

IMSERC User Manual for STOE StadiMP

CONTENTS

Introduction.....	3
Safety.....	3
Data management.....	4
Software.....	4
Default instrument status.....	4
General steps for sample preparation and packing in transmission.....	7
A. Pack sample in a flat disc transmission holder using polyimide or acetate tape.....	7
B. Pack sample in a flat disc transmission holder using acetate foils.....	8
General steps for data acquisition.....	9
Run samples with the flat disc transmission holder.....	13
Run samples in reflection mode.....	16
Run samples in capillary in Debye-Scherrer mode.....	19
A. With the goniometer head.....	19
B. With the capillary autosampler.....	22
C. With the permanently aligned capillary holder.....	25
Run samples at high temperature in capillary with the HT furnace.....	28
Run samples at low temperature (80-500 K) in capillary with the cryostat.....	36
Run samples at high temperature in capillary using the <i>in-situ</i> HT2 furnace.....	42
Publication.....	50
A. Experimental section.....	50
B. Acknowledgement.....	50
Troubleshooting.....	51
A. The computer screen will not turn on.....	51
B. Computer requires login and a password.....	51
C. There is no intensity or the diffraction is very weak during collection.....	51
D. Temperature of the HT furnace will not increase.....	51
E. Sample stage will not spin.....	52
F. Specific error messages.....	52
G. There is an error/problem with the instrument that is not addressed under the troubleshooting section..	53

Appendices	54
Appendix A: Alignment tools and position of adjustment screws on the Debey-Scherrer goniometer head....	54
Appendix B: Comparing powder patterns collected at different wavelengths.....	56
Revisions.....	57

IMSERC User Manual for StadiMP (v1.33)

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
- Compressed Gas Cylinders

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.

IMSERC User Manual for StadiMP (v1.33)

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/StadiP' every 10 minutes. 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 'WinXPow'. 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/STADI-MP-P'. 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

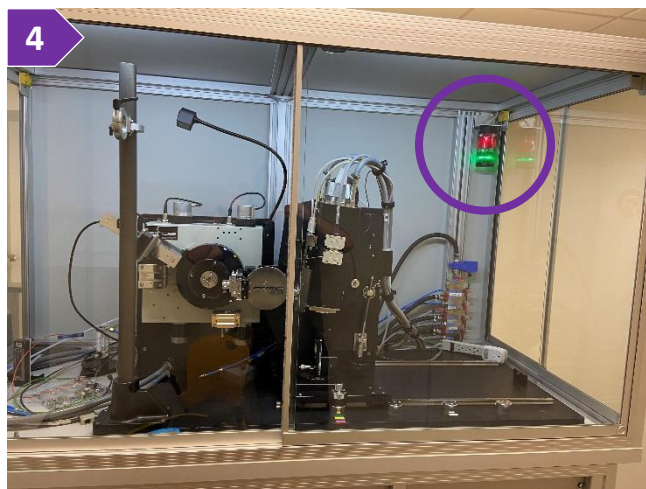
DEFAULT INSTRUMENT STATUS

The default measurement mode of StadiMP is **room temperature collections in transmission geometry with flat disc holders and pure MoK α 1-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 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 Crystallography staff member or check the corresponding capabilities section at <http://imserc.northwestern.edu/crystallography-instruments.html>.

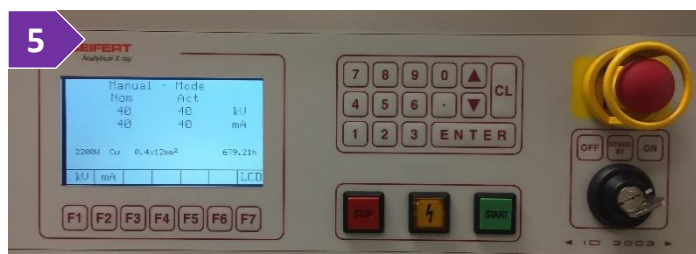
IMSERC User Manual for StadiMP (v1.33)

The default working condition of StadiMP 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 'StadiMP' user account should be logged in. In case the computer was restarted, the password for the 'StadiMP' account is
3. Acquisition software should be running. Leave the acquisition software open when you are done with the measurement



4. Green light on the safety beacon at the top left corner in the enclosure must be on (figure 4)
5. X-ray generator (located in the middle of the rack of the diffractometer, figure 5) should be on with voltage and current values of 50 kV and 40 mA (Mo-radiation) or 40 kV and 40 mA (Cu- or Ag-radiation), respectively



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

1. Leave the acquisition software running
2. End your reservation in NUcore
3. In case transmission holders were used, do not unscrew all the way the screws that fix the metallic mask onto the holders, one turn counterclockwise is sufficient
4. Leave lab tables clean and tools/accessories organized
5. Return any unused metallic masks into the compartmentalized container

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:

IMSERC User Manual for StadiMP (v1.33)

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

IMSERC User Manual for StadiMP (v1.33)

GENERAL STEPS FOR SAMPLE PREPARATION AND PACKING IN TRANSMISSION

Please follow these general steps as discussed during your training sessions. Pack your powdered sample into the appropriate holder depending on the type of measurement. In more details:

- Use the flat plate disc holder in transmission mode for qualitative analyses on powder sandwiched between acetate, polyimide, or mylar foils (high throughput option up to 30 flat plates). Use the appropriate foil based on the details discussed during training. Polyimide and acetate foils are available to all users and supplies are located on the bench near the instrument
- Use glass capillaries in Debye-Scherrer (transmission) mode for samples in liquid or air sensitive samples (high throughput option up to 10 capillaries). Capillaries can be either ordered by your group or provided by a Crystallography staff and billed as an accessory under your NUcore reservation
- Use polyimide (or glass) capillaries in Debye-Scherrer (transmission) mode for quantitative analyses (e.g., Rietveld). Capillaries can be either ordered by your group or provided by a Crystallography staff and billed as an accessory under your NUcore reservation
- Use glass capillaries (Pyrex, borosilicate, quartz) for high temperature experiments with any of the furnaces. Capillaries can be either ordered by your group or provided by a Crystallography staff and billed as an accessory under your NUcore reservation

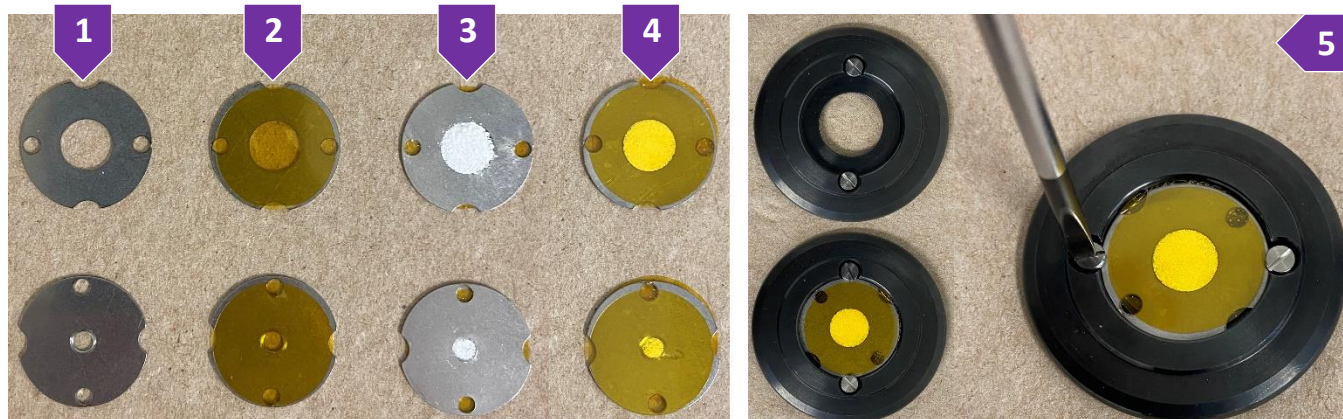
A. PACK SAMPLE IN A FLAT DISC TRANSMISSION HOLDER USING POLYIMIDE OR ACETATE TAPE

There are two different sizes of flat disc metallic masks for transmission experiments, i.e., 3 mm and 8 mm metallic masks. Use either size based on the availability of your samples. Note that the size of the X-ray beam is as wide as the 8 mm metallic mask, therefore use of a 3 mm metallic mask will result in an approximate 50% reduction in intensity in respect to the intensity obtained with 8 mm opening. Packing process of the sample into the metallic masks is the same regardless of the size of the metallic mask. Use the following steps to pack your powder into the holder for flat disc transmission measurements using polyimide or acetate tape with adhesive:

1. Select the appropriate size metallic mask based on the availability of your sample (figure 1)
2. Stick the polyimide or acetate tape on one side of the metallic mask (figure 2)
3. Flip the metallic mask with the side that has the tape facing down and pack your powder in the opening of the metallic plate (figure 3)
4. Stick the polyimide or acetate tape on the side of the metallic mask that is not covered with tape. Powder sample should be sandwiched between the metallic mask and two layers of tape (figure 4)

IMSERC User Manual for StadiMP (v1.33)

5. Take one of the black holders located in the plastic container by the instrument (top left of figure 5)
6. Place the metallic mask into the holder by aligning the half holes of the mask with the screws of the holder (bottom left of figure 5)
7. Rotate the mask by approximately 45 degrees and gently tighten the two screws (right of figure 5)



B. PACK SAMPLE IN A FLAT DISC TRANSMISSION HOLDER USING ACETATE FOILS

There are two different sizes of flat disc metallic masks for transmission experiments, i.e., 3 mm and 8 mm metallic masks. Use either size based on the availability of your samples. Note that the size of the X-ray beam is as wide as the 8 mm metallic mask, therefore use of a 3 mm metallic mask will result in an approximate 50% reduction in intensity in respect to the intensity obtained with 8 mm opening. Packing process of the sample into the metallic masks is the same regardless of the size of the metallic mask. Use the following steps to pack your powder into the holder for flat disc transmission measurements using acetate foils without adhesive:

8. Place one acetate foil into the black holder and under the screw heads of the black holder (figure 8)
9. Place your powder sample on top of the acetate foil and in the middle of the black holder (figure 9)
10. Place one acetate foil on top of your powder and under the screw heads of the black holder (figure 10)
11. Place the appropriate size metallic mask on top of the acetate foil and clamp both acetate foils and powder against the black holder. Rotate the mask approximately 45 degrees and gently tighten the two screws (figure 11). Sample powder should be well packed between the two acetate foils (figure 12, back side of holder)



IMSERC User Manual for StadiMP (v1.33)

GENERAL STEPS FOR DATA ACQUISITION

The software interface for measuring under different modes, such as transmission, reflection, and Debye-Scherrer, is very similar regardless of the mode selection. 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 sessions:

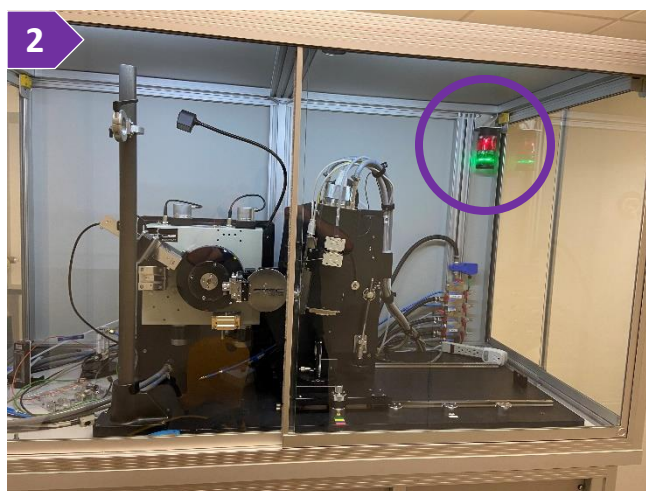
1. Pack your powdered sample into the appropriate holder as described under the '[General steps for sample preparation and packing](#)' section

2. Verify that instrument is idle by checking that the light on the safety beacon (figure 2) is green. Start your reservation in NUcore to have the screen of the computer turned on

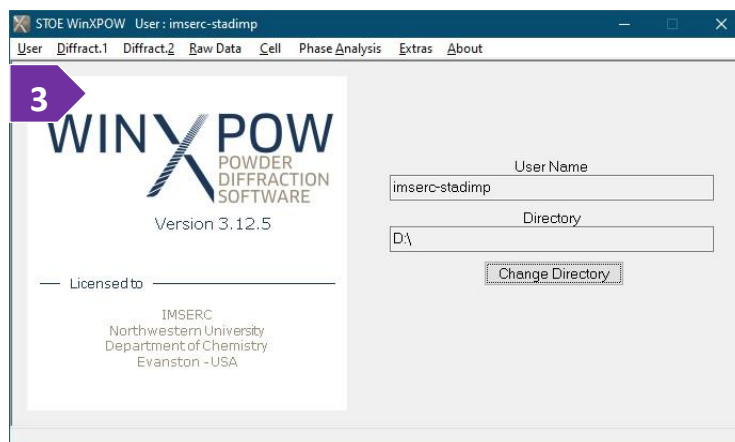
3. Start the 'WinXPOW' software (icon on the desktop) if it is not already running (figure 3), and

- a. (Optional) Press on 'Change Directory' button to select your personal working directory under your group's folder. Create a folder for the new files

- b. Under the 'Diffract.1' menu select 'Diffractometer Control'. It will take a few seconds to initialize hardware before the control window is accessible

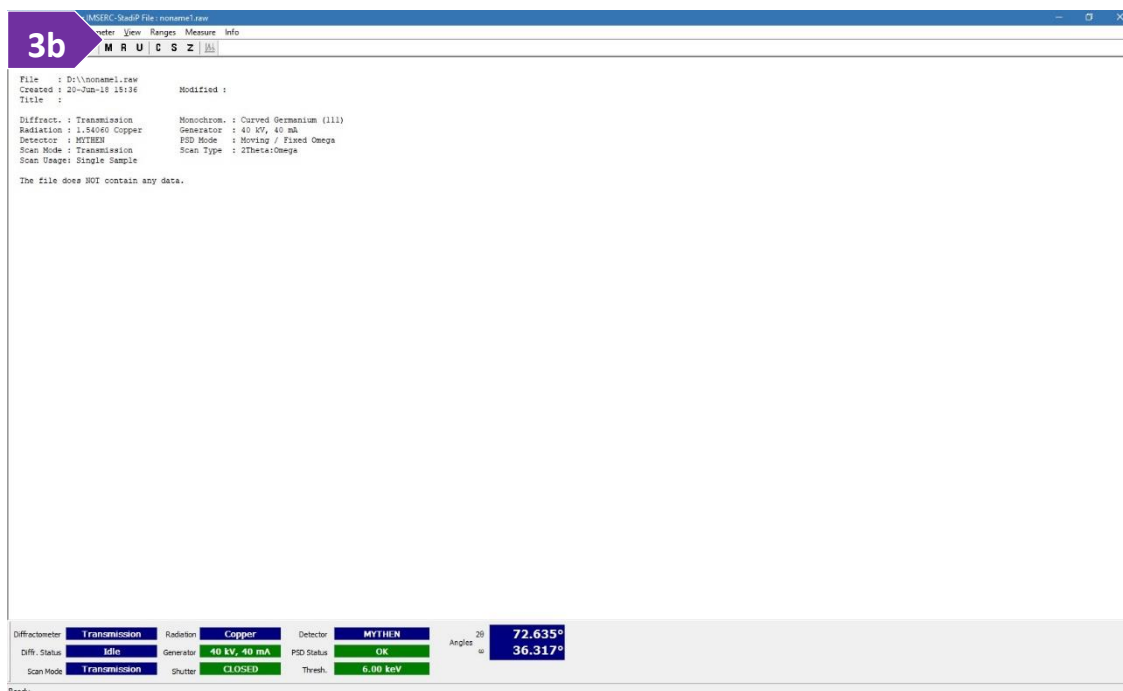


- i. 'Diffractometer Control' window ('PowDat' module) shown the acquisition parameters (figure 3b) of the loaded or newly created file. You can load a previous measurement (*.dat) from the menu 'File' and option 'Open', in case you want to reuse the same acquisition parameters every time



- ii. At the bottom of the 'Diffractometer Control' window, information about the wavelength, geometry, detector, position of omega and 2theta, and energy threshold of the detector are shown

IMSERC User Manual for StadiMP (v1.33)

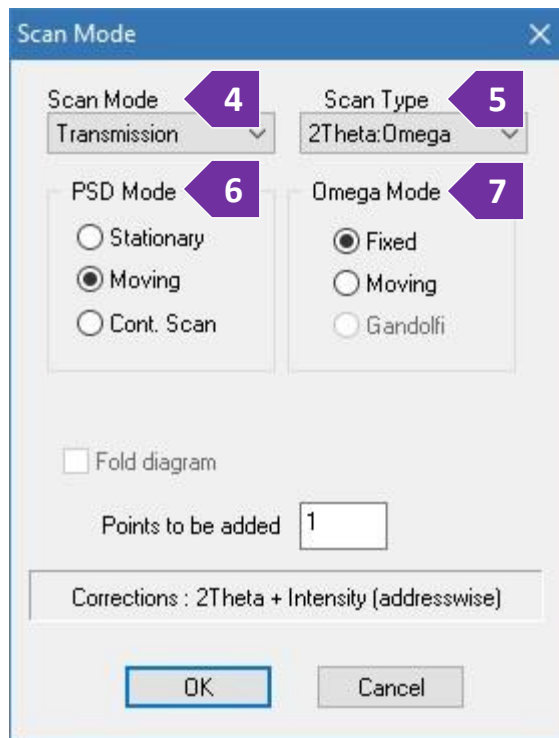


4. Define the 'Scan Mode' of your measurement by selecting the option under the 'Ranges' menu or you can press on the 'M' button. Specific configuration settings for each geometry (e.g., transmission plate, capillary, etc.) are described in the next pages. Generally, 'Scan Mode' (labeled as number 4 in the figure) relates to the type of sample holder in use:

- 'Transmission' (most common mode) scan mode for flat plate discs usually keeps a constant 2:1 between 2θ - and ω -position. However, the exact relation is defined with the 'Scan Type' option
- 'Reflection' scan mode adds a constant offset of 90° to the ω -position to account for the different orientation of the sample surface relative to the primary beam
- 'Debye-Scherrer' scan mode keeps the ω -position constant at 0° for every 2θ -position

5. 'Scan Type' option (labeled as number 5 in the figure) is activated only if the 'Scan Mode' parameter is not set to Debye-Scherrer. Options are:

- '2Theta/Omega' (most common type) invokes the normal 2:1 coupling between 2θ -and ω -position during the scan



IMSERC User Manual for StadiMP (v1.33)

