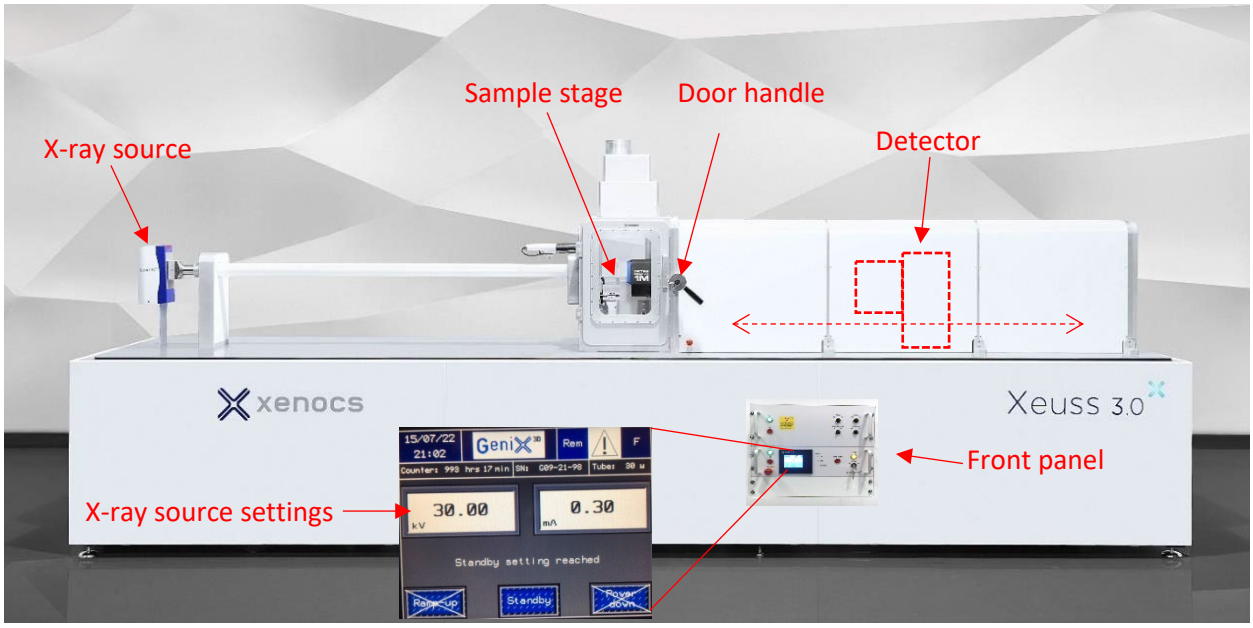
Xeuss Small Angle X-ray Scattering (SAXS)

1. Xeuss 3.0 Overview

Xeuss 3.0 is a versatile X-ray scattering system equipped with the GeniX 3D X-ray beam delivery system, which produces a monochromatic Cu K_a high intensity X-ray beam, and Dectris Eiger 2R 1M-pixel 2D detector. Variable detector positions allow a wide range of accessible q-values: from 3.4 $Å^{-1}$ (1.8 Å in d-space) in WAXS mode to ~0.0025 $Å^{-1}$ (2500 Å in d-space) in ESAXS mode. Wide selection of sample holders can be used to collect scattering data from solid and liquid samples, in transmission or grazing incidence (GISAXS) modes, at variable temperature if needed.



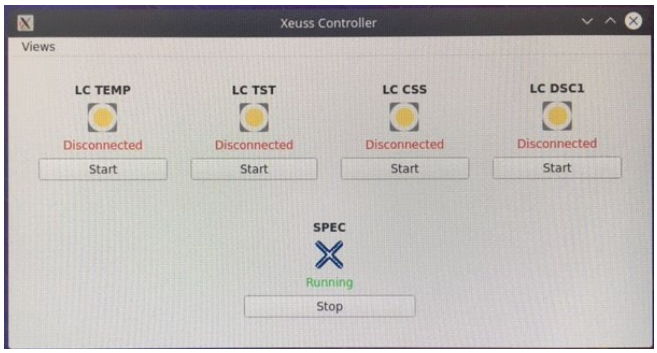
2. User Start Up

Normally, Raycam, X-controller, and XICC will be already running on the control computer. In case all software is closed, follow the instructions in the next paragraph to start the control software by using the icons on the desktop sidebar in the upper left corner of the screen (see Figure below).

