IMSERC User Manual for STOE StadiVari

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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.

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. Gloves are located by the Olympus optical microscope in room BG62.

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, otherwise 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/StadiVari' once every day. Please follow instructions at http://imserc.northwestern.edu/about-general-faq.html#data for details about data access.

SOFTWARE

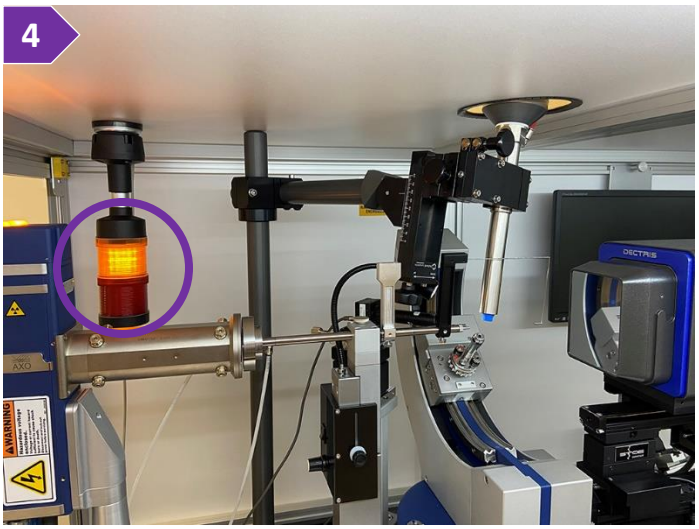
Data reduction and analysis can be performed with the 'X-Area' software. Software is installed on the instrument computer. For offline analysis, please use any the following resources:

- For registered IMSERC users, licensed to IMSERC software can be downloaded from 'imsercdata.northwestern.edu' under the folder 'public/Crystallography/Stoe/StadiVari'. Software is available for Windows only. Please follow instructions under 'Data Access' at http://imserc.northwestern.edu/about-general-faq.html#data on how to connect to the 'public' folder
- Software is installed on the communal computers located in the area outside room BG51
- You have the option to use the instrument computer for analyses, but you must reserve instrument time through NUcore

DEFAULT INSTRUMENT STATUS

The default measurement mode of StadiVari is ***room temperature measurements in transmission geometry and AgKα-radiation***. Please notify the right staff member well in advance if you would like to run an experiment in a different mode than the one listed above, and if you are not trained to perform the require mode-switch. Additionally, put a note on your NUcore reservation indicating the preferred mode of your measurement. 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.

The default working condition of StadiVari is as follows:



1. Computer screen is by default deactivated. You must start your reservation through NUcore to be able to turn on the computer screen. If screen is already on, start your reservation through NUcore
2. The default 'stadivari' user account should be logged in. In case the computer was restarted, the password for the 'stadivari' account is [see hardcopy]
3. The main panel of the acquisition software 'X-Area' should be running. Leave the acquisition software open when you are done with the measurement
4. Orange top light on the safety beacon at the top left corner in the enclosure must be on (circle in figure 4). Red bottom light on the beacon turns on only when a measurement is running
5. X-ray generator (located in the right cabinet of the instrument rack below the benchtop) should be on with voltage and current values of 65 kV and 0.68 mA, respectively (figure 5)

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

1. Leave the acquisition software 'X-Area' running
2. End your reservation in NUcore and add the appropriate accessories based on the measurements you ran
3. Leave lab tables clean and tools/accessories organized

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 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 microscopes.

A. OUR MICROSCOPES

IMSERC maintains three high magnification polarized light microscopes available for student use in the Crystallography facility. Our Nikon SMZ1500 and Leica S9i stereo-zoom microscopes are 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.

Nikon SMZ1500 stereoscope

Olympus SZ-PT stereoscope

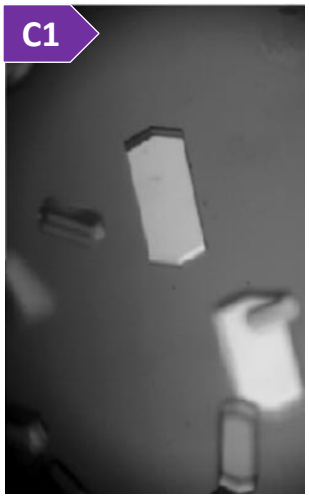
Leica S9i stereoscope

B. TURNING ON THE MICROSCOPE AND ILLUMINATOR

All microscopes have a timer (figure B) by the back wall for both the underneath light and the goose neck lights (when available). To turn on the light, press on the button on the timer which lasts for 30 minutes.

C. IDENTIFYING A GOOD CRYSTAL AND PREPARATION

Good crystals come in all shapes, sizes, and colors. Crystals might be transparent, translucent, opaque, air sensitive, etc. Different mounting tools might be needed depending on the types of crystals you have.



1. An optically 'good' crystal should (figure C1):
    - Extinguishes plane-polarized light
    - Uniform color if does not extinguish light
    - Smooth surfaces and sharp edges
    - Regular shape
    - Free of defects
    - On rotation of the polarized light will go from light to dark uniformly
    - For twin crystals, cut with a razor blade if possible
2. Choose the types of tools that have the best fit for you (figure C2):
    - 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 twinned and/or bigger than 0.3 mm:

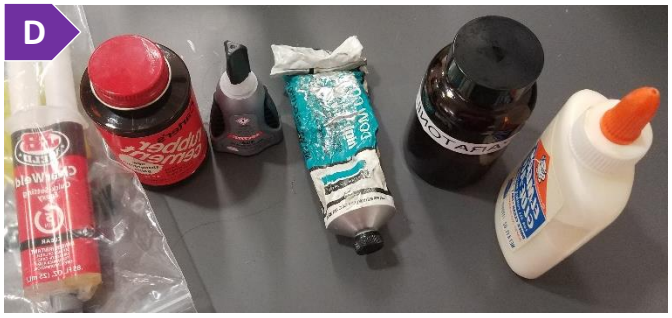
3. Move crystal to open space on slide in oil (figure C3)
4. Use razor blade to make cut, brace tip of razor on slide to reduce motion. Gently and smoothly press blade down to cut (figure C4)
5. Crystal should cleave cleanly

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

D. SELECTING AN APPROPRIATE MOUNT

The material you use to mount the crystal must harden at experimental temperature. Following are the adhesives depending on the temperature of data collection (figure D).

- Low temperature crystallography allows for easy handling of routine and air sensitive samples. Suitable compounds for low temperature measurements are:
  - Paratone-N
  - Grease
  - High-vacuum grease
  - Hydrocarbon oil
  - STP engine additive
  - Apiezon grease



- For room temperature mounting you can glue your sample onto a glass fiber. Suitable adhesives for room or high temperature measurements are:
  - Epoxy
  - Cyanoacrylate (Super Glue)
  - White glue
  - Rubber cement
  - Vacuum grease

Choosing a mount depends on the type of crystals and temperature of data collection. The options for mounts include:

1. Cryo-loops are typically attached on end of tapered metallic pin (figure D1). Cryo-loops give a minimal background due to scattering from C/H:
   a. Suspend crystal i) in mother liquor, or ii) affix with oil
   b. Place crystal i) inside (figure D1b-i), or ii) outside (figure D1b-ii) the loop

2. Glass fibers give a small amorphous background due to scattering from Si/O:
   a. Pulled from capillary using a capillary puller (figure D2a)
   b. Can be cut with stone
   c. Mount to copper pin with bee's wax (figure D2c)
   d. Affix crystal with small amount of grease or glue at end of fiber

D2a

D2c

3. MiTeGen mounts give a minimal background due to scattering from C/H:
   a. Affix with small amount of oil/grease
   b. Come in many different styles and sizes (figure D3)

D3

E. MOUNTING CRYSTALS

The general steps for attaching a crystal on a mount are:

1. Select specimen to be mounted
2. Move to clean part of slide or edge of oil
3. Slide mount under crystal (figure E3)
4. Push against crystal when using glass fiber
5. Center crystal on middle of loop or top of mount (figure E5)
6. Pick up crystal with minimum amount of adhesive, as the adhesive:
   - Makes hard to see to center the crystal
   - Creates difficulties to see and index faces
   - Cause crystal to slide/move
   - Creates additional scattering of an amorphous background

E3

E5

GENERAL STEPS FOR DATA ACQUISITION

The general steps for data acquisition described in this section illustrate the standard workflow of the software. The various sections listed in this user manual provide more details about each specific mode and its settings. Please follow these general steps as discussed during your training session:

1. Verify that instrument is idle by checking the instrument status, i.e., the bottom red safety light (circled in figure 1) in the beacon is off. Orange top light should be always on. Start your reservation in NUcore to have the screen of the computer turned on
2. Mount your crystal or pack your powder as instructed during training
3. Start the 'X-Area' software (icon on the desktop) if it is not already running, and
   a. Name and provide information about your project. See section 'A. Setting parameters for experiment'
   b. Set the temperature of the cryostat in case you need to collect at a temperature different than room temperature. See section 'B. Setting the temperature of your data collection'
   c. Align the crystal/capillary. See section 'C. Mounting and aligning your crystal on the goniometer'
   d. Close the safety door(s) and ensure that green door light is on (figure 3d). Watch the 'stadip-safety-windows' video for a visual demonstration (the safety enclosure system is identical between StadiVari, StadiP, and StadiMP)

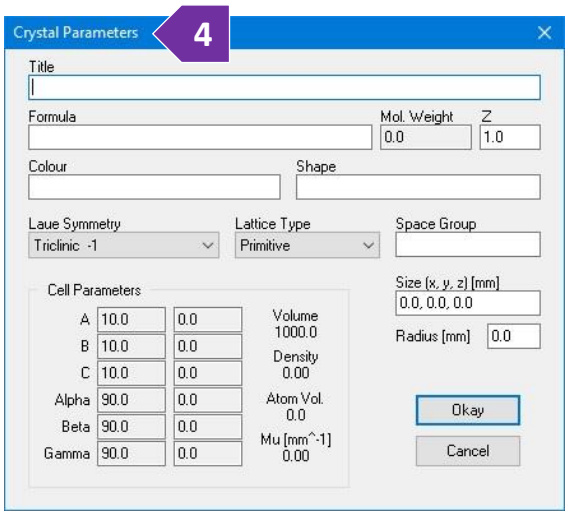
   e. Ensure that the 'CXS-XCU' software is running and the checkbox 'Shutter enable' is ticked. If program in not running, press on the 'CXS-XCU' icon on the desktop (figure 3e). See section 'D. Screening your crystal by running a pre-experiment' for more details

   f. Screen your crystal/powder and evaluate quality
   g. Define measurement parameters
   h. Start the full data collection
   i. Integrate and scale data after full data collection
   j. Ensure that data are okay and remove the sample
   k. End your reservation in NUcore and add the right accessories to your order

SETTING UP A SINGLE CRYSTAL MEASUREMENT

A. SETTING PARAMETERS FOR A SINGLE CRYSTAL EXPERIMENT

1. Verify that the instrument is idle (check 'Default instrument status' section)
2. In case the acquisition software ('X-Area') is not running, double-press on the 'X-Area' icon on the desktop
3. Create a new project file by pressing on the menu 'File' and then 'New'. Save the project file under a new folder in your personal folder, e.g., D:\SupervisorLastname\LastName-FirstName
4. Provide information about the crystal on the 'Crystal Parameters' window (figure 4). Most information on this window is optional and software will let you press 'Okay'. It is highly recommended to provide as much information as possible for each project, so this information is transferred to the final CIF:
   a. Title of the project, e.g., notebook number
   b. Formula of the compound in the form of chemical element followed by stoichiometry (no space). Separate each group of element + stoichiometry with a space, e.g., C10 H11 O S2. The information provided in the 'Formula' field will be used for analytical absorption correction. The molecular weight, density, atomic volume, and absorption coefficient (Mu) labels will be updated accordingly

