IMSERC User Manual for Synergy Systems

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INTRODUCTION

Use of this instrument is allowed only by qualified users after receiving training by a staff member. Do not run this instrument without approval from IMSERC staff. Failure to do so may cause damage to the instrument, produce invalid data, and result in additional fees and/or removal of all IMSERC privileges. This set of instructions is meant to serve as a guide for 'routine' data collection on the instrument. For custom experiments that are not covered in this user manual, contact a staff member. For the full list of modes, capabilities, and potential custom experiments that could be run on this instrument, please either contact a staff member or check the corresponding capabilities section at http://imserc.northwestern.edu/crystallography-instruments.html. Please read this user manual and acquaint yourself with the instrument.

A hard copy of this user manual can be found near the instrument. An electronic version of this user manual is linked to the desktop of the instrument computer and also available under the corresponding instrument section at http://imserc.northwestern.edu/crystallography-instruments.html by pressing on the 'User manual' button. If while using the system, something happens that you do not understand, please ***stop***, and ***get help***. In any event, be completely prepared to justify your actions. The cost of even minor repairs could be considerable.

Please remember to:

1. Leave the acquisition software open when you are done with the measurement
2. End your reservation from NUcore when you are done with the experiment
3. Leave lab tables clean and tools/accessories organized
4. Report problems with the instrument (see troubleshooting section for more details)

SAFETY

All users of IMSERC must review the general safety policies at http://imserc.northwestern.edu/about-policies.html and the Crystallography specific policies at http://imserc.northwestern.edu/crystallography-policies.html. To become an independent user of this instrument, you must have the following safety training and certificates under your LUMEN profile:

- Laboratory Safety
- Personal Protective Equipment
- X-Ray Safety

You need the above certificates to be able to reserve time for this instrument on NUcore. Online classes and certification are offered at https://learn.northwestern.edu. Upon completion of the certificate, it will take an overnight to filter through the different systems and get into the files that NUcore uses. Additionally, familiarize yourself with the location of standard safety stations like eye wash and shower stations found in outside of room BG70. Protective eyewear is required in this room, and gloves should be removed when using the computer.

DATA MANAGEMENT

Your personal data folder is created during training. Please save data under your personal folder, which must be located under your supervisor's group folder, either wise you might not be able to access your data remotely. See a staff member if you do not have a personal folder on this instrument yet. For users that prefer to name their data folders using dates, use the order of YYYY-MM-DD or YYYYMMDD in the name, so that folders can be sorted chronologically by the operating system if needed.

Data from this instrument are copied in your supervisor's group folder on 'imsercdata.northwestern.edu' under 'xrd/***X***-Synergy' (where ***X*** is specific to the model, i.e., Cu, Mo, or DW) every 15 minutes. Please follow instructions at http://imserc.northwestern.edu/about-general-faq.html#data for details about data access.

SOFTWARE

Data reduction and analysis are performed with the 'CrysAlisPro' software package. Software is installed on the instrument computer. For offline analysis after your instrument reservation is complete, please use the following resources:

- For registered IMSERC users, software can be downloaded from 'imsercdata.northwestern.edu' under the folder 'public/Crystallography/CrysAlisPro'. Software is available for Windows only
- Software is installed on the communal computers located in the area outside room BG51 and in the computer lab room B190
- You have the option to use the instrument computer for analyses, but you must reserve instrument time through NUcore

For a detailed list of all available crystallographic software for IMSERC-registered users, please check at http://imserc.northwestern.edu/crystallography-resources.html#software.

DEFAULT INSTRUMENT STATUS

In describing the steps involved in collecting a data set, several assumptions have been made regarding the status and conditions of the instrument. The default working condition of the diffractometer is listed below. Control panel is located at the bottom right side of the diffractometer

1. The HV ENABLE key should be "Enabled" (figure 1)
2. The following lights should be illuminated
   a. Green ON (figure 2a)
   b. HV ON/Start. If HV OFF is illuminated, press HV ON and wait ~1 min for the interlock status to update (figure 2b)
   c. Shutters: Red (open) or Green (closed) are both OK (figure 2c)
   d. Error = no light (figure 2d)
   e. Doors OK: Red or none are both OK (figure 2e)

When you are done with your measurement, please remember to:

1. Leave the acquisition software open when you are done with the measurement
2. End your reservation from NUcore when you are done with the experiment
3. Leave lab tables clean and tools/accessories organized
4. Report problems with the instrument (see troubleshooting section for more details)

If there is an error or problem with the instrument which is not addressed under the troubleshooting section, please report the issue by following at least one of the steps below:

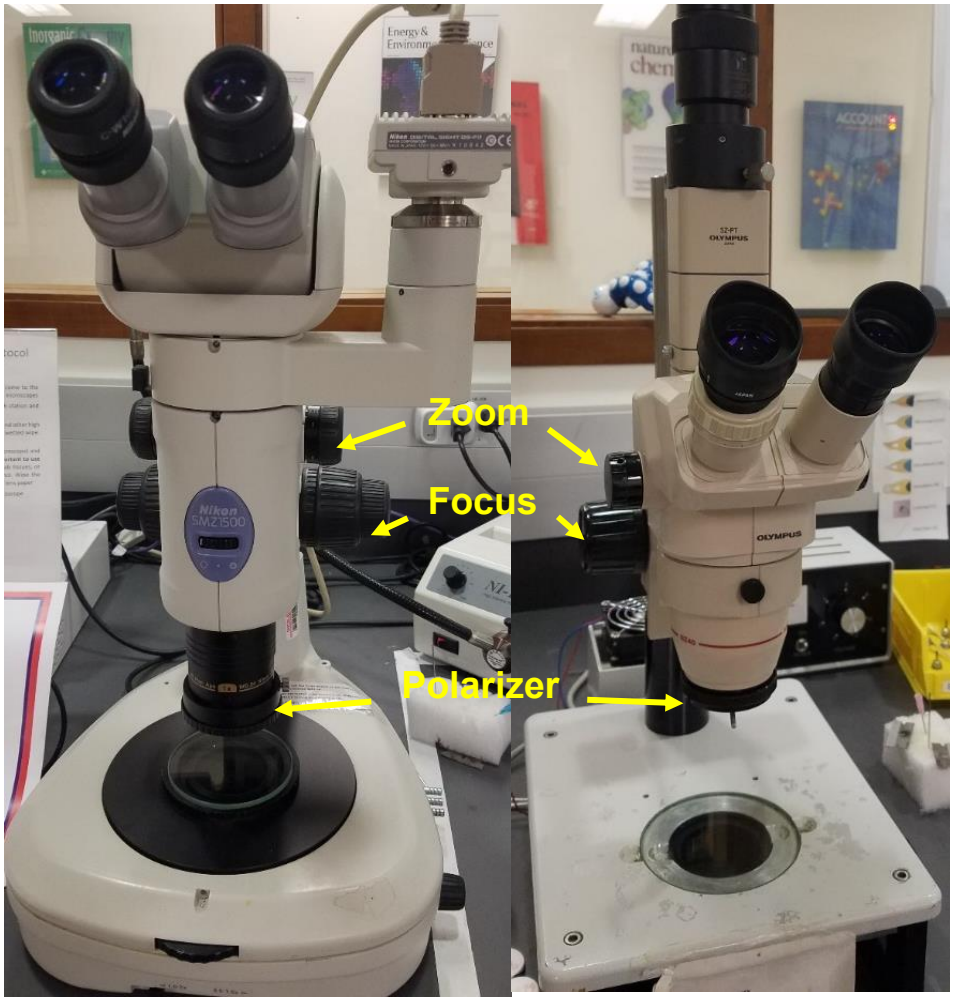
1. If you have already started your reservation using NUcore, please end your reservation and select the error reporting option with a brief description about the issue. Place the 'Stop' sign near the instrument computer to notify users immediately after you. 'Stop' signs are located on the shelf above the computers in BG51
2. If you have not started your reservation using NUcore, please report problems with the instrument at http://imserc.northwestern.edu/contact-issue.html and place the 'Stop' sign near the instrument computer
3. Contact a staff member for instructions

CRYSTAL MOUNTING UNDER A MICROSCOPE

This standard operating procedure is meant for training students/postdocs with the microscopes available at IMSERC. Do not run these microscopes without this training or approval from IMSERC staff. Failure to do so may cause damage to the instrument and result in additional fees and/or removal of all IMSERC privileges. This short set of instructions is meant to serve as a guide for 'routine' usage on the instrument.

A. OUR MICROSCOPES

IMSERC maintains two high resolution, polarized light microscopes available for student use in the Crystallography facility. Our Nikon SMZ1500 stereo-zoom microscope is equipped with a digital camera and video monitor for visualization of crystalline samples. Users can perform visual inspection of their samples with these instruments to assess crystal quality. High resolution photographs can be taken and used for publications or other presentations.

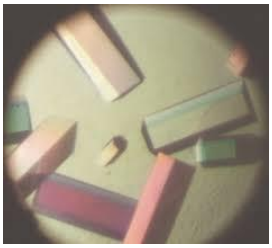


B. TURNING ON THE MICROSCOPE AND ILLUMINATOR

Both microscopes have a timer by the back wall for both the underneath light and the goose neck lights. This timer lasts for 30 minutes.

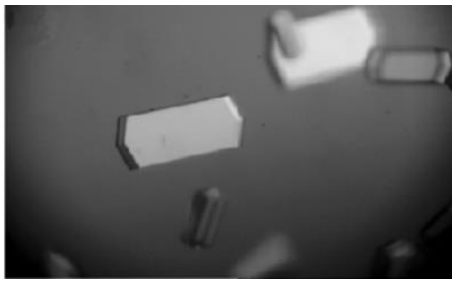
C. IDENTIFYING A GOOD CRYSTAL AND PREPARATION

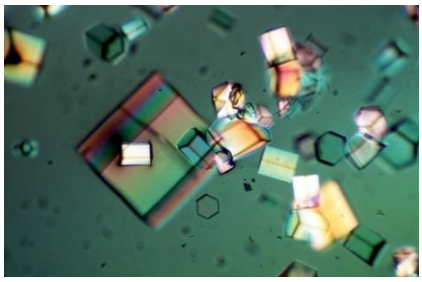
Crystals come in all shapes, sizes, colors and transparent, translucent, and opaque, air sensitive, solvent loss issues or stable. You will get used the types of crystals you usually grow and what tools and mounts you will usually need.



An optically 'good' crystal should:

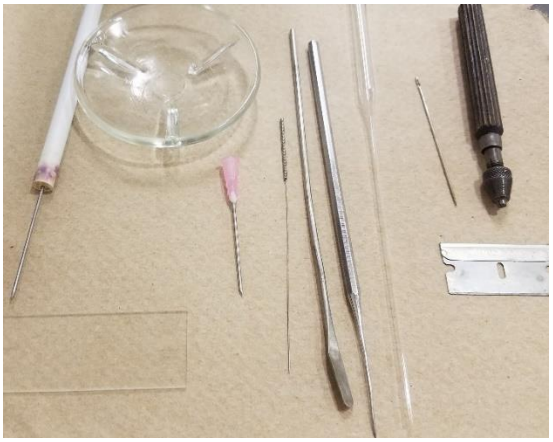
- Extinguishes plane-polarized light
- Uniform color if does not extinguish light
- Smooth surfaces and sharp edges
- Regular shape
- Free of defects
- On rotation will go from light to dark uniformly
- For twin crystals, Cut with a razor blade if possible





You will learn to choose the types of tools that has the best fit for you

- Ease of use
- Matched to sample
- Pipette if needed in solvent
- Smaller tools for small specimens
- Slide for most mounting
- Watch glass if mounting solely from mother liquor
- Razor blade for cutting crystals



Cut crystals if bigger than 0.5 mm

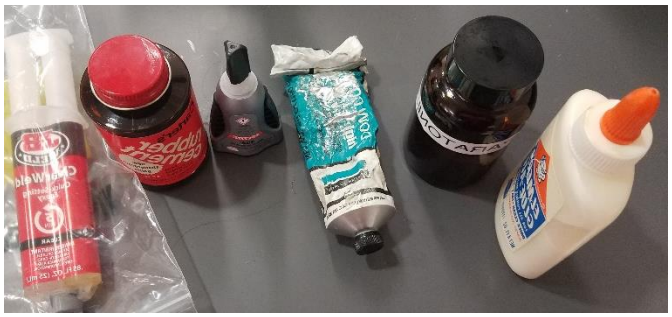
- Move crystal to open space on slide in oil
- Use razor blade to make cut
- Brace tip of razor on slide to reduce motion
- Gently and smoothly press blade down to cut
- Crystal cleaves cleanly

By sweeping crystal through oil, small crystallites and other debris can be separated from the crystal. Do not crush, crystals can shatter

D. MOUNTING CRYSTALS ON AN APPROPRIATE MOUNT WITH ADHESIVE

Low temperature crystallography allows for easy handling of routine and air sensitive samples. The material you use to mount the crystal must harden at experimental temperature. Following are the adhesives depending on the temperature of data collection.

