face Indexing (4)

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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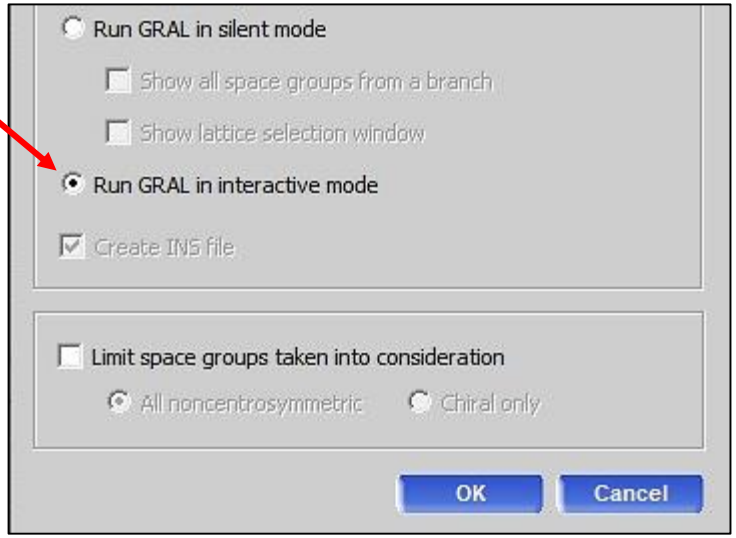
Refinalize Module – Manual Space Group Search

1. Click here to open the space group search dialog.

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



3. Click 'OK'. At the end of the next 'Refinalize' run, the space group search dialog will open.
   Note: GRAL is the equivalent of XPREP.

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Re-processing Data Manually in an Offline Version of CrysAlis^{Pro}

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Reprocess the Data Offline

Previously, we have refinalized data that have been automatically integrated by CrysAlis^{Pro}. The next, and final, section shows how to re-integrate data manually in CrysAlis^{Pro}, using user-input parameters, should you wish to do so.

1. You must open a processing-only instance of CrysAlis^{Pro}. Manual data integration cannot be performed in an online instance. The easiest is to open File Explorer and browse to the desired CrysAlis^{Pro} experiment directory. Then double-click on the experiment_name.par file. CrysAlis^{Pro} will open and display the current status of the experiment.

2. Open the Lattice Wizard and perform manual indexing, if desired (as described on Pages 14-16). Then close the Lattice Wizard.

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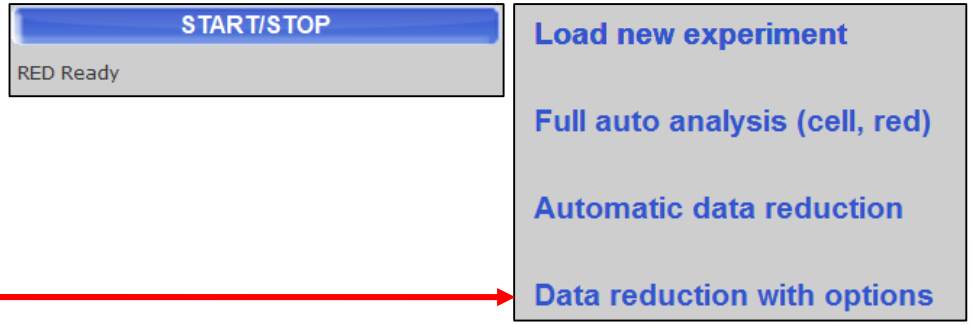
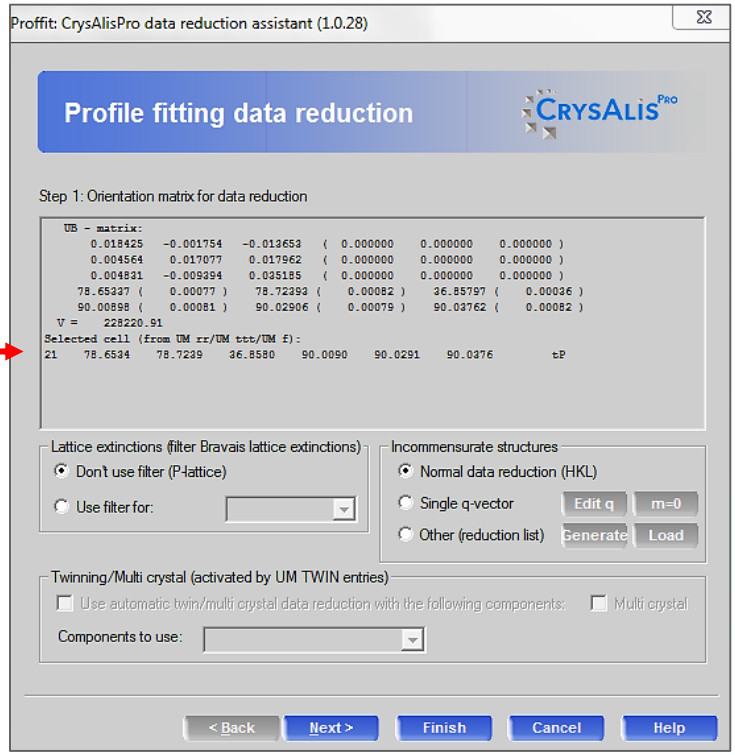
Step 1: Lattice Review