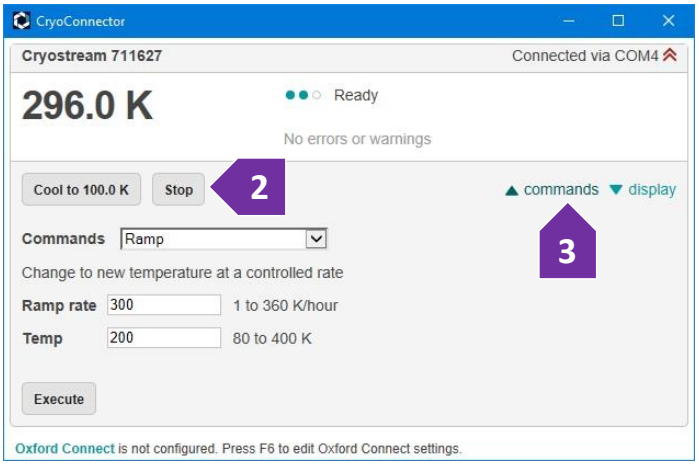


   c. 'Z' number of your compound. This number and formula will affect the absorption coefficient (Mu) every time that is updated. Once the structure is known, 'Z' and/or formula might need to be revised and analytical absorption correction re-calculated
   d. Color of the crystal/powder
   e. Shape of the crystal
   f. Symmetry related information, such as Laue, lattice type, and space group will be updated during the reduction process (using the 'X-Red32' module)
5. Press on the 'Okay' button to save the parameters of your crystal
6. In case you need to provide or edit crystal information, the 'Crystal Parameters' window can be accessed anytime by pressing on either:
   - The menu 'Edit' on the main panel and selecting 'Crystal Parameters', or
   - The 'Crystal Parameters' button on the 'X-red32' window which is accessible under the 'Extras' menu on the main panel

B. SETTING THE TEMPERATURE OF YOUR DATA COLLECTION

In case you are performing a non-ambient temperature measurement, you need to set the base temperature of the temperature device in use. When using the Cryostream device (for measuring between 80K and 500K):

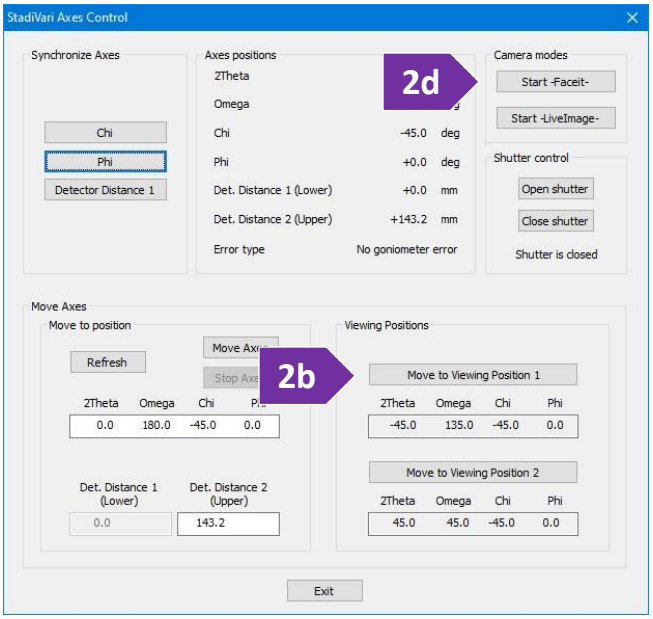
1. Connect to the controller by launching the 'CryoConnector' software by double-pressing on the icon on the desktop. It will take a few seconds for the software to connect to the controller
2. Depending on the state of the controller, you have the option to either run a command directly or restart the controller:
   - If the 'Cool to 100.0 K' button is enabled, then the controller is online, and you can run any command. If you just need to cool down to 100 K as fast as possible, then just press on the 'Cool to 100.0 K' button and proceed to the next section 'C. Mounting and aligning your crystal on the goniometer'
   - If the 'Cool to 100.0 K' button is disabled, then you need to press on the 'Restart' button next to it (figure 2 where the 'Stop' button is) and wait for a few seconds for the controller to restart



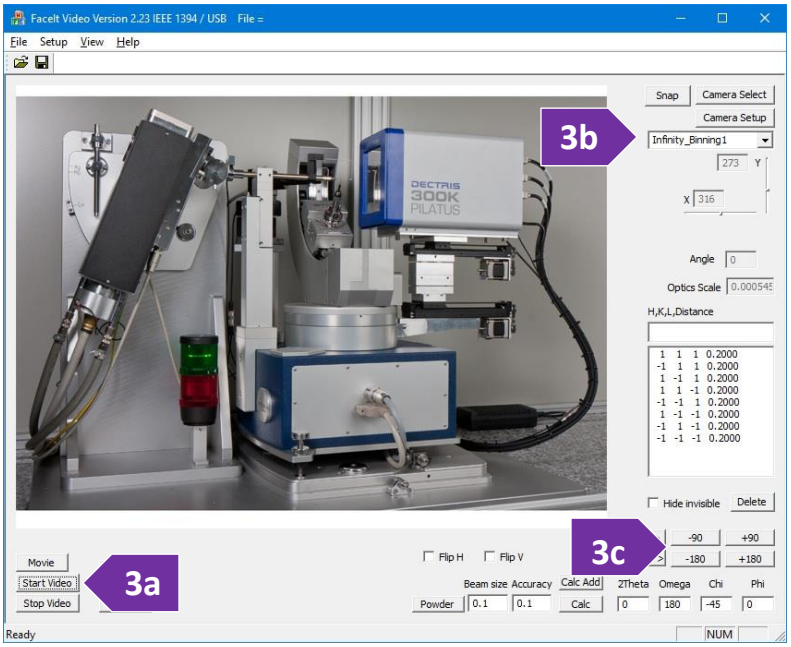
3. For changing/setting temperature, you have two options depending on if the temperature change must have a fixed rate (controlled) or not (uncontrolled):
   - [Uncontrolled] In case you want to cool down to a specific temperature as fast as possible, select from the 'Commands' drop-down menu the 'Cool' option. If list of commands is not visible, press on the 'commands' tab (figure 3). Provide the target temperature and press on the 'Execute' button
   - [Controlled] For changing the temperature at a controlled rate, select from the 'Commands' drop-down menu the 'Ramp' option, provide the target temperature and desired rate, and press on the 'Execute' button
4. You can proceed to the next steps, regarding sample mounting and screening, while the temperature is changing. The 'CryoConnector' software sends commands to the controller which controls the cryostat directly. Therefore, in case you accidently close the 'CryoConnector' software, the cryostat will keep operating as instructed
5. Remember to warmup the cryostat when done with your measurements. To warmup, under the 'Commands' dropdown menu, select 'End', provide the maximum rate (360K/hour), and press on the 'Execute' button

C. MOUNTING AND ALIGNING YOUR CRYSTAL ON THE GONIOMETER

1. On the main 'X-Area' window, press on the 'Instrument' menu, and select 'Measure'. It will take for a few seconds for the software to initialize and start. If the measure 'StadiVariPilatus' window is already open, skip this step
2. Drive all axis to the mounting position by:
   a. Pressing on the menu 'Extras' on the 'StadiVariPilatus' window, and selecting 'Synchronize Axes …'
   b. Pressing on the 'Move to Viewing Position 1' button on the 'StadiVari Axes Control' window (figure 2b). Target values for the viewing position are shown under the button
   c. Waiting for the goniometer to reach the viewing position before going to the next step



3. Start the optical camera software for aligning your sample by pressing on the 'Start -FaceIt-' button (figure 2d)
   a. Start the video by pressing on the 'Start' video button (figure 3a)
   b. Mount your sample on the goniometer head and start the alignment process. There are two different goniometer heads available depending on the type of mount you have used. Watch the '[stadivari-alignment-crystal]' video for a visual demonstration of the alignment process. In case you have to change the magnification of the optical camera, change the binning option from 'Binning1' (high magnification) to 'Binning4' (low magnification) (figure 3b)
   c. As instructed in the alignment video, toggle between the '+/- 180', and '+/- 90' buttons to adjust the centering (figure 3c)



d. Exit the optical camera software by pressing on the 'File' menu and selecting 'Exit'

e. Exit the 'StadiVari Axes Control' window

4. Close the safety windows of the enclosure and make sure the green light next to the top emergency button is on (figure 4). Watch the 'stadip-safety-windows' video for a visual demonstration (the safety enclosure system is identical between StadiVari, StadiP, and StadiMP)

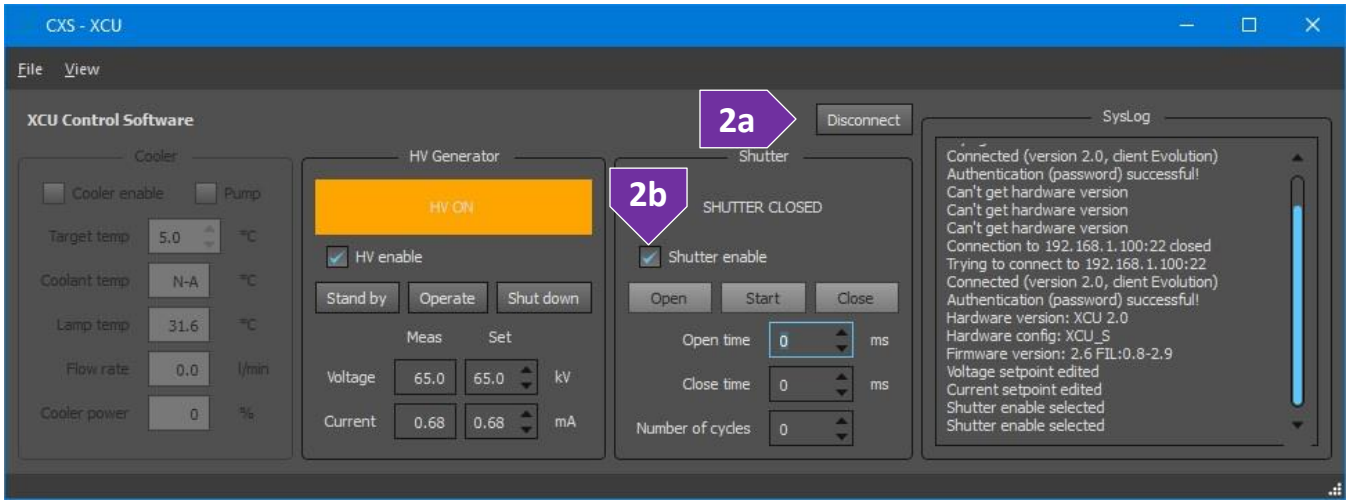
D. SCREENING YOUR CRYSTAL BY RUNNING A PRE-EXPERIMENT

After mounting and aligning your crystal on the diffractometer, you are ready to start the screening process by running a pre-experiment.