- Suitable compounds
  - Paratone-N
  - Grease
  - High-vacuum grease
  - Hydrocarbon oil
  - STP engine additive
  - Apiezon



For room temperature mounting you can glue your sample onto the glass fiber:

- Suitable adhesives
  - Epoxy
  - Cyanoacrylate (Super Glue)
  - White glue
  - Rubber cement

Choosing a mount:

- Cryoloop
  - Typically mounted on end of tapered copper pin
  - Place crystal at center of
  - Affix with oil
  - Suspend in mother liquor
- Glass fiber
  - Inexpensive
  - Pulled from capillary on capillary puller
  - Some background scatters
  - Cut with stone
  - Mount to copper pin with bee's wax

- Affix crystal with small amount of grease or glue at end of fiber

- MiTeGen mounts
  - Almost no background
  - Easy to use
  - Affix with small amount of oil
  - Many different styles and sizes

Mounting crystals:

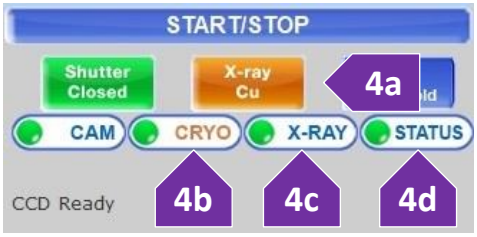
1. Select specimen to be mounted
2. Move to clean part of slide or edge of oil
3. Slide under crystal with mount
4. Push against crystal when using glass fiber
5. Center crystal on middle of loop or top of mount
6. Pick up crystal with minimum amount of adhesive
   a. Hard to see to center crystal
   b. Hard to index faces
   c. Crystal may slide in oil
   d. Creates background amorphous scattering

STARTING A NEW MEASUREMENT

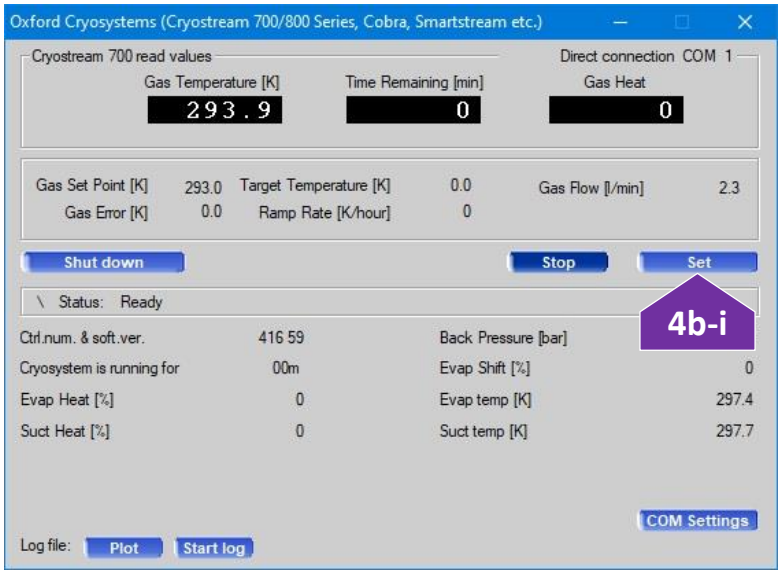
A. SETTING PARAMETERS FOR EXPERIMENT

1. Verify that the instrument is at a working state (see 'Default instrument status' session above)
2. In case the acquisition software (CrysAlisPro) is not running, double-click on the CrysAlisPro icon 

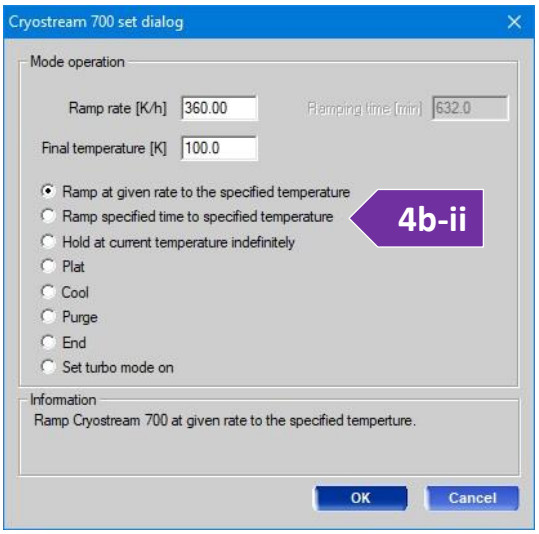
on the desktop. The software will go through an initialization procedure, which takes a few seconds. Status of the initialization procedure is visible at the top right side of the CrysAlisPro window
3. Control menus of the various components, such as, low temperature attachment ('CRYO', figure 4b), X-ray source ('X-RAY', figure 4c), and lights ('STATUS', figure 4d) in the enclosure can be accessed by pressing the corresponding buttons at the top right corner of CrysAlisPro



4. Before starting the screening process of your crystal, select the appropriate settings:
   a. When you are using the dual-wavelength system (DW-Synergy), press on the corresponding button (figure 4a) to change the wavelength if needed. The Cu- and Mo-synergy systems do not have this option/button since they are single source systems
   b. Set collection temperature by pressing on the CRYO button (figure 4b)
      i. Press on the 'Set' button (figure 4b-i) to change temperature. If button is not clickable, press on the 'Restart' button (label of the button toggles between 'Shut down' and 'Restart' depending on the state of the 'Set' button) and wait for a few seconds until the 'Set' button is activated



ii. At the set dialog window, depending on the current state of the cryostat and your target temperature, select (figure 4b-ii):



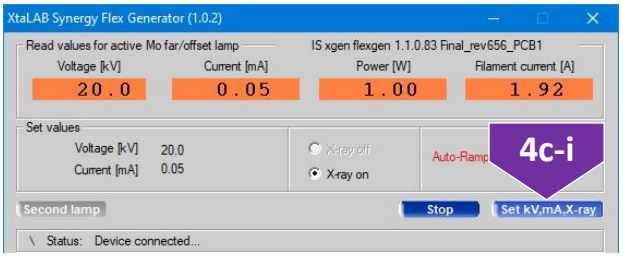
- ➢ 'Ramp at a given rate to the specific temperature' when you want to use a specific heating or cooling rate. Maximum rate is 360 K/h
- ➢ 'Cool' when you want to cool down as fast as possible without controlling the rate
- ➢ 'End' when you want to turn off the cryostat. ***Do not use the 'Stop' button on the controller*** of the Cryostream unless there is an emergency

c. Current power settings of the X-ray source are shown at the top right corner in the diffraction frame window. If settings are different than 50 kV, and 1 mA, press on the X-RAY button (figure 4c)

i. On the generator window, press on the 'Set kV, mA, X-ray' button (figure 4c-i)

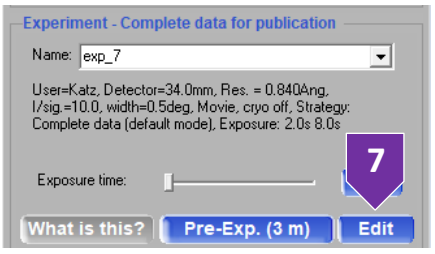
ii. At the set dialog window, select 'Auto-ramp' and press 'OK'

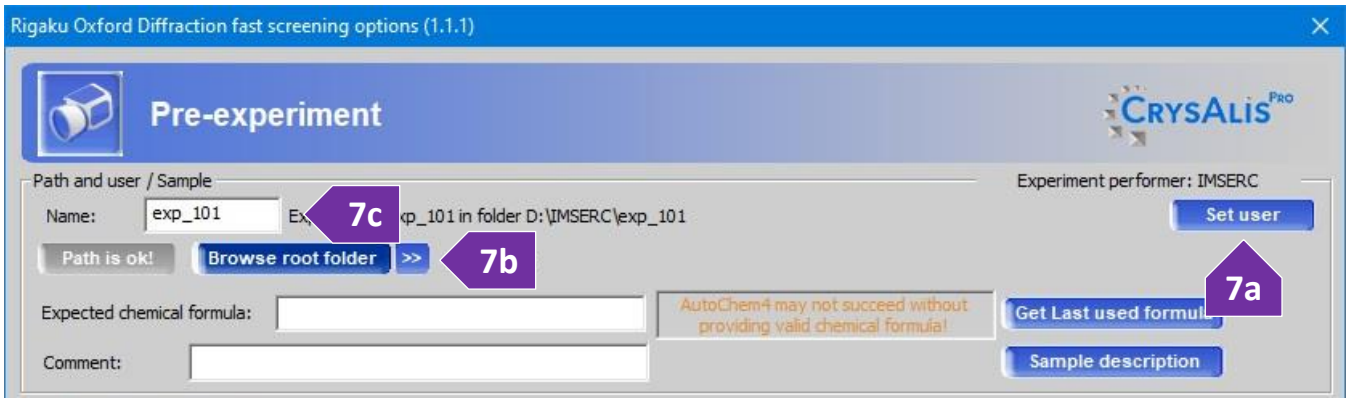
iii. Wait until the power ramps up to 50kV and 1 mA and then close the generator window



d. Press on the STATUS button (figure 4d) in case you want to adjust the intensity of the light in the enclosure and/or the intensity of the light for the optical camera used for aligning the sample. Light in the enclosure will turn on when the main door of the enclosure is opened

5. To start a new experiment, press on the START/STOP button in the top right-hand corner (above the CRYO, X-RAY, and STATUS buttons). This will open up a new window. Press on 'Start New' option
6. The SM (Small Molecule) Screening window will now show up on the right-hand side. It is worth noting that if this is not a new instance of CrysAlisPro (i.e., you are working on a data set after someone else has been using the instrument and the software is still open), then you may see some unit cell information in the Screening section. Do not worry, once you start collecting your data, this will reflect your crystal
7. Set your data collection folder and information about your sample by pressing on the 'Edit' button at the bottom right hand corner of the SM Screening window (figure 7)



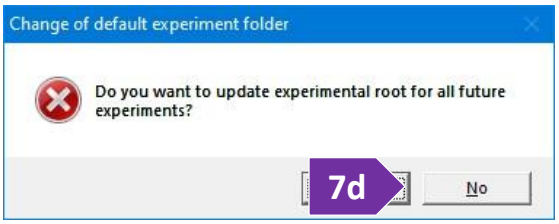


a. Press on the 'Set user' button (figure 7a) and select your group folder by selecting the name of your group and then selecting the option 'Set user'. Your group name should appear above the 'Set user' button and in the 'Experiment performer' label. In case your group folder does not exist in the list of groups, please talk to a staff to create the folder for you

b. Press on the 'Browse root folder' button (figure 7b) and select your personal folder, e.g., 'D:\SupervisorLastname\LastName-FirstName'

c. In the 'Name' text box (figure 7c), provide your experiment name. This experiment name will be also the name of the subfolder under your person folder

d. Software will prompt for changing the default folder to your personal and project folder. Press on the 'No' button (figure 7d) since all other group members will not be saving under your personal folder



B. MOUNTING YOUR CRYSTAL ON THE GONIOMETER

1. In the SM Screening panel, press on either the 'Mount' button (figure 1) or 'F12' on the keyboard. The crystal video window will open and the goniometer will move to the last mounting position. If the goniometer does not move, then ensure that either the door lock button is 'enabled' or you are holding down the 'motion enabled' buttons located inside the enclosure. When the door lock button is 'disabled', anytime you need to move all except the phi axis, i.e., either the omega, 2theta, or chi axes, you need to hold down on both the 'motion enabled' buttons. This is an extra safety feature, so that your fingers and/or hand are not near the moving parts of the instrument

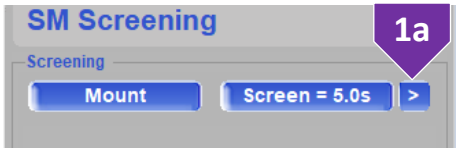
2. On the crystal video window, press on the 'HOME' button (figure 2) once all motion stops (all buttons will be blue again) and open the door of the enclosure

   a. Mount the sample pin on the goniometer
   b. Using the monitor and mouse in the enclosure, press on the 'Lower' button (figure 2b) to adjust the left/right direction. Hold down on both the 'motion enabled' buttons to position the goniometer. Toggle between '0° Arrow Down' and '180° Arrow Up' button to adjust the X-direction
   c. Center the crystal such that it rotates about its center of mass. This is done by adjusting the adjustment screw pointing on the left or right as you face the mounted goniometer head
   d. In case you need to adjust the light intensity on the crystal, press on the 'Lights' button (figure 2d) at the bottom right corner of the video window
   e. Toggle between '0° Arrow Down' and '180° Arrow Up' button to adjust the up/down direction (X-direction)
   f. Finally, toggle between 'Upper' and 'Lower' to verify the Z-height
   g. Press on 'Home', so the instrument goes back to home at the end of data collection
   h. Exit the Crystal video window

