User Manual
For Standard Operation of the Xeuss 3.0

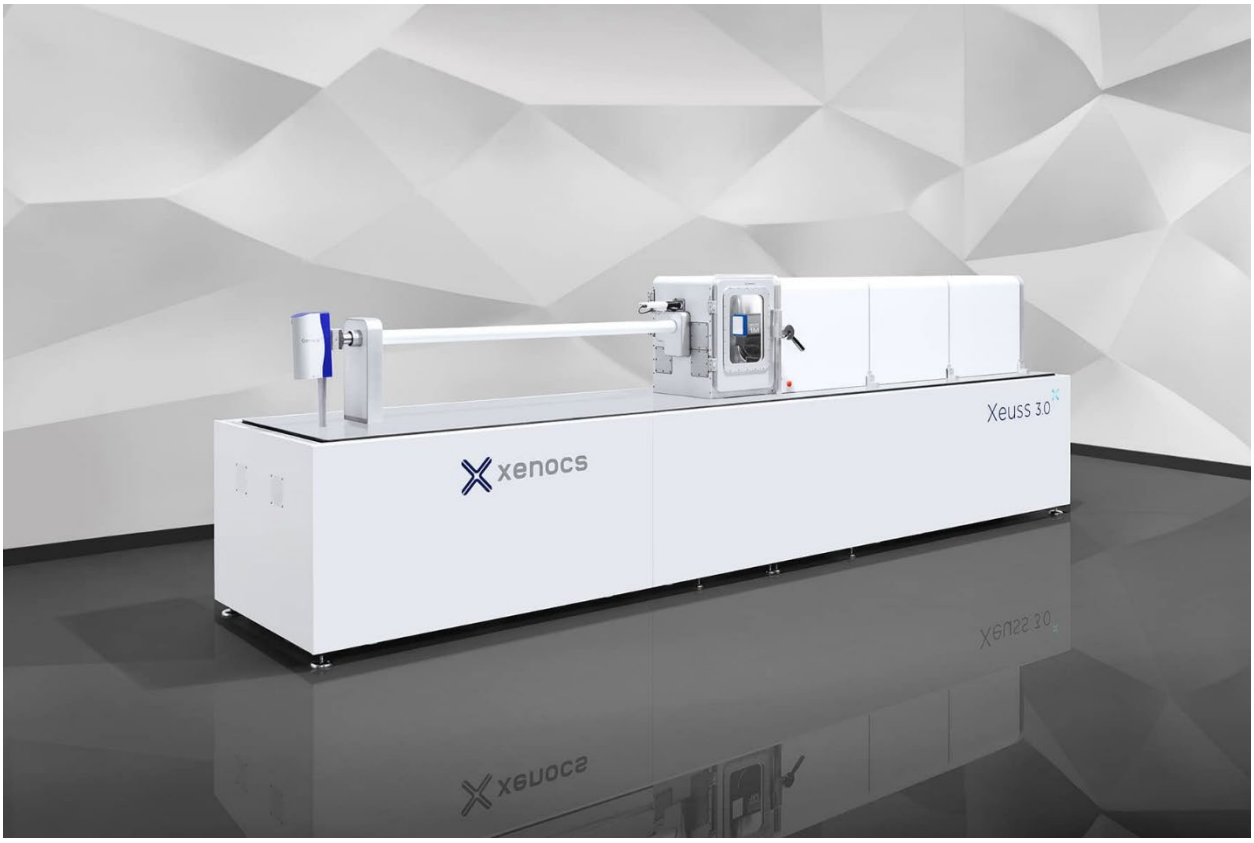


TABLE OF CONTENTS

01. PURPOSE

a. This shortened manual provides instruction for the general work flow and operation of the Xeuss 3.0 with the standard, under-vacuum SAXS stages, advanced GISAXS, linkam heater, and JSP reusable capillary stages, the biocube, and in-air SAXS measurements. For more detailed instructions refer to the General Manual found in the manuals folder on the main Xeuss computer.

02. SPECIFICATIONS

a. Xeuss 3.0 consists of the table, beam collimation parts, the beam path, data collection chambers, the Cu based Genix^{3D} µ-source X-ray beam, the slit collimation system, the sample chamber, an Eiger 2R 1M 2-D detector and chiller from Dectris, 3 electronic controller racks for the detector and motor computers, and a computer with SPEC, XICC, and XSACT installed for instrument control, data acquisition, and data processing.

03. REFERENCES

a. Xeuss 3.0 Operation Manual (located on the Xeuss 3.0 control computer) by Xenocs

b. XSACT Manual by Xenocs

c. Xeuss Small Angle X-ray Scattering (SAXS) SOP by the IAC at Princeton University

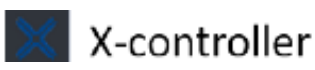
d. SAXSLab Website at Sapienza, University of Rome

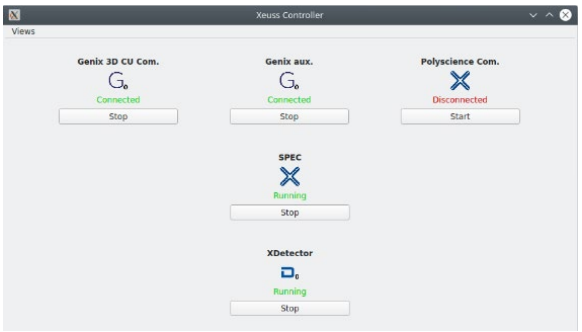
04. SYSTEM/PROGRAM START UP

a. Log on to your session using the FOM to turn on the monitors of the control computer

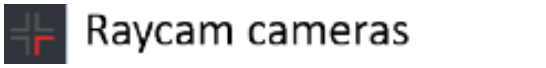
b. Usually, all the necessary control software should be always left on. In the case of all the software being closed, refer to the rest of this section. Otherwise, proceed to stage loading.