1. Ensure that the door safety light is green (figure 1).
2. Select the 'CXS-XCU' window from the taskbar to enable the shutter. If program in not running, press on the 'CXS-XCU' icon on the desktop:

a. If you just started 'CXS-XCU', press on the 'Connect' button (figure 2a, the 'Connect' button is at the same location with the 'Disconnect' button shown on the figure) for the software to connect to the X-ray generator

b. Enable the checkbox 'Shutter enable' option (figure 2b). This option needs to be ***re-checked/enabled every time you open the safety windows***. If the 'Shutter enable' option remains unchecked during a data collection, there will be no X-rays going through your sample and all frames will be empty

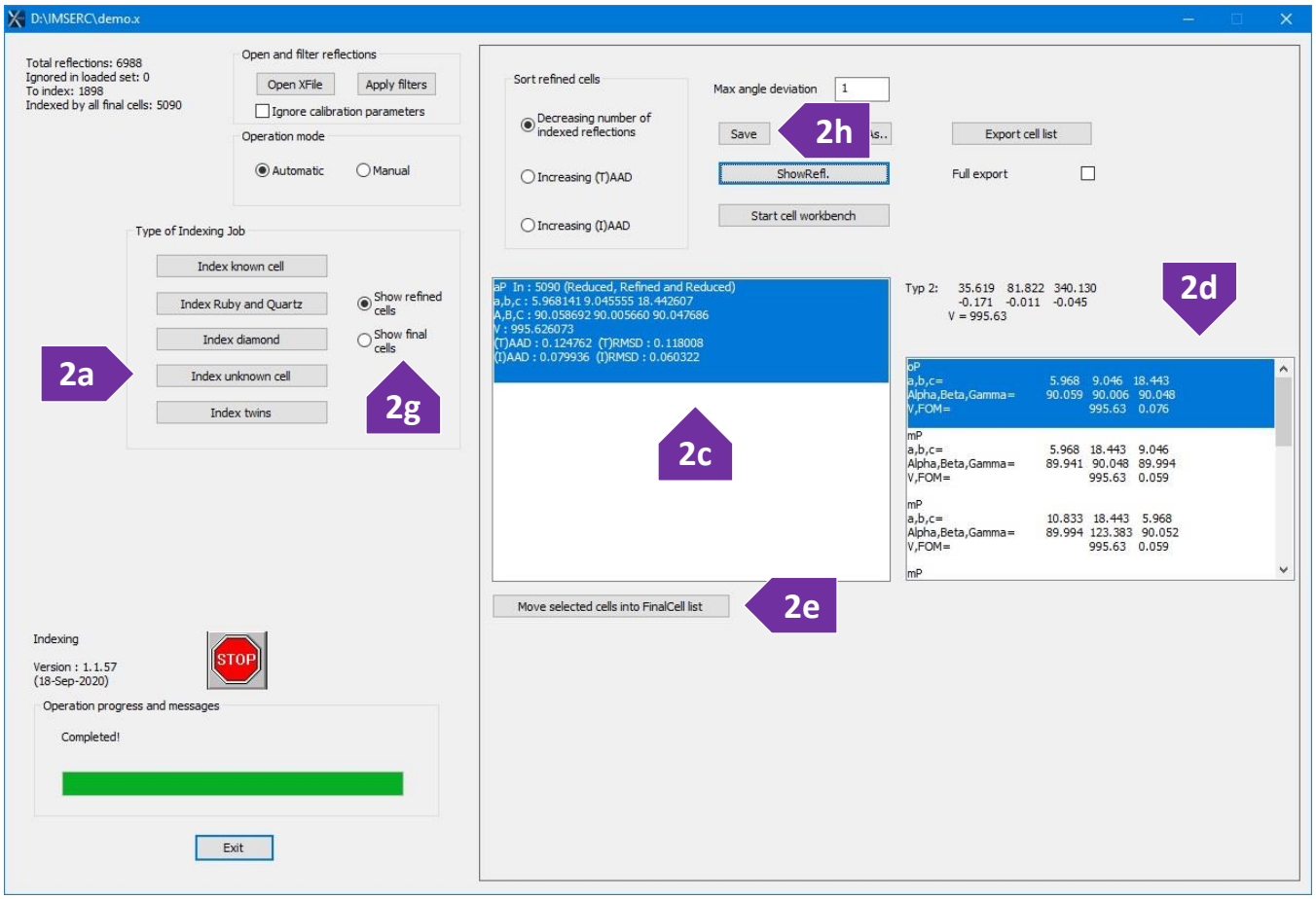


3. Select the 'StadiVariPilatus' window from the taskbar and go to the next step. If program is not running, on the main 'X-Area' window, press on the 'Instrument' menu, and select 'Measure'. It will take a few seconds for the software to initialize and start
4. For screening your crystal and find a unit cell, you need to collect a good number of unique reflections. You are going to use a pre-set number of runs for collecting data for indexing purposes:
   a. On the 'StadiVariPilatus' window, select the 'Runlist' menu, and press on the 'Edit' option. Alternatively, double press on the large white text box in the middle of the window
   b. On the 'Edit Runlist' window, press on the 'Template' button
   c. On the 'Select template' window, select the first template named as 'Ag_orient_1', and press on the 'Replace' button to add the template in your run list
   d. Press on the 'OK' button on the 'Edit Runlist' window
   e. Press on the 'Start' button on the 'StadiVariPilatus' window. This step will take you through a series of windows for finalizing your collection parameters:
      i. On the 'Conditions' window, provide the basic conditions of the setup. The only value you may have to check, and edit is the 'Temperature' value of your crystal collection. The default values are:
         1st. 'Generator voltage' is 65 kV
         2nd. 'Generator current' is 0.68 mA
         3rd. 'Collimator' is 0.3 mm
      ii. Press on the 'Next' button to go to the 'Frame Parameters and Detector settings' window. The detector distance set by the template is 40 mm. Change this value to something longer if you expect a large axis cell (> 30 Å). Leave all other settings related to detector energy and corrections as they are
      iii. Press on the 'Next' button to go to the 'Overflow handling …' window. You do not have to change any of the default values which are:
         1st. 'Overflow handling' is set to 'don't care'
         2nd. 'When a measurement is completed' is set to 'Do nothing'
      iv. Press on the 'Finish' and your screening measurement will start
   f. Progress of the measurement is shown on the 'Measurement' tab of the 'StadivariPilatus' window
   g. Diffraction can be monitored by viewing each new diffraction frame under the 'Current Frame' tab of the 'StadivariPilatus' window
   h. For a better picture of each frame, use the 'IPGraphics' window. To access 'IPGraphics', on the main 'X-Area' window, press on the 'Display' menu, and select 'IPGraphics'.

E. INDEXING YOUR CELL

Once you have some diffraction intensity data through either screening or full data collection, you are ready to start the indexing process for finding a unit cell:

1. Extract the position of reflections in each frame:
   a. On the 'X-Area' main window, press the 'Peak/Cell' menu, and select 'Find Peaks'
   b. By default, all collected runs will be selected on the 'Select Run(s)' window. Press on the 'OK' button to accept the selection
   c. On the 'PeakSearch' window, use the default 'Search Parameters', and press on the 'Start (fast)' button for the search process to start. Default parameters are 6 and 0 for the min and max I/sigma limits, respectively. Grid is set to 4, and N-skip to 0. The min 2theta limit is 0 and max depends on your collection
   d. After the search process is complete, press on the 'OK' button on the window that informs you about the number of peaks found
   e. Press on the 'Exit' button on the 'PeakSearch' window, select 'Yes' on the prompt about saving the modified Peaklist, and then select 'OK'



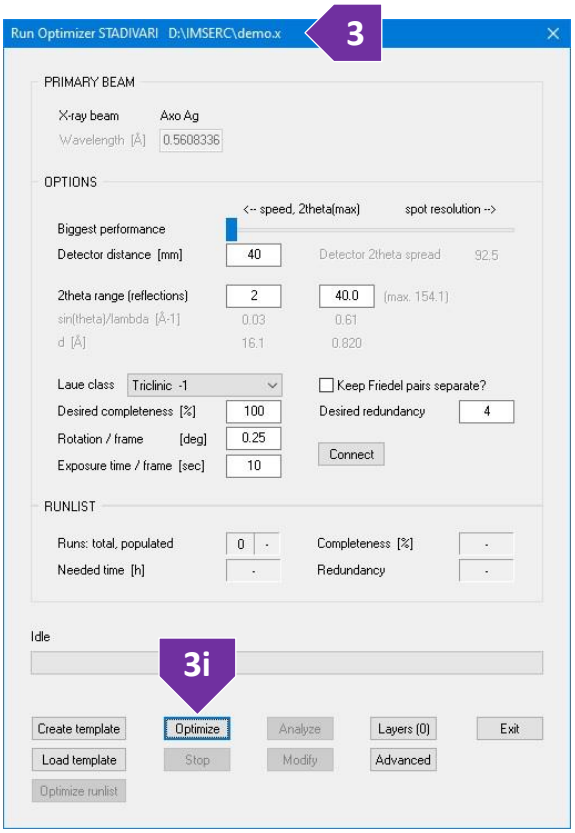
2. With the peak list generated, next step is the indexing process. On the 'X-Area' main window, press on the 'Peak/Cell' menu, and select 'Indexing':
   f. To start the indexing process, press on the 'Index unknown cell' button (figure 2a)
   g. Accept the default indexing threshold assuming a cell axis is not longer than 80 Å, otherwise change the threshold values accordingly and press on the 'Start' button to start indexing
   h. Possible cells found will be listed in the middle of the window (figure 2c). Select a suitable candidate by pressing on the list item
   i. A list of possible crystal systems for the cell selected in the previous step will be listed in the right list (figure 2d). Select a crystal system from the right list
   j. Once a crystal system is selected, press on the 'Move selected cells into FinalCell list' button to add your selection into the final list (figure 2e). Check the number of indexed and remaining reflection statistics shown on the top left corner of the window. A good quality single domain crystal should have a large fraction of reflections indexed (> 80%)
   k. In case of a twin crystal, repeat steps a-e by indexing the remaining reflections
   l. The list of final cells/orientation matrixes can be seen by selecting the 'Show final cells' radio button (figure 2g)
   m. When all orientation matrixes are defined, press on the 'Save' button to save all setting into the project file (figure 2h)
   n. Press on the 'Exit' button to exit the indexing window

F. CALCULATING COLLECTION STRATEGY

After the end of the pre-experiment and the indexing process, you are ready to determine the optimum collection strategy based on the crystal symmetry and mounting position:

1. On the 'X-Area' main window, press the 'Instrument' menu, and select 'Run Optimizer'
2. Software will prompt you to use the existing run list from the pre-experiment. Say 'No' (most common) if you do not want to keep the pre-experiment data. If you prefer keeping the pre-experiment frames, select 'Yes'
3. On the 'Run Optimizer' window (figure 3):
   a. Define the detector distance. As a rule of thumb, set the detector distance approximately at a value that is three-times the length of the longest cell axis of your crystal in a primitive setting. Minimum distance for the detector is 40 mm

b. Set the two-theta range according to the desired resolution. Given the short Ag-radiation wavelength, the large detector area, and assuming the minimum distance of 40 mm can be used, a maximum 2theta of 40 degrees will give you a resolution of 0.61 $Å^{-1}$ (0.6 $Å^{-1}$ is the minimum recommended resolution by IUCr). For Charge Density measurements or well diffracting crystals, increase the 2theta maximum to larger values. Inspect the high angle frames to see how well the crystal diffracts. In case it is needed, collect extra frames at high angles by taking single exposures using the single frame option



c. 'Laue class': 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 a lower Laue class from the drop-down list

d. 'Keep Friedel pairs separate?': If you think the compound is non-centrosymmetric (chiral or polar), then uncheck this option

e. 'Desired completeness [%]': Keep this value > 95%, ideally 100%

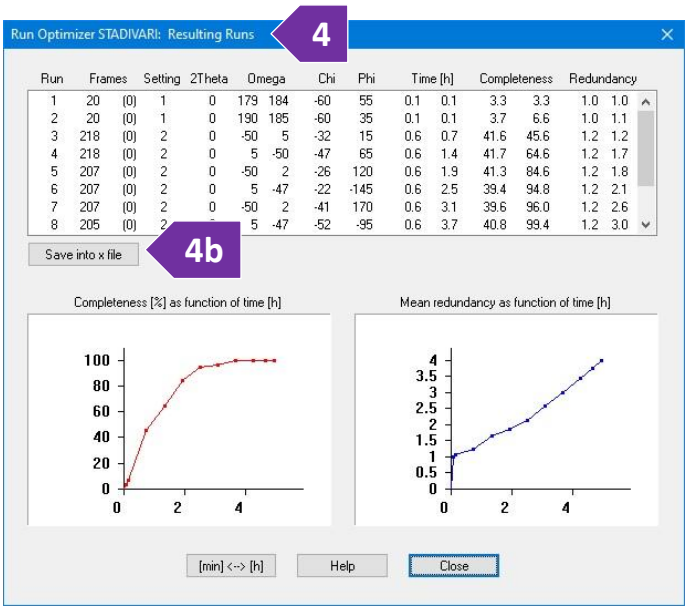
f. 'Desired redundancy': The higher the better. Redundancy of 4+ for monoclinic and triclinic systems is reasonable. Increase to 10+ for higher symmetry systems

g. 'Rotation / frame [deg]': Fine slicing is preferred, values of 0.5 or smaller are ideal

h. 'Exposure time / frame [s]': Change according to the diffraction properties of your sample. Given the short wavelength of Ag-radiation and large size of the detector, it is very possible that a single frame will have the entire 2theta range of interest. Therefore, select an exposure time that gives good intensity statistics (I/sigma > 15) for both the high-angle and low-angle reflections. As a rule of thumb, increase the exposure time by a factor of 4+ based on the exposure you have estimated from the low angle reflections, e.g., if you believe that an exposure of 10 s is good for the low angle reflections, then use at least 40 s of exposure for the entire frame

i. Press on the 'Optimize' button to start the calculation of the strategy (figure 3i) and acknowledge the information window that may appear