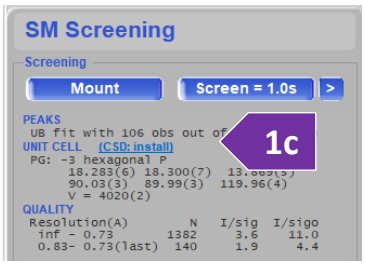
3. Close the door of the enclosure and press the "Door OK" on the Safety System Control Panel

C. SCREENING YOUR CRYSTAL

1. You are ready to start the screening process, which is divided into two steps. First step is called 'Screen' and the second step is called 'Pre-Exp'. At the first step, you should be able to evaluate/screen crystal quality and depending on the number of reflections collected, obtain a unit cell. At the second step, you will collect more data for CrysAlisPro to calculate an accurate strategy
   a. Press on the ">" button (figure 1a) just next to the 'Screen' button and select the exposure time that you feel would be appropriate for your sample. Last used exposure time is shown in the 'Screen' button as 'Screen = X.Xs'

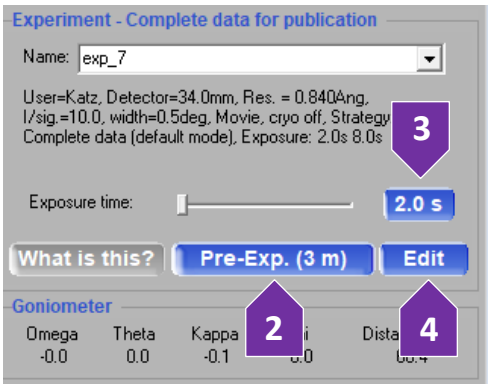


   b. CrysAlisPro will collect a set of 20 images and try to determine a unit cell
   c. If a unit cell is found, a search against the Cambridge Structural Database (CSD) is run and possible hits are available through the CSD link next to the 'UNIT CELL' caption (figure 1c)



   d. A percent of reflections that fit the suggested unit cell is shown on the right panel along with intensity statistics for the exposure time used
   e. You need to press START/STOP and 'Start New' every time you mount a new sample
   f. Note that the screening frames are not stored in your directory, but frames are saved in a temporary folder that gets deleted before every screening collection

2. The 'Pre-exp' option (figure 2) will collect 30 images (Mo-radiation) or 60 images (Cu-radiation) and the software will try to determine how well the crystal diffracts and calculate a data strategy. The overall collection time of the pre-experiment will appear in parentheses on the button, e.g., 3 minutes as shown in figure 2. The minimum setting you need for the pre-experiment is the exposure time per frame:

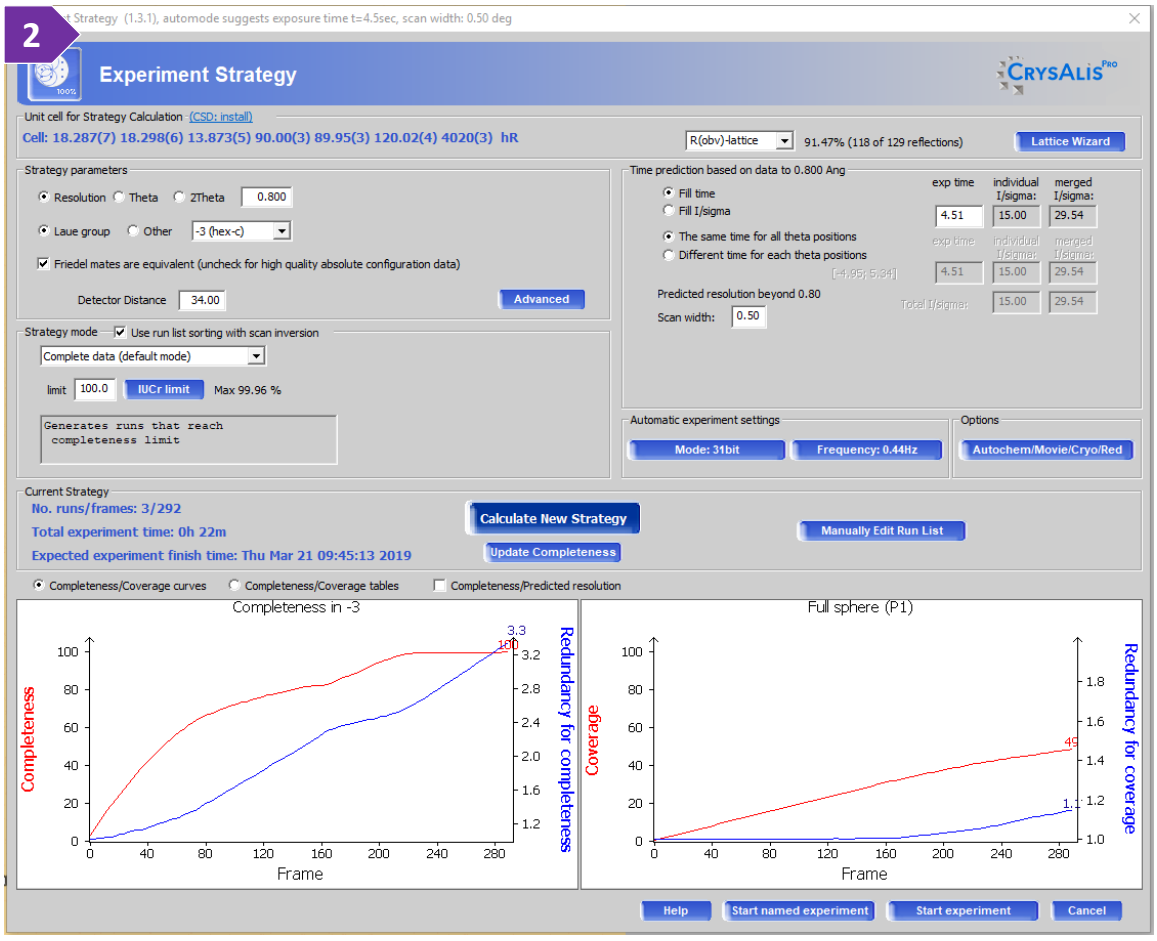


a. A recommended exposure time for the 'Pre-Exp' will be estimated from the 'Screen' step and pre-selected for you. Exposure time appears as a label of the 'Exposure time' button (figure 3)
b. (Optional) Adjust exposure time for the 'Pre-Exp' by either sliding the bar just above the 'Pre-Exp' button or pressing on the small button on the right of the sliding bar (figure 3). Exposure time depends on the size and quality of your crystal but in general:
   - Good scattering crystal: 1 s
   - Moderate scattering crystal: 5 s
   - Weakly scattering crystal: 30 s

3. (Optional) You have the option to re-edit crystal information (as described under the 'A. Setting parameters for experiment' section) by pressing on the 'Edit' button (figure 4)
   a. Leave the 'Interactive strategy after pre' option checked for launching the strategy method right after the pre-experiment
   b. Press on 'Exit & Start pre-experiment' and the software will start the pre-experiment collection
4. If you have skipped the optional step above, press on the 'Pre-Exp' button to start the pre-experiment measurement
5. As the images appear on the screen
   a. Analyze spots for quality. Spots should be single and circular. If multiple spots are clumped together or the spots are very broad in one dimension, stop the pre-experiment by pressing on the 'START/STOP' button and selecting the 'Stop All' option, and screen more crystals
   a. Turn on the resolution rings in case there are not visible by pressing on the  button under the frame window. Spots should be visible to around 0.80 Å resolution. If spots are not visible in this range, either re-screen with a longer exposure time or screen another crystal

D. COLLECTION STRATEGY

After the pre-exp ends, the 'Experiment Strategy' window will open if a unit cell has been found. A list of options and parameters are available for optimizing the collection strategy:



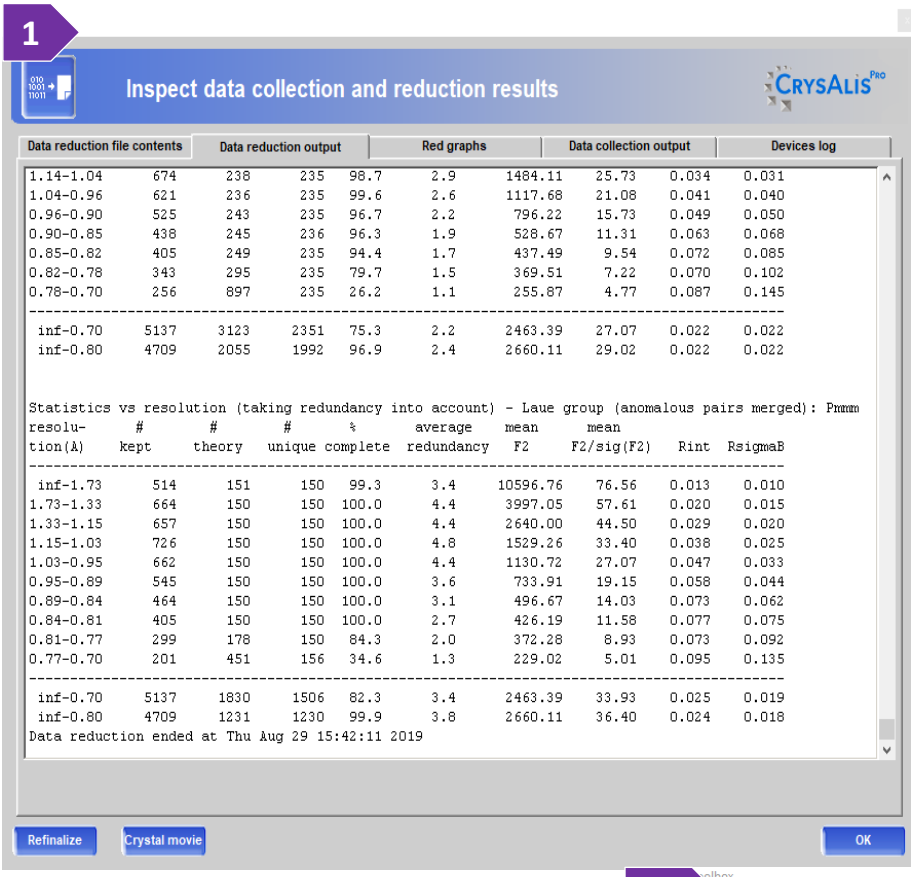
1. Cell constants will appear at the top left corner of the strategy window. If there are any hits in the CSD that match these cell constants, you will be able to see these reported structured by pressing on the corresponding link. Select the lattice type that you want to collect under
2. 'Resolution': The higher the resolution the better. Typically, 0.84 $Å^{-1}$ is the lowest publishable value for samples without getting any alerts during checkCIF and publication. However, that does not mean that you should just collect to that resolution (for Mo-radiation typically to 0.70). If your sample diffracts well, then go higher. If your sample does not diffract well, then only collect it to where it needs to be collected. Inspect the images to see how well it collects the data. In case is needed, collect extra frames at high angles by taking single exposures using the  tool

3. ‘Laue group’: If you are sure that the unit cell is correct and the point group is correct, then leave this unchanged. If you are worried that the actual sample will be of lower symmetry, then select other and lower the symmetry
4. ‘Friedel mates’: if you think the molecule is non-centrosymmetric (chiral or polar), then uncheck this option
5. ‘Detector Distance’: Minimum detector distance is 34 mm. If strategy gives a different value, accept the suggested value
6. ‘Strategy mode’: Typically use the ‘Complete data’ option for giving higher priority on completeness. Select one of the other options in case you want to optimize the strategy using additional priorities
7. ‘Time prediction’: Adjust the exposure time to achieve an estimated merged I/sigma greater than 15. As a rule, the ratio of the low and high angle exposure times should be about 1:4
8. Press on the ‘Calculate New Strategy’ button to (re-)calculate the list of runs that meet the criteria set above.
9. Adjust these values so the data collection ends at a reasonable time if necessary
10. Once you have finalized the strategy, press on the ‘Start named experiment’. Type in the expected chemical formula and any comment you have about the sample in the corresponding fields
11. Press on the ‘Sample Description’ button and fill in the entries for Sample color and Sample Shape. Leave sample size as default values. Press on the ‘OK’ button
12. Once any of the start buttons is pressed, the software will take a movie of your crystal and automatically start the data collection

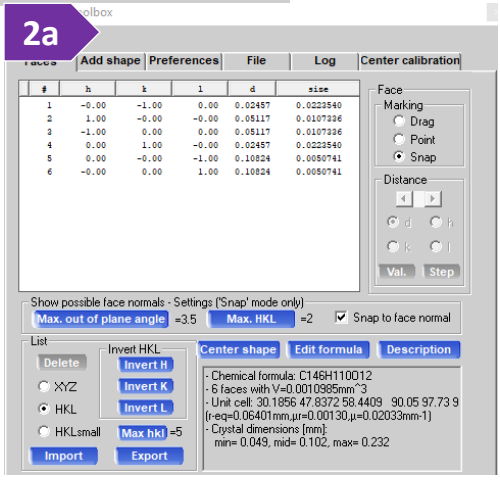
E. MEASURE CRYSTAL FACES AND REFINALIZE

If X-ray absorption is significant, a "face indexed" absorption correction must be performed. At minimum the size of the crystal must be provided for publication

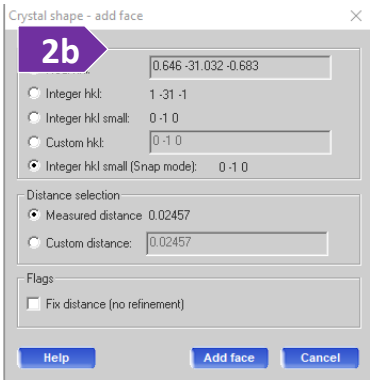
1. Click Data Reduction and then Inspect data reduction results or on from the side bar. And then go to crystal movie.