- '2Theta' defines a scan with moving detector, but the ω -position kept constant at a user-defined value
 - 'Omega' is a scan with fixed detector position and moving ω -position (rocking curve)
 - 'Independent' let you define 2θ and ω movements independently from each other, e.g., a certain ω -offset and/or a 1:1 ratio between 2θ - and ω -step width may be entered in the 'Edit Ranges' dialog box
6. Set the 'PSD Mode' (labeled as number 6 in the figure) which controls the movement of the detector during measurement. You can select between:
- 'Moving' (most common mode) where the detector will move in steps during a measurement, allowing a maximum scan range as wide as the diffractometer allows
 - 'Stationary' where the detector will stay at one defined by the user position. The width of the scan range in 2θ will be as wide as the detector itself ($\sim 19^\circ$)
7. Set the 'Omega Mode' (labeled as number 7 in the figure) for the position of the ω -angle. Options are:
- 'Fixed' means that for every PSD 2θ -position the ω -position will be set to $2\theta/2$ and will remain at this position for all the measuring time of this PSD step
 - 'Moving' means that the ω -circle is continuously moved during data collection which usually requires longer step times and thus should only be used if additional particle randomization is required
 - 'Gandolfi' requires a special attachment/holder which currently is not available at IMSERC. The 'Gandolfi' modes enables a Debye-Scherrer measurement at a fix χ -angle which is different than 0 or 180 degrees while changing the ω - and ϕ - angles during collection. 'Gandolfi' is the ultimate method to reduce preferred orientation during collection

8. Under 'Ranges' menu, select the 'Scan Range' option. On the 'Edit Range' window

a. Press on the '+' button to add a scan range

b. Double press on the new scan range you just added and

- i. Edit the '2theta range' accordingly (figure 8b) assuming a 'PSD Mode' of either 'Moving' or 'Cont.Scan' has been selected in step 6. In case a 'Stationary' 'PSD Mode' has

been selected, the 'Edit Scan Range' window will not let you change directly the '2Theta range' (figure 8b-i) but you can edit the center of the '2Theta range' in the next step

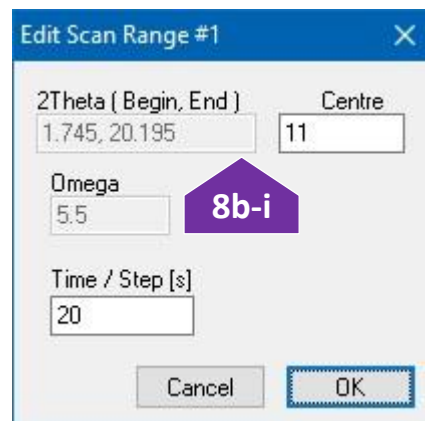
- ii. Define the 'Step' of the detector, typically 4° . This step is different than the resolution step (2θ values in your powder pattern) which is fixed to 0.015° for a single scan. Resolution can be improved with more special scans (see a Crystallography staff for more details). Our detector can cover a solid angle of $\sim 19^\circ$ with a single step, therefore with a step of 1° , the same 2θ region will be measured

2Theta (Begin, End)	Step
10, 94.105	4.005
Omega (Begin, End)	Step
5, 47.0525	2.0025
Time/PSD Step [s]	
20	

OK Cancel

IMSERC User Manual for StadiMP (v1.33)

19 times. In case a 'Stationary' 'PSD Mode' has been selected, the 'Step' is renamed to 'Centre' where you can select the center of the detector position in respect to the primary beam (2theta of zero). By changing the 'Center' value and pressing in a different field, e.g., 'Time' text box, the '2Theta' range will change accordingly



iii. Set the integration 'Time/PSD Step' in seconds, typically 10-20 s. Signal-to-noise ratio improves when you measure with a

longer exposure time or average multiple scans that have been collected with a long enough exposure (see 'Scan usage' below)

iv. The 'Omega' range will be automatically filled assuming you have selected the '2Theta/Omega' for 'Scan Type'. In case you have selected either '2Theta' or 'Independent' for 'Scan Type', provide the 'Omega' value you want for the experiment

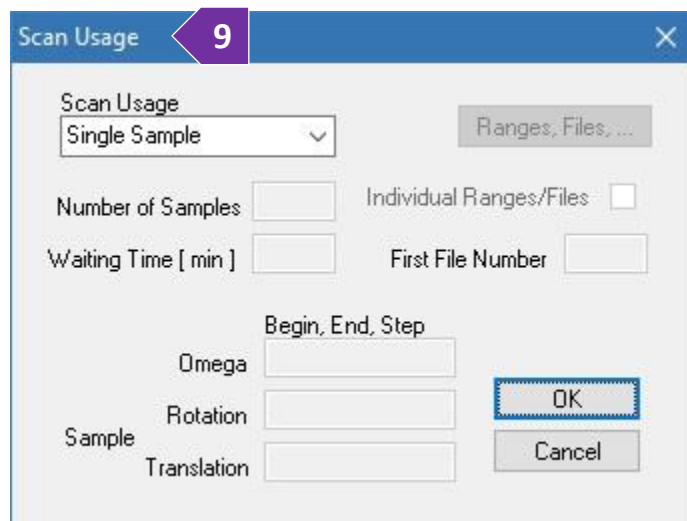
c. Keep adding more ranges according to your needs, e.g., add a range with longer exposure for the high angle reflections, etc.

9. Under 'Ranges' menu, select the 'Scan Usage' option (figure 9) accordingly:

o Select 'Single Sample' if you want to measure your sample/scan range only once, or

o Select 'Multiple Samples' if you would like to use any of the autosamplers (transmission or capillary), or

o Select 'Repetition' if you want to measure your sample/scan range multiple times for improving the signal-to-noise ratio. You have the option to add a 'Waiting time' in case you are interested in a time dependent measurement, e.g., transformation, decomposition, etc.



10. Start the measurement by pressing on either 'Data Collection' under the 'Measure' menu or the 'C' icon

11. Provide the location and filename of the collection

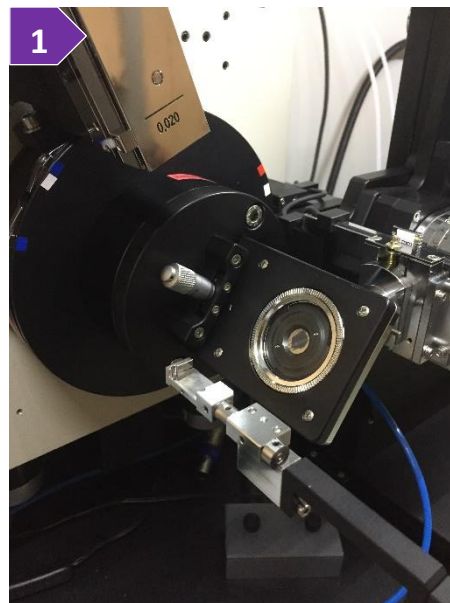
12. (Optional) Provide more info into the header of the file by typing a file title and/or some comments

13. Press on the 'OK' button and the measurement will start. An estimate time of completion will be shown on the screen during collection

IMSERC User Manual for StadiMP (v1.33)

RUN SAMPLES WITH THE FLAT DISC TRANSMISSION HOLDER

1. Verify that the transmission stage is mounted onto the diffractometer (figure 1), otherwise see a Crystallography staff
2. If stage is spinning and/or occupied with a sample holder, prior to mounting your sample, you need to

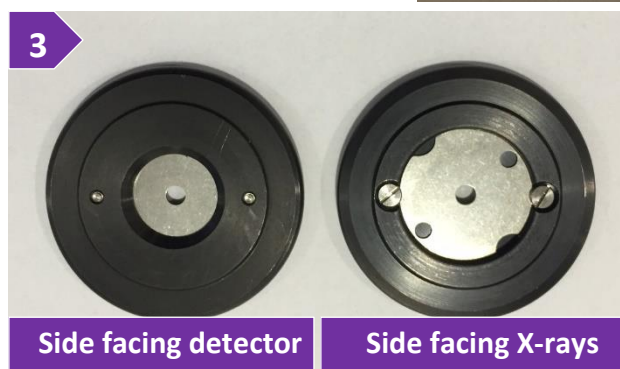


- a. Stop any rotation by turning the 'Sample spinner' dial counterclockwise until it clicks. Dials are located on the left side of the goniometer base behind the arm of the detector (figure 2a). Watch the ['stadimp-sample-spinner'](#) video for a visual demonstration. The ['stadimp-sample-spinner'](#) video is also part of the (un)mounting demonstration video ['stadimp-transmission-manually-mounting-unmounting-holder'](#) that most likely you need to watch for the next steps described below

- b. Remove the sample holder and leave it on the sample table outside the enclosure of the diffractometer. Transmission stage is different than the one on STADI P (the spring-loaded pins are missing). To unmount the holder from the stage, you need to rotate the front lock-ring counterclockwise while holding the back side of the lock-ring. Watch the ['stadimp-transmission-manually-mounting-unmounting-holder'](#) video for a visual demonstration

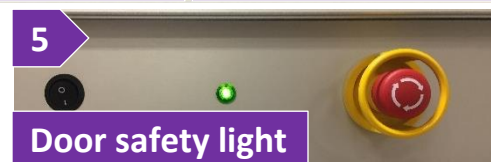


3. Mount your sample holder onto the stage. Refer to the attached picture (figure 2c) for the right orientation of the sample holder on the stage. The metallic mask should face the X-ray source and the side of the holder facing the detector should have only one hole visible in the middle of the holder/mask. Watch the ['stadimp-transmission-manually-mounting-unmounting-holder'](#) video for a visual demonstration



4. Turn on spinning by rotating the 'Sample spinner' clockwise (figure 2a). Watch the ['stadimp-sample-spinner'](#) video for a visual demonstration

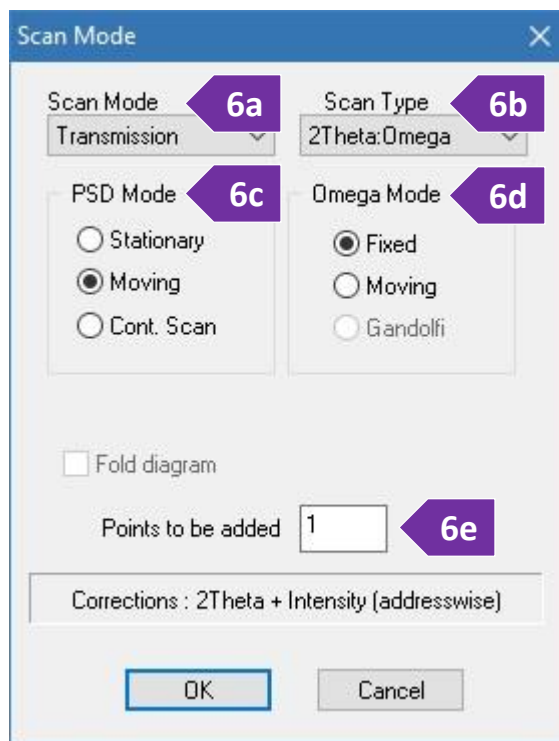
5. Close windows of the enclosure and make sure the green light next to the top emergency button is on (figure 2e). Watch the ['stadimp-safety-windows'](#) video for a visual demonstration



IMSERC User Manual for StadiMP (v1.33)

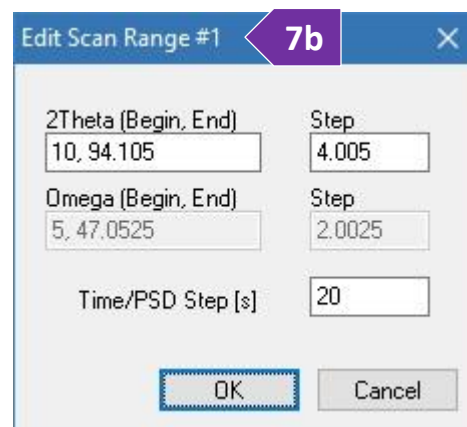
6. Under 'Ranges' menu, select the 'Scan Mode' option (or press on the button with the 'M' icon) and set (figure 7a-e)

- 'Scan mode' to 'Transmission'
- 'Scan Type' to '2Theta/Omega' (most common). Read the ['General steps for data acquisition'](#) section for details about the other custom options
- 'PSD mode' to 'Moving' (most common). Read the ['General steps for data acquisition'](#) section for details about the other custom options
- 'Omega mode' to 'Fixed' (most common). Read the ['General steps for data acquisition'](#) section for details about the other custom options
- 'Points to be added' to '1' (most common). Read the ['General steps for data acquisition'](#) section for details about the other custom options



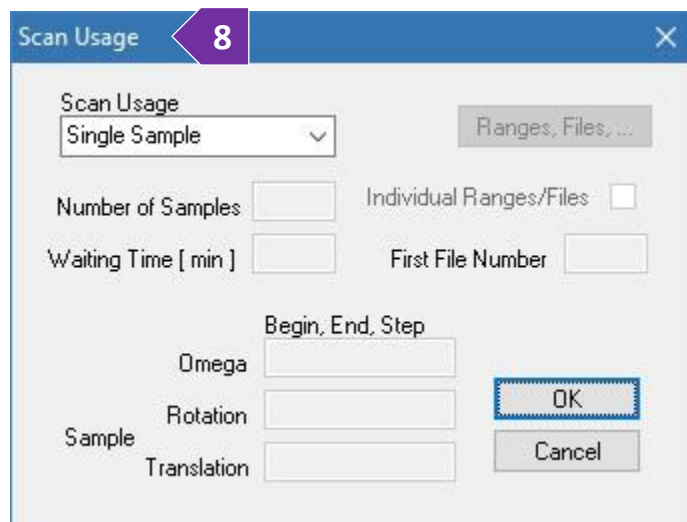
7. Under 'Ranges' menu, select the 'Scan Range' option. On the 'Edit Range' window

- Press on the '+' button to add a scan range
- Double press on the new scan range you just added and
 - Edit the '2theta range' accordingly (figure 7b)
 - Define the 'Step' of the detector, typically 4° . This step is different than the resolution step (2theta values in your powder pattern) which is fixed to 0.015° for a single scan. Resolution can be improved with more special scans (see a staff for more details). Our detector can cover a solid angle of $\sim 19^\circ$ with a single step, therefore with a step of 1° , the same 2theta region will be measured 19 times
 - Set the integration 'Time/PSD Step' in seconds, typically 10-20 s. Signal-to-noise ratio improves when you measure with a longer exposure time or average multiple scans that have been collected with a long enough exposure (see 'Scan usage' below)
- Keep adding more ranges according to your needs, e.g., add a range with longer exposure for the high angle reflections, etc.



IMSERC User Manual for StadiMP (v1.33)

8. Under 'Ranges' menu, select the 'Scan Usage' option (figure 8)
 - Select 'Single Sample' if you want to measure your sample/scan range only once
 - Select 'Repetition' if you want to measure your sample/scan range multiple time for improving the signal-to-noise ratio. You have the option to add a 'Waiting time' in case you are interested in a time dependent measurement, e.g., transformation, decomposition, etc.

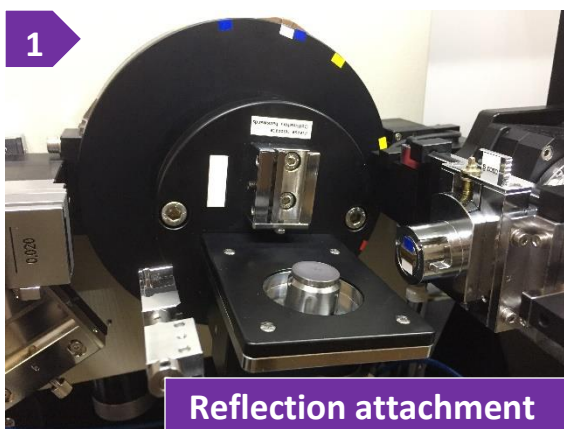


9. Start the measurement by pressing on either 'Data Collection' under the 'Measure' menu or the 'C' icon
10. Provide the location and filename of the collection
11. (Optional) Provide more info into the header of the file by typing a file title and/or some comments
12. Press on the 'OK' button and the measurement will start. An estimate time of completion will be shown on the screen during collection

IMSERC User Manual for StadiMP (v1.33)

RUN SAMPLES IN REFLECTION MODE

1. Verify that the reflection stage is mounted onto the diffractometer (figure 1), otherwise see a Crystallography staff



2. If stage is spinning and/or occupied with a sample holder, you need to

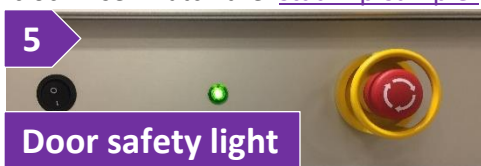
- Stop any rotation by turning the 'Sample spinner' dial counterclockwise until it clicks. Dials are located on the left side of the goniometer base behind the arm of the detector (figure 2a). Watch the '[stadimp-sample-spinner](#)' video for a visual demonstration

- Remove the sample holder and leave it on the sample table outside the enclosure of the diffractometer

3. Mount your sample holder onto the stage and align sample height using the spacer. Watch the '[stadimp-reflection-sample-alignment](#)' video for a visual demonstration

4. Turn on spinning by rotating the 'Sample spinner' clockwise. Watch the '[stadimp-sample-spinner](#)' video for a visual demonstration

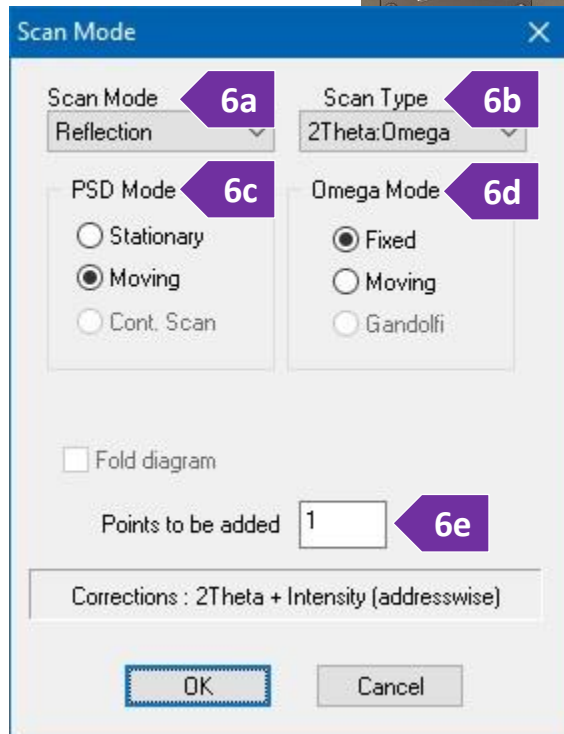
5. Close windows of the enclosure and make sure the green light next to the top emergency



button is on (figure 5). Watch the '[stadimp-door-safety](#)' video for a visual demonstration

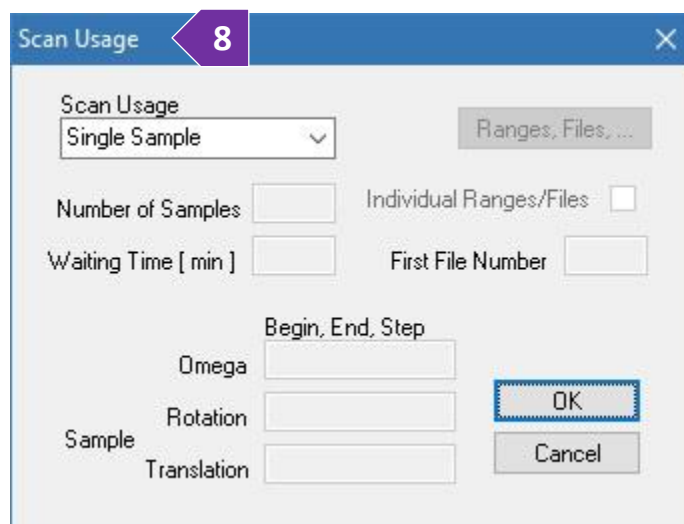
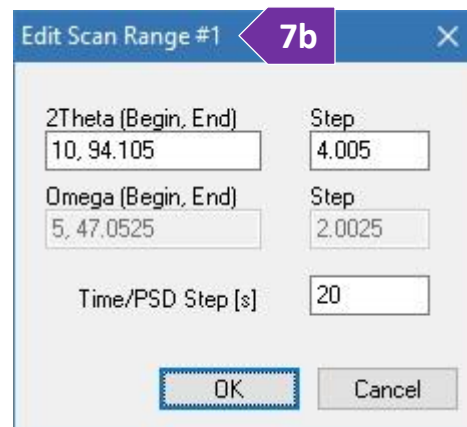
6. Under 'Ranges' menu, select the 'Scan Mode' option (or press on the button with the 'M' icon) and set (figures 6a-e)