4. When the optimization is complete, the list of suggested runs and plots of Completeness [%] and Mean Redundancy as a function of time will be shown (figure 4)
   a. Inspect the plots and re-run the calculation if needed based on the estimated time of completion and statistics
   b. If the optimized run list looks ok, press on the 'Save into x file' button to save the run list into your project (figure 4b)
   c. Press on the 'Close' button and acknowledge all information windows by pressing on the 'OK' button
5. On the 'Run Optimizer' window, press on the 'Exit' button
6. Your optimized run is saved into your project file as a 'funlist'. Next, you may either measure crystal faces (see section 'G. Measure crystal faces for analytical absorption') or start the collection (see section 'H. Start a single crystal measurement')



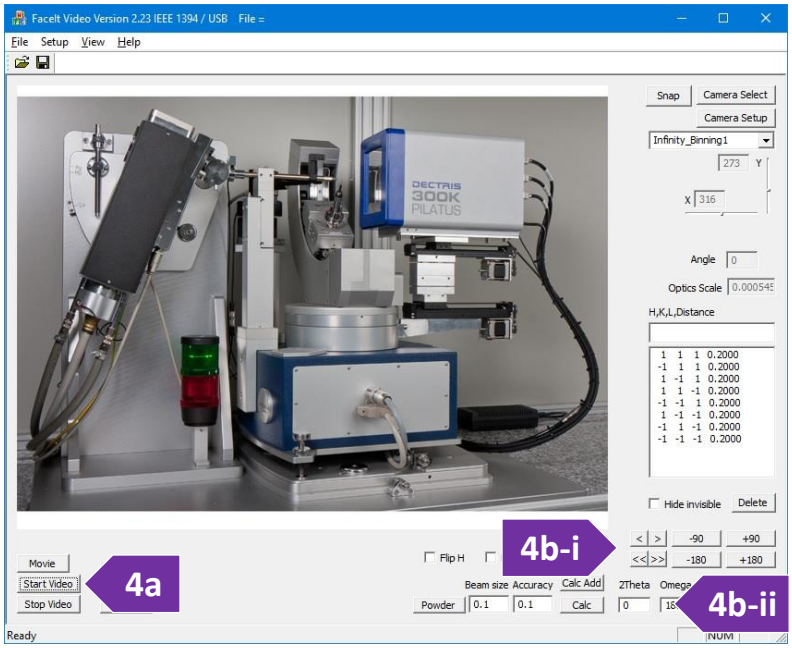
G. MEASURE CRYSTAL FACES FOR ANALYTICAL ABSORPTION

If the crystal mounted on the diffractometer has well-defined faces, it is recommended to measure the faces on the crystal in case an analytical absorption correction based of faces is needed. This type of analytical absorption is common for strongly absorbing crystals. Measurement of crystal faces can be done before or after the measurement while the crystal is still mounted on the diffractometer. In any case, at minimum, the size of the crystal must be provided for publication.

1. On the main 'X-Area' window, press on the 'Instrument' menu, and select 'Measure'. It will take a few seconds for the software to initialize and start. If the measure 'StadiVariPilatus' window is already open, skip this step
2. Pressing on the menu 'Extras', and selecting 'Synchronize Axes …'
3. Start the optical camera software for aligning your sample by pressing on the 'Start -FaceIt-' button
4. On the 'FaceIt Video' window
   a. Start the video by pressing on the 'Start' video button (figure 4a)
   b. Rotate ϕ and ω such that face to be measured is exactly perpendicular to the screen (parallel to line of vision)

i. Fine rotation of ϕ can be performed by pressing on the buttons with brackets (figure 4b-i)

ii. Rotation of ω can be performed by typing the desired value in the corresponding field (figure 4b-ii) and pressing the 'Enter' key on the keyboard



c. Left or right press on mouse and drag a line so it is parallel to the face

d. Press on 'Calc Add' button to index the face and add it to the list. Press on the 'Crystal' button to see the faces as you populate them

5. Although face indexes are saved into your project file, it is recommended to save a backup of the list with indexed faces in case you accidentally delete them during re-processing of the data. To save the face index list, on the main 'X-Area' window, press on the 'File' menu, select 'Save 'as', and select '*.crs' as the 'Save as type' option

H. START A SINGLE CRYSTAL MEASUREMENT

Once a collection strategy is calculated or a pre-experiment on a single crystal needs to be run, you are ready to start collecting intensity data. If you do not want to overwrite your screening frames, setup a new folder to separate the unit cell determination (screening frames) from the data collection.

1. On the main 'X-Area' window, press on the 'Instrument' menu, and select 'Measure'. It will take a few seconds for the software to initialize and start. If the measure 'StadiVariPilatus' window is already open, skip this step
2. Load the optimized strategy runs:
   a. On the 'StadiVariPilatus' window, select the 'Runlist' menu, and press on the 'Edit' option. Alternatively, double press on the large white text box in the middle of the window
   b. If the 'runlist' from the unit cell determination (pre-experiment) is still there, use the 'Reset' and 'Delete' button. You could keep the 'runlist' from the unit cell determination run in the same X file, but you must remember not to integrate those runs if exposure/conditions are different between the full collection and pre-experiment

c. On the 'Edit Runlist' window, press on the 'Load funlist' button

d. Press on the 'OK' button on the 'Edit Runlist' window

e. The optimized run list should appear in the list at the middle of the window. The rest of the instructions in this section are very similar to the instructions in section 'D. Screening your crystal by running a pre-experiment'

3. Press on the 'Start' button on the 'StadiVariPilatus' window. This step will take you through a series of windows for finalizing your collection parameters:

   a. On the 'Conditions' window, provide the basic conditions of the setup. The only value you may have to check, and edit is the 'Temperature' value of your crystal collection. The default values are: 'Generator voltage' is 65 kV, 'Generator current' is 0.68 mA, and 'Collimator' is 0.3 mm

   b. Press on the 'Next' button to go to the 'Frame Parameters and Detector settings' window. The detector distance set by the template is 40 mm. Change this value to something longer if you expect a large unit cell axis (> 30 Å). Leave all other settings related to detector energy and corrections as they are

   c. Press on the 'Next' button to go to the 'Overflow handling …' window. You do not have to change any of the default values which are:

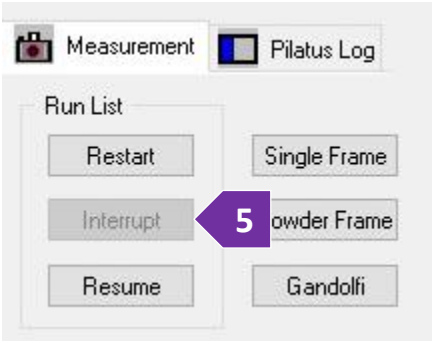
      i. 'Overflow handling' is set to 'don't care'

      ii. 'When a measurement is completed' is set to 'Do nothing'

   d. Press on the 'Finish' and your screening measurement will start

4. Progress of the measurement is shown on the 'Measurement' tab of the 'StadivariPilatus' window

5. If needed, you have the options to pause/interrupt, resume, or restart the measurement by pressing on the corresponding buttons (figure 5)



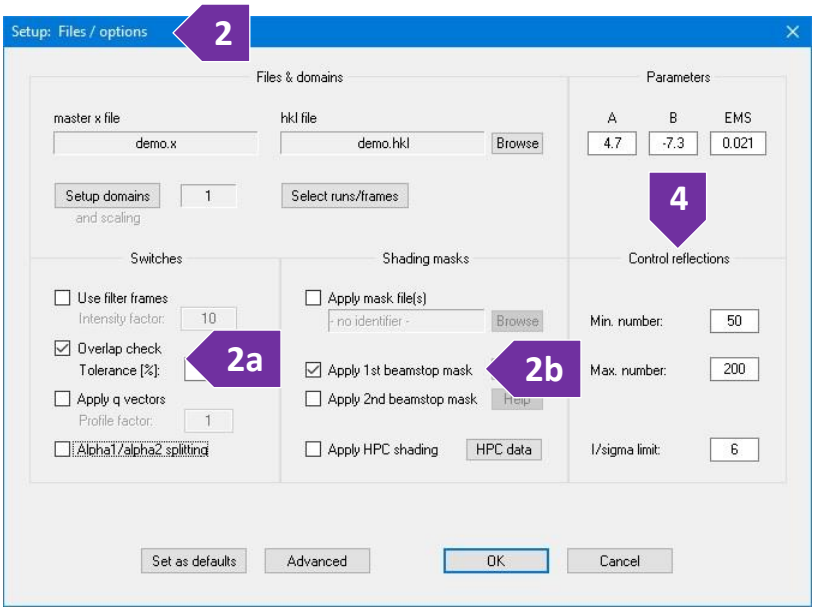
6. Once data collection is complete (or in progress), proceed to the data reduction procedure

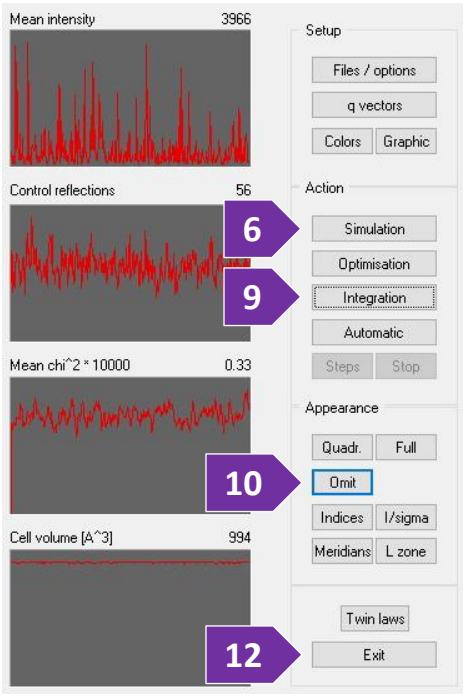
SINGLE CRYSTAL DATA REDUCTION PROCEDURE

To integrate and reduce your data to the final HKL file, you need to first integrate the data, refine the cell, determine the Laue group, scale, and apply absorption correction.