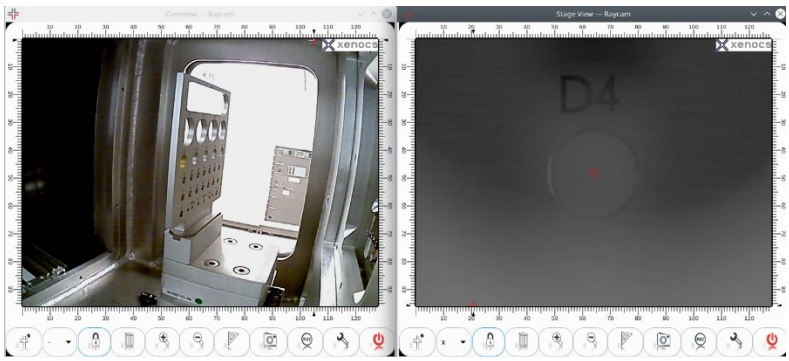
c. Open “X-controller” via the icon on the lefthand side of the screen. This is the main program for controlling the components of the Xeuss 3.0. In the Xeuss Controller window that pops up, turn on the controllers for your setup. A minimum operation will need Genix 3D CU Com, XDetector, and SPEC. If you need to access the Linkam controllers, you would need to click on views in the upper left corner and check the linkam controller option.





d. Open “Raycam cameras”. This will open 2 windows: Overview and Stage View. Use “Overview” to help you position the sample stage and the detector. Use “Stage View” and its red crosshair to help line a sample up with the x-ray beam.





e. Open “XICC”. This is the user interface where you can control your data acquisition parameters. You are now ready to load your stage and sample of choice.

06. SAMPLE LOADING FOR STANDARD SAXS STAGES – WEAR GLOVES

a. **IMPORTANT:** Open capillaries with liquids, wet samples, or closed cells with liquids or air inside may burst in a vacuum chamber and should not used if vacuuming the chamber down. This can damage the detector and will incur costly charges on your group for repairs.

b. SOLIDS: Use the provided Kapton tape to attach your sample to the desired slot.

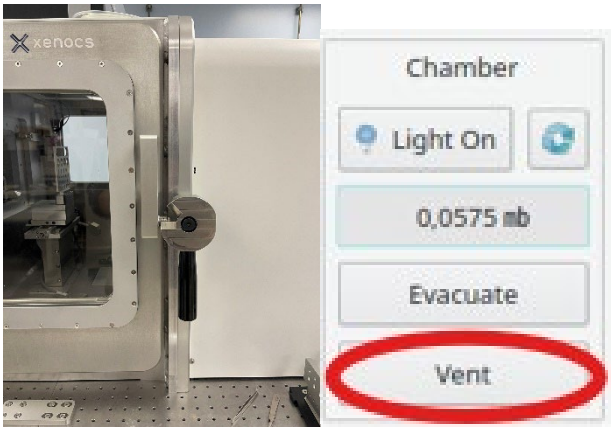
c. POWDERS: Load your sample into a washer with 2 sticky Kapton windows. Mount your washer on the loading rack, and then load the rack onto the Powder stage, with the conical depressions facing out and away from the source.

d. CAPILLARIES: The capillary holder can hold capillaries with an external diameter of 2mm, though larger capillaries can be mounted on the external face of the sample holder using tape (though the sample-detector distance will need to be adjusted for these scans, see the general manual for details). Make sure your capillaries are sealed in a vacuum proof manner. If unsure of your setup, consult the staff scientist.

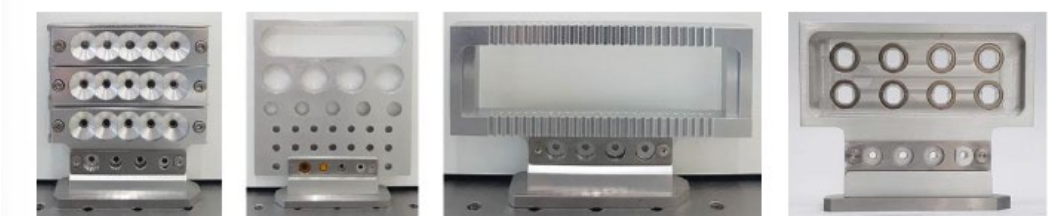
e. GELS/POWDERS: Gels/powders that need to be sealed from the vacuum of the main chamber can be encased in capsules for this stage. Each capsule will consist of the capsule body, 3 O-rings, 2 Kapton windows, a spacer, and a cap. Choose spacers and caps that are the appropriate size for your application. Follow the visual guide below to set up a capsule. Add your sample before the 2nd Kapton window is installed so that your sample will be between 2 windows in the final product.

05. STAGE LOADING – WEAR GLOVES WHEN HANDLING STAGES

f. The sample chamber will likely be under vacuum at the start of your session. Make sure that the door handle for the sample chamber is in the free position. Using XICC, vent the chamber. This process will take a few minutes and will be done when the console output disappears from the screen. This is a good time to load your sample on to your desired stage. Refer to the sample loading section (03) for directions for your specific stage.

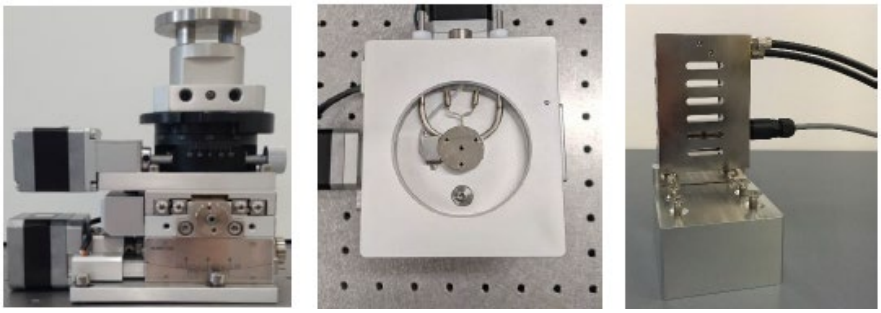


g. If you are using a standard SAXS stage (solids, powders, capillaries, or powder/gels stage) you will be able to freely swap out the loaded stage for yours. The standard stages have magnets that will hold the stage down during measurements, just make sure to align the stage with the standard stage insert. If unsure of the direction to place it, check the bottom of the stage for the RFI chip and line it up with its corresponding component on the baseplate.