- 'Scan mode' to 'Reflection'
- 'Scan Type' to '2Theta/Omega' (most common). Read the '[General steps for data acquisition](#)' section for details about the other custom options
- 'PSD mode' to 'Moving' (most common). Read the '[General steps for data acquisition](#)' section for details about the other custom options
- 'Omega mode' to 'Fixed' (most common). Read the '[General steps for data acquisition](#)' section for details about the other custom options



IMSERC User Manual for StadiMP (v1.33)

- e. 'Points to be added' to '1' (most common). Read the ['General steps for data acquisition'](#) section for details about the other custom options
7. Under 'Ranges' menu, select the 'Scan Range' option. On the 'Edit Range' window
 - a. Press on the '+' button to add a scan range
 - b. Double press on the new scan range you just added and
 - i. Edit the '2theta range' accordingly (figure 7b)
 - ii. Define the 'Step' of the detector, typically 4° . This step is different than the resolution step (2theta values in your powder pattern) which is fixed to 0.015° for a single scan. Resolution can be improved with more special scans (see a Crystallography staff for more details). Our detector can cover a solid angle of $\sim 19^\circ$ with a single step, therefore with a step of 1° , the same 2theta region will be measured 19 times
 - iii. Set the integration 'Time/PSD Step' in seconds, typically 10-20 s. Signal-to-noise ratio improves when you measure with a longer exposure time or average multiple scans that have been collected with a long enough exposure (see 'Scan usage' below)
 - c. Keep adding more ranges according to your needs, e.g., add a range with longer exposure for the high angle reflections, etc.
 8. Under 'Ranges' menu, select the 'Scan Usage' option (figure 8)
 - o Select 'Single Sample' if you want to measure your sample/scan range only once, or
 - o Select 'Repetition' if you want to measure your sample/scan range multiple times for improving the signal-to-noise ratio. You have the option to add a 'Waiting time' in case you are interested in a time dependent measurement, e.g., transformation, decomposition, etc.
 9. Start the measurement by pressing on either 'Data Collection' under the 'Measure' menu or pressing on the button with the 'C' icon



IMSERC User Manual for StadiMP (v1.33)

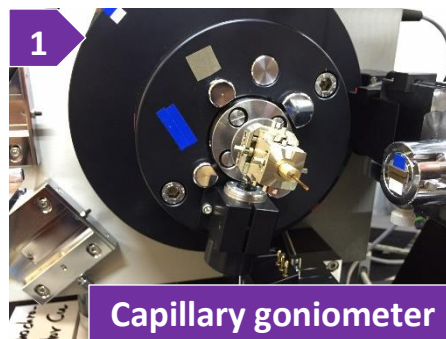
10. Provide the location and filename of the collection and press on the 'OK' button. (Optional) Provide more info into the header of the file by typing a file title and/or some comments. Press on the 'OK' button and the measurement will start. An estimate time of completion will be shown on the screen during collection

IMSERC User Manual for StadiMP (v1.33)

RUN SAMPLES IN CAPILLARY IN DEBYE-SCHERRER MODE

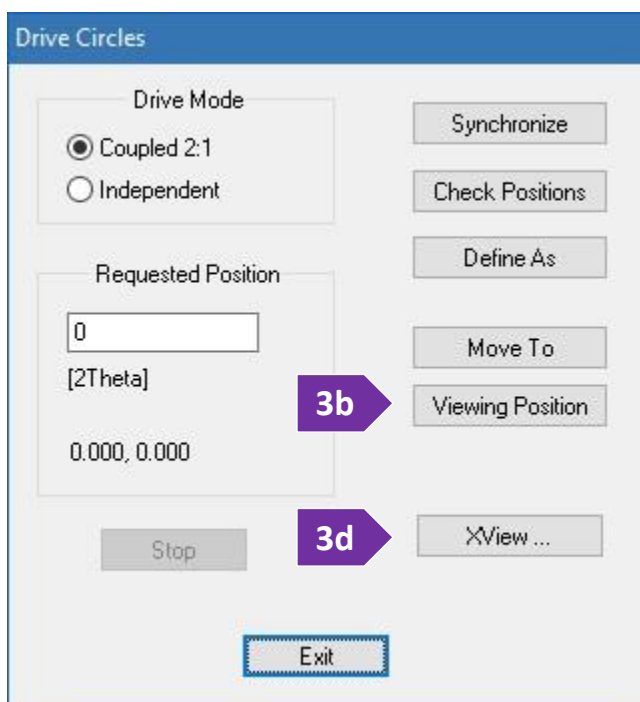
A. WITH THE GONIOMETER HEAD

1. Verify that the capillary stage with the goniometer (figure 1) is mounted onto the diffractometer, otherwise see a crystallography staff to set up the attachment for you. The autosampler for transmission flat plates may remain mounted onto the diffractometer
2. If stage is spinning and/or occupied with a sample holder, you need

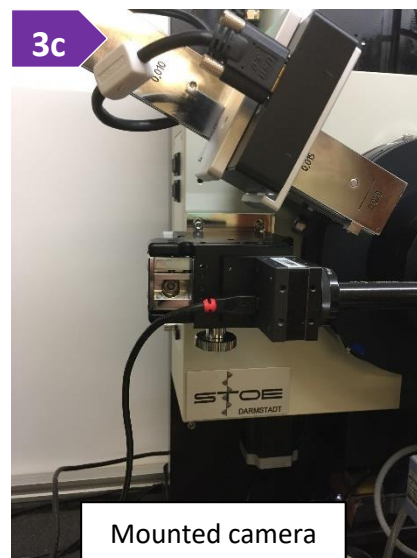


- a. Stop any rotation by turning the 'Sample spinner' dial counterclockwise
- b. Remove the beam stop. Watch the '[stadimp-beamstop](#)' video for a visual demonstration
- c. Remove the capillary (if any) and leave it on the sample table outside the enclosure of the diffractometer
- d. Mount your capillary onto the goniometer

3. Align the capillary on the rotation axis of the goniometer. Watch the '[stadimp-debye-scherrer-goniometer-capillary](#)' video for a visual demonstration. Please check '[Appendix A](#)' for the tools needed and location of the screws on the goniometer head that needs to be adjustment during this alignment process



- a. Under the 'Diffractometer' menu, select the 'Drive Circles' option or press on the button with the 'D' icon
- b. Press the 'Viewing Position' button to drive the 2theta above the dovetail mount for the camera (figure 3b)
- c. Camera is located next to the computer of StadiP. Mount the optical camera onto the dovetail and connect the USB cable (Figure 3c)
- d. Press the 'XView' button to launch the video software and press on the 'Start Video' button



IMSERC User Manual for StadiMP (v1.33)

- e. Adjust the X, Y, dX, and dY screws on the goniometer, so that the capillary is parallel and centered when rotated by hand 360°. Please check [‘Appendix A’](#) for the tools needed and location of the screws on the goniometer head that needs to be adjustment during this alignment process. Watch the [‘stadimp-goniometer-capillary-alignment’](#) video for a visual demonstration
- f. Close the ‘XView’ software, disconnect and unmount the camera from the dovetail, and put it back onto its holder next to the computer of StadiP

g. Mount the beamstop

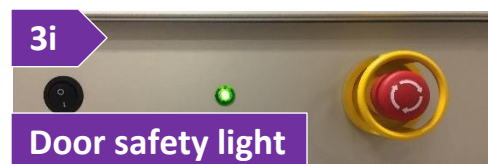
h. Turn on spinning by rotating the ‘Sample spinner’ clockwise.

The position of the knob should be between 3 and 4 o’clock

i. Close windows of the enclosure and make sure the green light

next to the top emergency button is on (Figure 3i).

Watch the [‘stadimp-door-safety’](#) video for a visual demonstration



4. Under ‘Ranges’ menu, select the ‘Scan Mode’ option (figure 4):

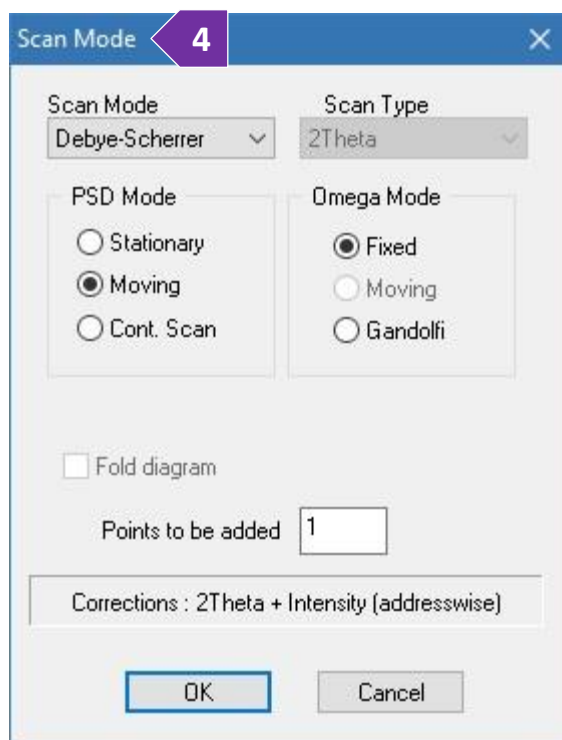
a. ‘Scan mode’ to ‘Debye-Scherrer’

b. ‘PSD mode’ to ‘Moving’ (most common). Read the [‘General steps for data acquisition’](#) section for details about the other custom options

c. ‘Scan Type’ is disable by default

d. ‘Omega mode’ to ‘Fixed’ (most common). Read the [‘General steps for data acquisition’](#) section for details about the other custom options

e. ‘Points to be added’ to ‘1’ (most common). Read the [‘General steps for data acquisition’](#) section for details about the other custom options

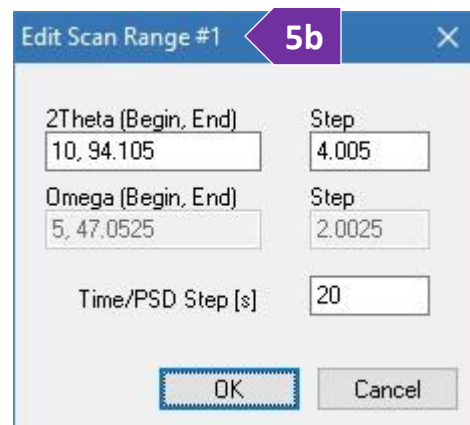


5. Under ‘Ranges’ menu, select the ‘Scan Range’ option. On the ‘Edit Range’ window

a. Press on the ‘+’ button to add a scan range

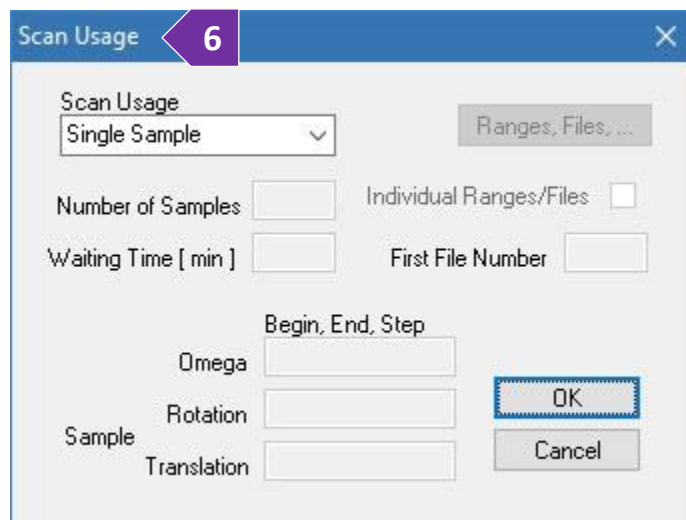
b. Double press on the new scan range you just added:

i. Edit the ‘2theta range’ accordingly (figure 5b)



IMSERC User Manual for StadiMP (v1.33)

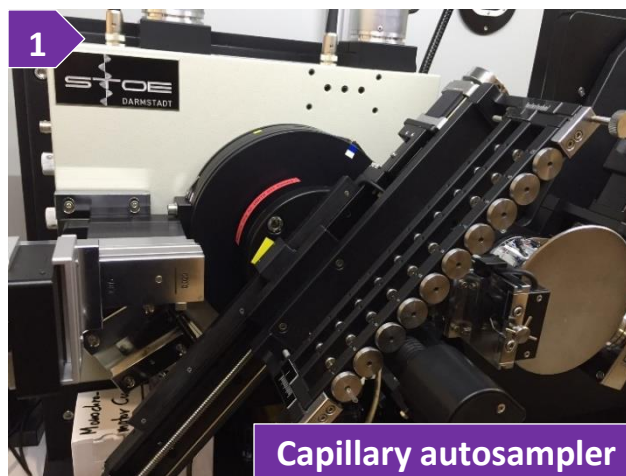
- ii. Define the 'Step' of the detector, typically 4° . This step is different than the resolution step (2theta values in your powder pattern) which is fixed to 0.015° for a single scan. Resolution can be improved with more special scans (see a staff for more details). Our detector can cover a solid angle of $\sim 19^\circ$ with a single step, therefore with a step of 1° , the same 2theta region will be measured 19 times
 - iii. Set the integration 'Time/PSD Step' in seconds, typically 20-60 s. For quantitative analyses, you need a very good signal-to-noise ratio especially at high angles. Exposure time in this case might be several minutes and overall collection time several hours. Signal-to-noise ratio improves when you measure with a longer exposure time or average multiple scans that have been collected with a long enough exposure
 - c. Keep adding more ranges according to your needs, e.g., add a range with longer exposure for the high angle reflections, etc.
6. Under 'Ranges' menu, select the 'Scan Usage' option
- a. Select 'Single Sample' if you want to measure your sample/scan range only once
 - b. Select 'Repetition' if you want to measure your sample/scan range multiple time for improving the signal-to-noise ration. You have the option to add a 'Waiting time' in case you are interested in a time dependent measurement, e.g., transformation, decomposition, etc.
7. Start the measurement by pressing on either 'Data Collection' under the 'Measure' menu or pressing on the button with the 'C' icon
8. Provide the location and filename of the collection
9. (Optional) Provide more info into the header of the file by typing a file title and/or some comments
10. Press on the 'OK' button and the measurement will start. An estimate time of completion will be shown on the screen during collection



IMSERC User Manual for StadiMP (v1.33)

B. WITH THE CAPILLARY AUTOSAMPLER

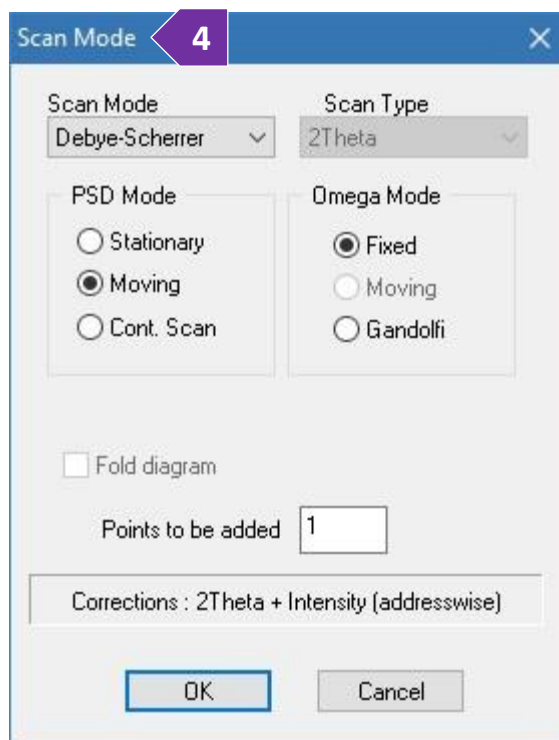
1. Make sure the capillary autosampler attachment is mounted onto the diffractometer (figure 1), otherwise see a Crystallography staff to set up the attachment for you
2. Insert the full-length glass capillary in one of the 10 positions
3. Close windows of the enclosure and make sure the green light next to the top emergency button is on (figure 3). Watch the '[stadimp-door-safety](#)' video for a visual demonstration



4. Under 'Ranges' menu, select the 'Scan Mode' option and set (figure 4)
 - a. 'Scan mode' to 'Debye-Scherrer'
 - b. 'PSD mode' to 'Moving' (most common). Read the '[General steps for data acquisition](#)' section for details about the other custom options



- c. 'Scan Type' is disabled by default
- d. 'Omega mode' to 'Fixed' (most common). Read the '[General steps for data acquisition](#)' section for details about the other custom options
- e. 'Points to be added' to '1' (most common). Read the '[General steps for data acquisition](#)' section for details about the other custom options

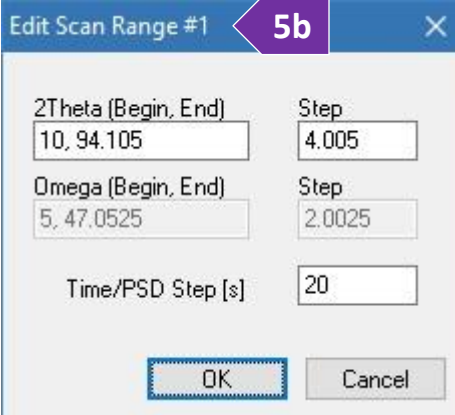


5. Under 'Ranges' menu, select the 'Scan Range' option. On the 'Edit Range' window
 - a. Press on the '+' button to add a scan range
 - b. Double press on the new scan range you just added and
 - i. Edit the '2theta range' accordingly (figure 5b)
 - ii. Define the 'Step' of the detector, typically 4° . This step is different than the resolution step (2theta values in your powder pattern) which is fixed to 0.015° for a single scan. Resolution can be improved with more special scans (see a staff for more

IMSERC User Manual for StadiMP (v1.33)

details). Our detector can cover a solid angle of $\sim 19^\circ$ with a single step, therefore with a step of 1° , the same 2θ region will be measured 19 times

- iii. Set the integration 'Time/PSD Step' in seconds, typically 10-60 s. Signal-to-noise ratio improves when you measure with a longer exposure time or average multiple scans that have been collected with a long enough exposure (see 'Scan usage' below)



5b

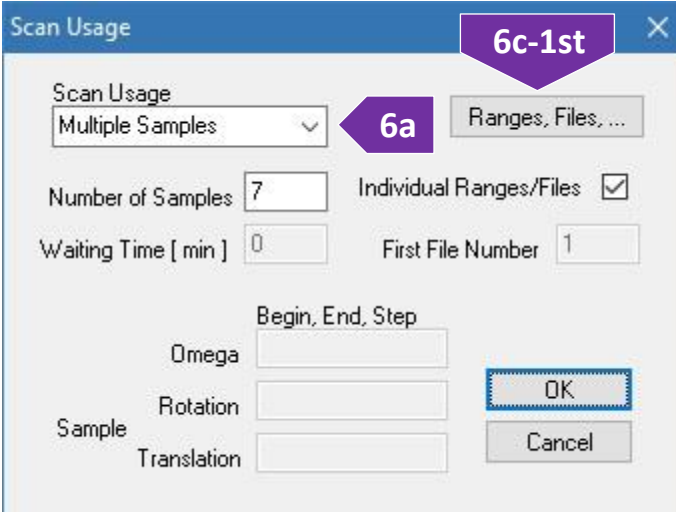
- c. Keep adding more ranges according to your needs, e.g., add a range with longer exposure for the high angle reflections, etc.