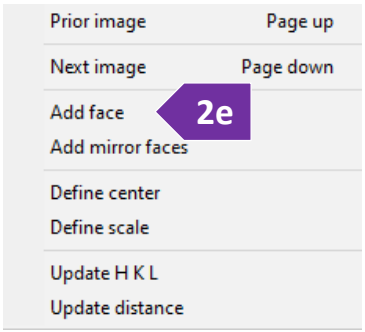


2. Index the faces of your crystal by the snap method
   a. Select Snap
   b. You can limit the number of HKL faces by clicking on "Max HKL"
   c. Rotate your crystal until you see a face with perpendicular lines to it
   d. Left click crosshairs along lines and face



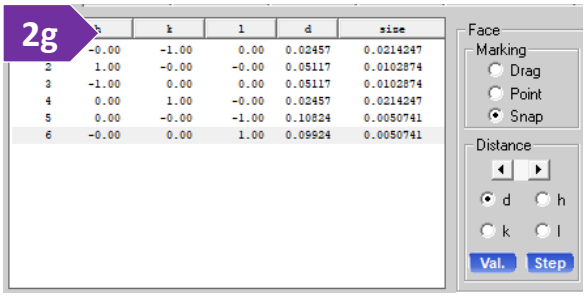
e. Right click and select “add face” you can also add mirror and we can correct size later





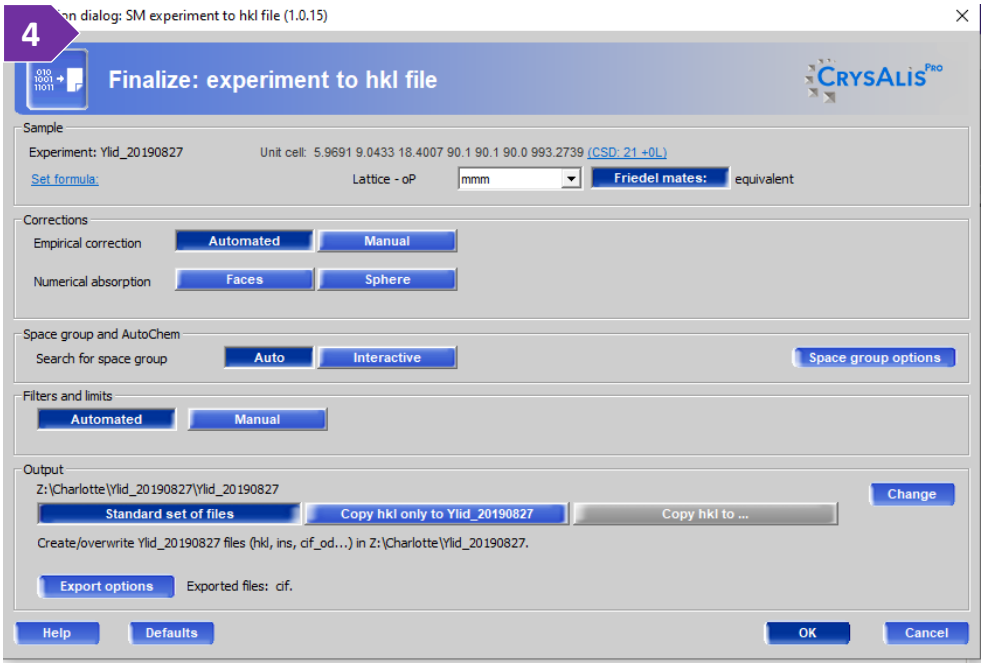
f. Rotate crystal and do it again until all faces are measured

g. You can now edit faces to match edge of crystal if used mirror

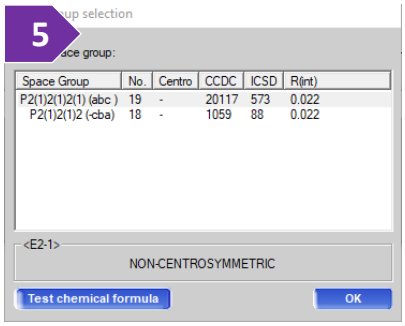


3. Click on the “Refinalize” button.

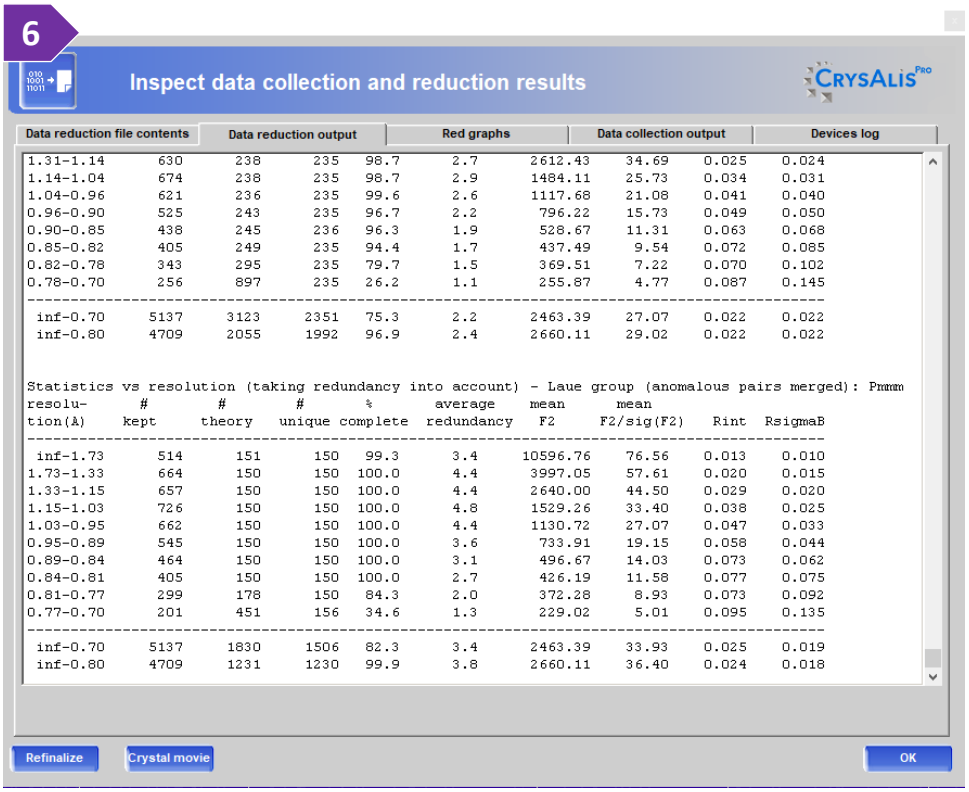
4. Under the “Corrections” section, verify that “Empirical correction” is set to “Automated”. This will apply the Blessing method of absorption correction to the reflections. You may also include faces. Click “OK”.



5. A new window will appear asking you to choose a space group. Choose the space group that is consistent with your previous choices. And press ok



6. Inspect the Rint and F2/sig(F2) to see how well the crystal diffracted vs resolution. Press OK



7. Click the OLEX2 button on the left toolbar to 

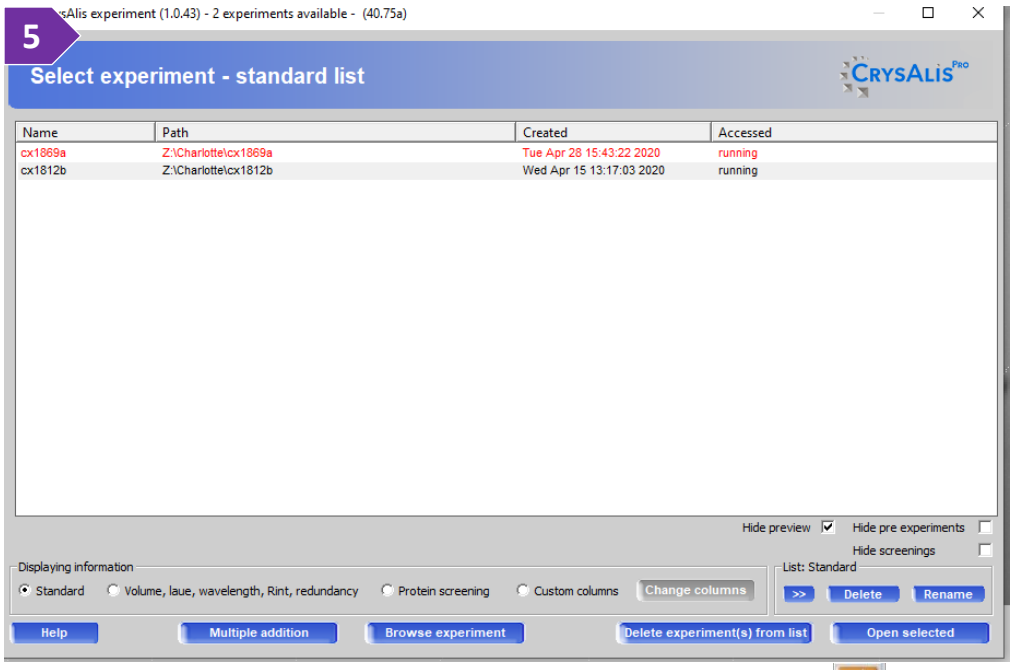
start solution and refinement. All your files needed will be in the struct folder under olex2_XXXXX

PROCESSING YOUR DATA OFFLINE

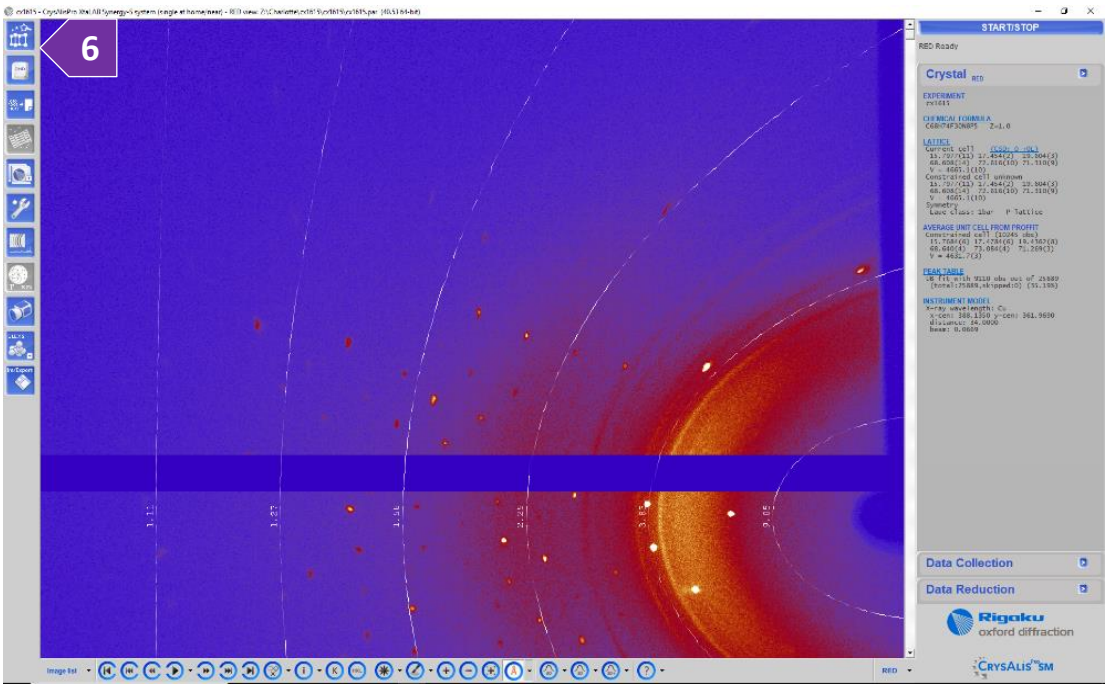
A. DATA INTEGRATION

CAP automatically integrates data online, this section shows how to do it manually.