1. Click on ‘START/STOP’.



2. Then select ‘Data reduction with options’.

3. Check unit cell and Bravais lattice are correct.



4. Click ‘Next’.

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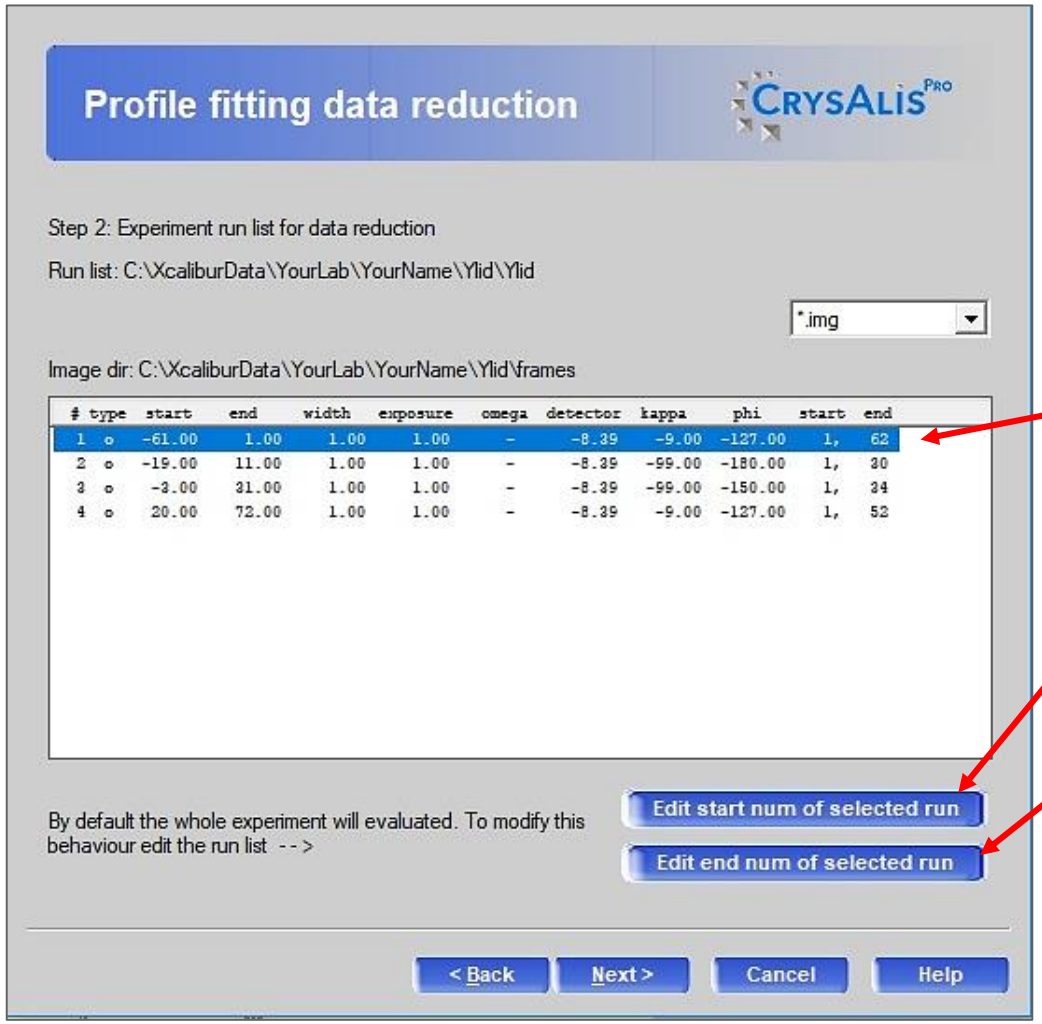
Step 2. Frame Range Selection



If you need to modify the number of frames to use within a scan:

1. Select the desired scan.

2. Click here and enter the number of the 1st frame to integrate.

3. Click here and enter the number of the last frame to integrate.

Note: entering 'zero' for either of these options removes the entire scan from integration.