Powders **Solids** **Capillaries** **Gels/Powders**

h. If you are using an advanced stage (GISAXS, BioCube, Linkam, JSP stage for refillable capillaries) please refer to the relevantly titled section towards the end of this SOP.

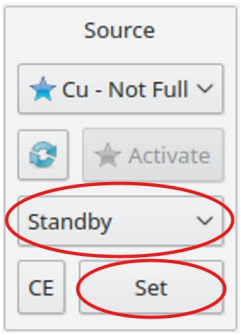


GISAXS **Linkam Heater Stage** **JSP Stage**

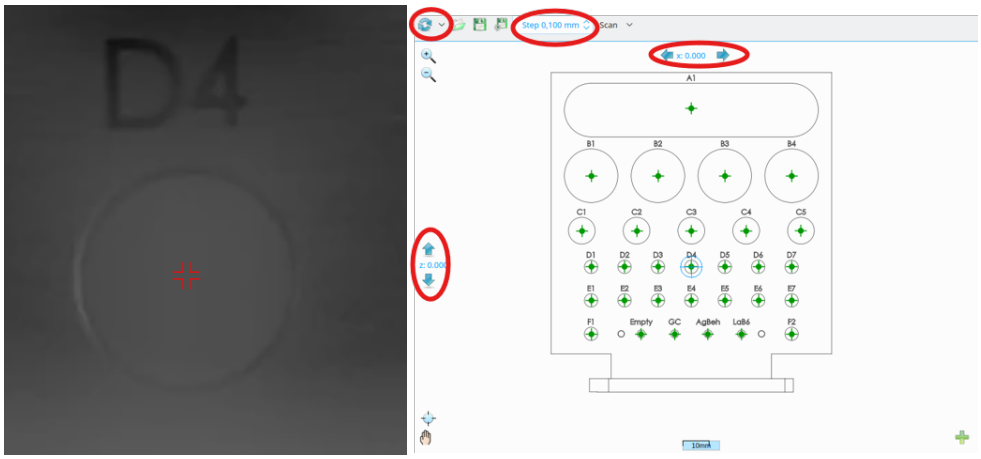
i. After your stage is loaded, place the sample chamber door handle in the lock position and select "Evacuate" in XICC. Check that the door handle drops to the free position after a few seconds of pumping. The chamber will pump down to ~0.03 mbar, which can take around 10 minutes.

07. SETTING UP AN ACQUISITION

a. When the chamber is back under vacuum, set the source from standby mode to full power by selecting the 'Standby' dropdown menu in the source window. Then select 'Set' to power up the source.

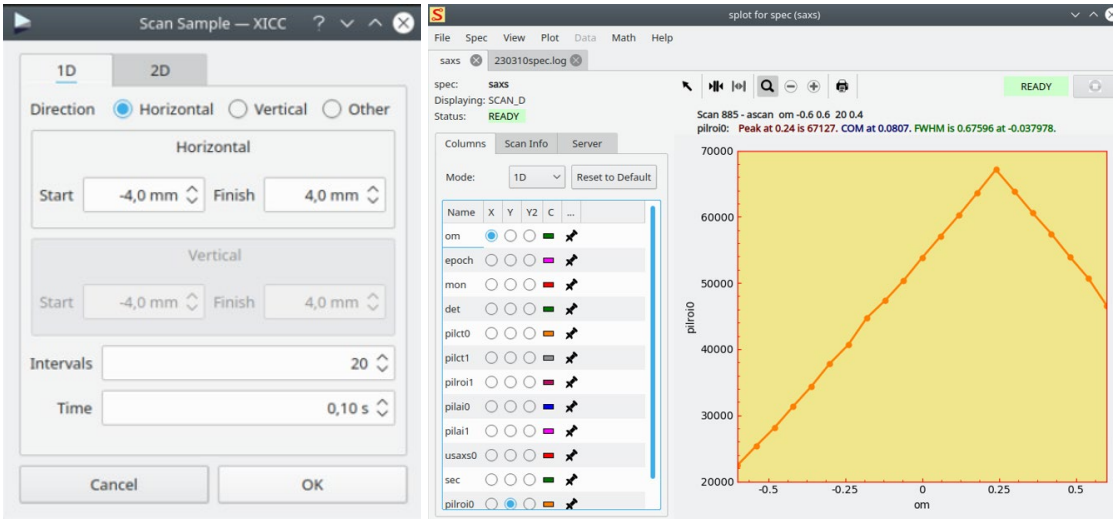


b. It is good practice to recalibrate where (0,0) is on your stage at the start of a session. To do this, select the refresh button at the top left of the window. This will autodetect the stage and center it at (0,0) relative to where the program thinks it is. Make note of where the program thinks (0,0) is and use the Stageview camera to confirm. If it is off, use the arrows on the window to move the stage up, down, left and right to move the stage until the crosshair in Stageview is centered where (0,0) should be for your stage. You can change the step size of the stage motors by typing a different number at the top. Once your stage is close to (0,0) you can select the dropdown menu next to the refresh button and select the "Calibrate to (0,0)" option. This will reset the (0,0) position within XICC.