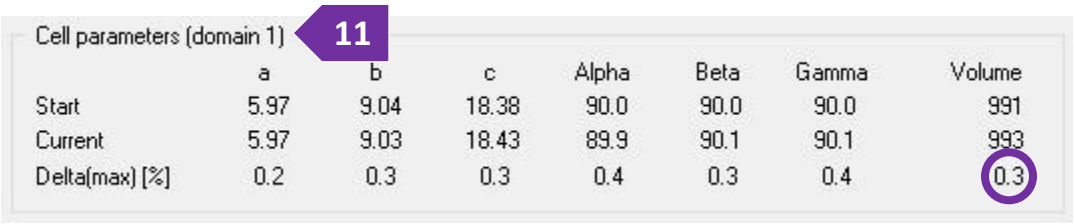
A. DATA INTEGRATION

1. On the main 'X-Area' window, press on the 'Integrate' menu, and select 'Integrate'
2. On the 'Setup: Files/Options' window that will appear, leave the default options as they are unless you are performing a non-standard integration, such as multi-twin domains, etc. (figure 2). Most options are unchecked and the only two basic options that are checked are:
   a. 'Overlap check Tolerance (%)' is set at '0' (figure 2a)
   b. 'Apply 1st beamstop mask' (figure 2b)
3. The A, B, and EMS integration parameters are sample specific and will be refined at a later step
4. The default values of control reflections that will be used for refining the orientation matrix and cell constants are 50, 200, and 6 for the minimum, maximum, and I/sigma parameters, respectively (Figure 4)
5. Press on the 'OK' button and the 'Integrate' window will be visible
6. On the 'Integrate' window, press on the 'Simulation' button to evaluate the A, B, and EMS parameters which control the shape, size, and density of ellipsoids that will be used for integration (figure 6)
7. Change the A, B, and EMS parameters by maximizing the number of useful reflections shown at the bottom of the 'Integrate' window. Useful reflections are defined as the total number of profiles minus the overlapped profiles and overflowed reflections





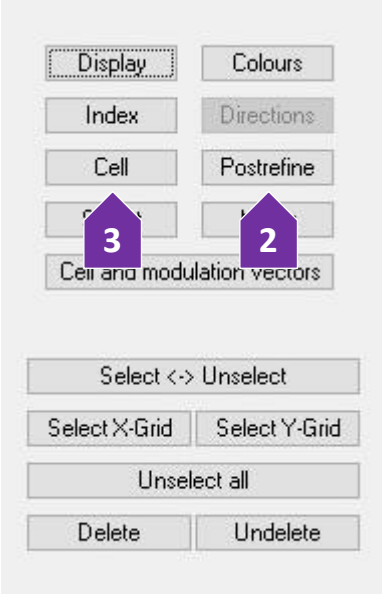
8. Repeat the A, B, and EMS parameter optimization using multiple representative frames, e.g., first, middle, and last frames. Provide an approximate average of each parameter determined for all frames and press on the 'Exit' button
9. Start the integration by pressing on the 'Integration' button (figure 9)
10. (Optional) Press on the 'Omit' button to speed up the integration process by hiding visual updates for each frame (figure 10)
11. Compare the values of the current cell (after integration) to starting cell to see if cell parameters deviated significantly. Typically, volume change should be less than 1% (circled in figure 11)



	a	b	c	Alpha	Beta	Gamma	Volume
Start	5.97	9.04	18.38	90.0	90.0	90.0	991
Current	5.97	9.03	18.43	89.9	90.1	90.1	993
Delta(max) [%]	0.2	0.3	0.3	0.4	0.3	0.4	0.3

12. Close the 'Integrate' window by pressing on the 'Exit' button (figure 12)

B. CELL REFINEMENT



1. On the main 'X-Area' window, press on the 'Peak/Cell' menu, and select 'Recipe/Index/Refine'
2. In the new window, press on the 'Postrefine' button; this will load the control reflection created during integration (figure 2)
3. Press on the 'Cell' button (figure 3), and press on the 'Refine' button on the 'Cell Parameters' window. This will refine cell parameters using the control reflections. Cycle between 'New HKL' and 'Refine' on the 'Cell Parameters' window until the refinement has converged, i.e., cell parameters do not change significantly when you press on the 'Refine' button
4. Press on the 'OK' button, then press on the 'Exit' button, and save the changes by pressing on the 'Save' button

C. SPACE GROUP DETERMINATION

Absolute configuration affects absorption correction, so please redo this step when the absolute configuration has been determined/refined.

1. On the main 'X-Area' window, press on the 'Extras' menu, and select 'X-red32'. The 'Crystal Parameter' window will appear where you can optionally modify some of the parameters

2. Check that I/σ as a function of 2θ is >3 for at least all data below 0.6 $Å^{-1}$. Above a certain 2θ value, you might see a drop in I/σ (indicates weak scattering which is usually more problematic than useful)
3. Press on the 'E-statistics' button to see if the structure is non-centrosymmetric or not. Look to see if |Z-1| is closer to theoretical value of either the non-centrosymmetric or centrosymmetric curves
4. Press on 'Spacegroup' button and according to the extinction conditions select an appropriate space group from the list:
   a. N(+): number of reflections violating symmetry elements shown on the left
   b. N(>4s): number of reflections above the 4σ level violating corresponding symmetry element
   c. Select a specific space group and press on the 'Check' button
   d. Press on the 'OK' button
5. In preparation for optimizing the shape of your crystal to perform an accurate analytical face absorption correction, you need to prepare an EQV input file for the optimization program 'X-Shape'
   a. On the 'X-Red32' main window, select the 'Extras' menu, and select 'Select X-Shape reflections'
   b. Use the default parameters of 500 reflections, min eq. 2, min I/σ 10, max relative intensity 95%
   c. Press on the 'OK' button to generate the EQV file that will be used by X-Shape

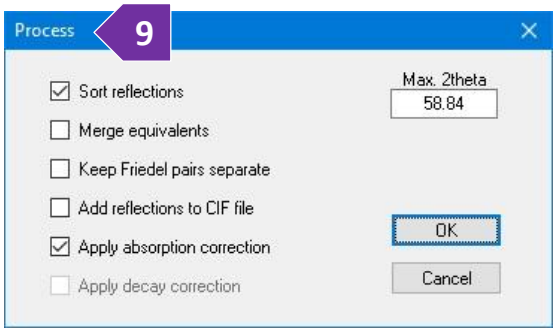
D. OPTIMIZING CRYSTAL SHAPE AND APPLYING ABSORPTION CORRECTION

Optimization of the measured faces might be needed especially when the morphology of a crystal is not well-defined, the sample absorbs X-rays strongly for the specific wavelength, and/or a precise analytical absorption is needed. For high values of μ (heavy elements and/or highly absorbing crystals), an absorption correction is critical.

1. On the main 'X-Area' window, press on the 'Extras' menu, and select 'X-Shape'
2. If a EQV input file was not created according to step #5 under 'C. Space group determination':
   a. On the main 'X-Area' window, press on the 'Extras' menu, and select 'X-red32'. The 'Crystal Parameter' window will appear where you can optionally modify some of the parameters
   b. On the 'X-Red32' main window, select the 'Extras' menu, and select 'Select X-Shape reflections'
   c. Use the default parameters of 500 reflections, min eq. 2, min I/σ 10, max relative intensity 95%
   d. Press on the 'OK' button to generate the EQV file that will be used by X-Shape
3. If faces were not indexed or erased during an integration, a 'Crystal model missing' message will appear, otherwise the measured faces will appear on the 'X-Shape' window. If you are confident that the size/distance of faces is well-measured, you can proceed to step #4 below, otherwise:

- If faces were saved into a CRS file, press on the 'File' menu, select 'Open', and load your CRS file by changing the 'Files of type' filter and browsing to the right folder
  - i. If faces were indexed for a well-defined crystal morphology but dimensions are not accurate:
  - ii. Press on the 'Single Step' button
  - iii. Select the 'Dimensions' option under 'Optimize' and press on the 'Start' button
  - iv. If lengths are off, may be due to incorrect Z value or stoichiometry
- If shape is known but no faces were measured:
  - i. Press on the 'Shape' menu and select an appropriate shape
  - ii. Press 'OK' to save the shape
  - iii. Press on the 'Automatic' button to refine all shape parameters, this process will take a few minutes

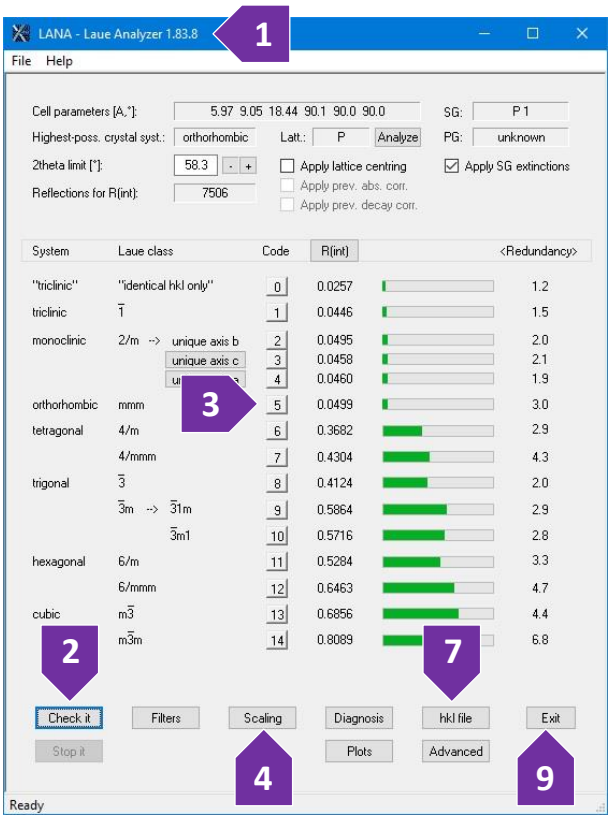
4. Close the 'X-Shape' window to save changes
5. On the main 'X-Area' window, press on the 'Extras' menu, and select 'X-red32'
6. Press on the 'Abs. Correction' button
7. Select 'Numerical' and press on the 'Start' button
8. It highly recommended to proceed to the scaling procedure described below to perform an outliner rejection
9. If for some reason an outliner rejection is not needed, press on the 'Process' button, select the desirable settings, and press on the 'OK' button to create the final HKC file. Recommended settings for the 'Process' window are (figure 9):
   - 'Sort reflections' enabled
   - 'Merge equivalents' disabled
   - 'Keep Friedel pairs separate' disabled
   - 'Add reflections to CIF file' disabled
   - 'Apply decay correction' disabled



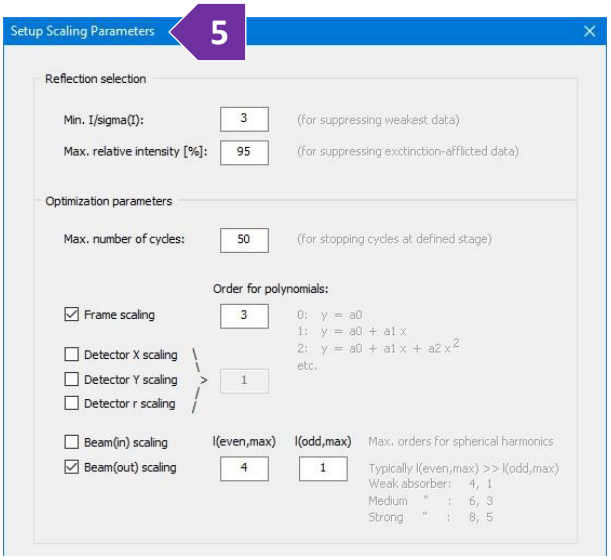
10. For solving and refining your structure, you will need the corrected HKL file, i.e., HKC. Cell parameters and other metrics are saved in the SUM file. Additional information about the experimental details and parameters used for your collection are saved in the cfx_LANA file
11. Close 'X-Red32' by pressing the 'Exit' button

E. SCALING AND HKL FILE CREATION

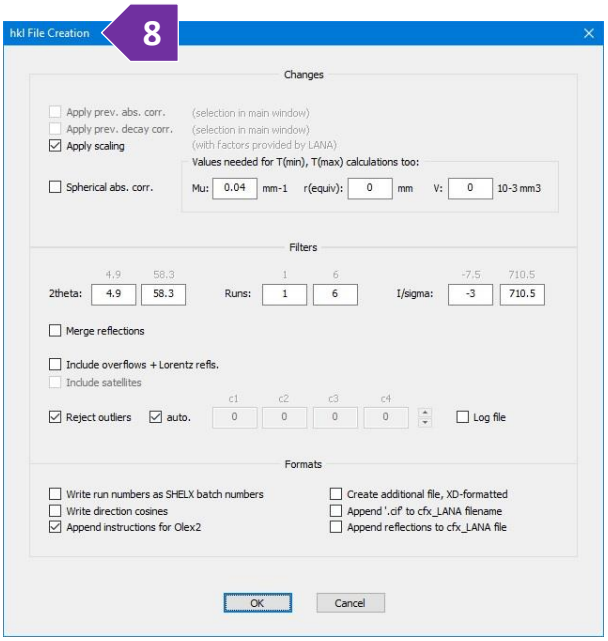
Scaling of the integrated intensity along with rejection of outlined reflection needs to be performed to generate the final HKL file that can be used for solving and refining your structure.