6. Under 'Ranges' menu, select the 'Scan Usage' option

- a. Select 'Multi-sample' in 'Scan Usage' (figure 6a)

- b. Set the total number of samples you have loaded on the autosampler in 'Number of Samples'

- c. You have two options for measuring the queue of samples in the autosampler. Options depend on the acquisition criteria of the samples, i.e., either i) measure all samples using the same acquisition



6a

6c-1st

parameters and with an automated file naming scheme (default option), or ii) define filenames and/or acquisition parameters manually for each sample in the autosampler queue:

- By default, measurements will be saved as a series of files having the same filename (to be provided in the next steps) plus a unique index number as a file extension whose starting value is set in 'First File Number'. For example, if the filename is 'sampleX', the 'First File Number' is '5', and the 'Number of Samples' is '9', the 9 individual files that will be saved in your folder will have filenames ranging from 'sampleX.005' to 'sampleX.013'
- If you want to give a unique filename and/or assign different acquisition parameters (ranges under step 5c), to each sample in the queue of the autosampler:

- 1st. Tick the 'Individual Ranges / Files' box and press on the 'Ranges, Files...' button (figure 6c-1st)

IMSERC User Manual for StadiMP (v1.33)

- 2nd. The 'Individual Ranges/Files/Titles' window will open with the list of samples prepopulated based on the 'Number of Samples' you provided (figure 6c-2nd)

Individual Ranges / Files / Titles

Entry

File: 7.raw

Title: & - Sample 2

Comment:

Set

Sample	Range	Filename	Title	Comment
1	2	1.raw	& - Sample 1	
2	1	2.raw	& - Sample 2	
3	1	3.raw	& - Sample 3	
4	2	4.raw	& - Sample 4	
5	0	5.raw	& - Sample 5	
6	1	6.raw	& - Sample 6	
7	0	7.raw	& - Sample 7	

+ -

Reset Reassign OK Cancel

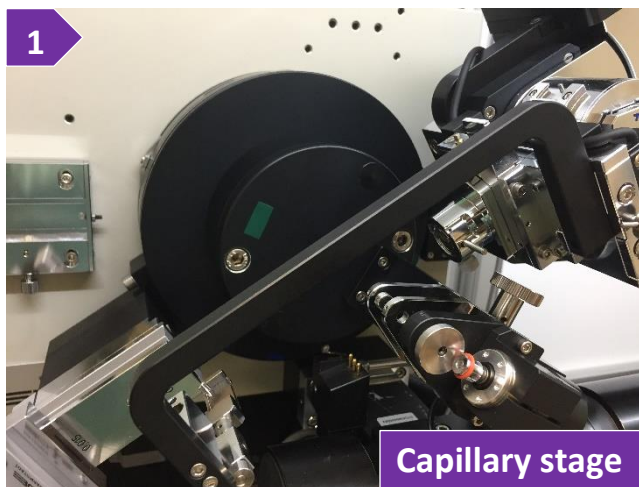
- 3rd. Provide a file name for each sample in the autosampler queue by either double pressing on the filename or single pressing, typing in the 'File' field, and pressing on the 'Set' button
- 4th. Assign a specific range per sample, in case you have more than one ranges defined (optional step 5c). Note that the default range of '0' will use all ranges in the list, i.e., if there are two ranges defined (step 5c), each sample will be measured twice, one measurement per range, when range is set to '0'
- 5th. (Optional) Edit the title option
- 6th. Press on the 'OK' button to close the 'Individual Ranges / Files / Titles' window when done editing
- 7th. Press on the 'OK' button to close the 'Scan Usage' window
7. Start the measurement by pressing on either 'Data Collection' under the 'Measure' menu or pressing the 'C' icon
8. Software will prompt for the location and filename of the collection. Since you are measuring with the autosampler, the filename provided here will contain only the information of the sample queue and acquisition parameters (step 4-6). The filename provided here will not contain any diffraction data since data for each sample in the autosampler queue will be saved in the individual files provided in step 6c. Provide a filename that is different than the filenames provided in the list of samples in the queue (step 6c)

IMSERC User Manual for StadiMP (v1.33)

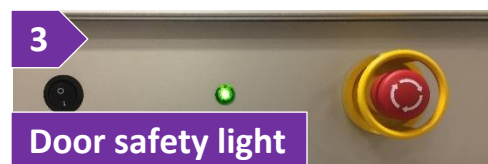
- (Optional) Provide more info into the header of the file by typing a file title and/or some comments
- Press on the 'OK' button and the measurement will start. An estimate time of completion will be shown on the screen during collection

C. WITH THE PERMANENTLY ALIGNED CAPILLARY HOLDER

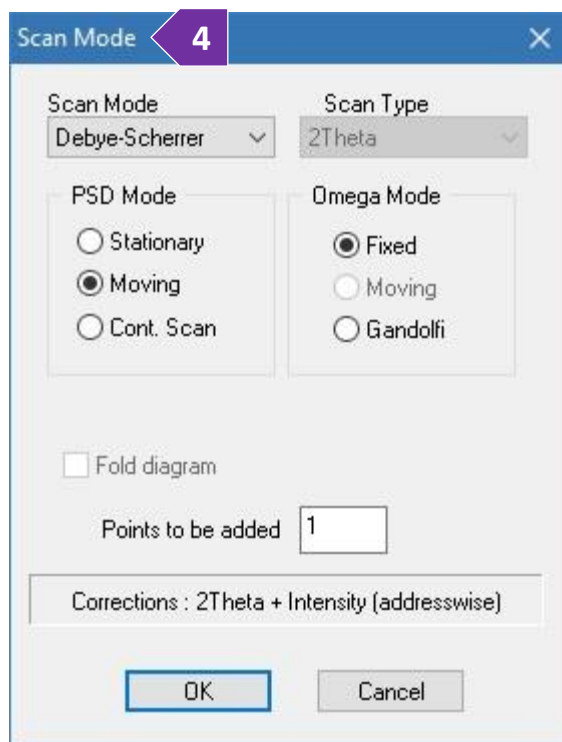
This pre-aligned capillary holder enables the measurement of full-length capillaries with a diameter ranging from 0.3 mm to 1.5 mm without the use of an optical camera for alignment. Capillary can be spun during the measurement. Make sure the permanently aligned capillary autosampler attachment is mounted onto the diffractometer (figure 1), otherwise see a Crystallography staff to set up the attachment for you. To perform a measurement with the permanently aligned holder, please follow the steps:



- If stage is spinning and/or occupied with a sample holder, you need to
 - Stop any rotation by turning the 'Sample spinner' dial counterclockwise
 - Remove the capillary and leave it on the sample table outside the enclosure of the diffractometer



- Insert your full-length glass capillary in the holder and rotate the 'Sample spinner' knob clockwise at a position between 3 and 4 o'clock
- Close safety windows of the enclosure and make sure the green light next to the top emergency button is on (figure 3). Watch the [stadimp-door-safety](#) video for a visual demonstration
- Under 'Ranges' menu, select the 'Scan Mode' option and set
 - 'Scan mode' to 'Debye-Scherrer' (figure 4)



IMSERC User Manual for StadiMP (v1.33)

- b. 'PSD mode' to 'Moving' (most common). Read the '[General steps for data acquisition](#)' section for details about the other custom options
 - c. 'Scan Type' is disabled by default
 - d. 'Omega mode' to 'Fixed' (most common). Read the '[General steps for data acquisition](#)' section for details about the other custom options
 - e. 'Points to be added' to '1' (most common). Read the '[General steps for data acquisition](#)' section for details about the other custom options
5. Under 'Ranges' menu, select the 'Scan Range' option. On the 'Edit Range' window
- a. Press on the '+' button to add a scan range
 - b. Double press on the new scan range you just added and
 - i. Edit the '2theta range' accordingly (figure 5b)
 - ii. Define the 'Step' of the detector, typically 4° . This step is different than the resolution step (2theta values in your powder pattern) which is fixed to 0.015° for a single scan. Resolution can be improved with more special scans (see a staff for more details). Our detector can cover a solid angle of $\sim 19^\circ$ with a single step, therefore with a step of 1° , the same 2theta region will be measured 19 times
 - iii. Set the integration 'Time/PSD Step' in seconds, typically 20-60 s. Signal-to-noise ratio improves when you measure with a longer exposure time or average multiple scans that have been collected with a long enough exposure (see 'Scan usage' below)
 - c. Keep adding more ranges according to your needs, e.g., add a range with longer exposure for the high angle reflections, etc.

5b

2Theta (Begin, End)	Step
10, 94.105	4.005
Omega (Begin, End)	Step
5, 47.0525	2.0025
Time/PSD Step [s]	20

OK Cancel

6. Under 'Ranges' menu, select the 'Scan Usage' option (figure 6)
- Select 'Single Sample' if you want to measure your sample/scan range only once
 - Select 'Repetition' if you want to measure your sample/scan range multiple time for improving the signal-to-noise ration. You have the option to add a 'Waiting time' in

6

Scan Usage

Single Sample

Number of Samples

Waiting Time [min]

Individual Ranges/Files

First File Number

Begin, End, Step

Omega

Rotation

Sample Translation

OK

Cancel

IMSERC User Manual for StadiMP (v1.33)

case you are interested in a time dependent measurement, e.g., transformation, decomposition, etc.

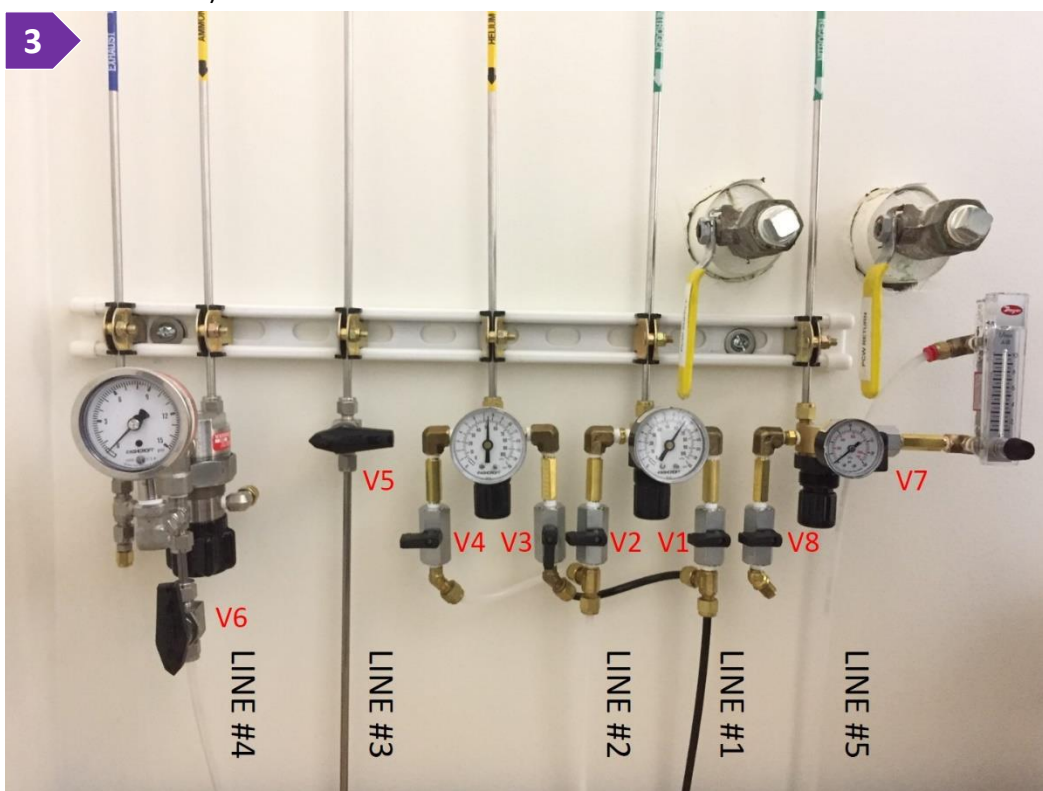
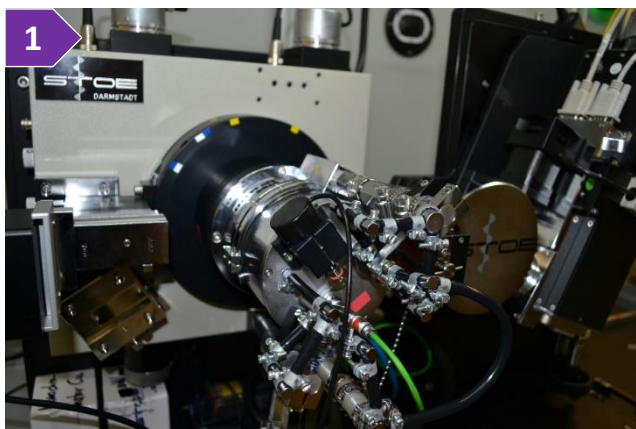
7. Start the measurement by pressing on either 'Data Collection' under the 'Measure' menu or the 'C' icon
8. Provide the location and filename of the collection
9. (Optional) Provide more info into the header of the file by typing a file title and/or some comments
10. Press on the 'OK' button and the measurement will start. An estimate time of completion will be shown on the screen during collection

IMSERC User Manual for StadiMP (v1.33)

RUN SAMPLES AT HIGH TEMPERATURE IN CAPILLARY WITH THE HT FURNACE

Running VT measurements requires 1) the creation of a data file that contains the acquisition parameters, and 2) the execution of the data file at specific temperature points defined in a customizable temperature profile.

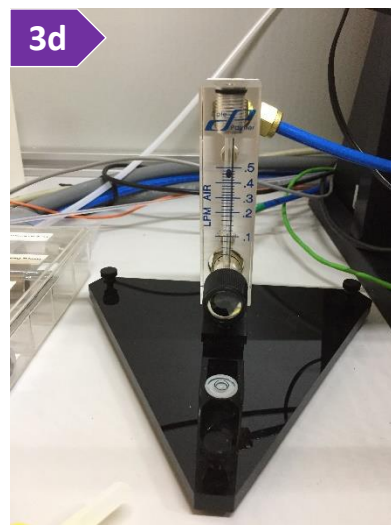
1. Verify that the HT furnace is mounted onto the diffractometer (figure 1), otherwise see a Crystallography staff
2. Heating element of the furnace is protected against oxidation by flowing inert gas through the blue tubing connected on the side of the furnace. Running the furnace without inert gas (especially above 400 C) will damage the heating element
3. Line #1 (black tubing in figure 3) connects to the flowmeter that provides inert gas to the furnace (blue tubing connected to the furnace)



- a. For using N₂ (recommended) as a protection gas, turn valve #1 (V1) on (black handle is parallel to the line) and turn V3 off (black handle is perpendicular to the line)

IMSERC User Manual for StadiMP (v1.33)

- b. For using He (special experiments) as a protection gas, turn valve #1 (V1) off and turn V3 on (as shown in the picture)
 - c. Pressure should be between 7-10 psi, do not adjust the regulators if pressure is lower, please see a staff
 - d. Check the flow on the flowmeter mounted on the black triangular base connected with the blue tubing (figure 3d), flow should be between 0.4 and 0.5 l/min. Adjust flow if necessary, using the knob on the flowmeter
4. Check the gas tanks located in the southwest corner of room BG66 next to the StadiVari (figure 4). Pressure of the gauge near the main valve (circled on figure 4) of the tank should be at least 100 psi. If pressure is low, replace the tank with a new cylinder located in the same corner, estimate 100 psi consumption per day. Complete the form at the end of this document with the pressure of the tank before and after your measurement



5. Pack your powdered sample into the appropriate capillary (i.e., polyimide, Glass, Borosilicate, Quartz, Sapphire)



- a. Insert the capillary into the furnace. Sample is located 7 cm below the surface of the furnace and the length of the heating profile is 3.5 cm. Size of the beam is 2-4 mm depending on the collimator used. Watch the [‘stadimp-debye-scherrer-furnace-ht-mounting-glass-capillary’](#) or [‘stadimp-debye-scherrer-furnace-ht-mounting-polyimide-capillary’](#) video for a visual demonstration on how to insert a glass or polyimide capillary respectively into the furnace
- b. Set the spinning on by turning the ‘Sample spinner’ dial clockwise between the 3 and 4 o’clock position. Capillary is pre-aligned with the furnace. In case you have a very small amount of powder into the capillary, use the optical camera to align the sample into the beam.