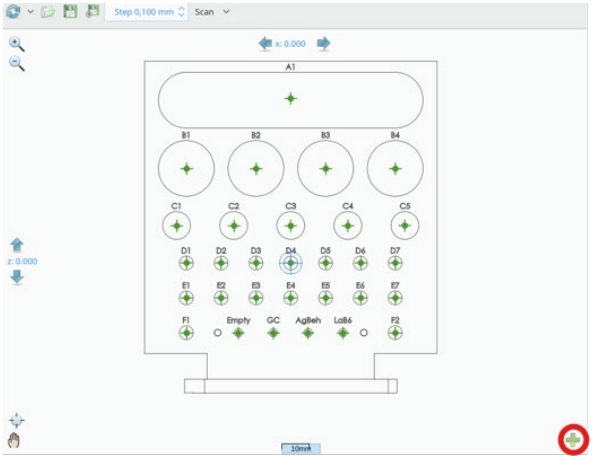


c. To align the beam with your sample, select the point on the stage control panel that matches your sample position. You can then select 'Go To' at the bottom of the window to move the stage to that position.

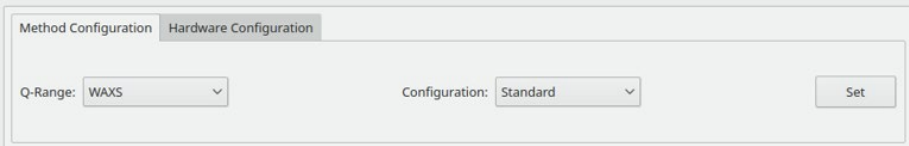
d. **SCAN SAMPLE:** If you need to align the beam with your sample further, there is a scan/align option at the top of the window that will let you take a scan of the hole with the beam. The start and end fields refer to distance around your current position. Intervals refers to how many data points will be taken between the defined start and end positions. Time refers to the time that the detector is exposed to the beam at each point. When you hit OK, a window called "splot for spec" will open and start graphing the detector counts vs position in real time. Use the generated plot to determine what position would be best for your scan (typically a good position is the FWHM of the detected peak for a hole, or the middle of a valley for a denser sample). The align hole/capillary scan options will automatically move the stage to the FWHM of a scan peak.



e. If you would like to make a new point of interest (POI) that you can return to later, select the green plus ✚ icon on the window to make a new POI. You can name this point and specify the exact position you want it to be within the information window that pops up when the point is selected.



f. Once your sample is aligned with the beam, you can set up the method configuration of your choosing. For Q-range, select the sample-to-detector (SDD) preset that corresponds to your Q-range of interest. Configuration determines the slit sizes for the beam (see table). The combination of sample detector distance and slit size has an impact on the X-ray flux values of the set up. Select a configuration that corresponds well with your sample. Press the set button to finalize your configuration choice.



Mode	Config	Slit Size (mm)	SDD (mm)	q-range (Å^-1)	d-space (Å)	I (Mph/s)
WAXS	Hi Int	1.4	55	~0.15-3.4	1.8-42	178
	Std	0.7	55	~0.11-3.4	1.8-57	114
	Hi Res	0.3	55	~0.08-3.4	1.8-78	64
MAXS	Hi Int	1.4	300	~0.022-0.9	7-280	114
	Std	0.7	300	~0.017-0.9	7-360	64
	Hi Res	0.3	300	~0.014-0.9	6-450	9.3
SAXS	Hi Int	1.4	900	~0.008-0.25	25-780	64
	Std	0.7	900	~0.055-0.25	25-1150	9.3
	Hi Res	0.3	900	~0.035-0.25	25-1780	1.2
ESAXS	Hi Int	1.4	1750	~0.0055-0.13	47-1150	64
	Std	0.7	1750	~0.0035-0.13	47-1780	9.3
	Hi Res	0.3	1750	~0.0025-0.13	47-2500	1.2

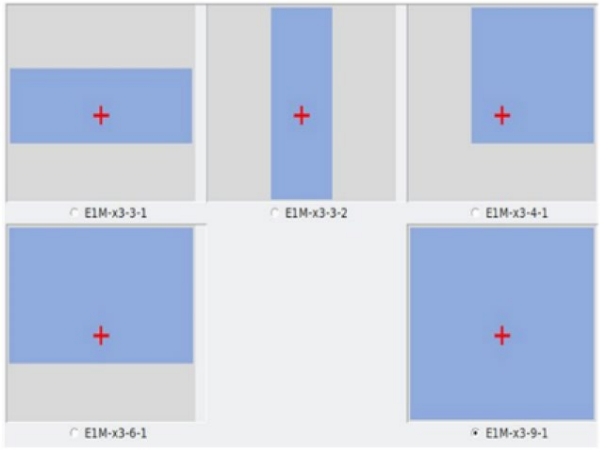
If you prefer a more advanced control over SDD and collimation configuration, you can select the hardware configuration mode. Here you can set a manual SDD and choose from a list of predefined slit apertures (see table below for details).

Table 10 –Slit apertures for the predefined collimation modes for C, HR and UHR Xeuss 3.0 systems.

C			HR and UHR		
Collimation mode	**S1 aperture**	**S2 aperture**	**Collimation mode**	**S1 aperture**	**S2 aperture**
Full open	8	8	**Full open**	8	8
Ultra high flux	4	2.5	**Ultra high flux**	4	2.5
Very high flux	2.5	1.4	**Very high flux**	2.5	1.4
High flux	1.5	0.9	**High flux**	1.6	0.9
High resolution	0.55	0.35	**High resolution**	0.8	0.5
Ultra high resolution	0.15	0.1	**Ultra high resolution**	0.3	0.15

g. Set your file save location using "Change" in the Image Location Panel. Use the pop up window to save file in a new folder under the data0/images folder under your name. You will also be able to use this window to name your scans. If you take multiple scans with the same name, there will be a number appended to the file name to help you distinguish scans.