4. Click Next

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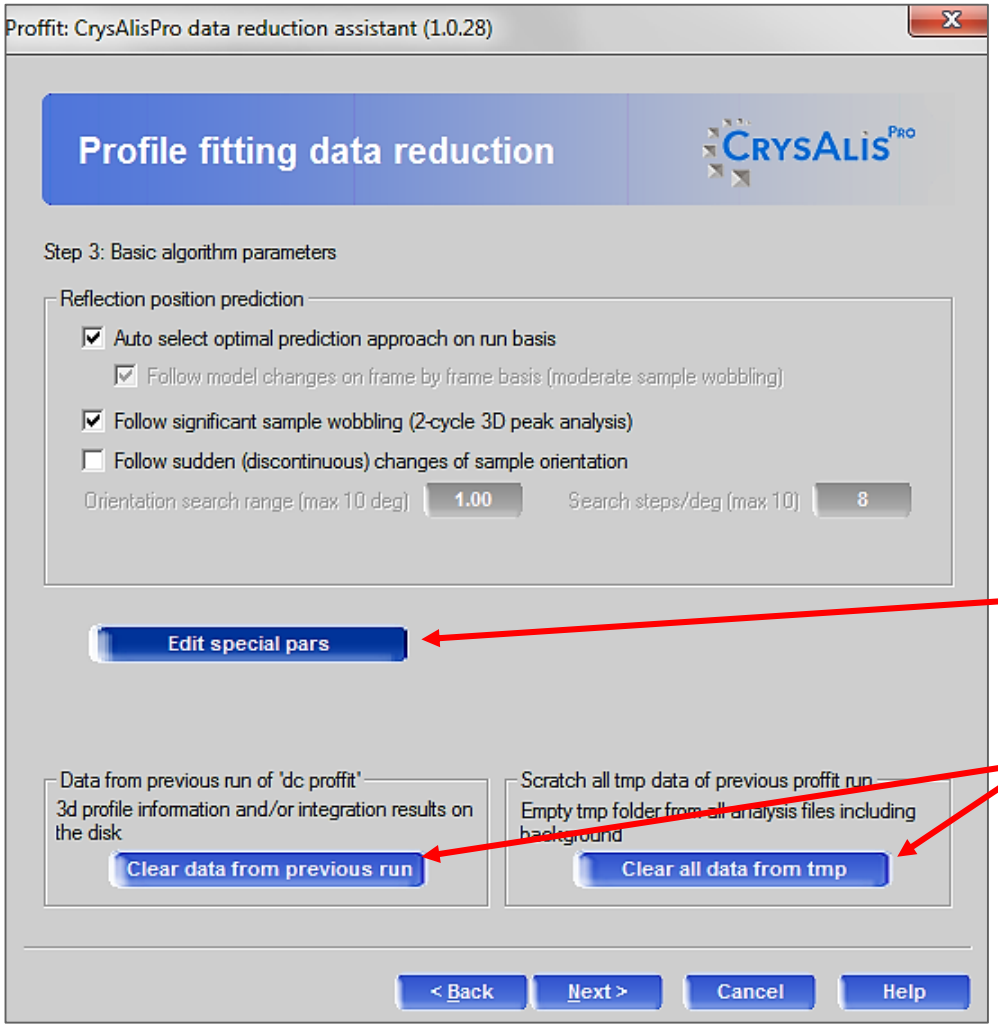
Step 3. Special Parameters



1. Click ‘Edit special pars’ (next page).
2. Click these buttons to delete all info from previous processing runs and start afresh.
3. Click ‘Next’.