Raycam cameras	-	Live camera views
XSACT	-	Data analysis software
XICC	-	Experiment control software
X-controller	-	Communication server

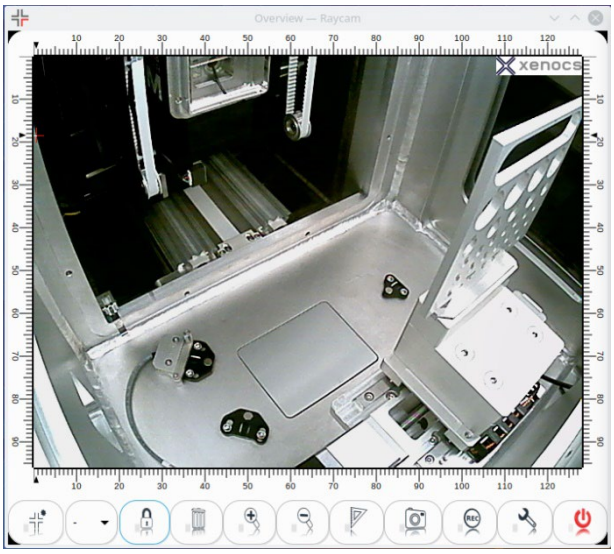
Begin by clicking on the "**X controller**" icon. The main program for Xeuss 3.0 control starts: the detector will automatically start up in Live View. A minimal operation will always include source controller (Genix 3D CU Com.), detector server (XDetector), and 'SPEC'. The first and the second will start automatically but SPEC needs to be started manually by pressing 'SPEC' button, which will bring up the underlying program for coordination of system resources. If successful, you will see 'Running' in green letters under the SPEC icon in the Xeuss Controller. A black-and-white 'Konsole' window will also appear, which can be used for entering direct command lines by advanced users.

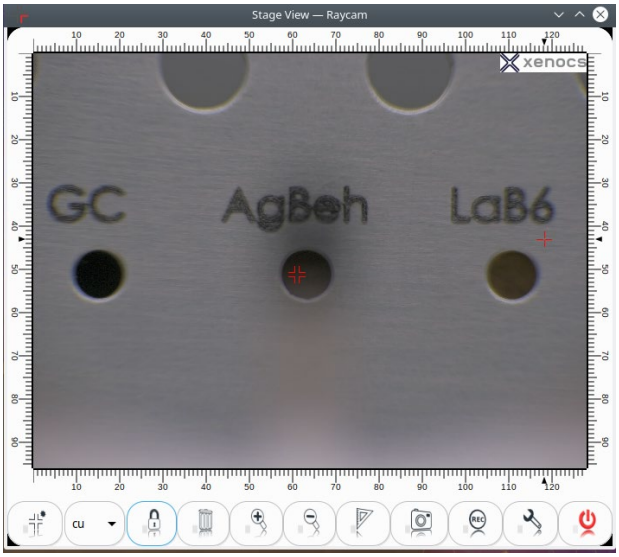
By pressing the ‘Views’ menu in the Xeuss Controller window, you can access controls of additional communication programs. For the use of Linkam stage, for example, you would need to open Linkam View and start additional drives. Below are examples of Xeuss Controller views



3. Raycam Cameras

There are two cameras in the system: Raycam overview window and Raycam stage view window. The two windows are opened by clicking the ‘Raycam’ icon located on the left side desktop taskbar. The overview camera shows the sample chamber, which displays the positioning of the sample stage, and the detector. The stage view shows an in-line view as seen from the incident x-ray side. When correctly calibrated, the red cross at the center of this window shows where the beam impinges on the sample.