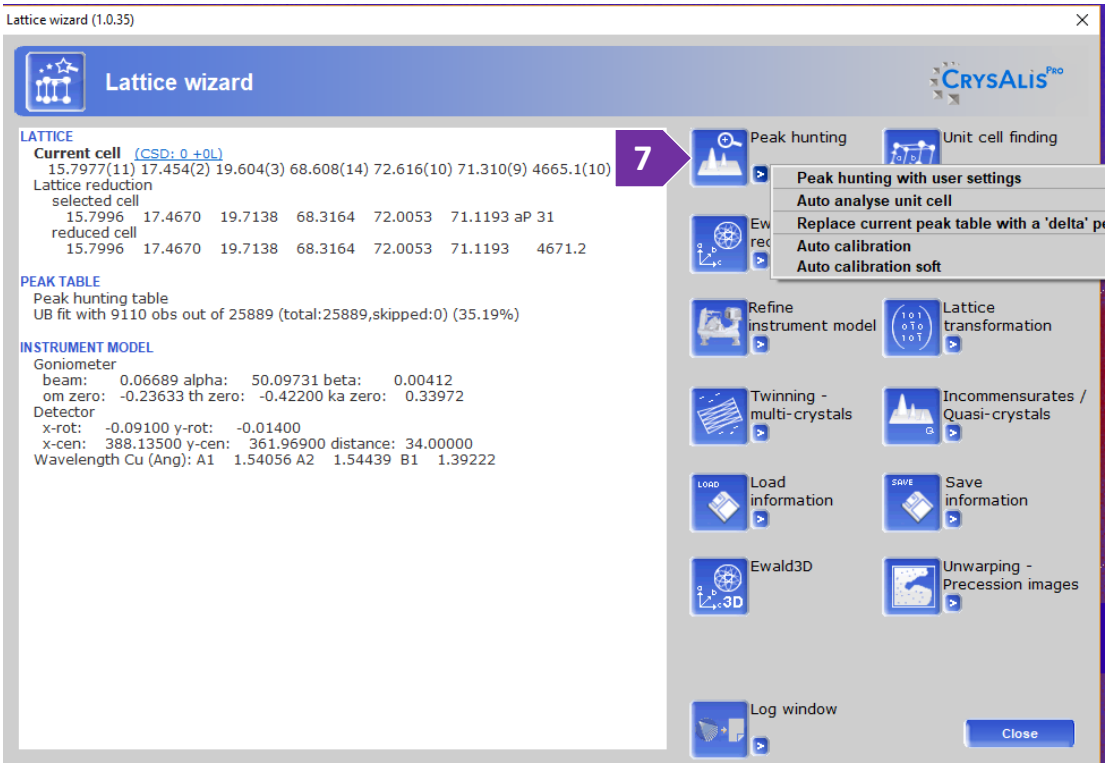
1. Copy the parent directory (i.e. the whole folder) for your data from the X-ray-live drive
2. Double click the CrysAlisPro (CAP) RED  shortcut icon on the desktop.
3. Double click the XXXXX.run file
4. Or in the Select Experiment menu, browse to your experiment. Select the XXXXX.par file.
5. Select the new "XXXXX" experiment file that has been created, and click "Open selected".



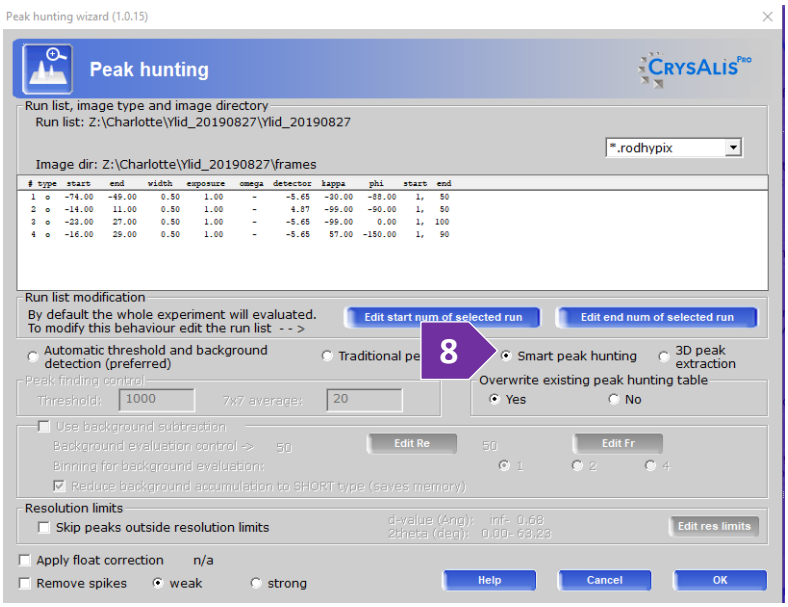
6. Your diffraction image should appear as below. Now select the "Lattice wizard"  button.



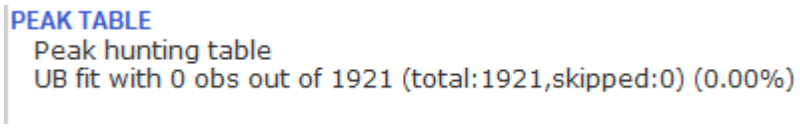
7. Click the “>” button under “Peak hunting”, and select “Peak hunting with user settings”.



8. In the next window, select “Smart peak hunting” and press ok.

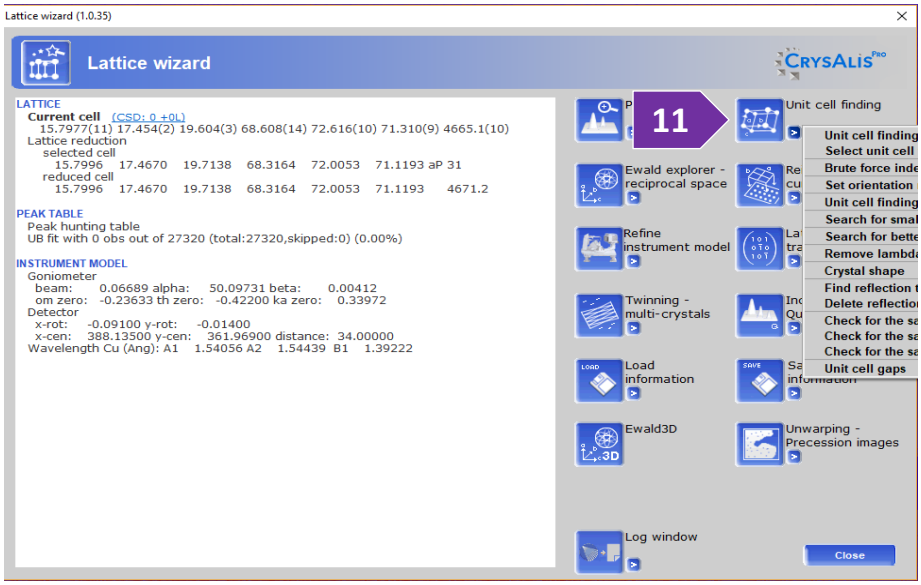


9. Say yes to overwriting the peak table. A window will pop up, and images will rapidly start to sequence as the peak hunting algorithm searches for diffraction peaks. You should see the strongest peaks being marked with "+"
10. At the end of the process, a unit cell is displayed, but 0% of the reflections have been fit to the unit cell. This

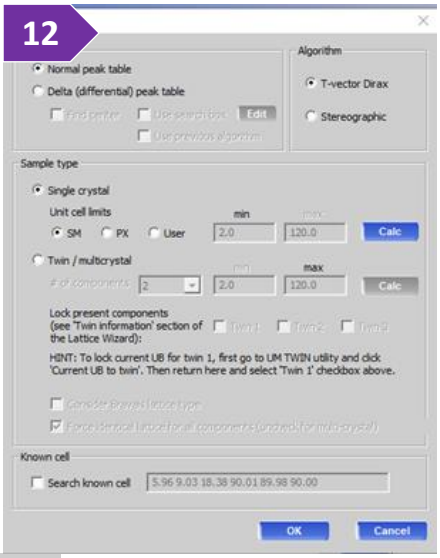


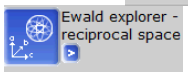
is because the displayed unit cell is from a previous peak hunt

11. Click the ">" button under "Unit cell finding", and select "Unit cell finding with options"

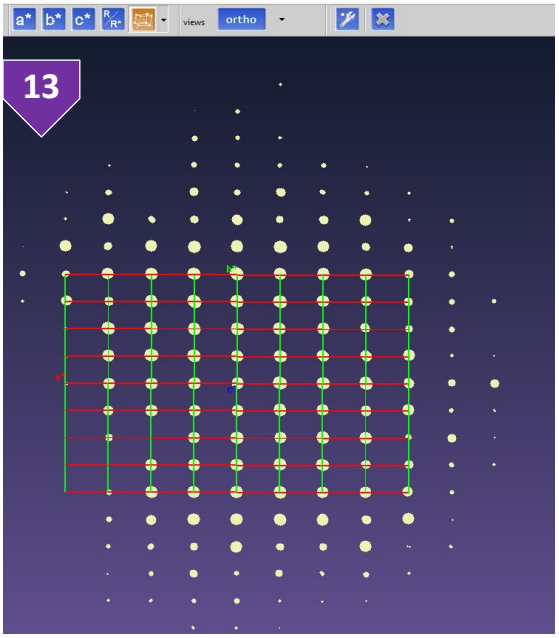


12. Ensure that “Normal peak table” and “T-vector Dirax” are selected. More importantly, “Single crystal” and “SM” should be selected in the Sample type filed. Here, you could also select “User” and set upper and lower bounds for unit cell parameters. For now, make sure your window looks like this image, and click “OK”



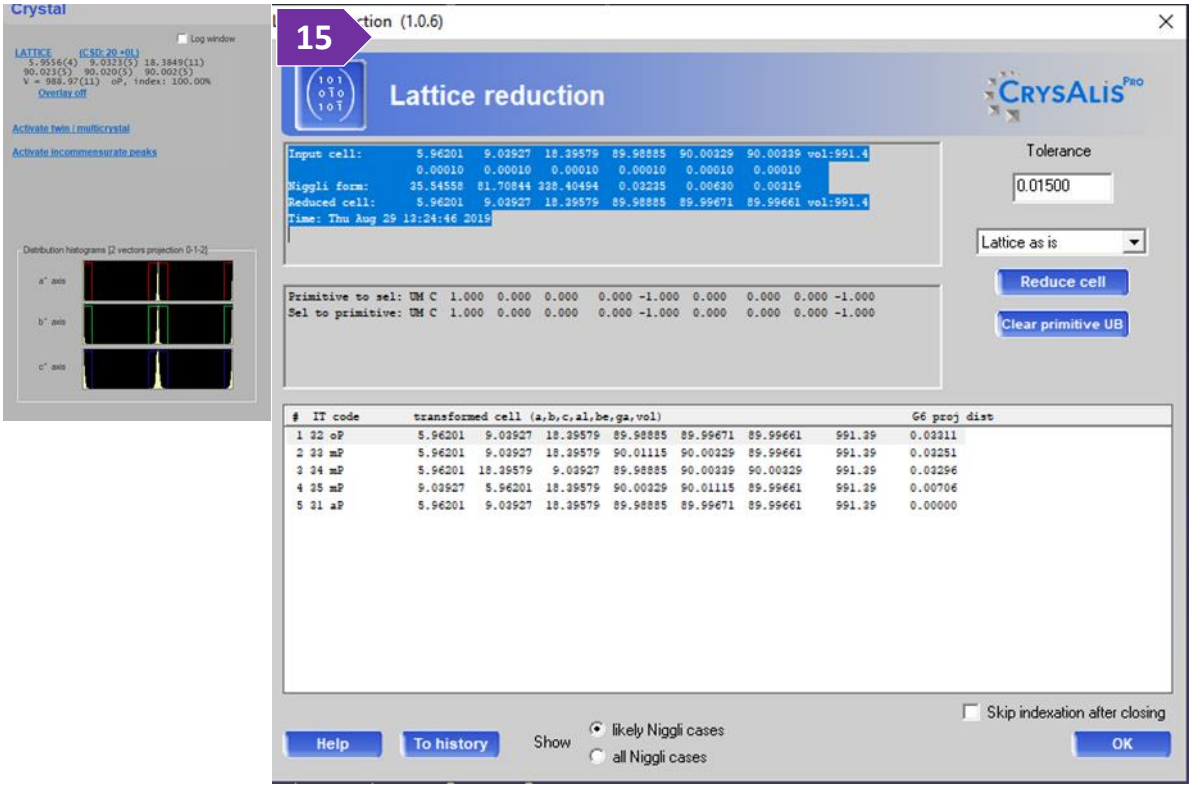
13. Click Ewald explorer 

and look at your reciprocal space. You may look down the different axis. You may turn the overlay on and off. If the cell is indexed properly, then the grid lines should be passing through reflections. If there are a lot of reflections NOT passing through the grid, or if there are grid lines NOT passing through reflections, then something is wrong.