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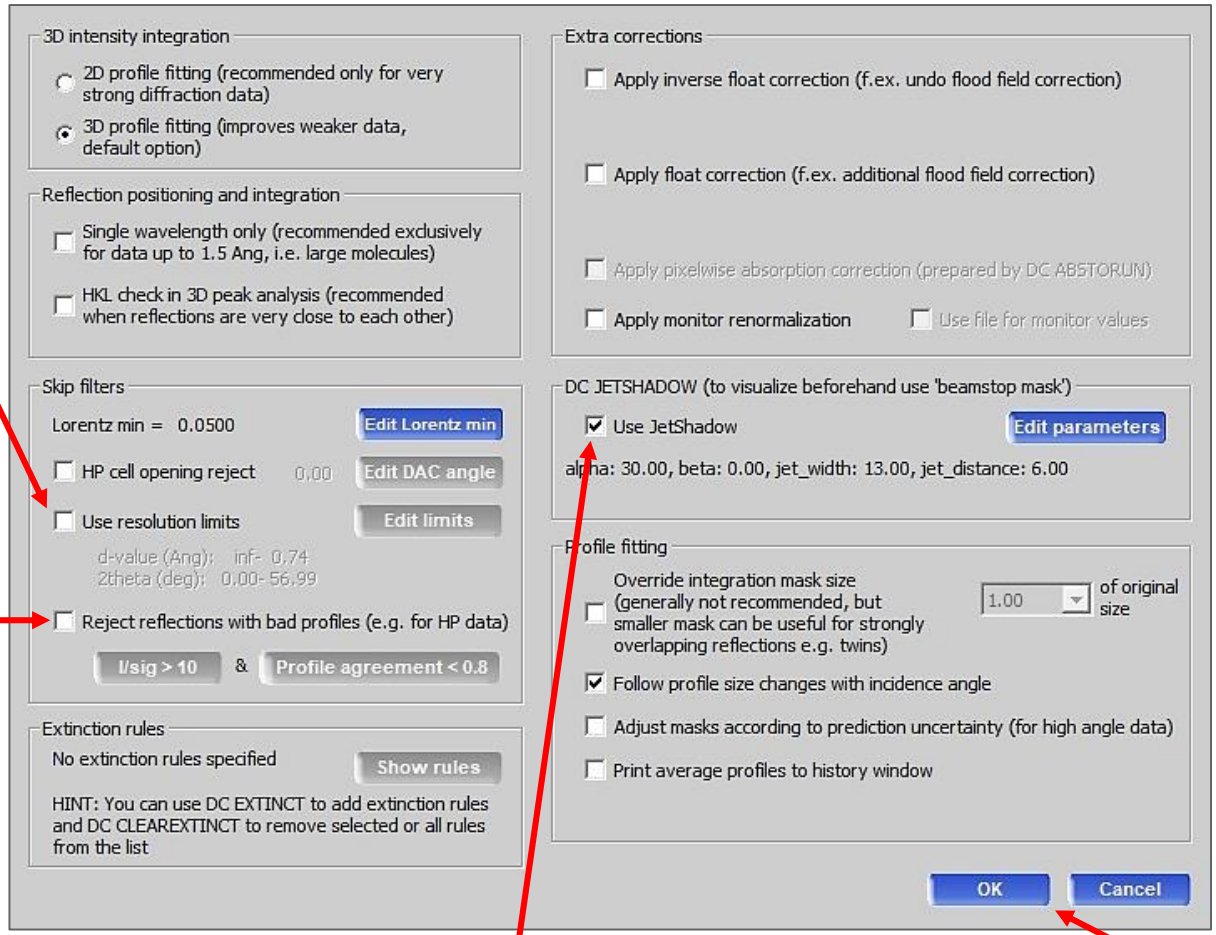
Special Parameters Options

1. To cut off the data set, check this box and enter a resolution limit.

2. To reject the worst shaped reflections or reflections overlapping with 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 only if Cu radiation was used for data collection

4. Click OK.

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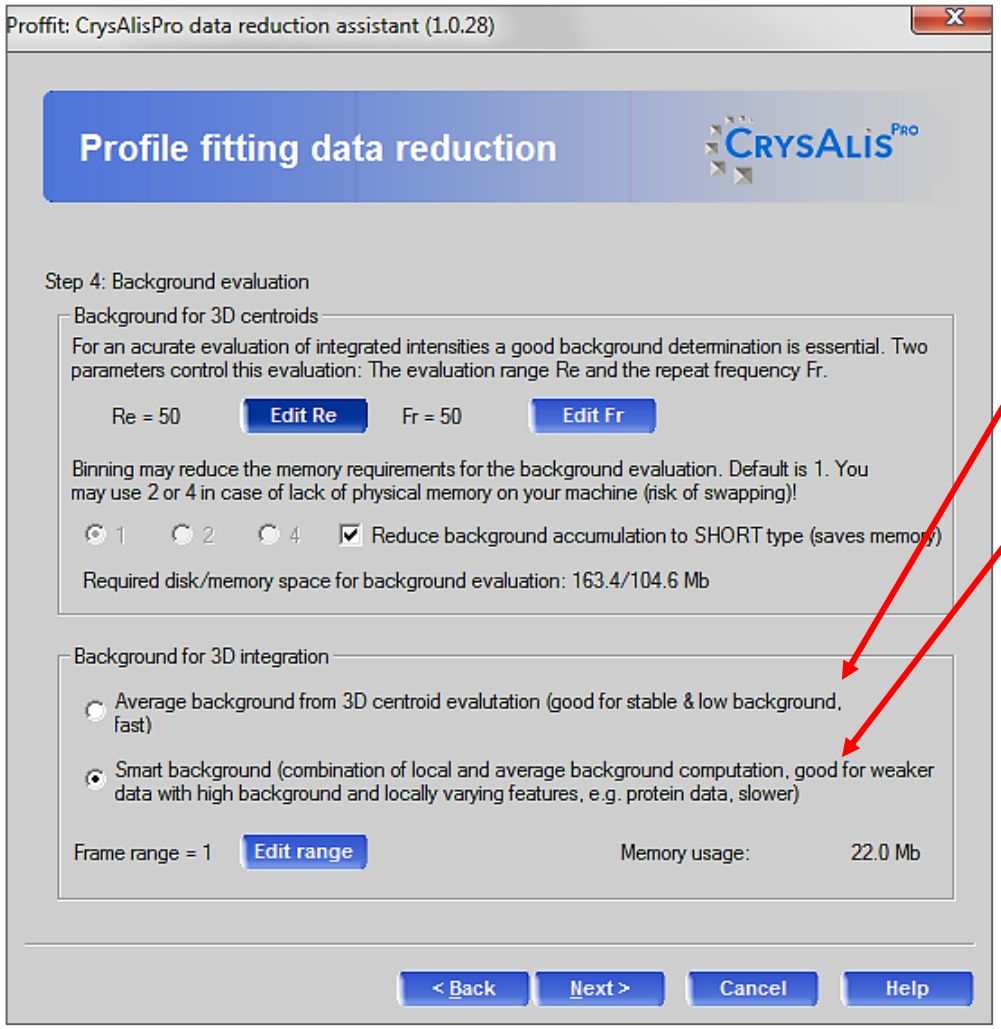
Step 4. Background Subtraction