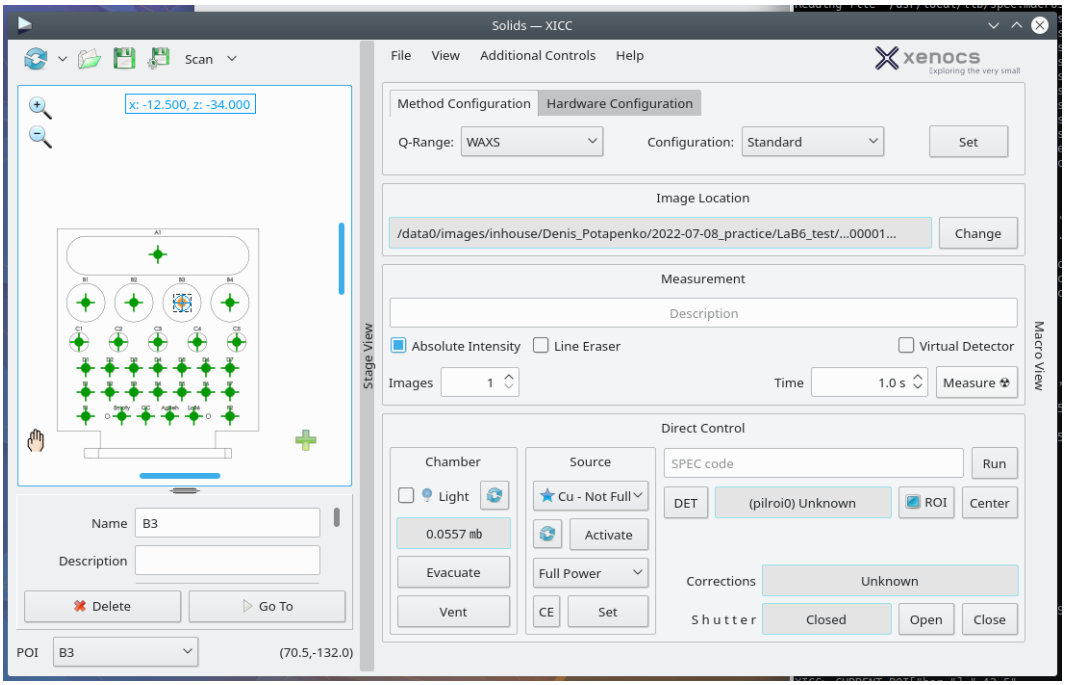


4. The XICC Control Software

Data collection is controlled by using the XICC panel. For the full description one should look at the Xenocs Xeuss 3 manual. There are several sample stages/holders available. The system recognizes these thanks to the RFI chip in the base plate. When sample holders are changed, push the ‘refresh’ button. When the stage is recognized a new graphic depiction of the sample holder will appear in the left section of the window. Click on one of the ‘holes’ or ‘capillaries’ and click ‘Go To’: that ‘hole’ will be placed in the X-ray beam.

In general, the red cross on the sample stage camera screen (above, right) indicates the beam position. However, this is a calculated value and in time might have to be recalibrated. For precise positioning select ‘Align Hole’ or ‘Align Capillary’ from ‘Scan’ pull down menu.

XICC window:



5. Venting/Evacuating the Chamber

Normally, the chamber is under vacuum in standby mode. In order to change a sample holder, and/or load a sample, you first need to vent the chamber by pressing 'Vent' button. Before venting the chamber be sure to check that the door handle is in the 'free position'. This door handle takes care that the door acts as the over pressure relief valve when venting the chamber. Please, refer to the Appendix at the end of this document for the list of available sample holders.

Once the sample cell holder is mounted, the vacuum chamber should be evacuated. Close the vacuum door, lock it with the handle, and press 'Evacuate' button in XICC. Check that the door handle drops to the 'free position' after some seconds of pumping. Open capillaries with liquids/wet samples/ closed cells with liquids or air that may burst in vacuum should not be placed in vacuum chamber. Once the chamber pressure is below 1 mbar, this takes ~450-480 seconds, the detector will turn back on.