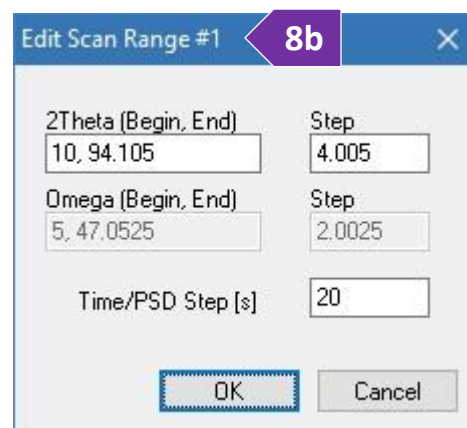
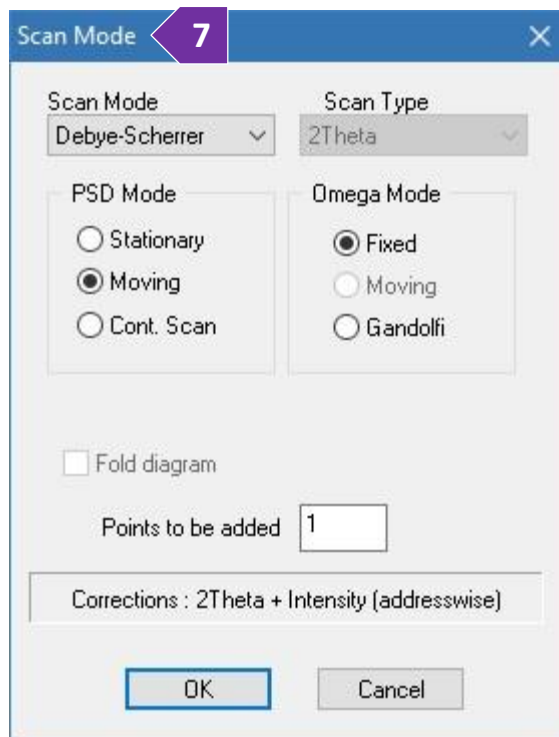
- c. Close windows of the enclosure and make sure the green light next to the top emergency button is on (figure 5c)



6. Start ‘WinXPOW’ software (icon on the desktop) and
- a. (Optional) Press on ‘Change Directory’ button to select your personal working directory under your group’s folder. Create a folder for the new files

IMSERC User Manual for StadiMP (v1.33)

- b. Under the 'Diffract.1' menu select 'Diffractometer Control'. It will take a few seconds to initialize hardware before control window opens
7. Under 'Ranges' menu, select the 'Scan Mode' option and set (figure 7)
 - a. 'Scan mode' to 'Debye-Scherrer' (this is the only option available)
 - b. 'PSD mode' to either 'Moving' or 'Stationary'. For collections that do not require a 2theta windows larger than 19 degrees, use the 'Stationary' option
 - c. 'Scan Type' is disable by default
 - d. 'Omega mode' to 'Fixed'
 - e. 'Points to be added' to '1'
8. Under 'Ranges' menu, select the 'Scan Range' option. On the 'Edit Range' window
 - a. Press on the '+' button to add a scan range
 - b. Double press on the new scan range you just added and:
 - i. Edit the '2theta range' accordingly (figure 8b) if you have selected 'Moving' for the 'PSD mode'. If 'Stationary' mode is used, define in degrees the center of the detector, and notice the '2theta range' values
 - ii. If you have selected 'Moving' for the 'PSD Mode', define the 'Step' of the detector, typically 4° . This step is different than the resolution step (2theta values in your powder pattern) which is fixed to 0.015° for a single scan. Resolution can be improved with more special scans (see a staff for more details). Our detector can cover a solid angle of $\sim 19^\circ$ with a single step, therefore with a step of 1° , the same 2theta region will be measured 19 times
 - iii. Set the integration 'Time/PSD Step' in seconds, typically 10-20 s. Signal-to-noise ratio improves when you measure with a longer exposure time or average multiple scans that have been collected with a long enough exposure
 - c. Keep adding more ranges according to your needs, e.g., add a range with longer exposure for the high angle reflections, etc.

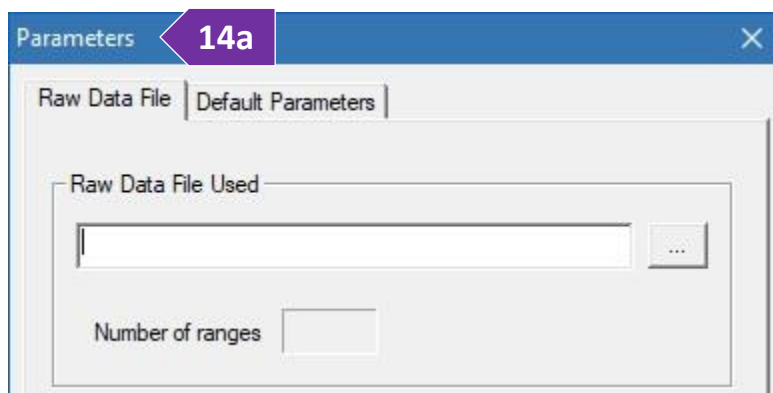


IMSERC User Manual for StadiMP (v1.33)

- Under the 'Ranges' menu, select the 'Scan Usage' option, and select 'Single Sample'
- Start the measurement by pressing on 'Data Collection' under the 'Measure' menu or pressing on the 'C' icon.
Verify that the exposure time is sufficient

- Once the acquisition parameters have been optimized, select the 'Scan Usage' option under the 'Ranges' menu, and select 'Temperature'

- Save the RAW file into your folder. The same filename and folder will be used for all files created during the variable temperature or time experiment



- Close/Exit the 'Diffractometer Control' window. Under the 'Diffract.1' menu select 'HTC Data Collection'. It will take a few seconds to initialize hardware before control window opens

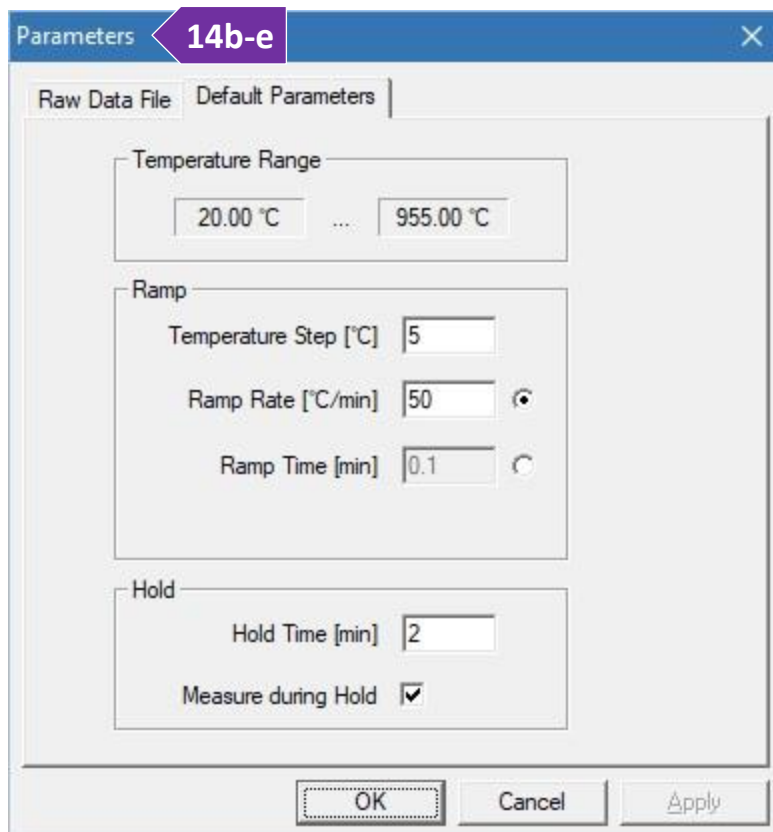
- Under the 'Setup' menu, select the 'Parameters' option

- Provide the path of the RAW file you created in step 12 (figure 14a). In case the RAW file has not been flagged as 'Temperature' (step 11), you will see an error message when you select the wrong RAW file
- Select the 'Default Parameters' tab in the 'Parameters' window (figure 14b-e)

- Temperature range is predefined based on the type of furnace and thermocouple installed

- 'Ramp' values are used as default parameters when a 'Ramp Step' is added in the temperature profile. These values can be modified afterwards when you build the temperature profile

- 'Temperature Step' is the spacing of the temperature points of your variable temperature collection. Spacing does not have to be the



IMSERC User Manual for StadiMP (v1.33)

same for the entire temperature range and 'Temperature Step' can be modified when you build the temperature profile

- You can define the heating or cooling speed by selecting either 'Ramp Rate' or 'Ramp Time'. Typical rates are 10-20 C/min. Heating/cooling speed can be different between temperature steps
- e. 'Hold' values are used for the isothermal steps. A measurement can be obtained only at a fixed temperature. If you need to measure dynamically during heating or cooling, talk to a Staff since this procedure requires advanced settings
- 'Hold Time' is the duration in minutes that the software will wait (soaking time) before taking a measurement or going to the next temperature step
 - 'Measure during Hold' triggers a measurement after the expiration of the 'Hold Time'. You need this option ticked if you want to take a measurement at a specific temperature point (most common)

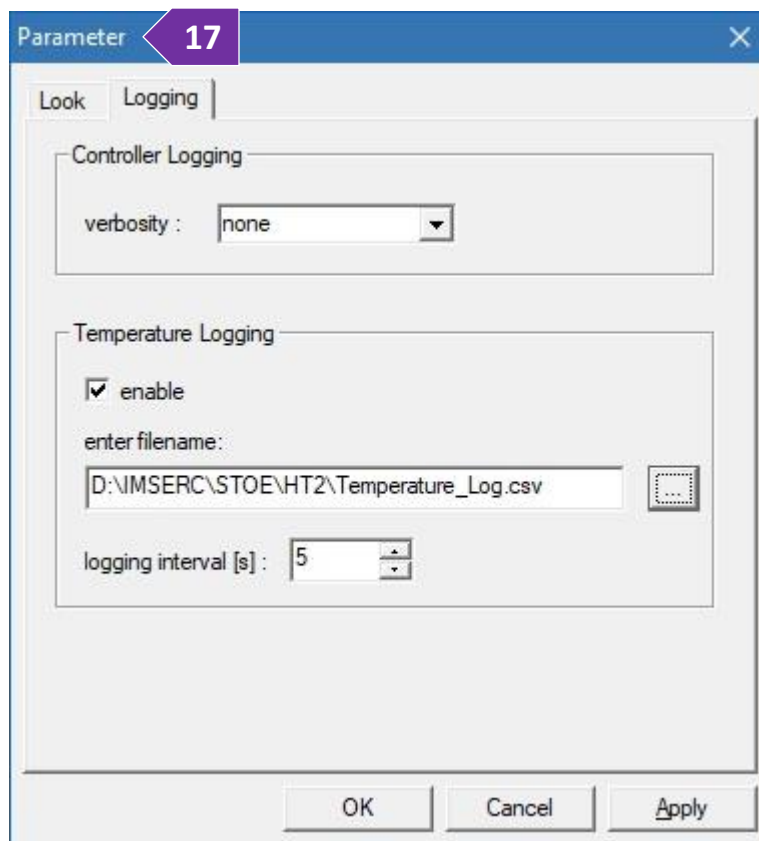
The screenshot shows the 'STOE Capillary Furnace' software window with a purple header bar containing the number '15'. The main area is a table with the following columns: Step, Temp. [...], Rate [°C/min], Wait, Meas, Range, Step Time, and Total Time. The table contains 24 rows of data, with the last row being 'Program End'. To the right of the table are several buttons: 'Add Ramp', 'Add Hold', 'Add Step', 'Add Steps', 'Delete', 'Parameters', and 'Load List...'. At the bottom of the window, there are two radio buttons: 'switch off controller' (selected) and 'hold last temperature'. The 'OK' and 'Cancel' buttons are at the bottom right.

Step	Temp. [...]	Rate [°C/min]	Wait	Meas	Range	Step Time	Total Time
0	25.00	4.00		No		00:01	00:01
1	25.00		00:01	Yes	1	00:09	00:10
2	100.00	4.00		No		00:18	00:29
3	100.00		00:01	Yes	1	00:09	00:38
4	150.00	4.00		No		00:12	00:50
5	150.00		00:01	Yes	1	00:09	00:59
6	175.00	4.00		No		00:06	01:05
7	175.00		00:01	Yes	1	00:09	01:14
8	200.00	4.00		No		00:06	01:21
9	200.00		00:01	Yes	1	00:09	01:30
10	225.00	4.00		No		00:06	01:36
11	225.00		00:01	Yes	1	00:09	01:45
12	250.00	4.00		No		00:06	01:51
13	250.00		00:01	Yes	1	00:09	02:00
14	275.00	4.00		No		00:06	02:06
15	275.00		00:01	Yes	1	00:09	02:15
16	300.00	4.00		No		00:06	02:22
17	300.00		00:01	Yes	1	00:09	02:31
18	325.00	4.00		No		00:06	02:37
19	325.00		00:01	Yes	1	00:09	02:46
20	350.00	4.00		No		00:06	02:52
21	350.00		00:01	Yes	1	00:09	03:01
22	375.00	4.00		No		00:06	03:07
23	375.00		00:01	Yes	1	00:09	03:16
24	400.00	4.00		No		00:06	03:23
Program End							

switch off controller hold last temperature

IMSERC User Manual for StadiMP (v1.33)

15. Under the 'Setup' menu, select the 'Profile' option for building your temperature sequence (figure 15)
 - a. First step is automatically added by the software. Double press on the first step to edit the initial values, such as starting temperature. If your first temperature point is near room temperature, ensure that the ramp rate is 10-20 C/min. If you prefer different temperature units than C, see step 17a below
 - b. Press on the 'Add Hold' button to add a 'Holding Time' and measurement (tick on the 'Measure during Hold' option)
 - c. 'Add Step' adds a pair of 'Add Ramp' and 'Add Hold' lines in the profile
 - d. 'Add Steps' adds a series of steps which is convenient if you want to run a variable temperature program with equally spaced temperature points
 - e. Use any combination of 'Add Ramp', 'Add Hold', and 'Add Steps' to create the desired temperature profile. There are not restrictions in terms of number of steps or duration of each step
 - f. For your final step, select the right option under the 'Program End' section at the bottom of the window
 - Most of the times you need to select 'switch off controller' to ramp down the temperature of the furnace to room temperature. Software will use an internally predefined cooling rate when you last step in the temperature profile is above 30 C
 - Select 'hold last temperature' only if you need to start your next measurement at a high temperature point. Heating source is localized in the center of the furnace while the walls of the furnace are water cooled. This enables the insertion or removal of a capillary at any temperature in case rapid heating or quenching is required
16. Press 'OK' to accept all values in the temperature profile
17. Under the 'Setup' menu, select the 'Preferences' option (figure 17)
 - a. Under the 'Look' tab menu, you can change the 'Temperature unit' and the color scheme of the temperature profile plot



IMSERC User Manual for StadiMP (v1.33)

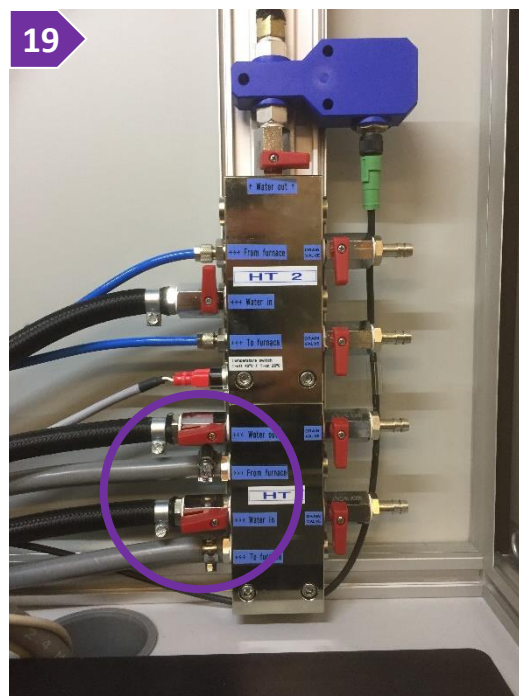
b. Select the 'Logging' tab in the 'Parameters' window. Enable 'Temperature Logging' for recording a detailed temperature log of the actual temperature as a function of time. Select the path and filename of where the temperature log will be saved

c. Press 'OK' to save the settings

18. Save the temperature profile (NATC file) by selecting 'Save as' under the 'File' menu. The NATC file contains the temperature steps and the path info for the RAW file. Logging information is not saved in the NATC file therefore you need to reset the setting of logging every time you perform a new measurement. If you need to repeat the same temperature profile for your future measurement, simply copy and paste the NATC file to the new folder and reset the path for the RAW file

19. Verify that the valves for the water lines to the furnace are on

a. There are two water valve boxes in the enclosure of the diffractometer at the lower right-hand corner labeled as 'HT' and 'HT2' (figure 19). The standard furnace uses the 'HT' box



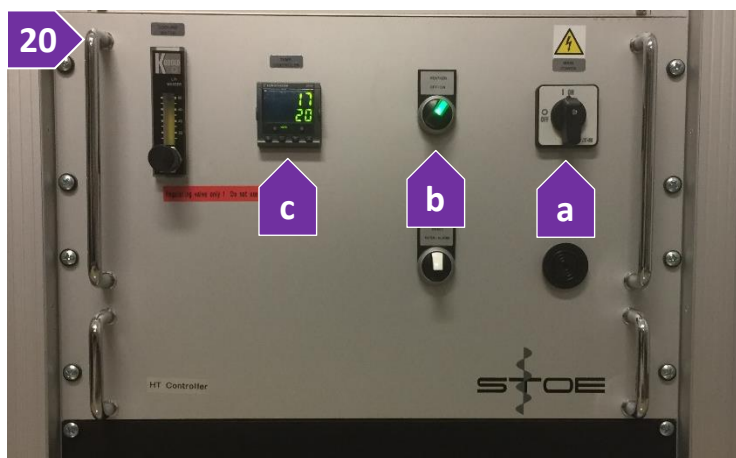
b. On the 'HT' box, turn the two water valves labeled as 'Water in' and 'Water out' on (circled on figure 19)

20. Turn the furnace controller on (figure 20)

a. Turn clockwise the main power switch (figure 20a)

b. Turn clockwise the power switch of the heater (figure 20b)

c. If all cables are connected properly on the furnace, you should see two temperature values on the temperature controller of the controller (figure 20c). If



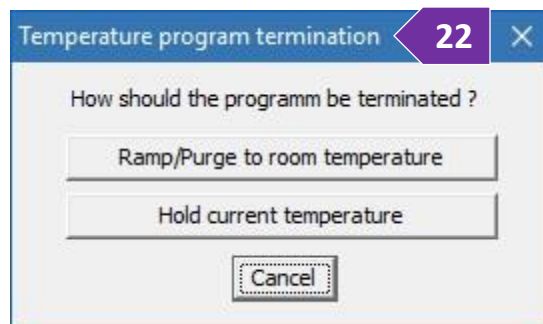
bottom temperature is missing, check if the thermocouple wire (green cable) is mounted on the furnace

21. Press the 'Execute' button or select 'Execute' under the 'Measurement' menu to start the variable temperature measurement

22. At any time during the variable temperature measurements, you can pause/stop the measurement by pressing the 'Shutdown' button. You have the option to (figure 22)

IMSERC User Manual for StadiMP (v1.33)

- Cool down the furnace to room temperature by pressing the 'Ramp/Purge to room temperature' button or
 - Hold at the current temperature
23. At the end of your measurement
- a. Stop the capillary spinning by rotating the 'Sample spinner' dial clockwise
 - b. Turn the power of the heater on the temperature controller off
 - c. Turn the main power of the temperature controller off
 - d. Close the water valves on the 'HT' box
 - e. Turn the inert gas valve off under the regulator behind the monitor. Do not close the main valve on the gas tanks
 - f. Complete the form at the end of this document with the pressure of the tank
 - g. End your reservation in NUcore

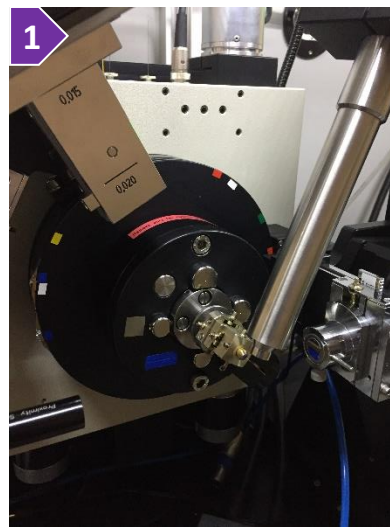


IMSERC User Manual for StadiMP (v1.33)

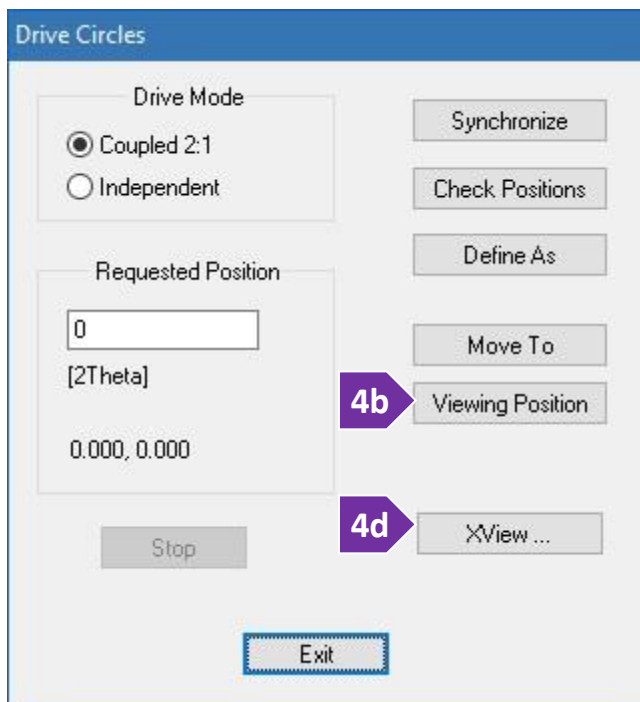
RUN SAMPLES AT LOW TEMPERATURE (80-500 K) IN CAPILLARY WITH THE CRYOSTAT

Running VT measurements requires 1) the creation of a data file that contains the acquisition parameters, and 2) the execution of the data file at specific temperature points defined in a customizable temperature profile.