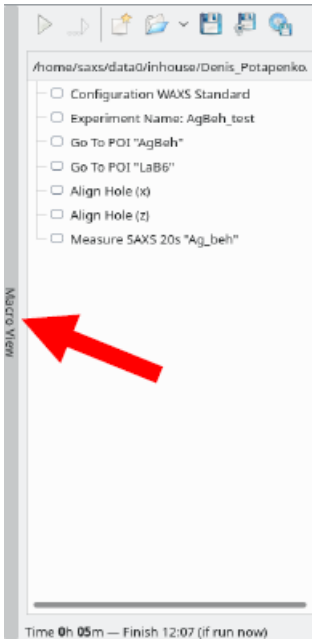
h. In the Scattering Measurement panel, you can set the exposure time of a scan next to the measure button. If "Absolute Intensity" is selected, the transmitted intensity is automatically measured and stored in the header of the .edf files and produces 1D profiles (.dat files) that can be used to compare the validity of different SDDs for your sample. If you want to remove the black line in your 2D images (this results from the tiling of the 2 modules of the detector), you can select "Line Eraser". This mode will approximately double data collection times. The "Virtual Detector" will take extra scans while moving the detector to different positions, with the intent of extending your Q-range to wider angles. The image shape and extended scan time depends on the virtual detector collected (see image below for a guide). You are now good to hit "Measure" and take your scan.



Virtual Detector Options

08. MACRO VIEW

a. You can set multiple samples on a stage to automatically run by selecting “Macro View”. When this view is open, click through XICC in the order of events that you want the program to perform. Instead of the command immediately performing, it will appear as a command line in the Macro panel. You can edit the script by right clicking on a command. When you are satisfied with the macro you have set up, click the play button and XICC will execute the commands in order. Progress through the script will be indicated by the oval bullets filling with color.

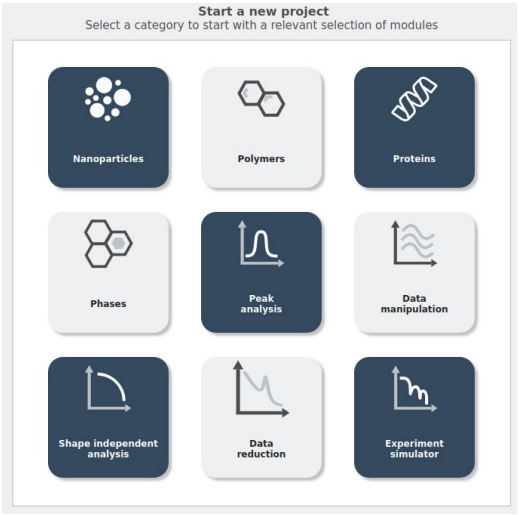


09. VIEWING YOUR DATA WITH XSACT

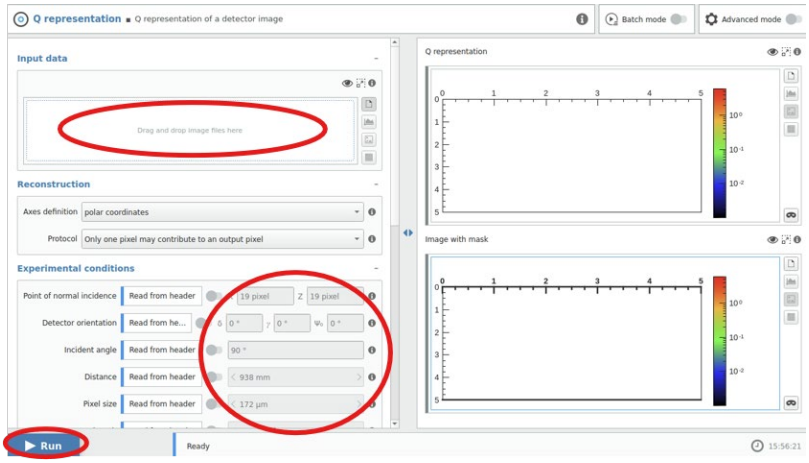
a. As your data is gathered, you can look at it using XSACT.

b. XSACT has 3 selections at the top: Home, Analyze, and View.

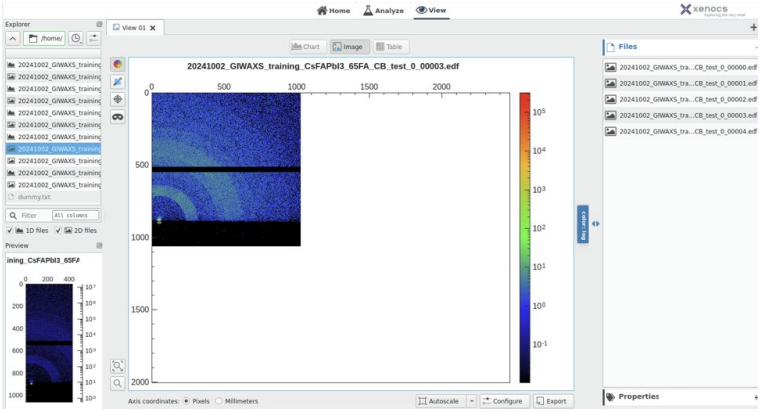
c. “Home” lets you choose different saxs applications (analysis for nanoparticles vs polymers) and will prepopulate your Analyze tabs with relevant analysis modules.



d. In the “Analyze” screen you can pick specific analysis modules. In each module tab, you can drag and drop relevant files into the “Input Data” boxes and then fine tune the chosen algorithm. When you click run, the algorithm runs on your data set and will populate the results panel.



e. You can use "View" to quickly evaluate your 2D data and plot 1D data. Simply double click to open a new window with that data, or drag and drop multiple data sets from the explorer panel and it will display onscreen. You may need to auto-scale your data, which you can do by right-clicking the image and selecting auto-scale. The View tab only lets you display either .edf data or .dat data, not both concurrently. Clear the selected data pane if you need to go from one file type to another.



f. To export any data file, image, or data table in XSACT, simply right click your selection and choose export. A window will open and provide you with options for file type and export location. You can also save your XSACT experiment file for future analyses (it is recommended to pick up all of your data at the end of each session as the Xeuss computer will be wiped periodically for performance). To collect your raw data, use the file manager (Dolphin) of the main computer to export all data you wish to keep onto a flash drive.