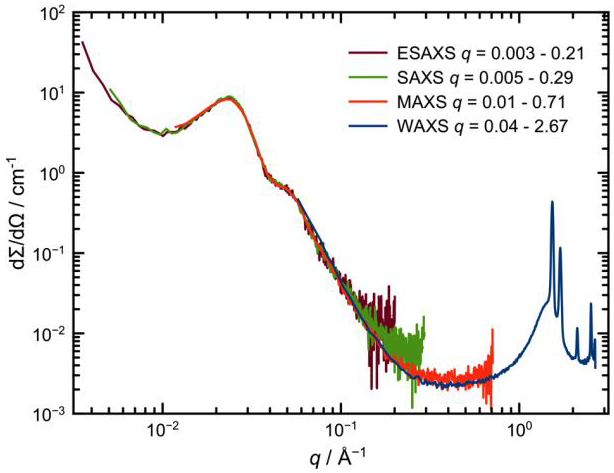
6. Source Power

In standby mode the X-ray source is running at **30 kV** and **0.3 mA**. For data acquisition, select 'Full Power' in the dropdown menu in 'Source' box in XICC window, and press 'Set'. The source will be powered to **50 kV** and **0.6 mA**. At the end of your experimental session bring the source back to 'Standby' mode.

7. Method Configuration

The sample-detector distance is set by the 'Q-Range' box. There are 4 pre-set detector positions designated WAXS, MAXS, SAXS, and ESAXS. The beam diameter, controlled by variable slits, is selected in 'Configuration' box from 'High Intensity', 'Standard', and 'High Resolution' presets (1.4, 0.7, and 0.3 mm respectively). After choosing the settings, you will need to push the 'Set' button. The corresponding q-ranges and approximate X-ray flux values are given in the plot and the table below.

Effect of different instrument configurations on photon statistics:



Measurement configurations for a Xeuss 3.0 with Eiger 1M detector in single shot mode:

Mode	Config	SD (mm)	*q*-range (Å^{-1})	*d*-space (Å)	I (Mph/s)
WAXS	Hi Int	55	~0.15 – 3.4	1.8 – 42	178
	Std	55	~0.11 – 3.4	1,8 – 57	114
	Hi Res	55	~0.08 – 3.4.	1.8 – 78	64
MAXS	Hi Int	300	~0.022 – 0.9	7 – 280	114
	Std	300	~0.017 – 0.9	7 – 360	64
	Hi Res	300	~0.014 – 0.9	7 – 450	9.3
SAXS	Hi Int	900	~0.008 – 0.25	25 – 780	64
	Std	900	~0.055 – 0.25	25 – 1150	9.3
	Hi Res	900	~0.035 – 0.25	25 – 1780	1.2
ESAXS	Hi Int	1750	~0.0055 – 0.13	47 – 1150	64
	Std	1750	~0.0035 – 0.13	47 – 1780	9.3
	Hi Res	1750	~0.0025 – 0.13	47 – 2500	1.2

As the table shows, the intensity of the direct beam decreases when the detector distance is increased. This is because longer distances require higher resolution, and as a result you will probably need to plan for longer measurement times when using longer distances. As an example, for a typical plot, if WAXS is measured for 60 seconds, MAXS would be measured for 120, SAXS for 240, and ESAXS for 360 seconds, for consistent data statistics. The measurement times should be adjusted in accordance with the choice of configuration as well.

8. Location of data files

Set the location and the filename for the data files by pressing ‘Change’ button in the ‘Image location’ panel. For each session you may create a new folder. The data are located at **/data0/images/inhouse**. Create your own folder, for instance **/data0/images/inhouse/PI_name/Your_name**. The box at the bottom of the pop-up window indicates a subfolder and also the default name for the file. Please note that the “Description” in the panel below is a sample attribute that shows up as comment in the header of the data file.

9. Measurement options

If the ‘Absolute Intensity’ has been selected, the transmitted intensity is automatically measured and then used in the automatic production of the 1D profiles.

The Eiger detector is a tiling of two individual modules that results in a black horizontal line in 2D images. For isotropic scatterers, where the intensity will be radially integrated, this poses no problem.

But if, for better visual presentation, you do not want the black line in the image, you can use the ‘Line Eraser’ mode. In this mode two patterns are taken at two shifted detector positions to account for the gap. These two patterns will be used to create a single composite image without the gap in between. This mode will increase the data collection time by a factor of 2.1.

The ‘Virtual Detector’ mode involves lateral translations of the detector with the purpose of extending the q-range to the wider angles. The pattern that will be produced is the composite of up to 3 x 3 = 9 individual exposures (depending on choice of virtual detector) that will go up to 18 if line eraser is also selected. The data collection time is proportionally increased. Unless there is a strong reason, it is only useful for data in WAXS mode.