1. On the main 'X-Area' window, press on the 'Extras' menu, and select 'Laue Analyzer (LANA)' (figure 1)
2. Press on the 'Check It' button to check the R_{int} of your data against all possible Laue groups (Figure 2). This process will give you an idea about the proper Laue group for your data. A good starting choice is usually the highest Laue group before the R_{int} jumps (e.g., in figure 1, this would be orthorhombic)
3. Press on the button with the number next to the proper Laue group to select the desired point group (figure 3). Choose acentric or centrosymmetric, according to the space group selection as described in section 'C. Space group determination'
4. Once the point group is selected, press on the 'Scaling' button (figure 4) to set the scaling parameters and to open the 'Setup Scaling Parameters' window (figure 5)
5. Some scaling parameters might be data set specific, but the suggested starting values (figure 5) for most collections are:
   a. 'Min I/sigma(I)' set at 3
   b. 'Max. relative intensity [%]' set at 95
   c. Max. number of cycles set at 50
   d. Frame scaling checked and set at 3
   e. Beam(out) scaling checked and set at 4 [I (even,max)] and 1 [I(odd, max)]
6. Press on the 'OK' button to save the scaling parameters
7. On the 'LANA' main window, press on the 'hkl File' button (figure 7) to set the final parameters for creating the HKL file and to open the 'hkl File Creation' window



8. On the 'hkl File Creation' window (figure 8):
   a. Ensure that the 'Apply scaling' option is selected
   b. If an analytical (using crystal faces) or empirical absorption correction have been applied (see section 'D. Optimizing crystal shape and applying absorption correction'), the 'Apply prev. abs. corr' will be selected but still inactive for you
   c. Transmission coefficient and 2theta limits are set by the software based on the formula, crystal size, and data collected. Modify 2theta limits only when you want to exclude data from the HKL file
   d. Enable the 'Reject outliers' option and select 'auto.'
   e. Check the 'Append instructions for Olex2' option if you are using Olex2
   f. Press on the 'OK' button to save the HKL file, and select 'Yes'/'OK' on any pop-up windows



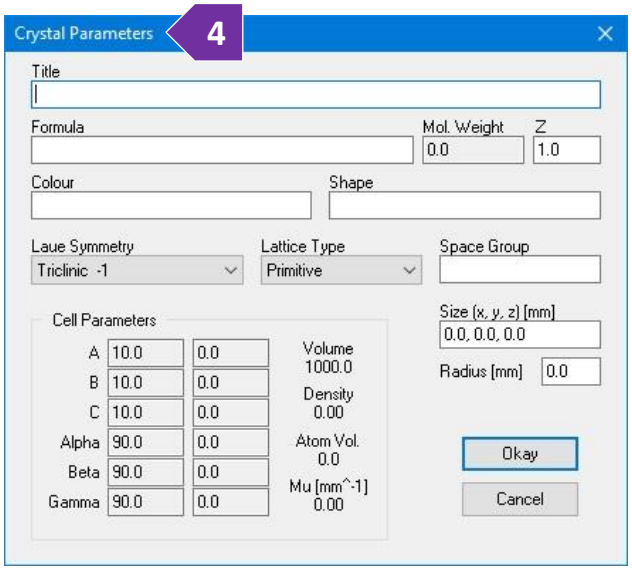
9. Exit 'LANA' by pressing on the 'Exit' button (figure 9)
10. For solving and refining your structure, you will need the corrected HKL file, i.e., HKC. Cell parameters and other metrics are saved in the SUM file. Additional information about the experimental details and parameters used for your collection are saved in the cfx_LANA file. In case you are using Olex2, you can open directly the created HKL file (assuming option described in section 8e is selected)

SETTING UP A POWDER MEASUREMENT

The process of setting up a powder measurement is very similar to the process followed for single crystal collection with the only difference being that there is no need to index the pattern to calculate a strategy.

A. SETTING PARAMETERS FOR A POWDER EXPERIMENT

1. Verify that the instrument is idle (check 'Default instrument status' section)
2. In case the acquisition software (X-Area) is not running, double-press on the 'X-Area' icon on the desktop
3. Create a new project file by pressing on the menu 'File' and then 'New'. Save the project file under a new folder in your personal folder, e.g., D:\SupervisorLastname\LastName-FirstName
4. Provide information about the powder on the 'Crystal Parameters' window (figure 4). Most information on this window is optional and software will let you press 'Okay'. It is highly recommended to provide as much information as possible for each project, so this information is transferred to the final CIF:

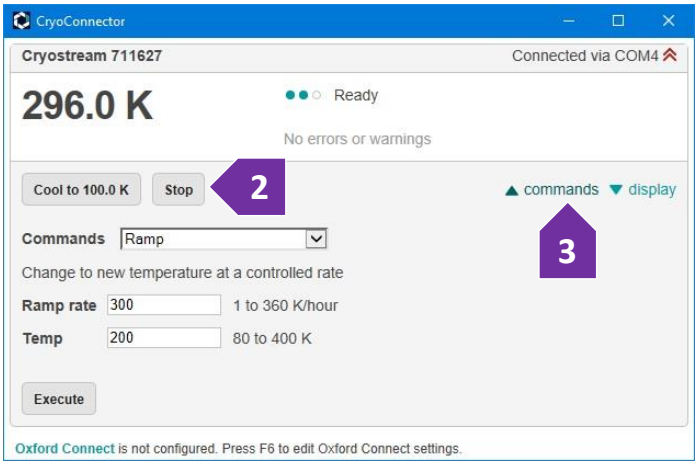


   a. Title of the project, e.g., notebook number
   b. Formula of the compound in the form of chemical element followed by stoichiometry (no space). Separate each group of element + stoichiometry with a space, e.g., C10 H11 O S2. The information provided in the 'Formula' field will be used for analytical absorption correction. The molecular weight label will be updated accordingly
   c. 'Z' number of your compound. This number will affect the absorption coefficient (Mu) every time that is updated
   d. Color of the powder
   e. Shape of the crystallites
   f. Symmetry related information, such as Laue, lattice type, and space group will be updated during the reduction process (using the 'X-Red32' module)
5. Press on the 'Okay' button to save the parameters of your crystal

B. SETTING THE TEMPERATURE OF YOUR DATA COLLECTION

In case you are performing a non-ambient temperature measurement, you need to set the base temperature of the temperature device in use. When using the Cryostream device (for measuring between 80K and 500K):

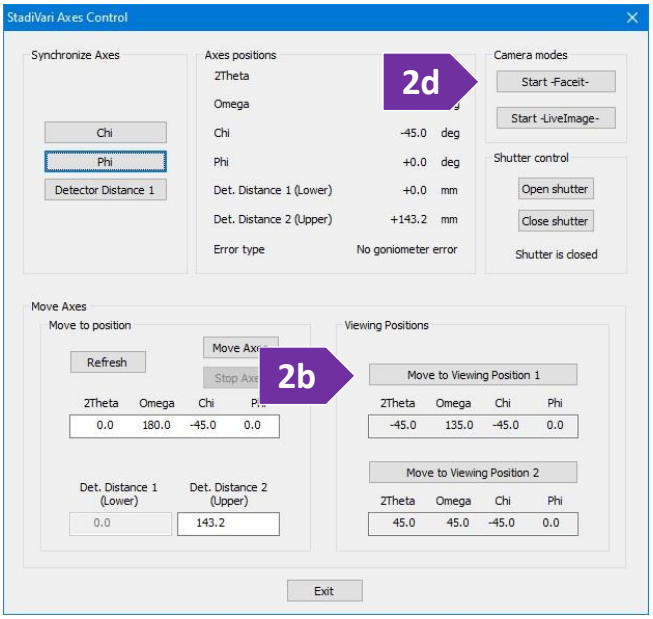
1. Connect to the controller by launching the 'CryoConnector' software by double-pressing on the icon on the desktop. It will take a few seconds for the software to connect to the controller
2. Depending on the state of the controller, you have the option to either run a command directly or restart the controller:
   - If the 'Cool to 100.0 K' button is enabled, then the controller is online, and you can run any command. If you just need to cool down to 100 K as fast as possible, then just press on the 'Cool to 100.0 K' button and proceed to the next section 'C. Mounting and aligning your powder sample on the goniometer'
   - If the 'Cool to 100.0 K' button is disabled, then you need to press on the 'Restart' button next to it (figure 2 where the 'Stop' button is) and wait for a few seconds for the controller to restart

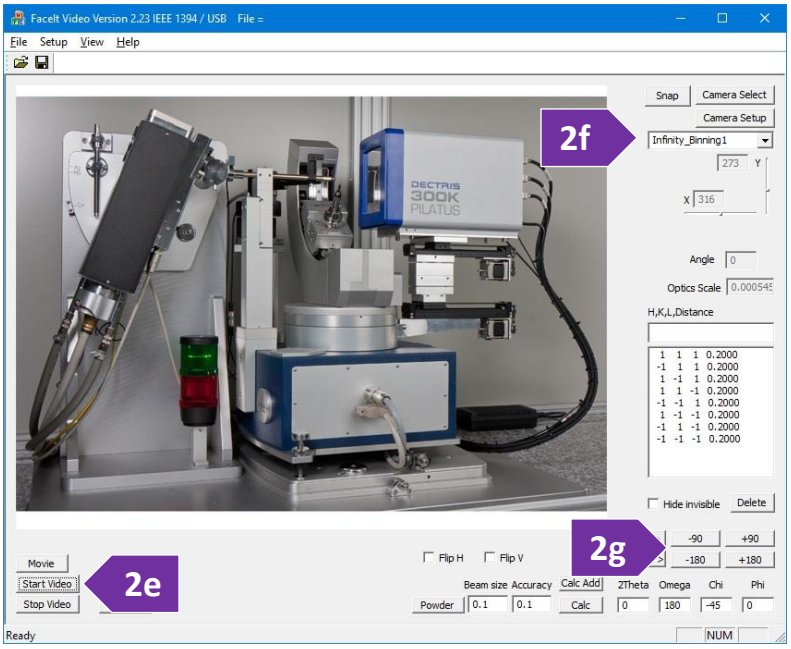


3. For changing/setting temperature, you have two options depending on if the temperature change must have a fixed rate (controlled) or not (uncontrolled):
   - [Uncontrolled] In case you want to cool down to a specific temperature as fast as possible, select from the 'Commands' drop-down menu the 'Cool' option. If list of commands is not visible, press on the 'commands' tab (figure 3). Provide the target temperature and press on the 'Execute' button
   - [Controlled] For changing the temperature at a controlled rate, select from the 'Commands' drop-down menu the 'Ramp' option, provide the target temperature and desired rate, and press on the 'Execute' button
4. You can proceed with the next steps, regarding sample mounting and screening, while the temperature is changing. The 'CryoConnector' software sends commands to the controller which controls the cryostat directly. Therefore, in case you accidently close the 'CryoConnector' software, the cryostat will keep operating as instructed
5. Remember to warmup the cryostat when done with your measurements. To warmup, under the 'Commands' dropdown menu, select 'End', provide the maximum rate (360K/hour), and press on the 'Execute' button

C. MOUNTING AND ALIGNING YOUR POWDER SAMPLE ON THE GONIOMETER

1. On the main 'X-Area' window, press on the 'Instrument' menu, and select 'Measure'. It will take a few seconds for the software to initialize and start
2. Drive all axis to the mounting position by:
   a. Pressing on the menu 'Extras', and selecting 'Synchronize Axes …'
   b. Pressing on the 'Move to Viewing Position 1' button on the 'StadiVari Axes Control' window (figure 2b). Target values for the viewing position are shown under the button
   c. Waiting for the goniometer to reach the viewing position before going to the next step
   d. Start the optical camera software for aligning your sample by pressing on the 'Start -FaceIt-' button (figure 2d)
   e. Start the video by pressing on the 'Start' video button (figure 2e)
   f. Mount your sample on the goniometer head and start the alignment process. Watch the '[stadivari-alignment-crystal]' video for a visual demonstration of the alignment process. In case you have to change the magnification of the optical camera, change the binning option from 'Binning1' (high magnification) to 'Binning4' (low magnification) (figure 2f)
   g. As instructed in the alignment video, toggle between the '+/- 180', and '+/- 90' buttons to adjust the centering (figure 2g)
   h. Exit the optical camera software
   i. Exit the 'StadiVari Axes Control' window