14. You can look for twins here too and look at the histograms

15. From within the Ewald Explorer, check the predicting crystal system and lattice centering: Left click lattice and select “Modify lattice type”. A window appears listing possible unit cells from highest to lowest.



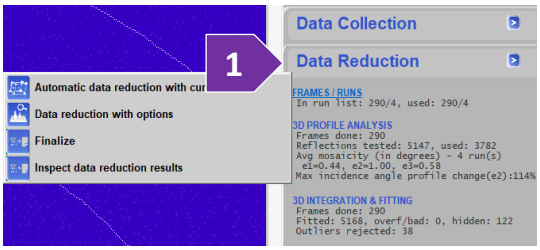
symmetry. The relative small figure of merit of 0.03 suggests that oP is correct. Click OK.

16. Close the Ewald viewer and Lattice Wizard.

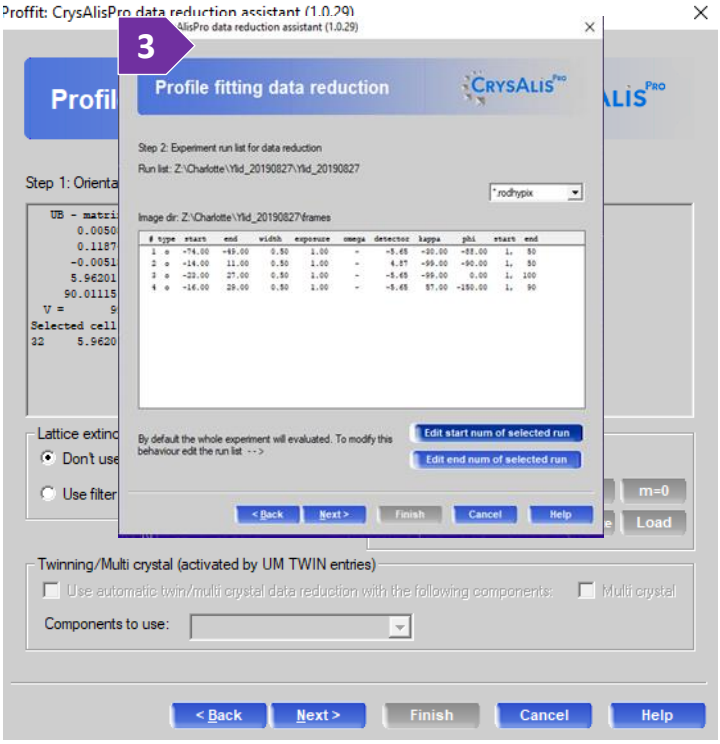
17. Click overlay spot prediction and play through your frames with the 10-foward button. Make sure the + marks are appearing on the peaks. Note: peaks marked ◊ and □ correspond to approaching and receding peaks, respectively. Click on to toggle the frame information on and off

B. DATA REDUCTION

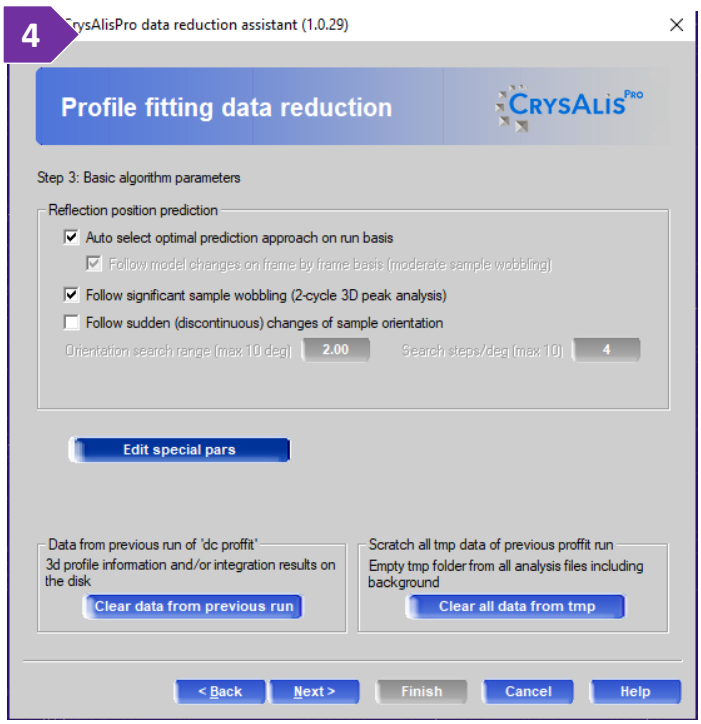
1. Click on “Data Reduction”, (this time not the carrot next to it). Click on it again and select “Data reduction with options”



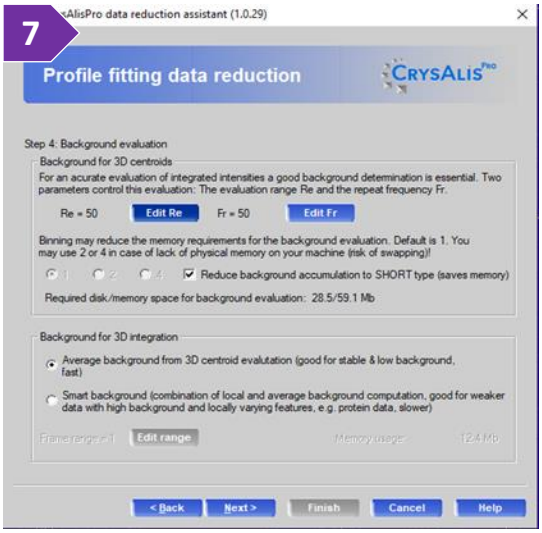
2. Ensure that don’t use filter is checked if lattice is not known. If the lattice is centered (A, B, C, I, F, R), click *use filter for*: and select the correct centering. Make sure Normal data reduction is checked and then click “Next”.



3. Check that CrysAlis^{Pro} has found all runs. This list will dictate which images are integrated. If there were bad frames, omit them here. To delete bad frames, click on the run '#'. Then select edit start [end] number of selected run, depending on whether the bad frames are at the beginning or the end of the run. Change the start [end] number to exclude the bad frames. To omit an entire run, enter 0 (zero) as the start number. Press next.
4. You can change some parameters here. You can correct for sample wobble or sudden movement. Clear data from previous run and Clear all data from tmp. Make sure you click yes in warning windows.



5. Edit special pars “Use resolution limits”. Click “Edit limits” will enable you to reset your resolution.
6. Click “Edit high limit”, and enter “0.77” in the Editing high-resolution limit window. You should already have an idea on how far the crystal diffracts based on your unit cell analysis. Click “OK”, and then click “OK” on the “Resolution limits” window.
7. Click “Next” to Background evaluation window.



8. You can change to “Smart background” option for weaker data. Your window should appear as below.
   a. Re = # of frames used in background calculation
   b. Fr = How often calculation restarts.
   c. Make #s smaller if sudden changes between frames

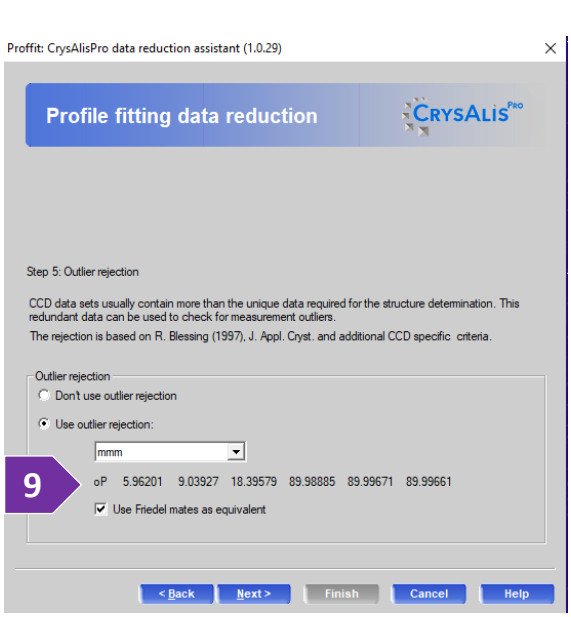
d. Background for 3D integration for average

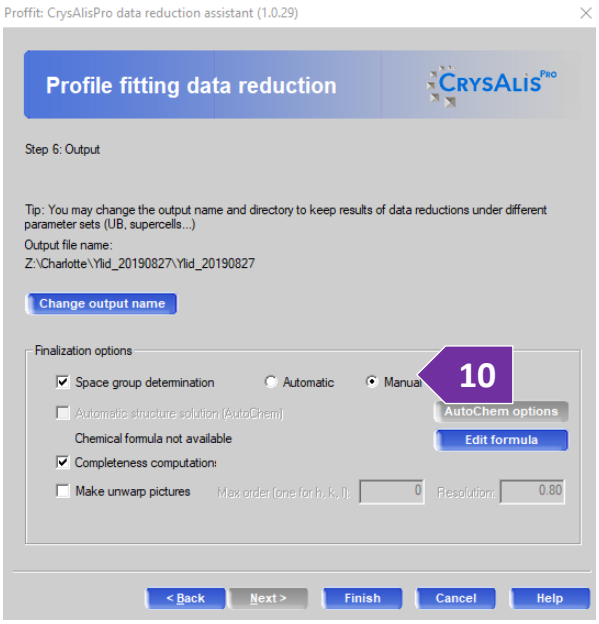
e. Smart: weak data or Large variation w/in frame

f. Click “Next” to proceed.

9. In the outlier rejection window, use the pull-down menu to choose the correct Laue group. Unless you know you have non-centrosymmetric space group make sure Use Friedel mates as equivalent is checked.

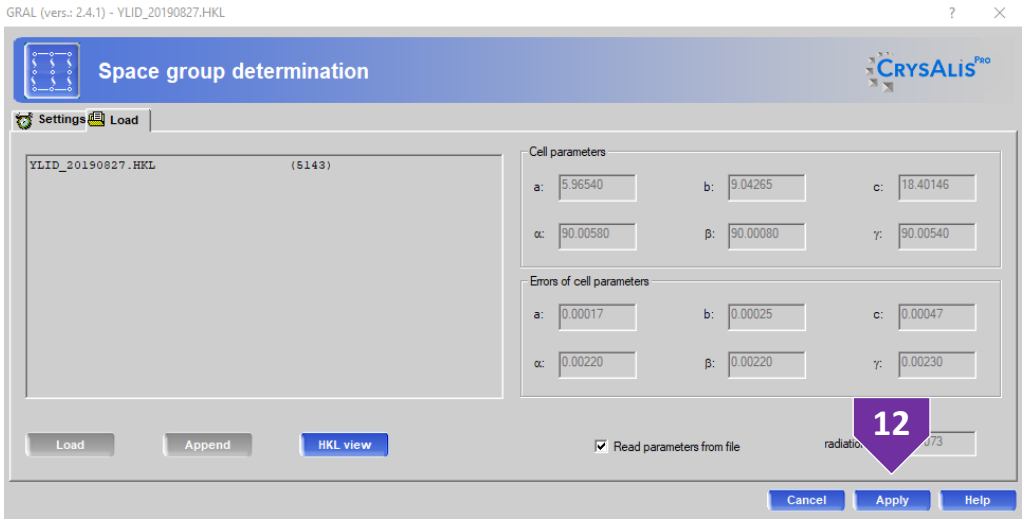
10. Change output name to something new, make sure that the Space group determination is set to “Manual”. Check the formula and Z.