10. ENDING YOUR SESSION

a. Once you are at the end of your session, set the source from Full to standby mode using the Source panel. If you used WAXS mode, set the SDD back to SAXS mode. This will protect the detector from accidental bumping.

b. Vent the sample chamber, collect your samples, and return one of the original standard sample stages to the chamber. If you used an advanced stage, you must remove it and return the standard sample stage back to the chamber, making sure to turn off spec and the motion controller before any cables are unplugged or plugged in.

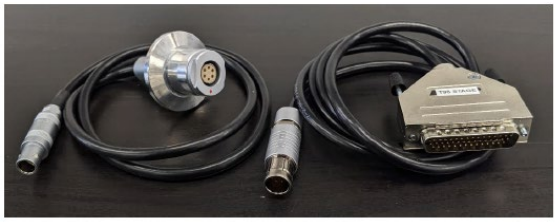
c. Follow the chamber evacuation procedure to return the chamber back down to 0.3 mbar. This is a good time to clean up your work space, analyze your graphs, and transfer your data to a thumb drive. Once the chamber is under vacuum again, you are free to use the FOM to sign out of your session.

11. LINKAM STAGE USE

The linkam heater stage uses liquid nitrogen for its active cooling. Make sure that you have LN2 on hand prior to use and setup. Make sure you are trained on the heater stage for your setup before use. Make sure chamber is vented (not under vacuum) before installation of all flanges and stage components.

a. Set up the heater stage specific flanges on the sample chamber. There are 3 to be connected: the LN2 tube that connects the stage to the LNP96 pump, the second LN2 connection to a dewar, and the cable that connects to the T96 controller. Make sure all screws are wrist tight and all O-rings are flush against their surfaces and aren't cracked.

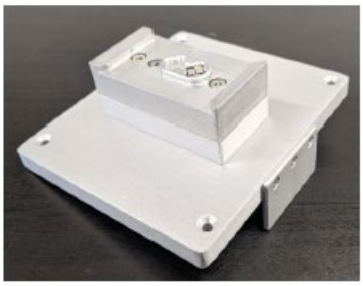
LN2 Tube



T96 controller cable with flange adapter

b. Select the sample stage mount for temperature controlled SAXS or temperature controlled GISAXS in the sample chamber. For GISAXS stage setup, follow the instructions in the next section, titled "GISAXS STAGE MOUNTING AND USE".

Transmission Mode



GISAXS MODE

c. Mount your sample onto the heater stage with the relevant adapter. If in transmission mode, place your sample in the center of the circular heating element and then attach one of the sample holders to the threaded holes to clamp down your sample (GISAXS mode uses a spacer attachment instead of a holder. You can place your sample onto the center of the spacer in the sample chamber after making all the needed connections.) For gels, use 2 kapton windows and an O-ring between the gel clamps to protect your gel from vacuum.

Top row, left to right: Lollipop holder, film clamp, capillary clamp, gel clamp

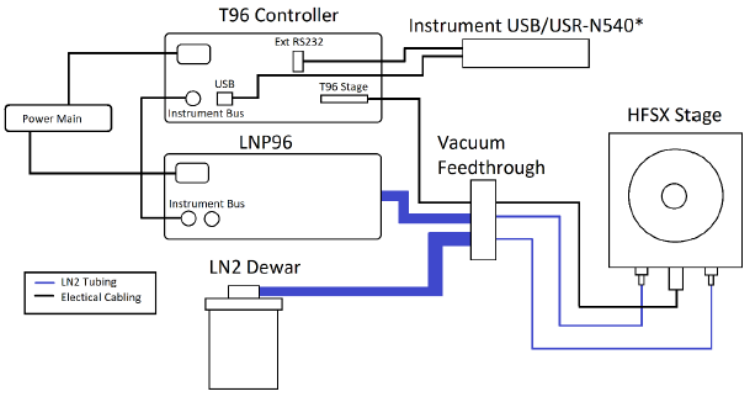
Bottom row, left to right: kapton window for gel clamp, O-rings for gel clamp, GISAXS spacer

d. Attach the heater stage to the relevant transmission/GISAXS adapter. Mount the whole stage in the sample chamber.

e. The Linkam stage has two controllers, the T96 controller (no white tubes) and the LNP96 controller (white tubes). The T96 controls the stage temperature and connects to the stage via cable. The LNP96 is the liquid nitrogen pump that cools the stage via the white tubes. Liquid nitrogen being used by the stage is supplied via the provided dewar. Attach the cables/tubing from these controllers and the dewar to the corresponding ports on the newly installed flanges. Attach the cables/tubing to the heater stage directly.

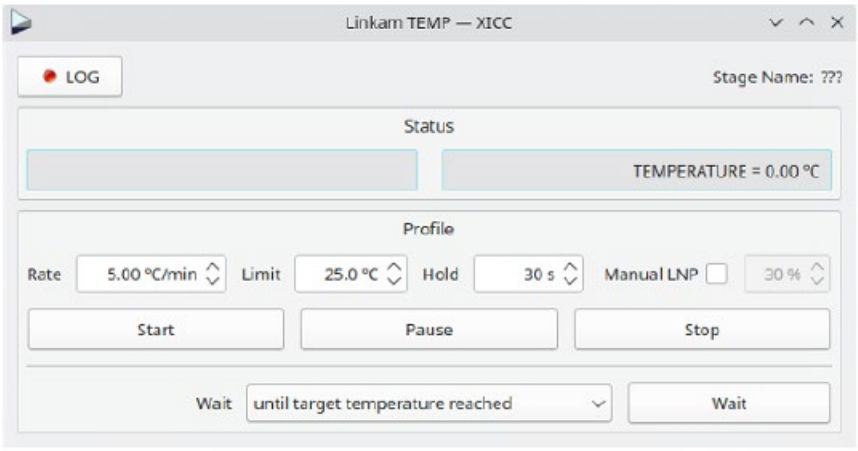
T96 LNP96 Dewar

f. Once all connections are made, turn on the T96 controller first. Wait a minute or two, then turn on the LNP96 pump. Start LC TEMP in X-Controller. Check that a connection to the controller and stage has been established in XICC.



g. When communication is enabled, fill the LN2 dewar. DO NOT USE ANY COOLANT OTHER THAN LN2. Other coolants will gum up the lines and lead to damage and may incur repair charges.

h. Use the Linkam TEMP tab (found under “Additional Controls”) to access the screen shown below. Use this screen to manually set temperatures, ramp rates, and hold times for the stage. This screen works with macro view if you need automated runs. Use the wait section to force XICC macros to wait until specific conditions are fulfilled. You may now take measurements as normal.