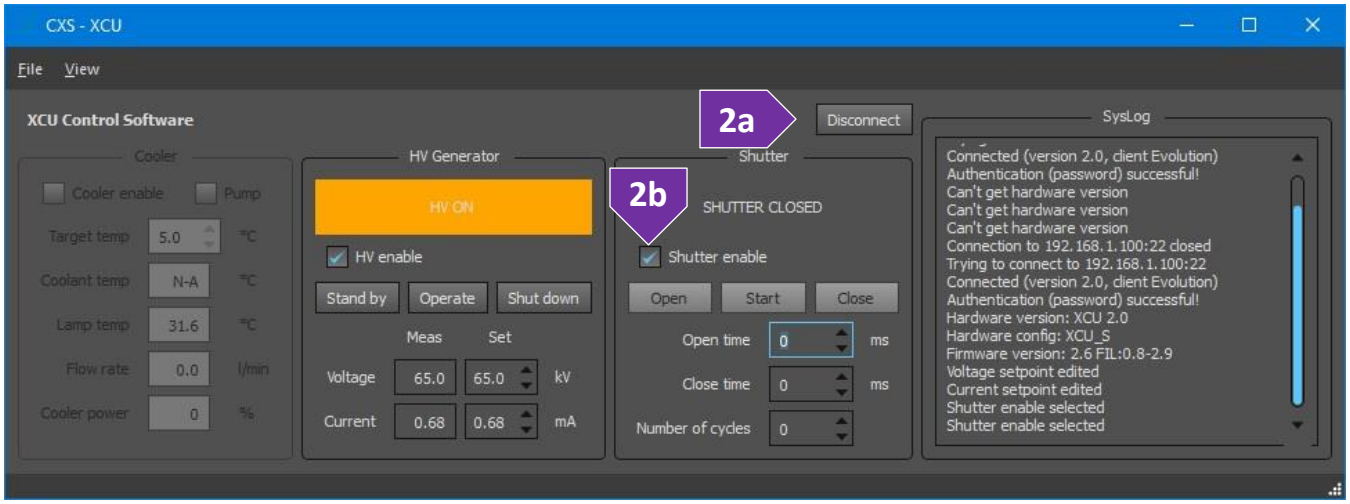


3. Close the safety windows of the enclosure and make sure the green light next to the top emergency button is on (figure 3). Watch the 'stadip-safety-windows' video for a visual demonstration (the safety enclosure system is identical between StadiVari, StadiP, and StadiMP)

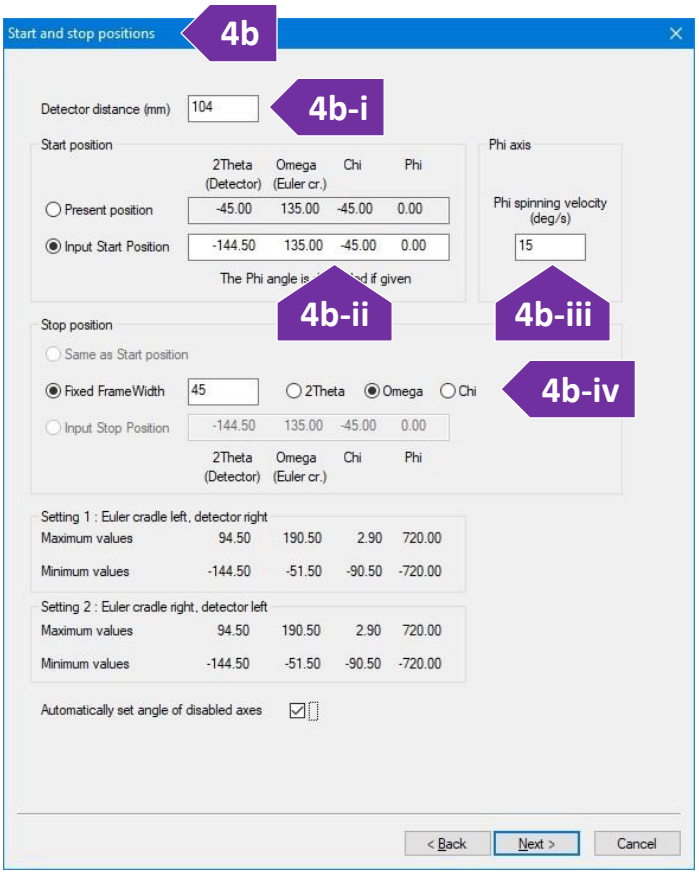
D. START A POWDER MEASUREMENT

There are two main ways for measuring powder samples on the single crystal diffractometer. One option is to keep most axes fixed and spin Phi ('Powder' button), and the other option is to change either omega (most common) or 2theta or chi at the same time with Phi ('Gandolfi' button). The 'Powder' method works well for most of polycrystalline powder samples. For samples that might be highly oriented and textured even when packed in capillaries, the 'Gandolfi' method is highly recommended.

1. Ensure that the door safety light is green (figure 1).
2. Select the 'CXS-XCU' window from the taskbar. If program is not running, press on the 'CXS-XCU' icon on the desktop:
   a. If you just started 'CXS-XCU', press on the 'Connect' button (figure 2a, the 'Connect' button is at the same location with the 'Disconnect' button shown on the figure) for the software to connect to the X-ray generator
   b. Enable the checkbox 'Shutter enable' option (figure 2b). This option needs to be ***re-checked/enabled every time you open the safety windows***. If the 'Shutter enable' option remains unchecked during a data collection, there will be no X-rays going through your sample and all frames will be empty



3. On the main 'X-Area' window, press on the 'Instrument' menu, and select 'Measure'. It will take a few seconds for the software to initialize and start
4. Press on either the 'Powder' or 'Gandolfi' button on the 'StadiVariPilatus' window. This step will take you through a series of windows for finalizing your collection parameters:
   a. On the 'Conditions' window, provide the basic conditions of the setup. The only value you may have to check, and edit is the 'Temperature' value of your crystal collection. The default values are:
      i. 'Generator voltage' is 65 kV
      ii. 'Generator current' is 0.68 mA
      iii. 'Collimator' is 0.3 mm
   b. Press on the 'Next' button to go to the 'Start and stop positions' window (figure 4b)
      i. Change the detector distance value to something longer than 40 mm in case you expect a large unit cell (figure 4b-i)
      ii. Set the 'Start Position' of all axes for the powder measurement. The most common starting values are 0, 180, -45, and 0 for the 2theta, Omega, Chi, and Phi axes, respectively (figure 4b-ii). See Appendix A for more information about the directions of the various axes of the Eulerian goniometer
      iii. Set the 'Phi spinning velocity' to 15 deg/s or higher in case your sample does not contain fine (< 50 µm) grains (figure 4b-iii)
      iv. In case you are using the 'Gandolfi' method, provide the number of degrees ('Fixed FrameWidth') and specific axes you would like to move during the powder collection. Typical values are 45 degrees for Omega (figure 4b-iv)
   c. Press on the 'Next' button to go to the 'Frame Parameters and Detector settings' window.



i. Set the exposure time based on the diffraction properties of your samples. You might want to do a quick collection (30-60 sec) and then increase exposure if needed

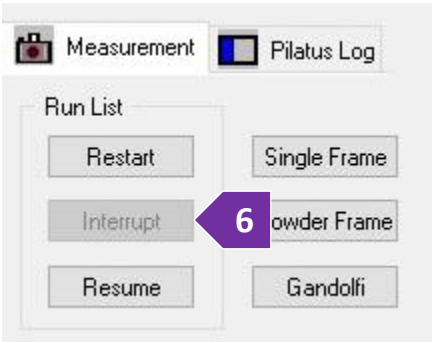
ii. Detector distance is set by the previous step, but you have the option to change it under this menu too

iii. Leave all the energy related options on their default values. These energy settings are used only when your sample fluoresces with Ag-radiation

d. Press on the 'Next' button, and provide the path and filename for the powder collection

e. Press on the 'Finish' button, and your powder measurement will start

5. Progress of the measurement is shown on the 'Measurement' tab of the 'StadivariPilatus' window
6. If needed, you have the options to stop/interrupt the measurement by pressing on the corresponding button (figure 6)
7. For a quick inspection of the powder frame (raw 2D data) after collection is over, on the main 'X-Area' window, press on the 'Display' menu, select 'IPGraphics', and load the frame

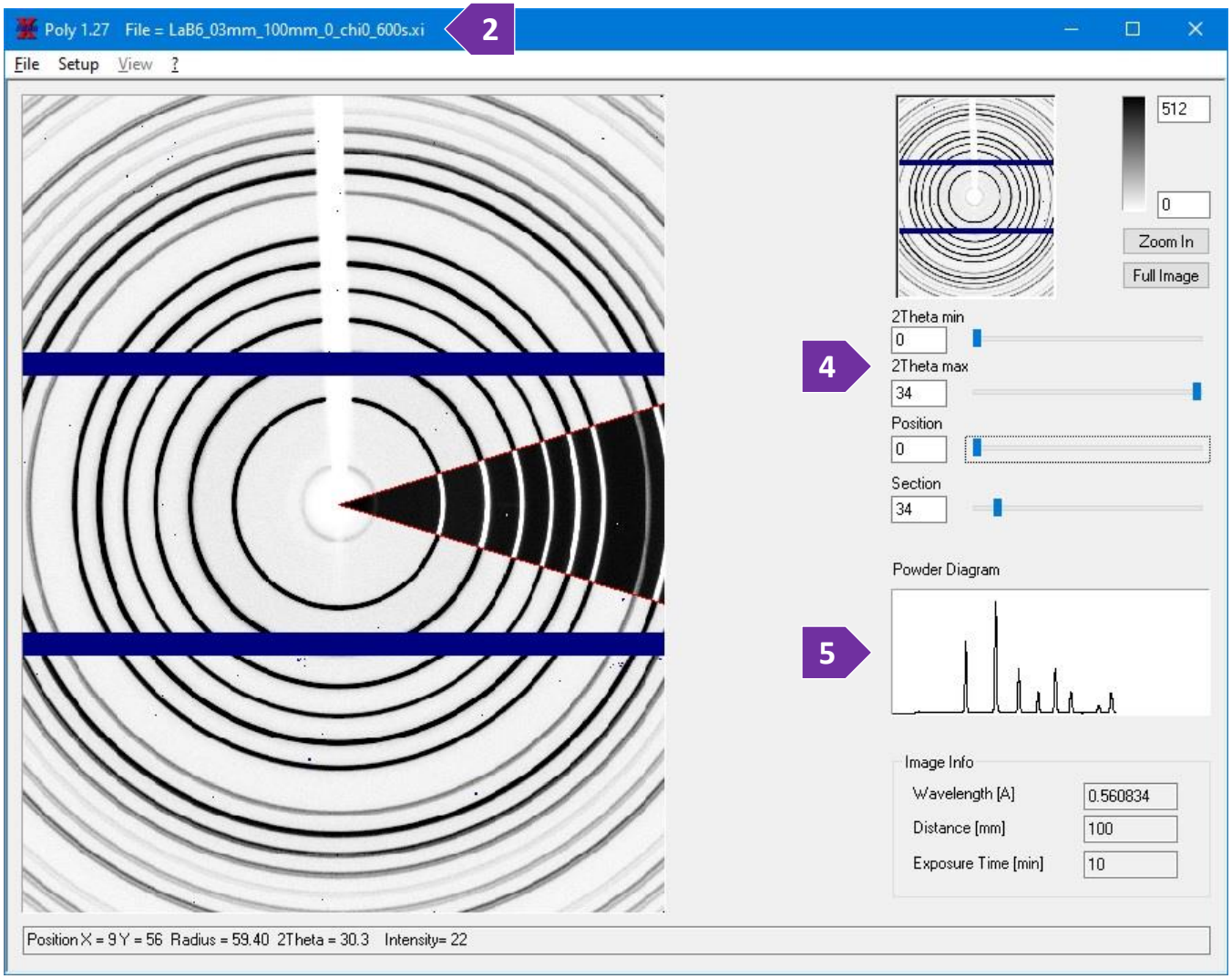


POWDER DATA REDUCTION PROCEDURE

This process will help you integrate the 2D powder frames and export the corresponding 1D powder patterns.