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 Rint and <I/σ(I)>.

2. Click ‘Next’.

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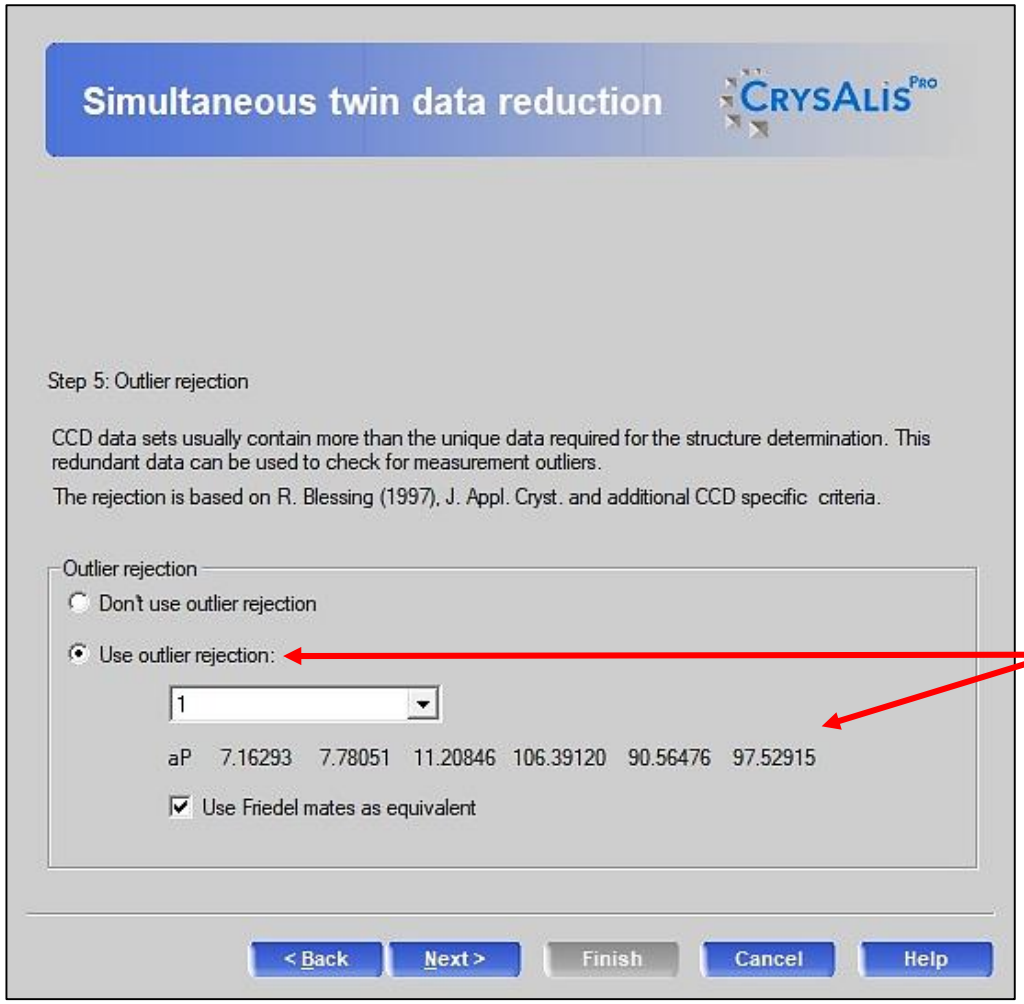
Step 5. Outlier Rejection