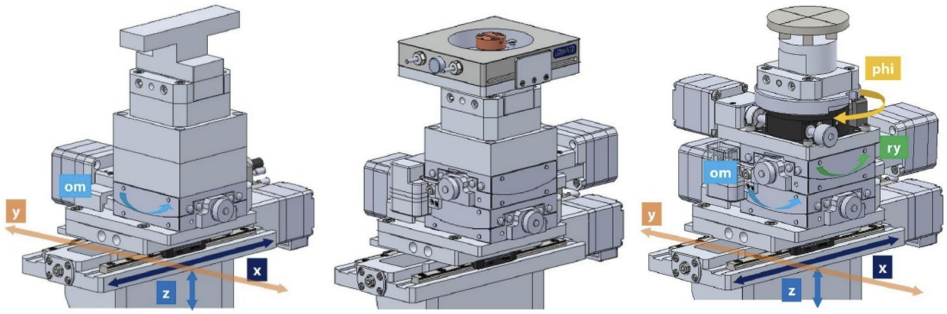
i. At the end of your session, turn off the Linkam controllers and disconnect all wires and tubing related to the set up. Reinstall the normal flanges and place all attachments in their respective boxes.

12. GISAXS STAGE MOUNTING AND USE

Installation of the GISAXS stage is more hands on and you need to be trained on it by the facility engineer before use. The following steps are in addition to the standard venting and evacuation steps of the standard SAXS stages. Advanced GISAXS measurements can accommodate samples+wafers with a thickness lower than 1-2mm.

a. After venting the sample chamber, turn off SPEC in Xeuss Controller by pressing "Stop" and then turn off the "Motion Controller" switch on the front panel of the Xeuss.

b. Set up the GISAXS stage in accordance with your experiment needs. Use Allen wrenches to interchange the basic and advanced GISAXS stages with the GISAXS mount plate. You can also install the Linkam Temperature Controlled stage using the standard GISAXS stage mount.

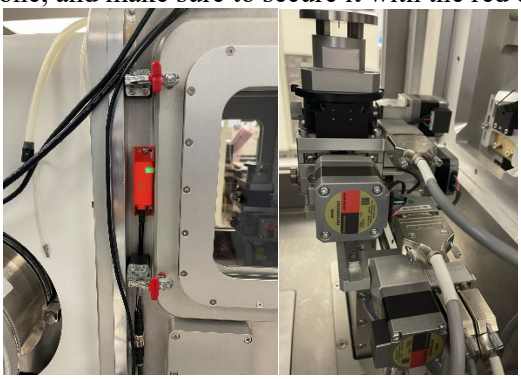


Standard GISAXS Linkam Attachment W/ GISAXS Advanced GISAXS

c. You are now free to mount the stage in the sample chamber. Use the Allen wrenches to unscrew the standard SAXS stage mount. Set the standard stage to the side, and then use the Allen wrenches to mount the GISAXS stage (make sure to line up the RFI chip on the stage with the RFI reader in the platform). Do not overtighten the screws.

d. Next, you will need to circle around to the back window of the Sample chamber and open the window by flipping up the red clamps. On this side, you will need to plug in the VGA cables in the chamber that correspond to the VGA ports on the stage (the heads are different sizes, so there is little chance of a mix-up). You will likely need to lightly tighten the VGA cables in with the short-stemmed flathead screwdriver if you can't get a good hold with your gloves. DO NOT OVERTIGHTEN THE CABLES, THIS CAN DAMAGE THE STAGE.

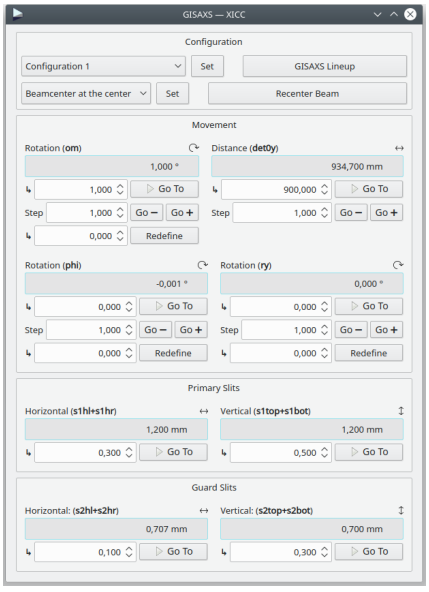
Make all cables are out of the path of the stage and detector motors. Close the window when done, and make sure to secure it with the red clamps.



e. You are now good to turn the motion controller and then SPEC back on.

f. Mount your samples onto the GISAXS stage. The advanced stage needs your sample to be in the center of the circle. If you are worried about your samples shifting during evacuation or alignment, you can use double-sided Kapton tape to secure your sample (though most samples are fine without tape). Evacuate the chamber and set the source to full-power.