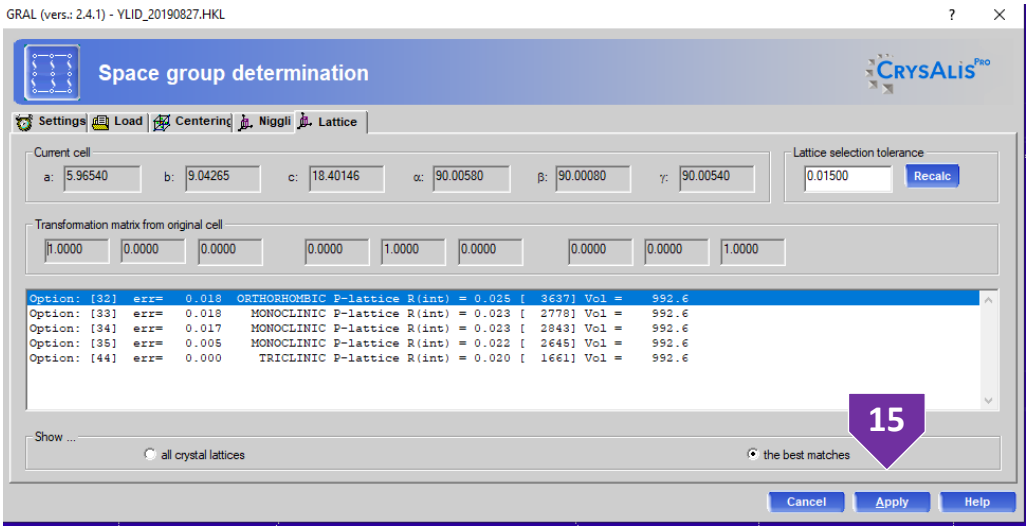
11. Click “Finish”, Watch the integration and monitor the output in the tab. The software runs through the dataset twice. First, the software locates the peaks (marked “+”) and develops a 3D peak profile. Second, the UB matrix and 3D profile are used to calculate the position and intensity of each reflection (3D integration & fitting). The peaks are marked by “integration masks” that give a visual idea of the size and possible overlap of the peaks.

12. Once the integration is finished, the GRAL window will pop-up (Like XPREP) and ask you to assign the space group. Click “Apply”.

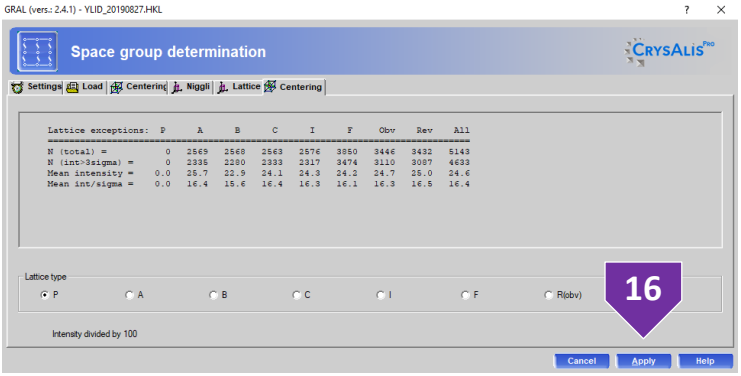


13. Investigate the centering absences. Recall that the first row corresponds to the total number of reflections you would expect to collect for the condition listed in the column. The second row indicates how many of the symmetry equivalent reflections that you collected violate that condition. This cell appears to be primitive. Click “Apply”.
14. The Niggli cell test will look for any unit cell transformation matrices that produce a reduced cell. Click “Apply”

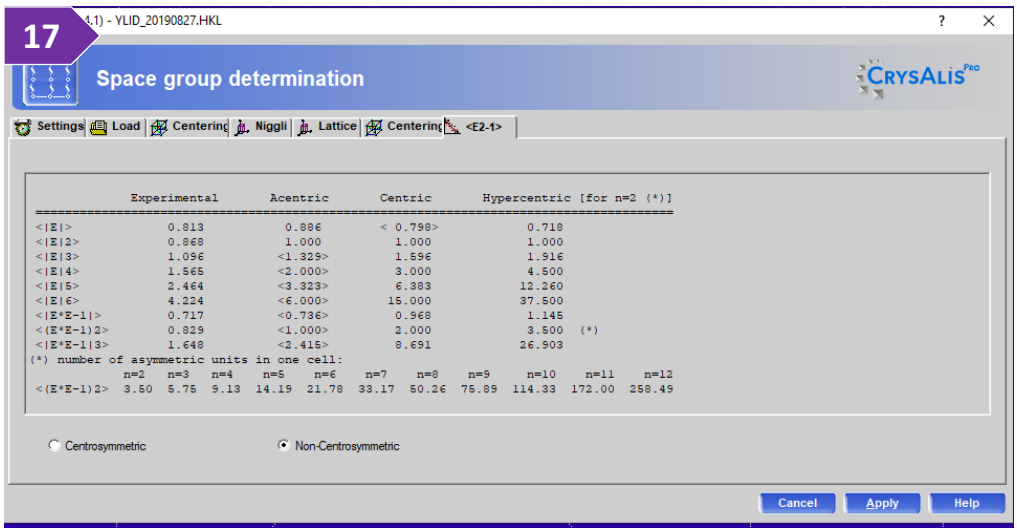
15. Pay close attention to the Rint value, recall that this value should be below 0.10 for good data. A low Rint value suggests that your integration strategy and unit cell assignments are correct. Click “Apply”.



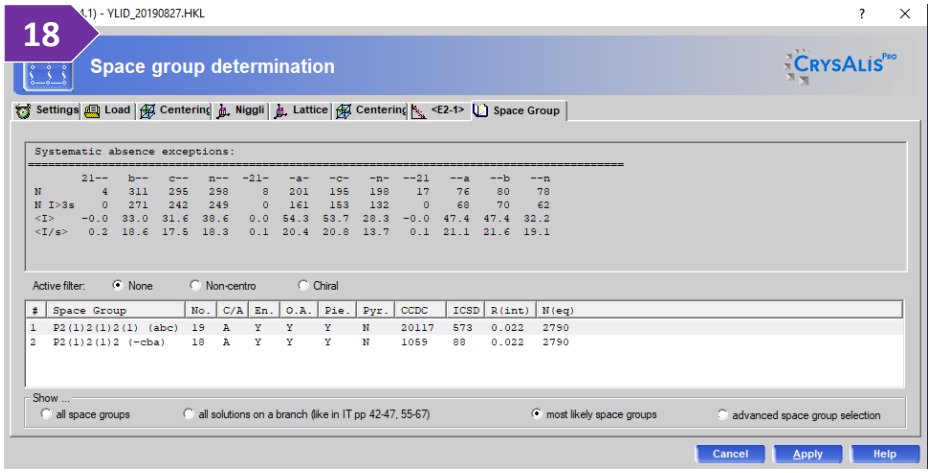
16. Now GRAL will search for higher metric symmetry and additional centering conditions. Click “Apply”.



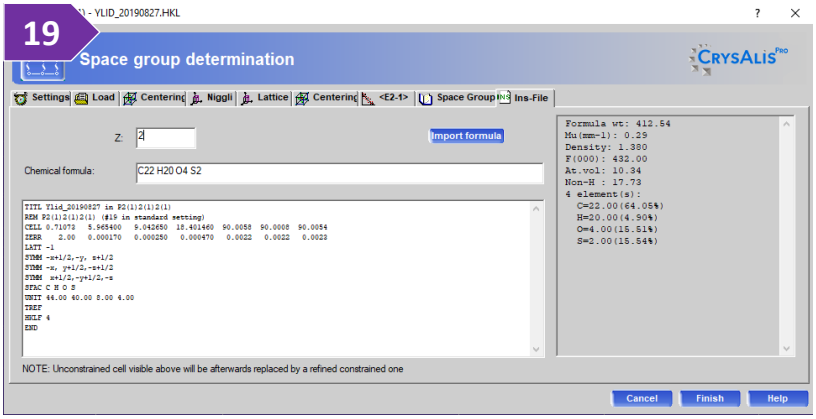
17. The |E2-1| analysis indicates a center symmetric structure. Look at the “Experimental” column, and the |E2-1| value. Click “Apply”



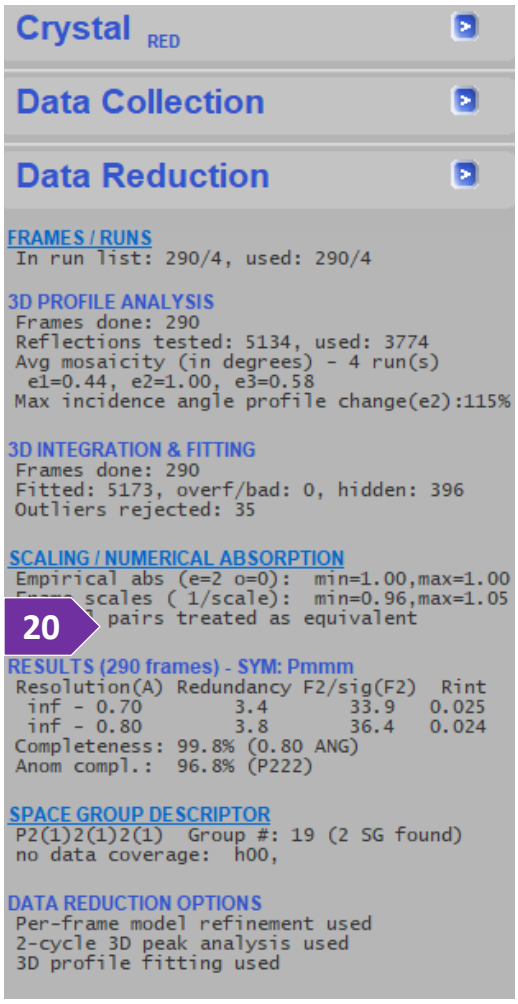
18. Space groups are presented.



19. Now GRAL will produce your HKL and .ins files. Click “Apply”.



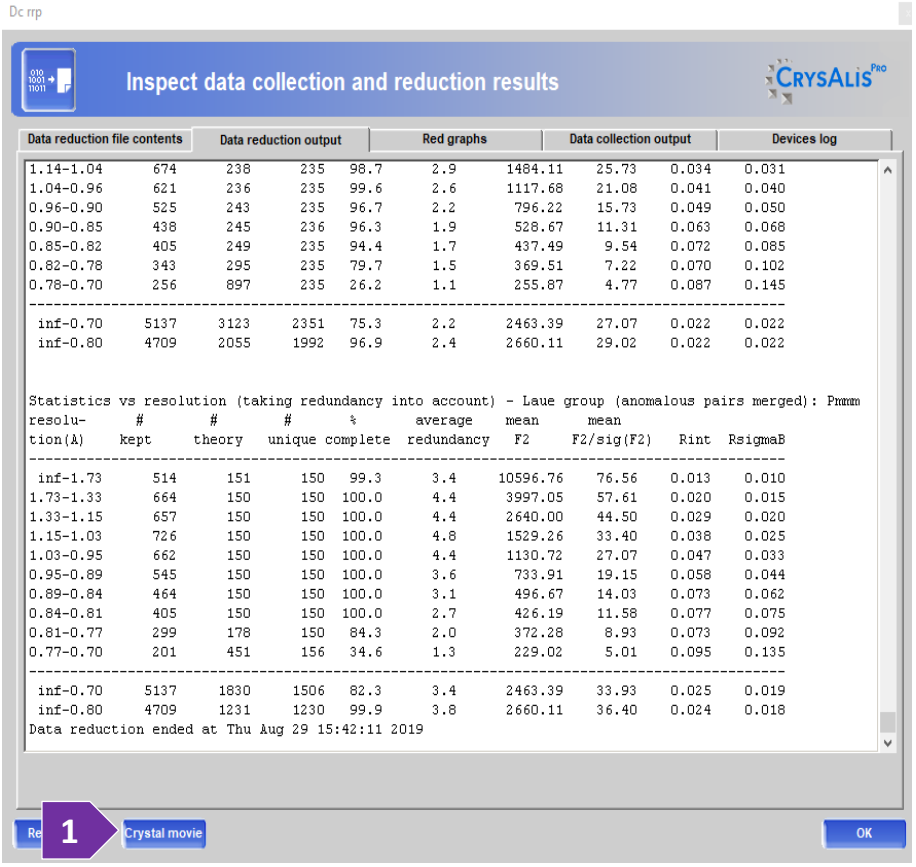
20. Notice the side bar of CrysAlisPro: resolution, redundancy, intensity (F2/σ(F2), Rint, and completeness values are listed for the reflection list. As is mosaicity values (e1,e2,e3), the min/max Empirical abs and Frame scales values. Mosaicity gives an idea of peak widths and crystal quality. For a good quality organic sample, values <1 are normal. The frame scaling and the empirical absorption correction correct for inconsistencies in the X-ray beam, absorption, and other anomalies. Ideally, both the min/max empirical absorption correction and the frame scaling should be close to 1. If a crystal is of poor quality, or a crystal absorbs X-rays strongly and a more accurate absorption correction based on crystal shape and size is necessary, then these values will deviate more substantially from 1.