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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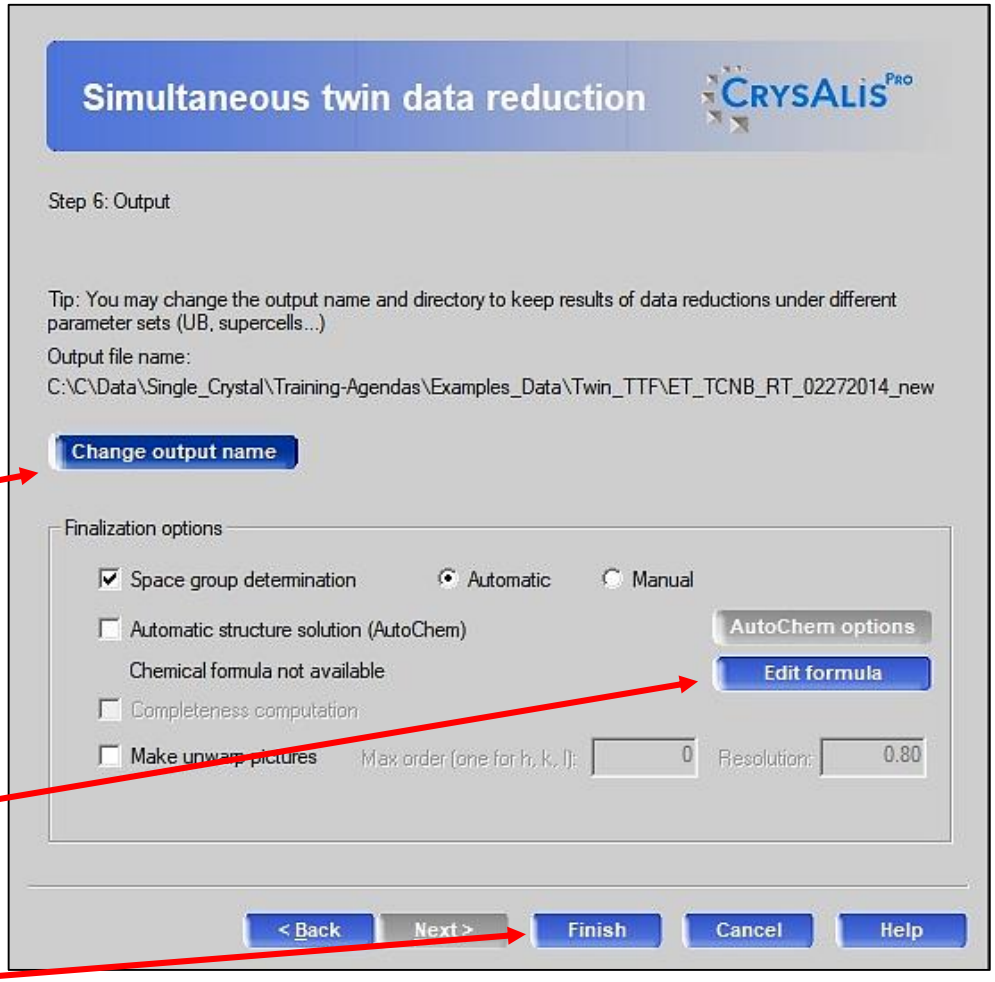
Step 6. Output