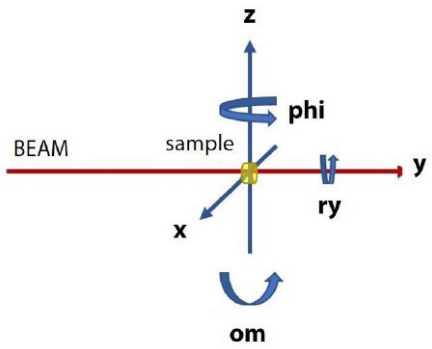
g. Along with standard stage controls, you can manipulate the GISAXS stage by selecting the "Additional Controls" tab at the top of XICC and selecting GISAXS. This will open a window that can control the GISAXS stage.



h. Choose "GISAXS Lineup". This will automatically align the surface of your sample with the beam. It uses plots generated from ROI counts vs different positions to determine where to best position the stage for a standard grazing incidence scan.

i. Choose a preferred Beamcenter positioning. This will determine where the beam spot shows up on your generated 2D image and will be accounted for in your q-transforms.

j. Once your sample is aligned and your beamcenter is in a preferred spot, you are good to set the GISAXS stage to an orientation that works for your scans. A diagram depicting what each variable corresponds to in relation to the beam is depicted below. Make sure to choose an om that is appropriate for your experiment. An om (or sometimes called alpha angle, or incidence angle) of less than 1 degree (usually around 0.1, 0.2, or 0.3) is typical for most GISAXS/GIWAXS tests. Angles higher than the critical angle of your material will start to interrogate the bulk of your sample instead of the surface.



k. Once your sample is lined up and oriented, you are good to proceed with the standard measurement and data acquisition procedures.

l. When finished with your experiment, make sure to uninstall the GISAXS stage and reinstall the standard SAXS stage. Remember to turn off SPEC in Xeuss Controller and the motion controller switch before unplugging any cables, and to turn them back on when uninstallation is completed.

13. JSP REUSABLE CAPILLARY STAGE

SAFETY NOTICE

The capillaries are intended to hold non-viscous, non-aggressive, non-corrosive fluids. Users should always be aware of actual capillary content and take proper precautions, accordingly.

The glass capillaries are fragile and break easily either from external touching and internal touching or scratching. Therefore, at always take care to avoid any physical contact with the glass.

If broken, the ultra-thin glass may easily cut skin and small pieces may enter the body and be taken up by internal fluid streams. Extreme care must therefore be taken in handling broken capillary glass, with local glass disposal practices strictly followed.

a. Replace one of the plain sample chamber flanges with the Julabo Feedthrough flange, making sure the O-ring is flush and not cracked and that all screws are wrist tight.

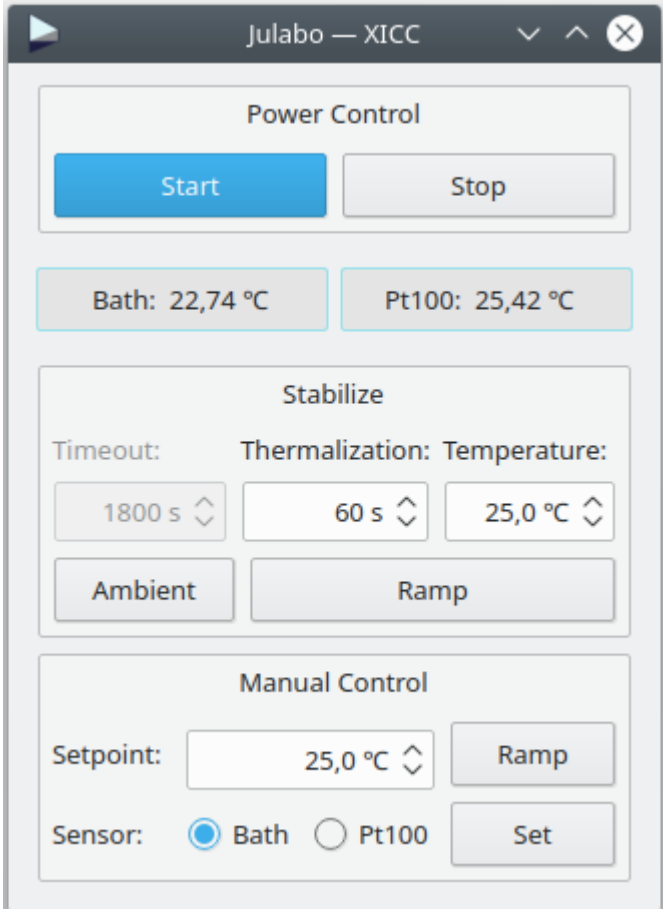
b. Connect the Julabo feedthrough flange to the Julabo chiller (which by default is connected to the Biocube setup). Make sure that the Julabo is turned on (by default it should always be on).

c. Install the JSP stage in the sample chamber. Connect the tubing to the Julabo feedthrough flange.

d. Connect the PT100 cable from the Julabo to the sample chamber. Then connect the PT100 probe to the inside sample chamber connection. Insert the PT100 probe into the JSP stage close to the sample positions where your samples will be.

e. Sample loading: Select the relevant capillary for SAXS or WAX measurements. Follow the visual guide for standard sample loading. Make sure o-rings are present between connections in the capillary. 50-100 uL of sample is recommended. If you have less sample than that, try pre/post filling the capillary with a non-miscible liquid (e.g. oil for water) to keep your sample in place during the measurement. If you are underfilling, the meniscus may wander during the measurement and ruin the sample data.

f. Load the capillary into the JSP stage in the correct orientation, then proceed to sample collection. In XICC, use the additional controls marked with “Julabo” to control the stage. Press start to begin temperature control. This screen can be used to control temperatures and ramp rates. This screen can be used when designing a macro for your experiment.



g. When your experiment is over, stop the julabo control in XICC. Disconnect all wires and tubing, and reinstall the standard plain flanges. Reconnect the julabo back to the biocube setup. Use relevant cleaning solution to rinse out the capillary of sample (do not use acetone or a sonicator to clean the capillaries).

14. HUMIDITY STAGE

Note: Use distilled water for the humidity controller. Sediment buildup in the lines will impact the stage's effectiveness.

a.

15. BIOCUBE AND PIPETTING ROBOT

a.