1. Verify that the cryostat (Oxford Cryostream) and the capillary stage with the goniometer are mounted onto the diffractometer (figure 1), otherwise see a Staff
2. Pack your powdered sample into the appropriate capillary (i.e., polyimide, Glass, Borosilicate, Quartz, Sapphire)
3. If stage is spinning and/or occupied with a sample holder, you need to
 - a. Stop any rotation by turning the 'Sample spinner' dial counterclockwise



- b. Remove the beam stop. Watch the '[stadimp-beamstop](#)' video for a visual demonstration
 - c. Remove the capillary (if any) and leave it on the sample table outside the enclosure of the diffractometer
 - d. Mount your capillary onto the goniometer
4. Align the capillary on the rotation axis of the goniometer. Watch the '[stadimp-debye-scherrer-goniometer-capillary](#)' video for a visual demonstration
 - a. Under the 'Diffractometer' menu, select the 'Drive Circles' option or press on the 'D' icon



- b. Press the 'Viewing Position' button (figure 4b) to drive the 2theta above the dovetail mount for the camera
- c. Mount the optical camera onto the dovetail. Verify the USB cable is connected to the camera (figure 4c)
- d. Press the 'XView' button to launch the video software and press on the 'Start Video' button (figure 4d)
- e. Adjust the X, Y, dX, and dY screws on the goniometer, so that the capillary is parallel and centered when rotated by hand 360°
- f. Close the 'XView' software, unmount the camera from the dovetail, and put it back onto its holder
- g. Mount the beamstop

IMSERC User Manual for StadiMP (v1.33)

9. Start the measurement by pressing on 'Data Collection' under the 'Measure' menu or pressing on the 'C' icon. Verify that the exposure time is sufficient
10. Once the acquisition parameters have been optimized, select the 'Scan Usage' option under the 'Ranges' menu, and select 'Temperature'
11. Save the RAW file into your folder. The same filename will be used for all files created at the variable temperature or time points



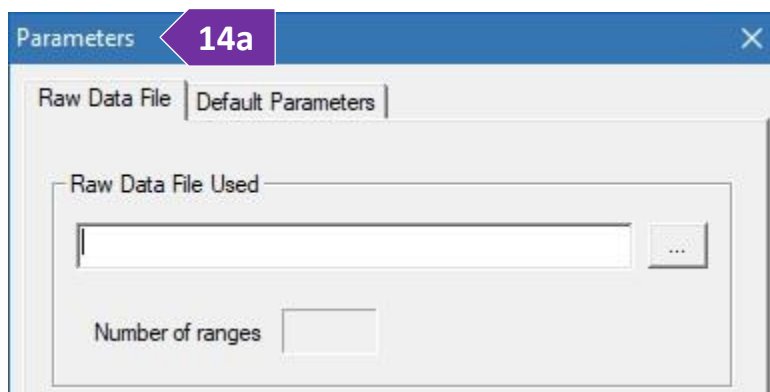
12. Turn on the nitrogen gas valve V7 located under the regulator on the wall behind the computer monitor (figure 12). A room temperature protective nitrogen gas is used to prevent ice formation on the capillary. Flow in the flowmeter should be 10L/min

13. Close/Exit the 'Diffractometer Control' window. Under the 'Diffract.1' menu select 'HTC Data Collection. It will take a few seconds to initialize hardware before control window opens

14. Under the 'Setup' menu, select the 'Parameters' option

- a. Provide the path of the RAW file

(figure 14a) you created in step 11. In case the RAW file has not been flagged as 'Temperature' (step 11), you will see an error message when you select the wrong RAW file



IMSERC User Manual for StadiMP (v1.33)

- b. Select the 'Default Parameters' tab in the 'Parameters' window (figure 14b-e)
- c. Temperature range is predefined based on the type of cryostat installed

d. 'Ramp' values are used as default parameters when a 'Ramp Step' is added in the temperature profile. These values can be modified afterwards when you build the temperature profile

- i. 'Temperature Step' is the spacing of the temperature points of your variable temperature collection. Spacing does not have to be the same for the entire temperature range and 'Temperature Step' can be modified when you build the temperature profile

- ii. You can define the heating or cooling speed by selecting either

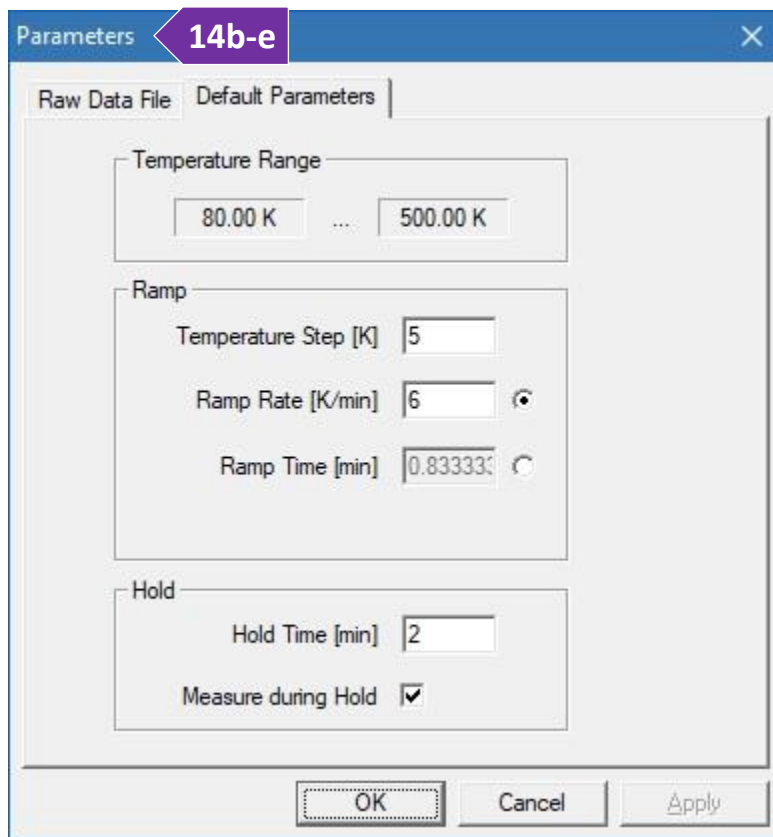
'Ramp Rate' or 'Ramp Time'. Maximum rate is 6 C/min. Heating/cooling speed can be different between temperature steps

e. 'Hold' values are used for the isothermal steps. A measurement can be obtained only at a fixed temperature. If you need to measure dynamically during heating or cooling, talk to a Staff since this procedure requires advanced settings

- i. 'Hold Time' is the duration in minutes that the software will wait (soaking time) before taking a measurement or going to the next temperature step
- ii. 'Measure during Hold' triggers a measurement after the expiration of the 'Hold Time'. You need this option ticked if you want to take a measurement at a specific temperature point

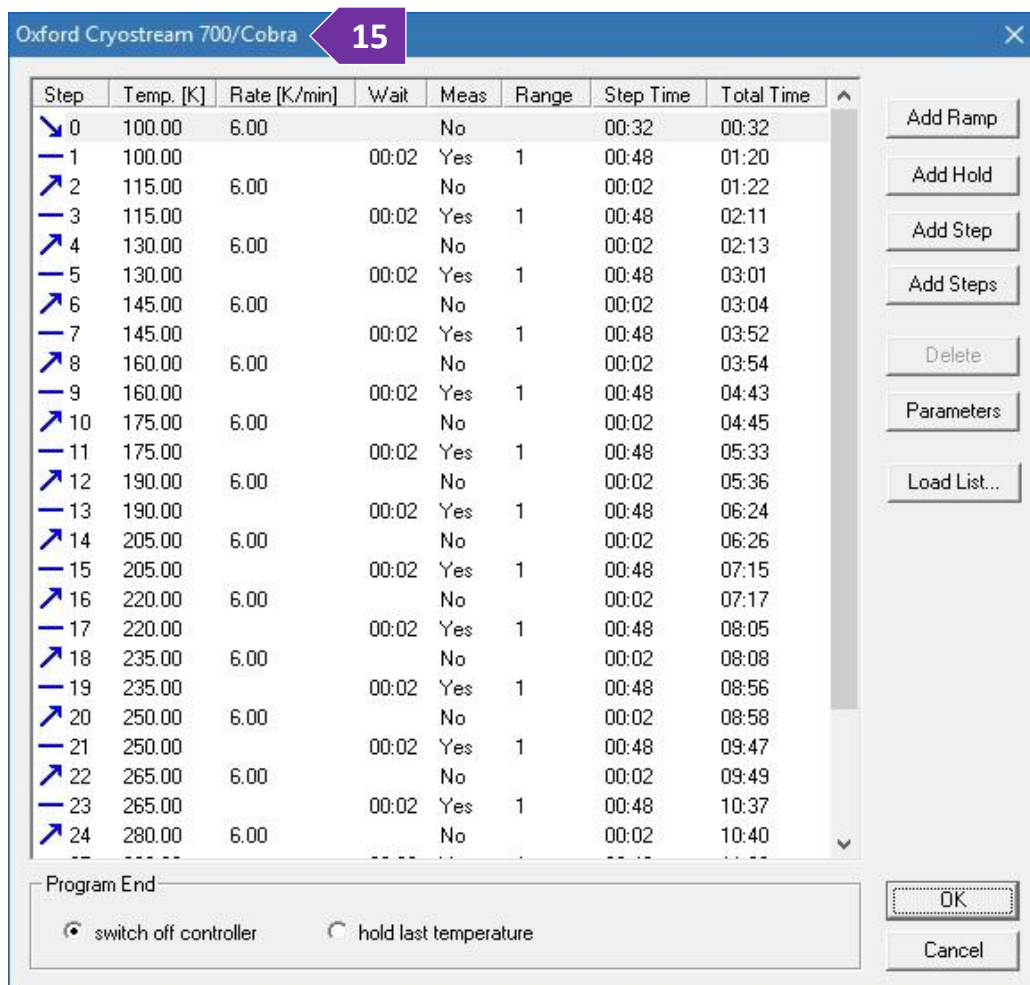
15. Under the 'Setup' menu, select the 'Profile' option for building your temperature sequence (figure 15)

- a. First step is automatically added by the software. Double press on the first step to edit the initial values, such as starting temperature
- b. Press on the 'Add Hold' button to add a 'Holding Time' and measurement (tick on the 'Measure during Hold' option)



IMSERC User Manual for StadiMP (v1.33)

- c. 'Add Step' adds a pair of 'Add Ramp' and 'Add Hold' lines in the profile
- d. 'Add Steps' adds a series of steps which is convenient if you want to run a variable temperature program with equally spaced temperature points



- e. Use any combination of 'Add Ramp', 'Add Hold', and 'Add Steps' to create the desired temperature profile. There are not restrictions in terms of number of steps or duration of each step.
- f. For your final step, select the right option under the 'Program End' section at the bottom of the window
 - i. Most of the times you need to select 'switch off controller' to ramp down the temperature of the furnace to room temperature. Software will use an internally predefined cooling rate when you last step in the temperature profile is different than room temperature
 - ii. Select 'hold last temperature' only if you need to start your next measurement at a low temperature point

16. Press 'OK' to accept all values in the temperature profile

17. Under the 'Setup' menu, select the 'Preferences' option (figure 17)

IMSERC User Manual for StadiMP (v1.33)

- a. Under the 'Look' tab menu, you can change the 'Temperature unit' and the color scheme of the temperature profile plot
- b. Select the 'Logging' tab in the 'Parameters' window (figure 17). Enable 'Temperature Logging' for recording a detailed temperature log of the actual temperature as a function of time. Select the path and filename of where the temperature log will be saved
- c. Press 'OK' to save the changes

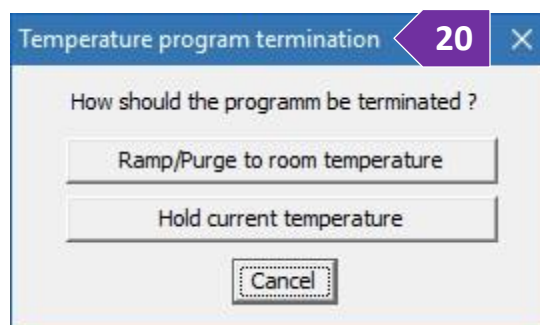
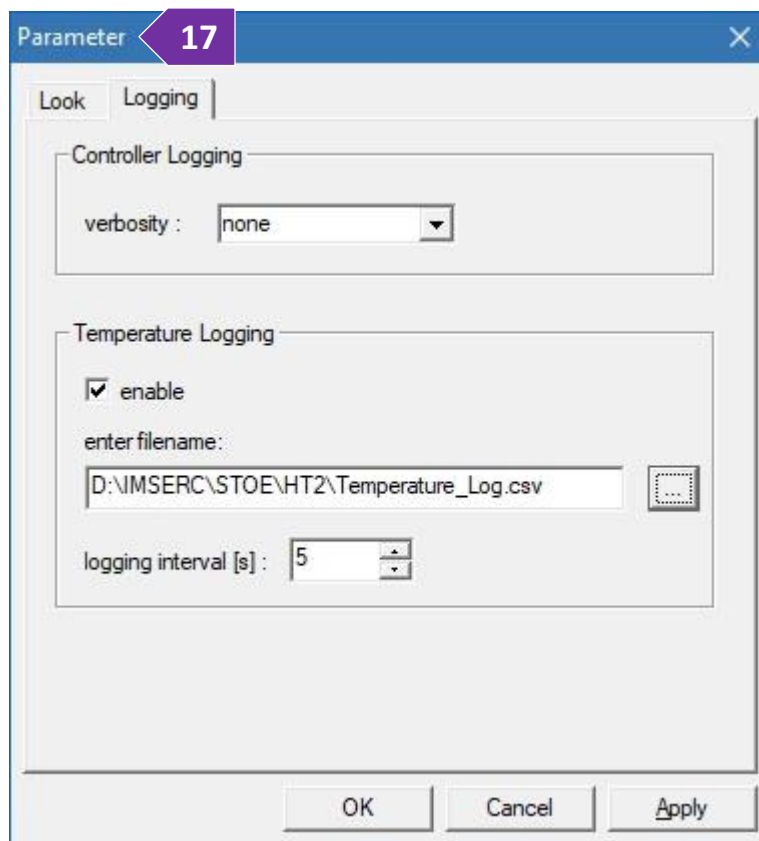
18. Save the temperature profile (NATC file) by selecting 'Save as' under the 'File' menu. The NATC file contains the temperature steps and the path info for the RAW file. Logging information is not saved in the NATC file therefore you need to reset the setting of logging every time you perform a new measurement. If you need to repeat the same temperature profile for your future measurement, simply copy and paste the NATC file to the new folder and reset the path for the RAW file

19. Press the 'Execute' button or select 'Execute' under the 'Measurement' menu to start the variable temperature measurement

20. At any time during the variable temperature measurements you can pause/stop the measurement by pressing the 'Shutdown' button. You have the option to either (figure 20)
- Cool down the furnace to room temperature by pressing the 'Ramp/Purge to room temperature' button or
 - Hold at the current temperature

21. At the end of your measurement

- a. Stop the capillary spinning by rotating the 'Sample spinner' dial clockwise
- b. Turn the nitrogen gas valve off (V7) under the regulator behind the monitor
- c. End your reservation in NUcore

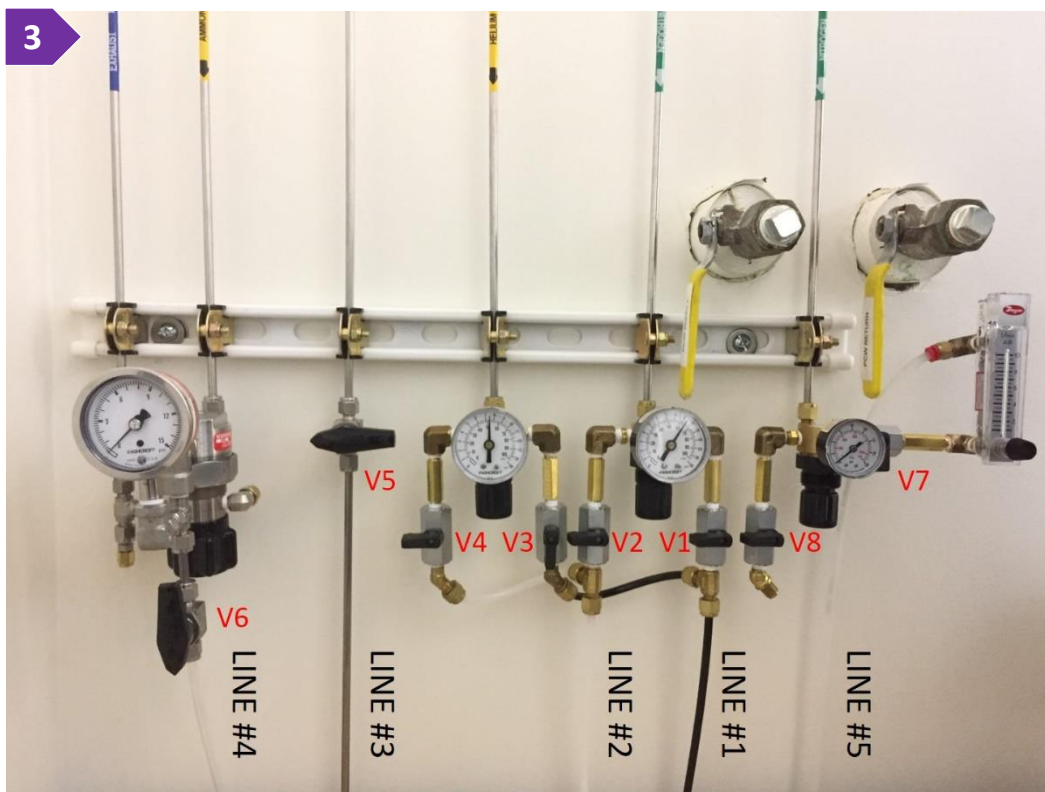
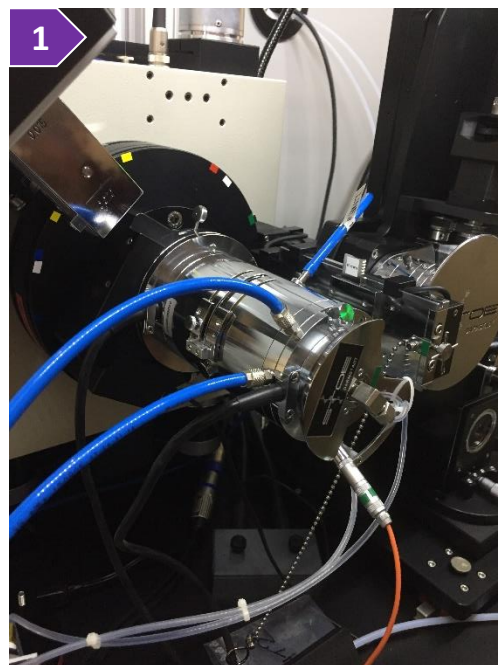


IMSERC User Manual for StadiMP (v1.33)

RUN SAMPLES AT HIGH TEMPERATURE IN CAPILLARY USING THE *IN-SITU* HT2 FURNACE

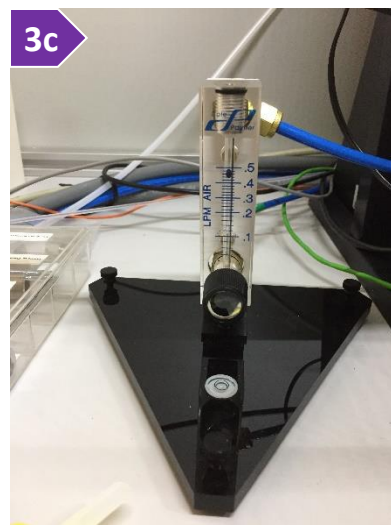
Running VT measurements requires 1) the creation of a data file that contains the acquisition parameters, and 2) the execution of the data file at specific temperature points defined in a customizable temperature profile.