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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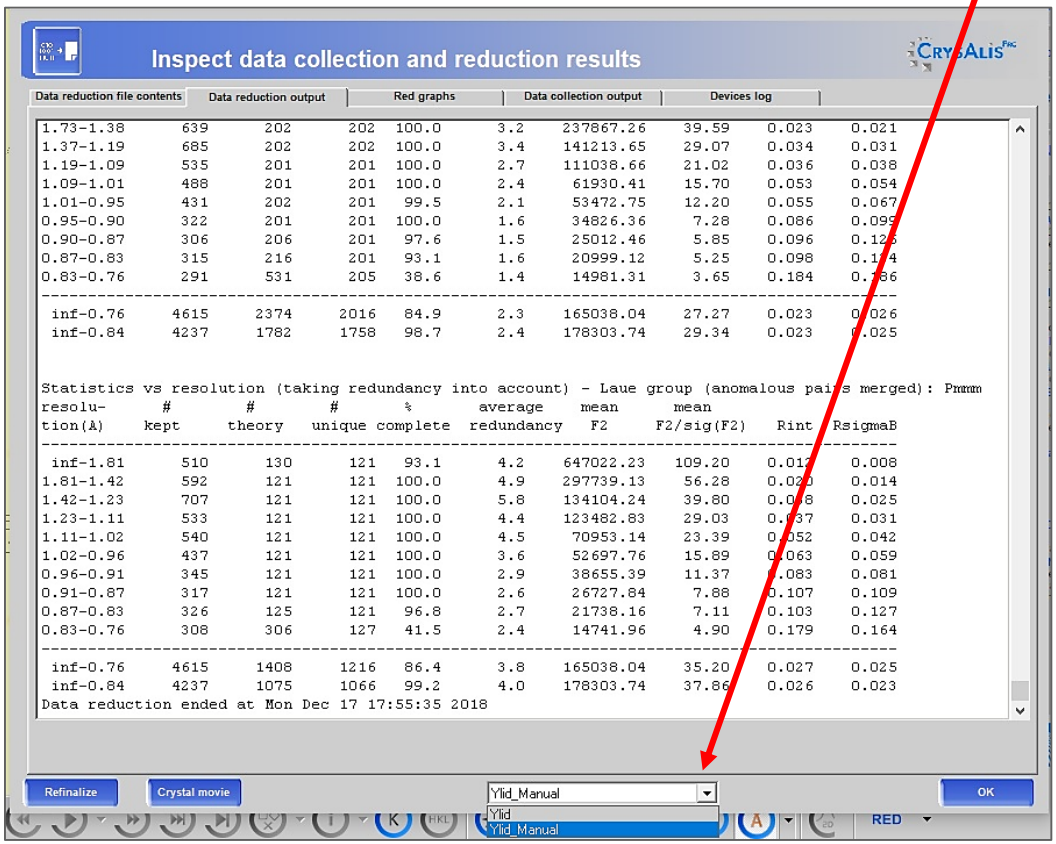
Re-finalize and Compare Statistics from Automatic and Manual Processing

1. Refer to pages 26-39 to refinalize the data manually integrated.
2. Click  to open the Data Inspection Module. From the bottom pull-down menu, you can select any of the processing runs that have been completed and compare the statistics.



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Where Are My Files?

Go to the experiment data directory: 

- The raw diffraction frames are in the ‘frames’ directory.
- The visible crystal pictures are in the ‘movie’ directory.
- All log files for the current experiment are in the ‘log’ directory.
- The reflections files are named ‘Experiment_name.hkl’ (from automatic processing) or ‘Your_Filename.hkl’ (from your manual processing) and are located in the experiment directory. The input files for structure solution have the same filenames, but with the extension ‘.ins’.
- For structure solution, click  at the bottom of the Power Tools panel on the left and select the desired .ins file in the next dialog. Upon opening a 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’.
  - transfers the corresponding .hkl and .ins files to the Olex2 sub-folder.