A. 2D POWDER DATA INTEGRATION WITHOUT TEXTURE ANALYSIS

1. On the main 'X-Area' window, press on the 'Extras' menu, and select 'Create Powder Diagrams'
2. On the 'Poly' window that appears (figure 2), load your powder frame by pressing on the 'File' menu, and selecting 'Open'



3. Mask any unwanted area, e.g., section blocked by the beamstop, by loading the corresponding mask file. To load a mask file, under the 'File' menu, select the 'Load Mask' option. Various standard masks files can be found under the 'D\Procedures\Masks' folder on the instrument computer. You can also create your own mask by using the corresponding module located under the 'Instrument' menu of the main X-Area panel, and selecting the 'Define Shading Marks' option

4. Select the entire 2D area or a wedge by adjusting the 2theta limits, position, and section parameters (figure 4). Look at the shaded section on the 2D pattern for a visual representation of the parameter settings you are adjusting
   - 'Position' sets the starting point of the integration
   - 'Section' sets the wedge in degrees that will be integrated
5. The preview of the integrated 1D powder pattern for the selected above parameters will be displayed in the 'Powder Diagram' window (figure 5). You can double-press on the graph to see a magnified view of the pattern
6. To save the 1D pattern, under the 'File' menu, select the 'Save as' option, and provide the path and filename for the exported 1D pattern file. You have two export options: 1) FullProf file that can be opened from the third-party FullProf software, and 2) Powder RAW file that can be ready with the WinXPow software.

B. 2D POWDER DATA INTEGRATION FOR PDF ANALYSIS

For the reduction of powder data for PDF analysis, the use of GSAS-II is recommended. The process is very similar to the workflow used at the Advance Photon Source (APS) at Argonne National Laboratory (ANL).

1. Use GSAS-II and import the CBF files with powder data
2. On the StadiVari instrument computer, under the 'D\Procedures\Standards' folder, copy the LaB_6 collection that matches the detector distance you used for your measurement. Use the LaB_6 file for calibrating the distance and calculating the other geometrical corrections needed by GSAS (see detailed GSAS-II tutorial)
3. For every imported file, ensure that the 'Detector 2-theta' value is the nominal 2theta value as shown on the file name of your collection, e.g., '$LabB_6$_-80_chi45_3600s.cbf' was collected at -80 degrees 2theta. The imported from the header value is '-80.3060' which needs to be set to '-80'
4. For each sample CBF file, under 'Image Controls' set the 'Dark image' to the corresponding background image file of air. Data for air scattering as a function of 2theta are under the 'D\Procedures\Backgrounds\Air' folder
5. Integrate all 2D images for each sample and export the 1D patterns
6. Stich the individual 1D patterns into one pattern. Scaling may be needed
7. Repeat steps 1-6 for the empty Kapton capillary using the corresponding data under the 'D\Procedures\Backgrounds\Kapton' folder

C. 2D POWDER DATA INTEGRATION WITH TEXTURE ANALYSIS

Texture analysis of 2D powder data can be performed with GSAS-II by loading the CBF files you have collected on the diffractometer. Please read the detailed tutorial in GSAS-II on how to perform the analysis.

PUBLICATION

A. EXPERIMENTAL SECTION

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 a STOE StadiVari diffractometer equipped with an AXO Ag Kα micro-focus sealed X-ray A-MiXS source (λ = 0.560834 Å), running at 65 kV and 0.68 mA, and a Dectris Pilatus3 R CdTe 300K Hybrid Photon Counting detector. **[Temperature of the crystal was controlled with an Oxford Cryosystems low-temperature device.]** Data reduction was performed with the X-Area software package using an ***(empirical | numerical)*** absorption correction **[using X-Shape]**. 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

"This work made use of the IMSERC Crystallography facility at Northwestern University, which has received support from the Soft and Hybrid Nanotechnology Experimental (SHyNE) Resource (NSF ECCS-2025633), and Northwestern University. Purchase of the Ag-microsource diffractometer used to obtain results included in this publication was supported by the Major Research Instrumentation Program from the National Science Foundation under the award CHE-1920248"

TROUBLESHOOTING

A. THE COMPUTER SCREEN WILL NOT TURN ON

Begin your reservation in NUcore to initiate access to the instrument

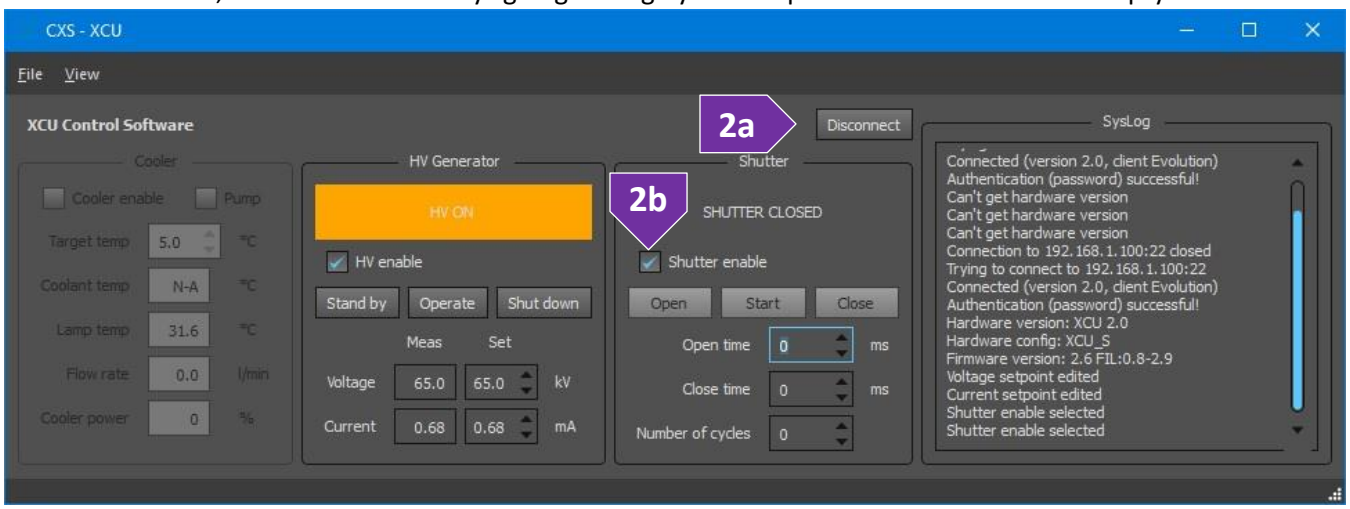
B. COMPUTER REQUIRES LOGIN AND A PASSWORD

The default 'stadivari' user account should be logged in. In case the computer was restarted, the password for the 'stadivari' account is . See 'Default instrument status' section for more details

C. THERE IS NO DIFFRACTION INTENSITY

1. Verify that all six safety doors are closed and aligned, and the safety green light is on (figure 1). If the green safety light is not on, reseat all safety doors by opening and closing each door. Watch the 'stadip-safety-windows' video for a visual demonstration

2. Ensure that the 'CXS-XCU' software is running and the checkbox 'Shutter enable' is ticked. If program in not running, press on the 'CXS-XCU' icon on the desktop:
   a. If you just started 'CXS-XCU', press on the 'Connect' button (figure 2a, the 'Connect' button is at the same location with the 'Disconnect' button shown on the figure) for the software to connect to the X-ray generator
   b. Enable the checkbox 'Shutter enable' option (figure 2b). This option needs to be ***re-checked/enabled every time you open the safety windows***. If the 'Shutter enable' option remains unchecked during a data collection, there will be no X-rays going through your sample and all frames will be empty



3. Check the voltage and current settings of the X-ray generator. Generator is in the right side of the rack, behind the door cabinet. Default operating settings are 65 kV and 0.68 mA (figure 3). In case values on the generator are zero, contact a Crystallography Staff

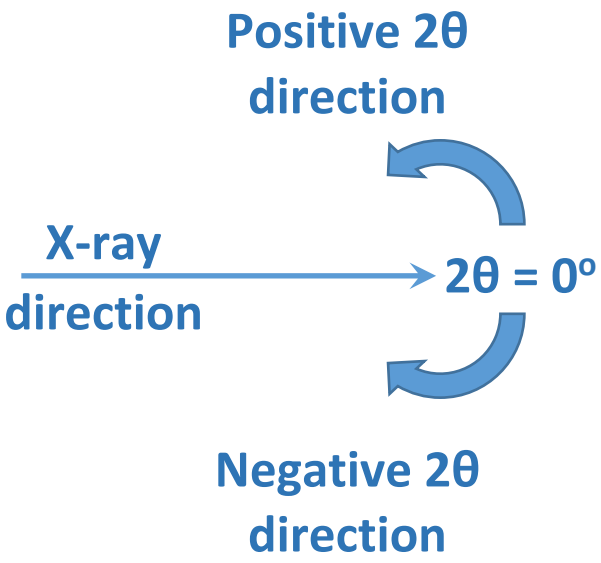
D. THERE IS AN ERROR OR PROBLEM WITH THE INSTRUMENT NOT ADDRESSED UNDER THIS TROUBLESHOOTING SECTION

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

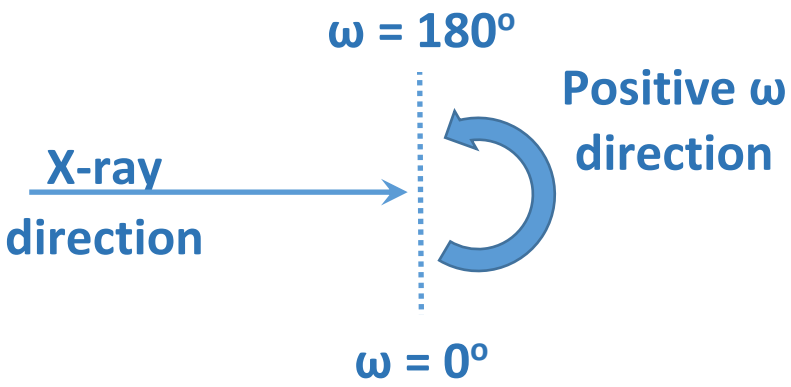
1. If you have already started your reservation using NUcore, please end your reservation and select the error reporting option with a brief description about the issue. Place the 'Stop' sign near the instrument computer to notify users immediately after you. 'Stop' signs are located on the shelf above 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

APPENDICES

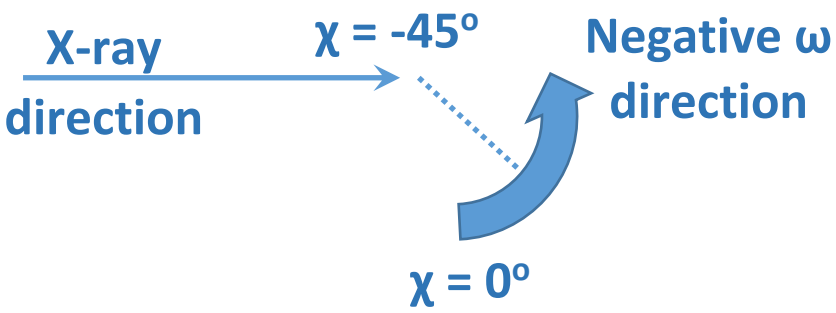
APPENDIX A: DIRECTIONS OF GONIOMETER AXES



Direction of 2θ axis in respect to the X-ray beam direction



Direction of ω axis in respect to the X-ray beam direction



Direction of χ Eulerian axis in respect to the X-ray beam direction

REVISIONS

v1.03 2021/09/22	• Training video files transferred from Box to OneDrive, and all relevant links were updated
v1.02 2021/06/27	• Release of original version of the user manual for XCU 2.0, MainMenu 1.90, StadiVariPilatus 1.31.170, IPGraphic 2.6.0.4, PeakSearch 1.34.12, Indexing 1.1.57, Recipe 1.36, Peaklist 2.06, Integrate 1.78.3, LANA 1.83.8, X-Red32 1.65.2, X-Red32(TwinAbs) 1.64.1, X-Shape 2.21, Decay 1.54.3, Scale 1.55.2, Poly 1.27, Mask 1.33