1. Verify that the furnace is mounted onto the diffractometer (figure 1), otherwise see a Staff
2. Heating element of the furnace is protected against oxidation by flowing inert gas through the blue tubing connected on the side of the furnace. Running the furnace without inert gas (especially above 400 C) will damage the heating element. Turn on the inert gas valve under the regulator located on the wall behind the computer monitor
3. Line #1 (black tubing in figure 3) connects to the flowmeter that provides inert gas to the furnace (blue tubing connected to the furnace)



IMSERC User Manual for StadiMP (v1.33)

- a. For using N₂ as a protection gas, turn valve #1 (V1) on (black handle is parallel to the line) and turn V3 off (black handle is perpendicular to the line)
- b. Pressure should be between 7-10 psi, do not adjust the regulators if pressure is lower, please see a staff
- c. Check the flow on the flowmeter mounted on the black triangular base connected with the blue tubing (figure 3c), flow should be between 0.4 and 0.5 l/min. Adjust flow if necessary



4. Check the gas tanks located in the southwest corner of room BG66 next to the StadiVari (figure 4). Pressure of the gauge near the main valve of the tank (circled on the figure) should be at least 100 psi. If pressure is low, replace the tank with a new cylinder located in the same corner, estimate 100 psi consumption per day. Complete the form at the end of this document with the pressure of the tank before and after your measurement



5. Pack your powdered sample in the middle of the appropriate tube (i.e., polyimide, Glass, Borosilicate, Quartz, Sapphire)
 - a. Insert the tube into the furnace. Size of the beam is 2-4 mm depending on the collimator used
 - b. Set the rocking on by pressing the ON/OFF button on the switch connected under the 'Sample spinner' dial. Tube is pre-aligned with the furnace. In case you have a very small amount of powder into the capillary, use the optical camera to align the sample into the beam

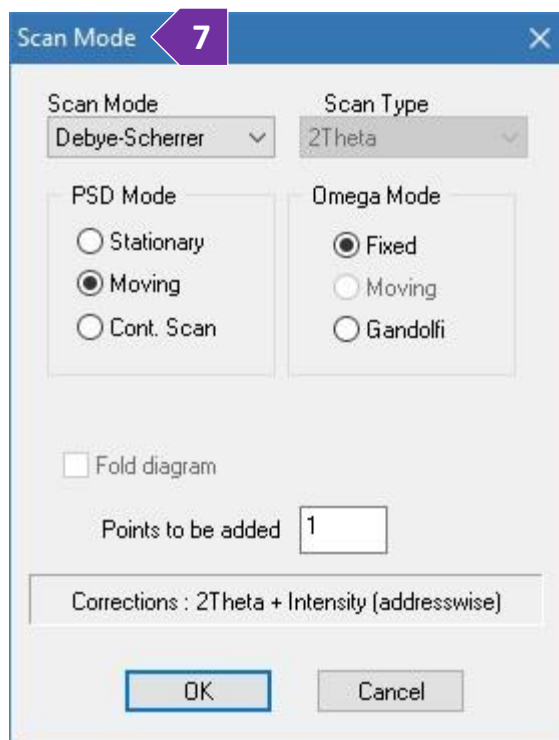
- c. Close windows of the enclosure and make sure the green light next to the top emergency button is on



6. Start 'WinXPOW' software (icon on the desktop) and
 - a. (Optional) Press on 'Change Directory' button to select your personal working directory under your group's folder. Create a folder for the new files
 - b. Under the 'Diffract.1' menu select 'Diffractometer Control'. It will take a few seconds to initialize hardware before control window opens
7. Under 'Ranges' menu, select the 'Scan Mode' option and set (figure 7)
 - a. 'Scan mode' to 'Debye-Scherrer' (this is the only option available)

IMSERC User Manual for StadiMP (v1.33)

- b. 'PSD mode' to either 'Moving' or 'Stationary'. For quick collections (10-120s) that do not require a 2theta windows larger than 19 degrees, use the 'Stationary' option
 - c. 'Scan Type' is disable by default
 - d. 'Omega mode' to 'Fixed'
 - e. 'Points to be added' to '1'
8. Under 'Ranges' menu, select the 'Scan Range' option. On the 'Edit Range' window
- a. Press on the '+' button to add a scan range
 - b. Double press on the new scan range you just added and
 - i. Edit the '2theta range' accordingly if you have selected 'Moving' for the 'PSD mode'. If 'Stationary' mode is used, define in degrees the center of the detector, and notice the '2theta range' values
 - ii. If you have selected 'Moving' for the 'PSD Mode', define the 'Step' of the detector, typically 4° . This step is different than the resolution step (2theta values in your powder pattern) which is fixed to 0.015° for a single scan. Resolution can be improved with more special scans (see a staff for more details). Our detector can cover a solid angle of $\sim 19^\circ$ with a single step, therefore with a step of 1° , the same 2theta region will be measured 19 times
 - iii. Set the integration 'Time/PSD Step' in seconds, typically 10-20 s. Signal-to-noise ratio improves when you measure with a longer exposure time or average multiple scans that have been collected with a long enough exposure (see 'Scan usage' below)
 - iv. Keep adding more ranges according to your needs, e.g., add a range with longer exposure for the high angle reflections, etc.
9. Under the 'Ranges' menu, select the 'Scan Usage' option, and select 'Single Sample'
10. Start the measurement by pressing on 'Data Collection' under the 'Measure' menu or pressing on the 'C' icon. Verify that the exposure time is sufficient
11. Once the acquisition parameters have been optimized, select the 'Scan Usage' option under the 'Ranges' menu, and select 'Temperature'
12. Save the RAW file into your folder. The same filename will be used for all files created at the variable temperature or time points

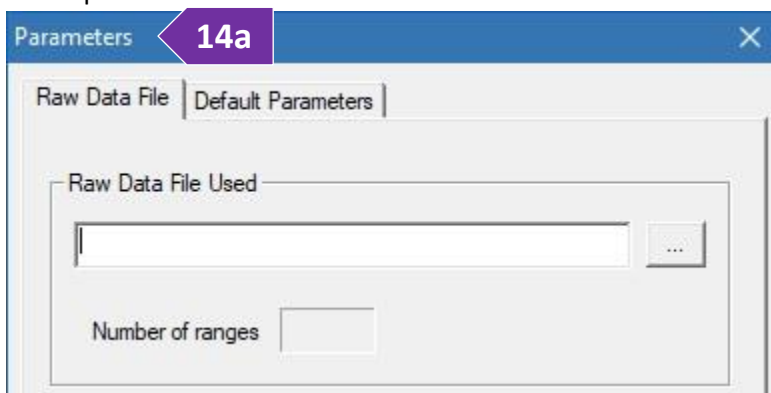


IMSERC User Manual for StadiMP (v1.33)

13. Close/Exit the 'Diffractometer Control' window. Under the 'Diffract.1' menu select 'HTC Data Collection. It will take a few seconds to initialize hardware before control window opens

14. Under the 'Setup' menu, select the 'Parameters' option

a. Provide the path of the RAW file you created in step 12 (figure 14a). In case the RAW file has not been flagged as 'Temperature' (step 11), you will see an error message when you select the wrong RAW file



b. Select the 'Default Parameters' tab in the 'Parameters' window (figure 14b-e)

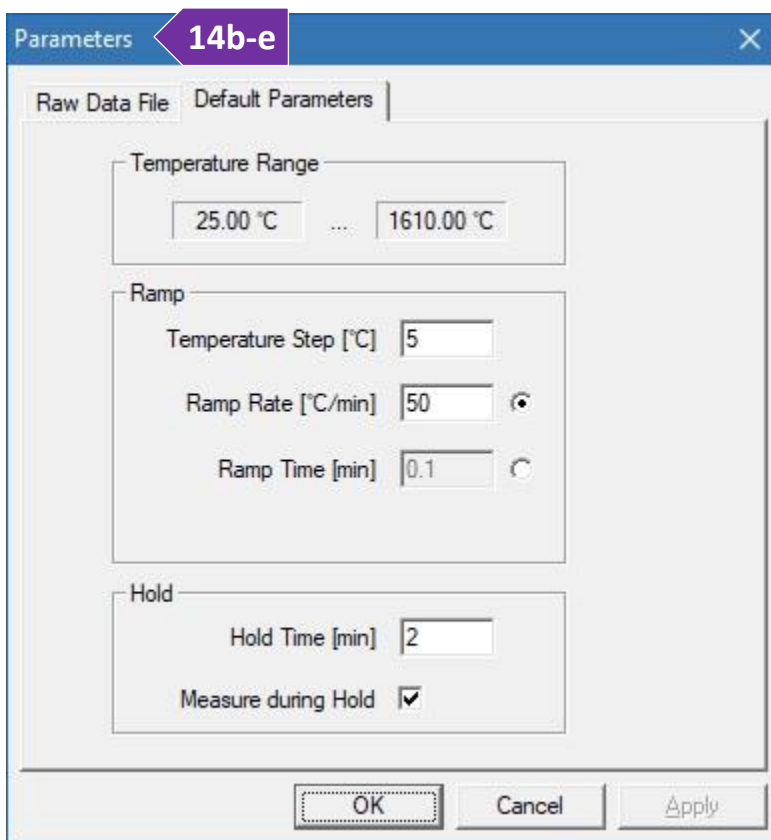
c. Temperature range is predefined based on the type of furnace and thermocouple installed

d. 'Ramp' values are used as default parameters when a 'Ramp Step' is added in the temperature profile. These values can be modified afterwards when you build the temperature profile

i. 'Temperature Step' is the spacing of the temperature points of your variable temperature collection. Spacing does not have to be the same for the entire temperature range and 'Temperature Step' can be modified when you build the temperature profile

ii. You can define the heating or cooling speed by selecting either 'Ramp Rate' or 'Ramp Time'. Maximum rate is 50C/min. Heating/cooling speed can be different between temperature steps

e. 'Hold' values are used for the isothermal steps. A measurement can be obtained only at a fixed temperature. If you need to measure dynamically during heating or cooling, talk to a Staff since this procedure requires advanced settings



IMSERC User Manual for StadiMP (v1.33)

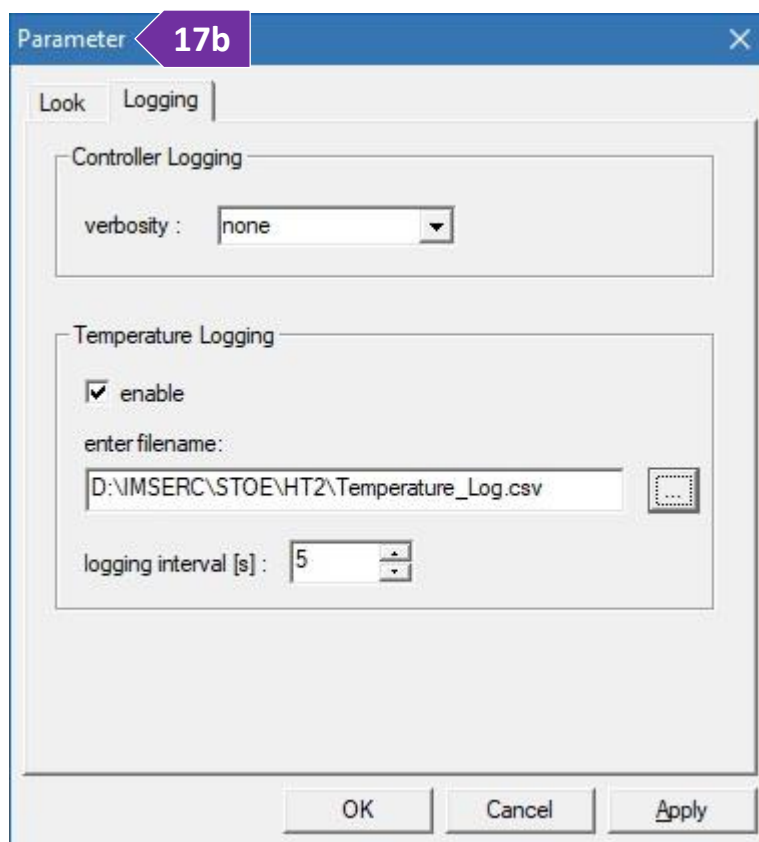
- i. 'Hold Time' is the duration in minutes that the software will wait (soaking time) before taking a measurement or going to the next temperature step
 - ii. 'Measure during Hold' triggers a measurement after the expiration of the 'Hold Time'. You need this option ticked if you want to take a measurement at a specific temperature point
15. Under the 'Setup' menu, select the 'Profile' option for building your temperature sequence (figure 15)
- a. First step is automatically added by the software. Double press on the first step to edit the initial values, such as starting temperature
 - b. Press on the 'Add Hold' button to add a 'Holding Time' and measurement (tick on the 'Measure during Hold' option)
 - c. 'Add Step' adds a pair of 'Add Ramp' and 'Add Hold' lines in the profile

The screenshot displays the 'STOIE InSitu HT2 Furnace' software window, labeled '15'. It features a table with columns for Step, Temp. [K], Rate [K/min], Wait, Meas, Range, Step Time, and Total Time. The table contains 24 rows of data, alternating between ramping and holding steps. To the right of the table are buttons for 'Add Ramp', 'Add Hold', 'Add Step', 'Add Steps', 'Delete', 'Parameters', and 'Load List...'. At the bottom, there are radio buttons for 'switch off controller' (selected) and 'hold last temperature', along with 'OK' and 'Cancel' buttons.

Step	Temp. [K]	Rate [K/min]	Wait	Meas	Range	Step Time	Total Time
0	298.15	4.00		No		00:01	00:01
1	298.15		00:01	Yes	1	00:09	00:10
2	373.15	4.00		No		00:18	00:29
3	373.15		00:01	Yes	1	00:09	00:38
4	423.15	4.00		No		00:12	00:50
5	423.15		00:01	Yes	1	00:09	00:59
6	448.15	4.00		No		00:06	01:05
7	448.15		00:01	Yes	1	00:09	01:14
8	473.15	4.00		No		00:06	01:21
9	473.15		00:01	Yes	1	00:09	01:30
10	498.15	4.00		No		00:06	01:36
11	498.15		00:01	Yes	1	00:09	01:45
12	523.15	4.00		No		00:06	01:51
13	523.15		00:01	Yes	1	00:09	02:00
14	548.15	4.00		No		00:06	02:06
15	548.15		00:01	Yes	1	00:09	02:15
16	573.15	4.00		No		00:06	02:22
17	573.15		00:01	Yes	1	00:09	02:31
18	598.15	4.00		No		00:06	02:37
19	598.15		00:01	Yes	1	00:09	02:46
20	623.15	4.00		No		00:06	02:52
21	623.15		00:01	Yes	1	00:09	03:01
22	648.15	4.00		No		00:06	03:07
23	648.15		00:01	Yes	1	00:09	03:16
24	673.15	4.00		No		00:06	03:23

IMSERC User Manual for StadiMP (v1.33)

- d. 'Add Steps' adds a series of steps which is convenient if you want to run a variable temperature program with equally spaced temperature points
 - e. Use any combination of 'Add Ramp', 'Add Hold', and 'Add Steps' to create the desired temperature profile. There are not restrictions in terms of number of steps or duration of each step
 - f. For your final step, select the right option under the 'Program End' section at the bottom of the window
 - i. Most of the times you need to select 'switch off controller' to ramp down the temperature of the furnace to room temperature. Software will use an internally predefined cooling rate when you last step in the temperature profile is above 30 C
 - ii. Select 'hold last temperature' only if you need to start your next measurement at a high temperature point. Heating source is localized in the center of the furnace while the walls of the furnace are water cooled. This enables the insertion or removal of a capillary at any temperature in case rapid heating or quenching is required
16. Press 'OK' to accept all values in the temperature profile
17. Under the 'Setup' menu, select the 'Preferences' option
- a. Under the 'Look' tab menu, you can change the 'Temperature unit' and the color scheme of the temperature profile plot
 - b. Select the 'Logging' tab in the 'Parameters' window (figure 17b). Enable 'Temperature Logging' for recording a detailed temperature log of the actual temperature as a function of time. Select the path and filename of where the temperature log will be saved
 - c. Press 'OK' to save the settings
18. Save the temperature profile (NATC file) by selecting 'Save as' under the 'File' menu. The NATC file contains the temperature steps and the path info for the RAW file. Logging information is not saved in the NATC file therefore you need to reset the setting of logging every time you perform a new measurement. If you need



IMSERC User Manual for StadiMP (v1.33)

to repeat the same temperature profile for your future measurement, simply copy and paste the NATC file to the new folder and reset the path for the RAW file

19. Verify that the valves for the water lines to the HT2 furnace are open

a. There are two water valve boxes in the enclosure at the lower right-hand corner labeled as 'HT' and 'HT2'. The standard furnace uses the 'HT' box and the in-situ furnace uses the 'HT2' box

b. On the 'HT2' box, turn the two water valves labeled as 'Water in' and 'Water out' on (circled on figure 19)