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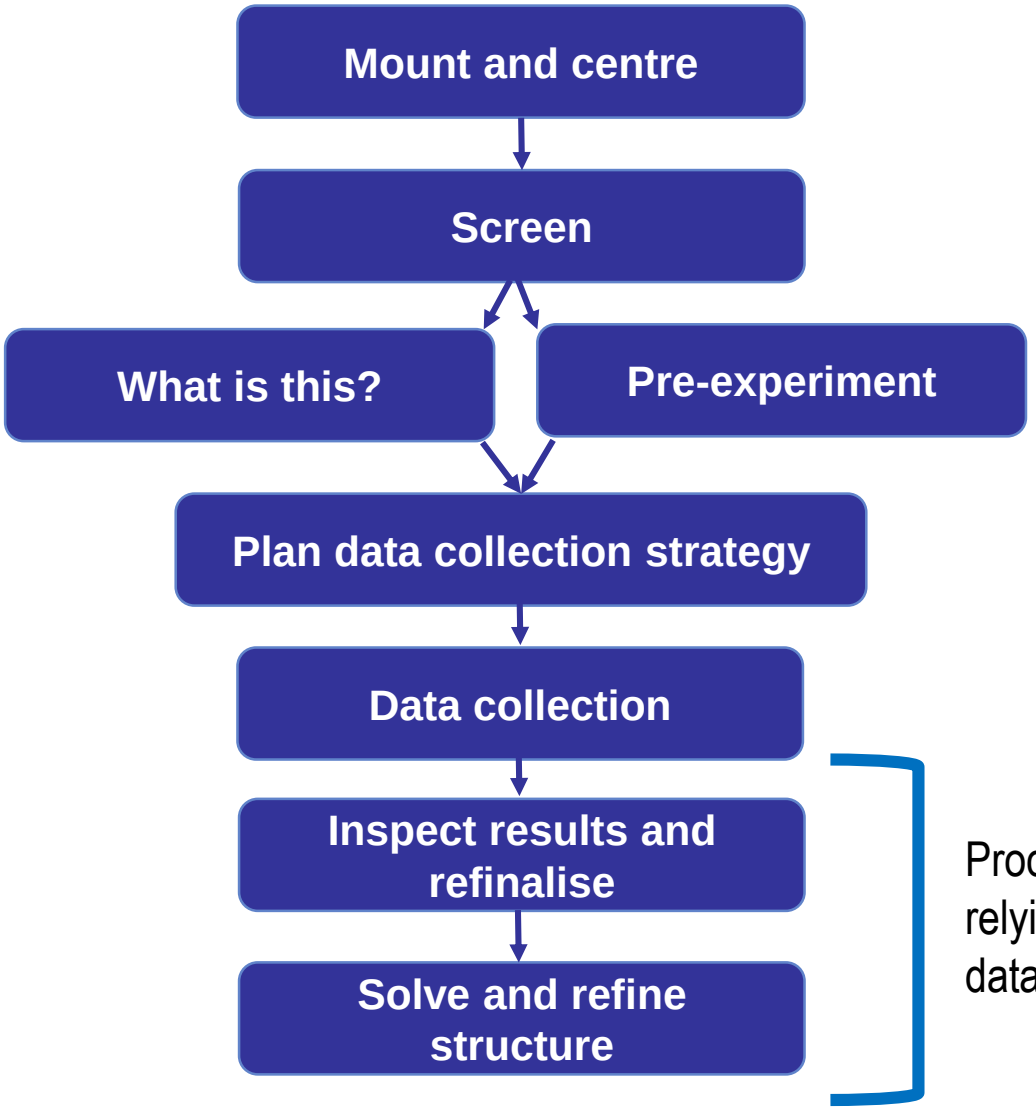
Typical SMX Workflow: Relying on automatic results



Procedure when relying on automatic data reduction results

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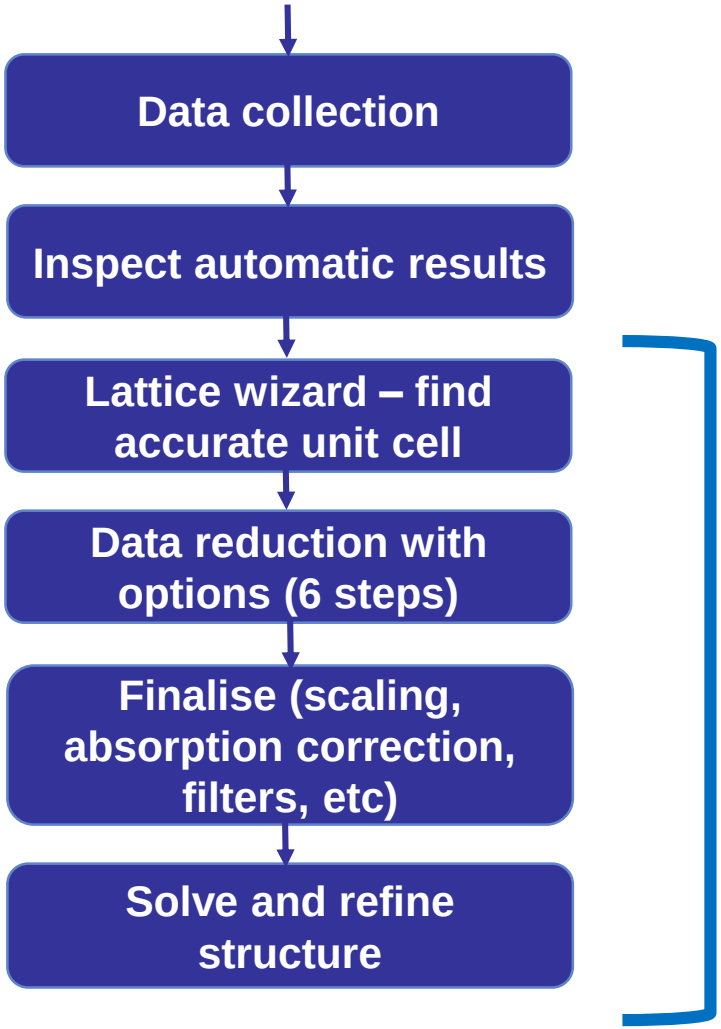
Typical SMX Workflow: after data collection, manual data reduction



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