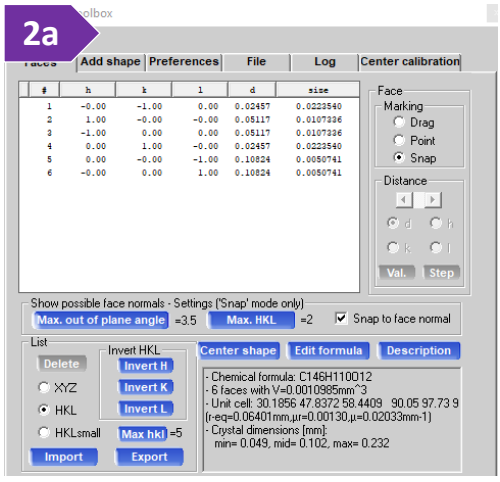
C. MEASURE CRYSTAL FACES AND REFINALIZE

If X-ray absorption is significant, a “face indexed” absorption correction must be performed. At minimum the size of the crystal must be provided for publication

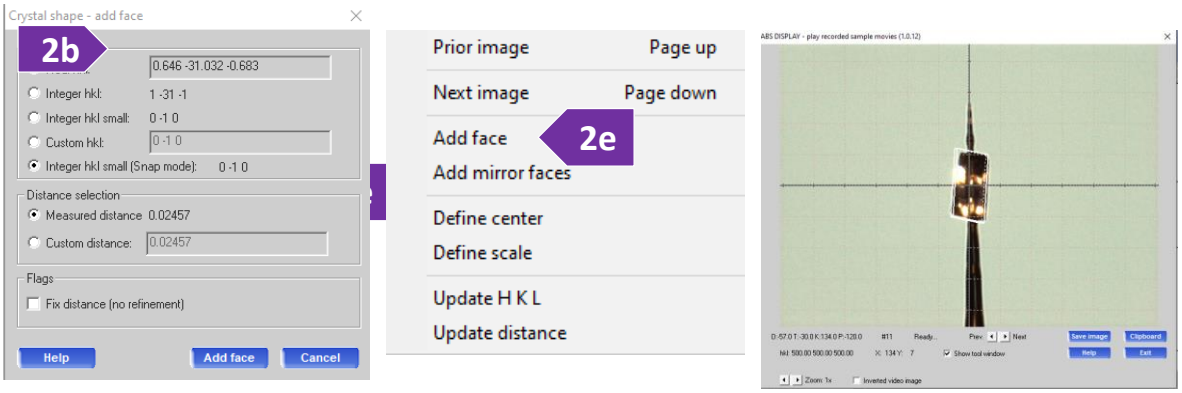
1. Click on  from the side bar. And then go to crystal movie.



2. Index the faces of your crystal by the snap method
   a. Select Snap
   b. You can limit the number of HKL faces by clicking on “Max HKL”
   c. Rotate your crystal until you see a face with perpendicular lines to it
   d. Left click crosshairs along lines and face

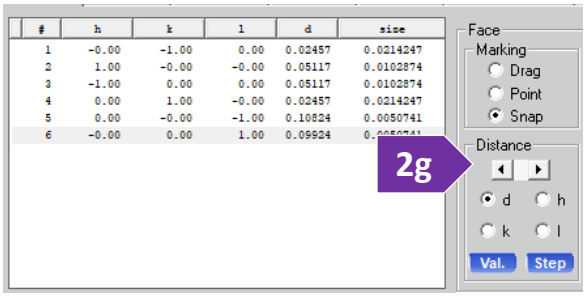


e. Right click and select “add face” you can also add mirror and we can correct size later

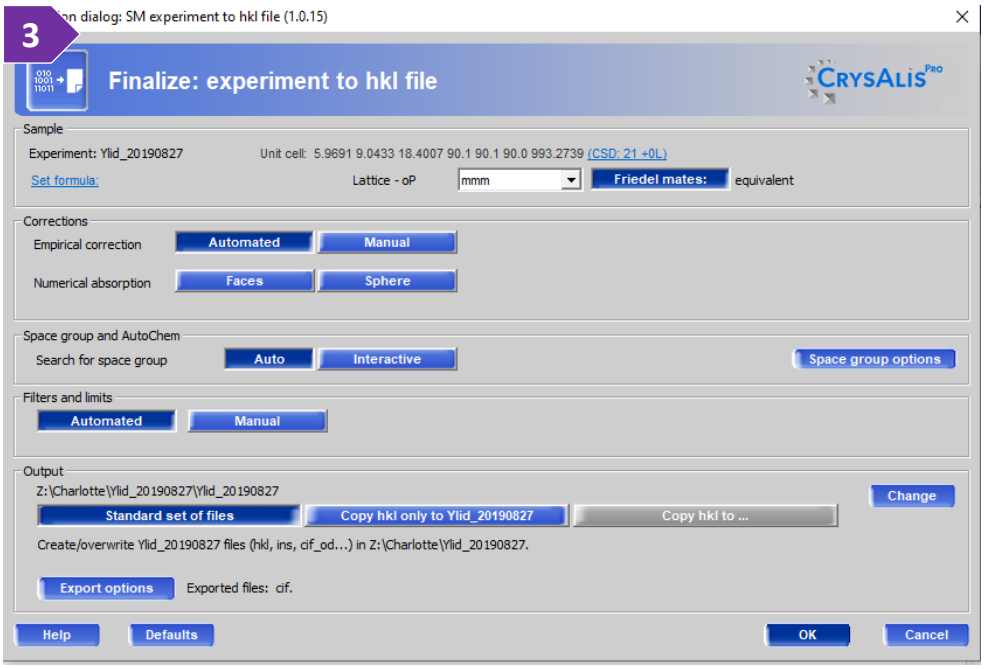


f. Rotate crystal and do it again until all faces are measured

g. You can now edit faces to match edge of crystal if used mirror

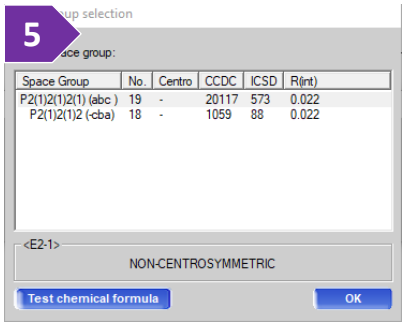


3. Click on the Refinalize button.

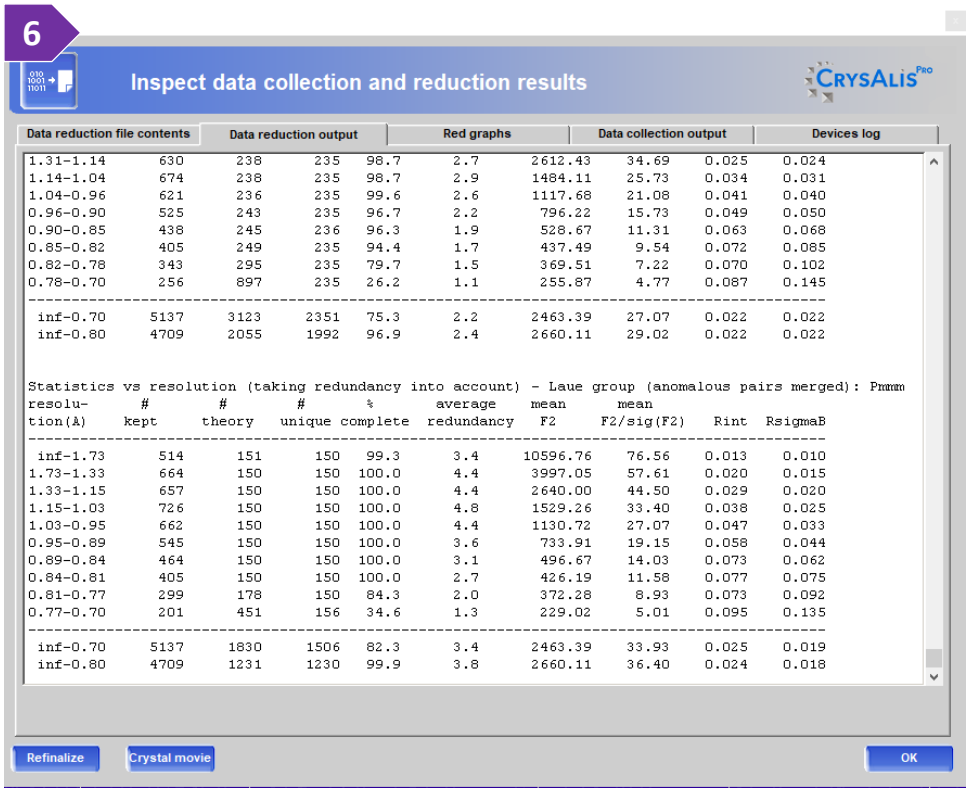


4. Under the “Corrections” section, verify that “Empirical correction” is set to “Automated”. This will apply the Blessing method of absorption correction to the reflections. You may also include faces. Click “OK”.

5. A new window will appear asking you to choose a space group. Choose the space group that is consistent with your previous choices. And press ok



6. Inspect the Rint and F2/sig(F2) to see how well the crystal diffracted vs resolution. Press OK



7. Click the OLEX2 button on the left toolbar to 

start solution and refinement. All your files needed will be in the struct folder under olex2_XXXXX

D. IMPORTANT FILES

- Frames directory has the images
- XXX.par file has the experiment for the instrument. (this is the one you open when starting CAP)
- XXX.rrprof is the data integration results
- XXX.run has the scans table for the data collection
- XXX.cif_od has all the experimental data for your cif file
- Log directory has all current experiment log files
- Quick overview of control buttons

image list	Image list		Find hkl
	Previous run		Look up table
	Jump back 10 frames		Colour table
	Previous image		Zoom in
	Play/stop image movie		Zoom out
	Next image		Zoom localiser window
	Jump forward 10 images		Resolution rings
	Next run		2D Peak profile (a line profile)
	Predictions		3D Peak profile (rocking curve)
	Pixel / area information		Help
	Image header information	CCD	View (CCD/RED/USER)

PUBLICATION

A. EXPERIMENTAL SECTION

Project-specific details and setup information are saved in the '*.cif_od' file located under your project folder. ***Modify the text below according to the setup and conditions you used during the measurement:***

"Intensity data of a ***(color and shape)*** single crystal of ***(project name)*** were collected at ***XXX(Y)*** K. A suitable single crystal with dimensions of ***X×Y×Z*** mm^3 was mounted on a ***(loop | MiTeGen loop | glass fiber | etc.)*** with ***(paratone oil | glue | grease | etc.)*** on an XtaLAB Synergy diffractometer equipped with a ***(micro-focus sealed X-ray tube PhotonJet (Mo) X-ray source | micro-focus sealed X-ray tube PhotonJet (Cu) X-ray source | micro-focus rotating-anode X-ray tube Rigaku (Cu|Mo) X-ray source)*** and a Hybrid Pixel Array Detector (HyPix) detector. Temperature of the crystal was controlled with an Oxford Cryosystems low-temperature device. Data reduction was performed with the CrysAlisPro software using an ***(empirical | numerical)*** absorption correction. The structure was solved with the ***(ShelXT | ShelXD | ShelXS | etc.)*** structure solution program using ***(the Intrinsic Phasing | direct methods | Patterson | Dual space | charge flipping)*** solution method and by using ***(Olex2 | Jana2006 | ShelXle | etc.)*** as the graphical interface. The model was refined with ***(ShelXL | Jana2006 | etc.)*** using least squares minimization."

B. ACKNOWLEDGEMENT

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TROUBLESHOOTING

1. If the goniometer does not move, then ensure that the door lock button is enabled or you are holding down the "motion enabled" buttons in the enclosure
2. Restart CAP to home the goniometer

REVISIONS

v1.20 2020/8/9	• Reformatted according to the latest template. Sections about 'Safety', 'Data management', 'Software', 'Publication', and 'Troubleshooting' were added • Section about 'Crystal mounting under a microscope' was added • Data analysis section was expanded
v1.06 2019/10/24	• Release of original version of the user manual