20. Turn the furnace controller on (figure 20)

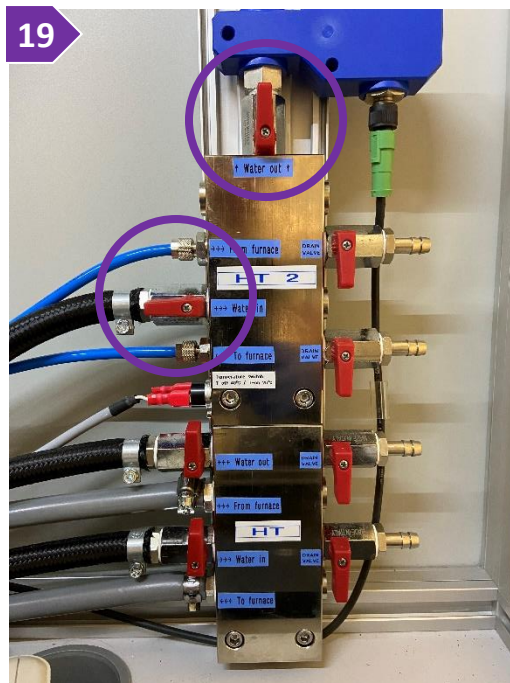
a. Verify that the cooling water valves are open

b. Verify that the inert gas for the furnace (Line #1) is on

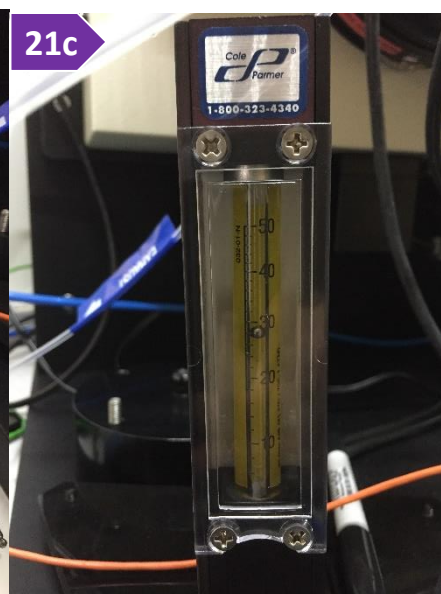
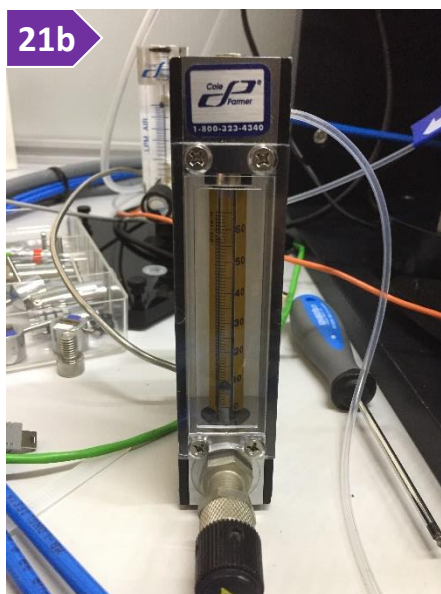
c. Turn the ON/OFF power switch on the controller ON

d. Press the 'F1' button on the controller to reset any error messages

e. If you see no temperature reading, ensure that the thermocouple wire (orange cable) is connected on the furnace

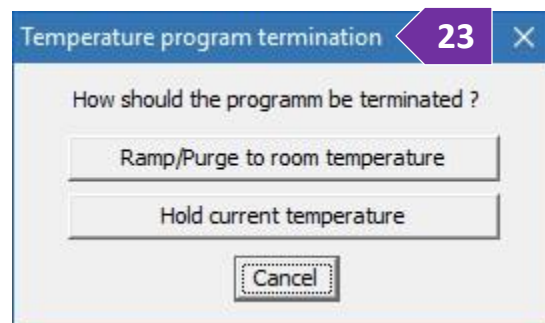


21. Turn the gas in contact with the sample on



IMSERC User Manual for StadiMP (v1.33)

- a. Turn the appropriate valves (see figure 3) on. Use Line #2 (Valves V2 or V4) for inert gas and/or line #4 (Valve V6) for reactive gas
 - b. Adjust the inlet flowmeter for the desired flow (figure 21b)
 - c. Observe the outlet flowmeter for a flow reading (figure 21c), depending on the packing of the tube flow might be reduced in respect with the inlet flow
22. Press the 'Execute' button or select 'Execute' under the 'Measurement' menu to start the variable temperature measurement
23. At any time during the variable temperature measurements, you can pause/stop the measurement by pressing the 'Shutdown' button. You have the option to (figure 23)
- o Cool down the furnace to room temperature by pressing the 'Ramp/Purge to room temperature' button or
 - o Hold at the current temperature
24. At the end of your measurement
- a. Stop the capillary spinning by rotating the 'Sample spinner' dial clockwise
 - b. Turn the power of the heater on the temperature controller off
 - c. Turn the main power of the temperature controller off
 - d. Close the water valves on the 'HT2' box
 - e. Turn the inert gas valve off under the regulator behind the monitor. Do not close the main valve on the gas tanks
 - f. Complete the form at the end of this document with the pressure of the tank
 - g. End your reservation in NUcore



IMSERC User Manual for StadiMP (v1.33)

PUBLICATION

A. EXPERIMENTAL SECTION

Modify the text below according to the setup and conditions you used during the measurement:

“PXRD data were collected at room temperature on a STOE-STADI-MP powder diffractometer equipped with an asymmetric curved Germanium monochromator (***CuK α 1 radiation, $\lambda = 1.54056 \text{ \AA}$ | MoK α 1 radiation, $\lambda = 0.70930 \text{ \AA}$ | AgK α 1 radiation, $\lambda = 0.559407 \text{ \AA}$***) and one-dimensional silicon strip detector (MYTHEN2 1K from DECTRIS). The line focused ***Cu X-ray tube was operated at 40 kV and 40 mA | Mo X-ray tube was operated at 50 kV and 40 mA | Ag X-ray tube was operated at 40 kV and 40 mA***. Powder was packed in ***a 3 or 8 mm metallic mask | X mm capillary and sandwiched between two polyimide or acetate layers of tape | capillary***. The as received powder was packed in ***a X mm glass | borosilicate | quartz*** capillary and placed into the furnace. Temperature stability is typically 0.1 C. Intensity data from ***YY*** to ***ZZ*** degrees two theta were collected over a period of ***XX*** mins. Instrument was calibrated against a NIST Silicon standard (640d) prior the measurement.”

B. ACKNOWLEDGEMENT

“This work made use of the IMSERC X-RAY facility at Northwestern University, which has received support from the Soft and Hybrid Nanotechnology Experimental (SHyNE) Resource (NSF ECCS-2025633), and Northwestern University.”

IMSERC User Manual for StadiMP (v1.33)

TROUBLESHOOTING

A. THE COMPUTER SCREEN WILL NOT TURN ON

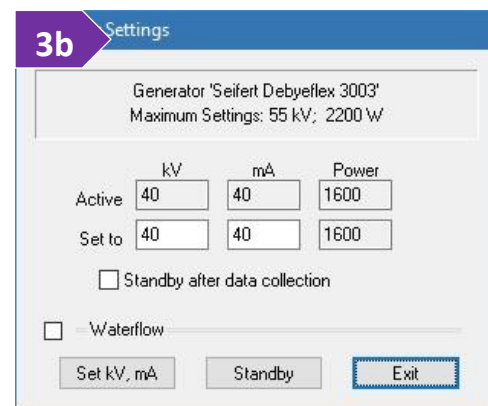
Begin your reservation in NUcore to initiate access to the instrument

B. COMPUTER REQUIRES LOGIN AND A PASSWORD

The default 'StadiMP' user account should be logged in. In case the computer was restarted, the password for the 'StadiMP' account is . See '[Default instrument status](#)' section for more details

C. THERE IS NO INTENSITY OR THE DIFFRACTION IS VERY WEAK DURING COLLECTION

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, reseal all safety doors by opening and closing each door. Watch the '[stadimp-door-safety](#)' video for a visual demonstration
2. The sample is heavily absorbing X-rays. Repack the sample and do not fill-up the metallic masks with material. Use only a thin layer of powder for heavily absorbing samples
3. Check the voltage and current settings of the X-ray generator (middle of the rack with electronics/controllers). Default operating settings for Cu-radiation are 40 kV and 40 mA (figure 3a). In case:
 - a. Values on the generator are zero, contact a Staff
 - b. Settings are different than the default values, under the 'Setup' menu select 'Generator'. At the 'Generator' window (figure 3b) put the default values at the 'Set to' fields and press the 'Set kV, mA' button. It takes a few seconds for the generator to apply the new values



D. TEMPERATURE OF THE HT FURNACE WILL NOT INCREASE

Verify that you have turned on the power of the heater ([step 20b](#), section 'Run samples at high temperature in capillary with the HT furnace'). The green light on the heater switch must be on. You need to ramp the software temperature option to room temperature before turning on the heater

IMSERC User Manual for StadiMP (v1.33)

E. SAMPLE STAGE WILL NOT SPIN

Sample spinning might not start right away after rotation of the sample spinner, but sample spinning will start with data collection. Please check the following steps:

4. Verify that the sample spinner dial is rotated all the way clockwise. Panel with the dial is located on the left side of the goniometer (figure E1). Watch the '[stadimp-sample-spinner](#)' video for a visual demonstration. When dial is not turned all the way clockwise, autosampler will either not start or start and load only the first sample in the queue
5. Verify that the acquisition software is running. Acquisition software controls the spinning function and if software is closed, spinning will not start when turning the sample spinner dial clockwise. If acquisition software is already running, please restart the acquisition software. To (re)start the acquisition software, under the 'Diffract.1' menu of 'WinXPow', select 'Diffractometer Control'. Check [step 3](#) under the '[General steps for data acquisition](#)' section for more details about restarting the acquisitions software



F. SPECIFIC ERROR MESSAGES

Please check the error list below with the suggested actions:

1. Job stopped by an error! Error while starting data collection!

Verify that all settings for using the autosampler are correct. Verify that the sample spinner dial is rotated all the way clockwise. Panel with the dial is located on left side of the goniometer (figure D)

2. Job stopped by an error! Error while reading PSD intensities

Restart the acquisition software. If after restarting the error is persistent, please contact a staff

3. Unable to init the PDT-Board! Error: Unable to connect to MYTHEN interface! ('-get version' failed)

Restart the acquisition software. If after restarting the error is persistent, please contact a staff

4. Unable to communicate with the generator! Generator does not respond. The program will terminate!

Restart the acquisition software. If after restarting the error is persistent, please contact a staff

IMSERC User Manual for StadiMP (v1.33)

G. THERE IS AN ERROR/PROBLEM WITH THE INSTRUMENT THAT IS NOT ADDRESSED UNDER THE TROUBLESHOOTING SECTION

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

APPENDICES

APPENDIX A: ALIGNMENT TOOLS AND POSITION OF ADJUSTMENT SCREWS ON THE DEBEY-SCHERRER GONIOMETER HEAD

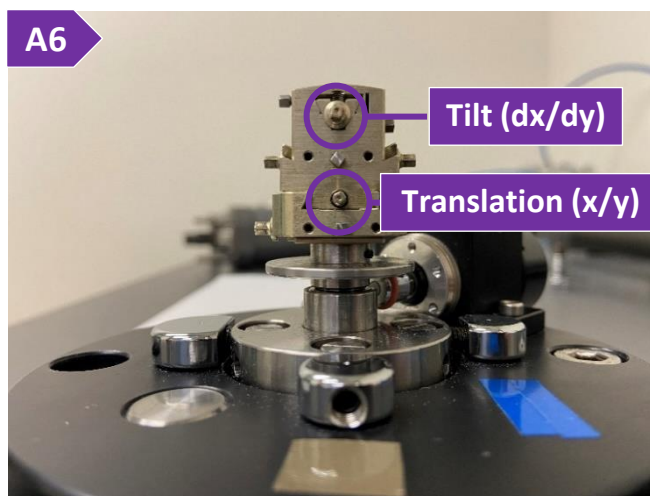
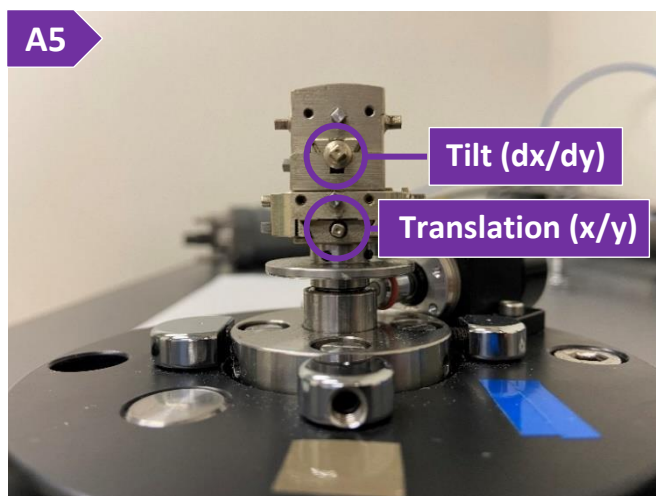
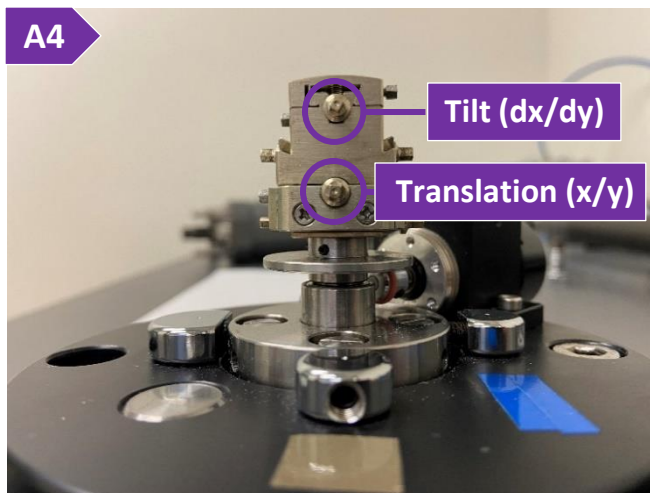
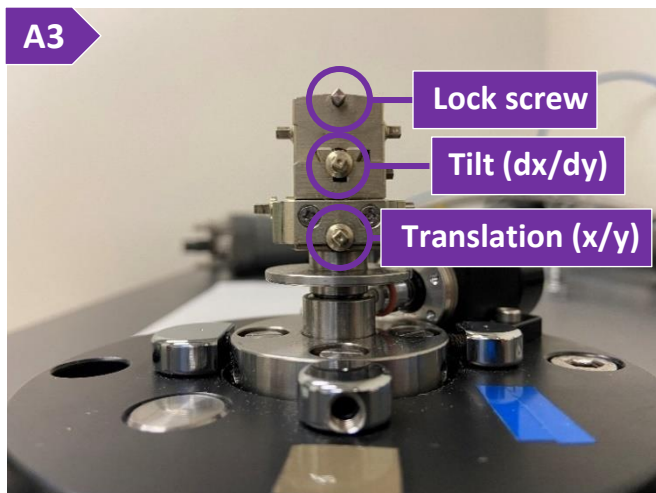
Locate the toolbox (figure A1) in the enclosure of the diffractometer and take one of the two wrenches (figure A2) needed for adjusting the alignment screws.



Figure A1. Toolbox containing the various accessories needed for the alignment of the goniometer head. **Figure A2.** Wrenches that can be used for adjusting the alignment screws on the goniometer head

First, locate the 'Lock screw' as shown in figure A3, and secure the pin with your sample onto the goniometer head using the wrench. With the wrench, adjust the X, Y, dX, and dY screws (figures A3-A6) on the goniometer head, so that the capillary is parallel and centered when rotated by hand 360°. Watch the '[stadip-debye-scherrer-goniometer-capillary](#)' video for a visual demonstration of the alignment process.

IMSERC User Manual for StadiMP (v1.33)



IMSERC User Manual for StadiMP (v1.33)

APPENDIX B: COMPARING POWDER PATTERNS COLLECTED AT DIFFERENT WAVELENGTHS

To compare powder patterns collected with different wavelengths/sources, such as Cu-, Mo, Ag, synchrotron-radiation, you need to convert the 2θ axis (which is wavelength dependent) to a different unit. Two popular options for wavelength-independent units are:

- $Q = 4\pi\sin(\theta)/\lambda$, where λ is the wavelength of the source (CuK α 1 radiation, $\lambda = 1.54056 \text{ \AA}$ | MoK α 1 radiation, $\lambda = 0.70930 \text{ \AA}$ | AgK α 1 radiation, $\lambda = 0.559407 \text{ \AA}$), and
- $1/d = 2\sin(\theta)/\lambda$

To use the software that came with the diffractometer ('WinXPow'):

1. Start the 'Graphics' program by selecting 'Graphics' under 'Raw Data' menu on 'WinXPow'
2. Load the patterns collected with different wavelengths by either opening (File->Open) a selection of files or pressing the 'Add raw Data' option under the 'Window' menu
3. Select 'Scaling axes' under the 'Options' menu and select either 'Q' or '1/D' for the 'X-axis' option

IMSERC User Manual for StadiMP (v1.33)

REVISIONS

v1.33 2021/09/22	<ul style="list-style-type: none">• 'Appendix' section added• 'Troubleshooting' section updated• Training video files transferred from Box to OneDrive, and all relevant links were updated
v1.31 2020/08/23	<ul style="list-style-type: none">• Links to online video module added for demonstrating sample alignment using a capillary goniometer head
v1.30 2020/07/27	<ul style="list-style-type: none">• Improvements and more pictures added for facilitating remote training:<ul style="list-style-type: none">○ More pictures were added under the sample preparation section which has its own section now (used to be under 'General steps for sample packing and data acquisition' section). The 'General steps for data acquisition' section was modified accordingly○ Links to online video modules added for demonstrating holder (un)mounting processes using modes of 'transmission with flat disc holders', 'reflection', and 'mounting capillaries on the HT furnace'. Additionally, references to the videos for proper verification of alignment of safety doors and un(mounting) the beamstop were added in the corresponding sections• Details about custom runs using the autosampler (Debye-Scherrer) were added
v1.20 2020/02/18	<ul style="list-style-type: none">• Reformatted according to the latest template. Sections about 'Safety', 'Data management', and 'Software' were added• Added more pictures for each section• Updated figures to reflect the new options and layout in windows for MainMenu 3.12.5, PowDat 3.6.0.6, Graphic 3.6.0.1, and RawDat 3.6.0.1
v1.18 2018/06/24	<ul style="list-style-type: none">• Updated figures to reflect the Win10 theme and new options for MainMenu 3.12, PowDat 3.6.0.1, Graphic 3.6.0.0, RawDat 3.6.0.0, FIT 3.6.0.0, Calibrate 3.6.0.0, Index 3.6.0.0, Theo 3.6.0.0, SystEval 3.6.0.0, Cryst 3.6.0.0, and Retaust 3.6.0.0• Added sections for variable temperature measurements• Added more pictures for each section• Added 'Publication' section• Added 'Troubleshooting' section
v1.16 2016/08/30	<ul style="list-style-type: none">• Release of original version of the user manual for MainMenu 3.4.6, PowDat 3.4.8, Graphic 3.4.4, RawDat 3.4.4, FIT 3.4.0, Calibrate 3.4.2, Index 3.4.1, Theo 3.4.2, SystEval 3.4.2, Cryst 3.4.2, and Retaust 3.4.0