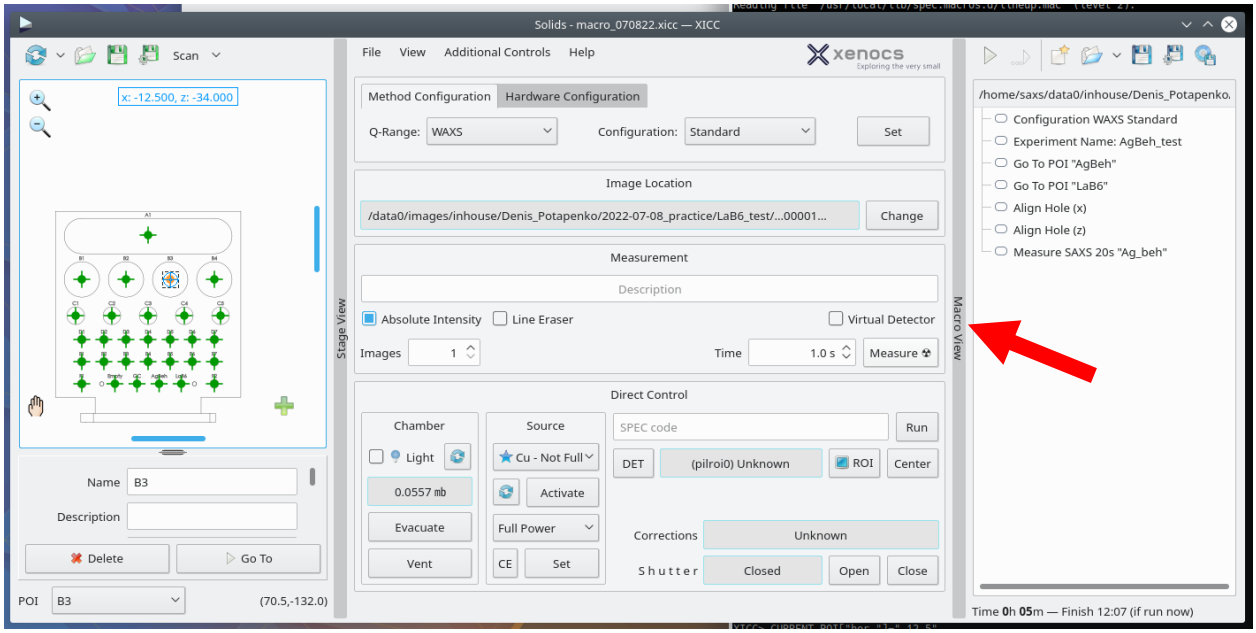
10. Data Acquisition

In ‘Time’ box the user indicates the exposure time for a single frame measurement, i.e., in the case of ‘line eraser’ or ‘virtual detector’ this value will be multiplied by the number of detector positions that is required to obtain the final data set plus some overhead time. To start the measurement, press ‘Measure’ button. A separate window will appear showing the progress of the experiment.

11. Creating Scripts

To create a series of commands in which several samples are measured, aligned, etc. the ‘Macro View’ (below, marked with the red arrow) should be clicked. The side panel will open. Then any command selected in XICC window will not be immediately performed but instead will appear as a command line in the script in Macro View panel. Estimated time for the whole script will show at the bottom of the Macro panel. Start the script by clicking ‘play’ button above. The progress of the script will be indicated by color filling the oval bullets next to the command lines.

Macro View example:



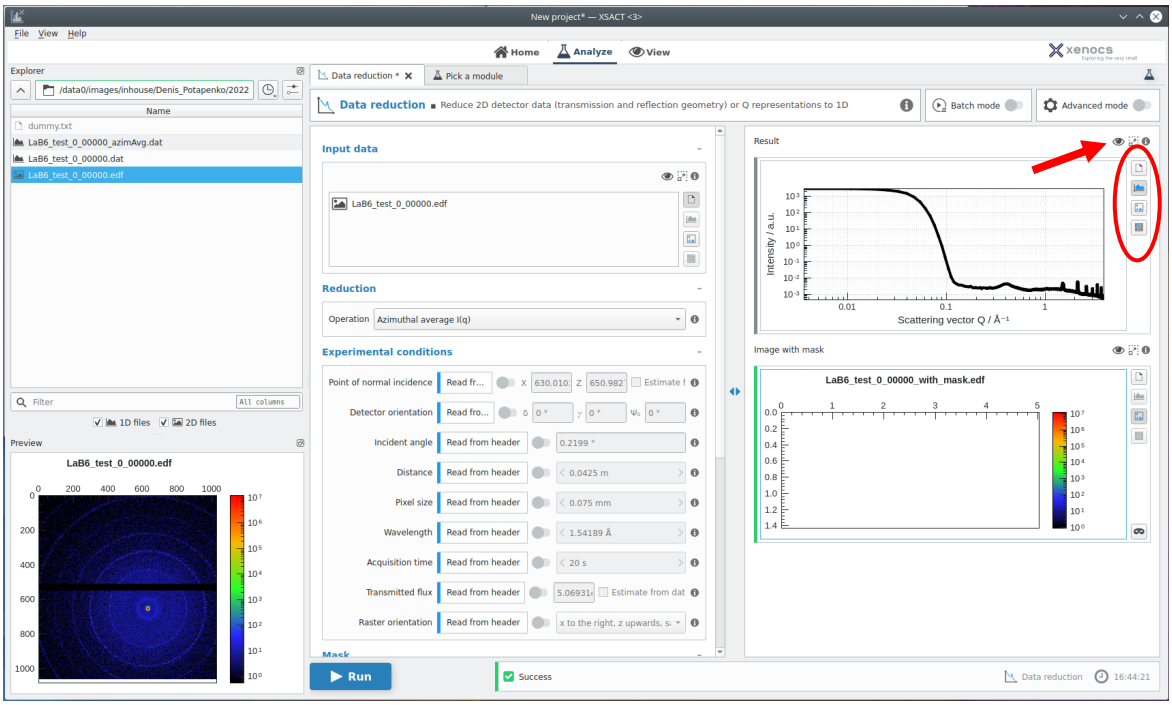
You can save a new script or open an existing one using options on top of the macro panel marked. Each of the macros in the scripts can be individually edited as well as looped. Please refer to XICC manual for more detail in this regard.

12. XSACT Analysis Software

To process and analyze X-ray scattering data, you can use XSACT software. At the very top you will have three screen selections: 'Home', "Analyze', and 'View'. In '**Home**' screen on the left panel you can choose a specific SAXS application, i. e. "Nanoparticles', 'Polymers', etc. and the appropriate data analysis modules will appear as new tabs in the "Analysis' screen. Also, you can open an existing project that you have saved earlier. After performing your analysis, you can save the combination of all open images, graphs, and results as a new project.

In the '**Analysis**' screen you always have 'Pick a module' tab where you can activate one of the data analysis modules from four groups: 'Data analysis', '1D data operations', 'Direct modeling', and 'Image operations'. A new tab will appear with controls similar to shown in the image below (this example is 'Data reduction' module). Here at the left panel you can browse through your data files and preview both 2D images and 1D data in the section below. Once you have selected the file for processing, you can drag-and-drop the file(s) into 'Input data' box. Each algorithm can be fine-tuned using controls below the 'Input data' box. 'Batch mode' allows simultaneous processing of many datasets according to the same algorithm and 'Advanced mode' opens extra controls in the 'Experimental condition' section. Press 'Run' button at the bottom to perform the calculations. The results will appear in the right panel.

XSACT window example:



Using the buttons on the right (circled in the red oval above), each result item can be switched between four representations, if applicable: file, graph, image, and table. Then the result can be saved, respectively, as *.dat file, and exported as *.pdf or *.png image/graph, or as *.csv data table by right-clicking on the result window and selecting 'save' or 'export'. If you press an eye button (marked with the red arrow above) then the selected result window will appear in '**View**' screen for further inspection and comparison. For specifics about mathematics used in each of the analysis modules please refer to XSACT manual.

13. Finishing experimental session.

At the end of your experiments follow these steps: set the X-ray source power back to standby level (see section 6.), vent the chamber (see section 5.), remove your samples, clean the sample holder, place it back. In case you used one of the advanced sample stages and you are not planning to use it again in the near future – please, install back one of the standard sample holders. Evacuate the chamber.

14. Appendix. Sample Holders and Sample Stages.

Four standard sample holders are provided with Xeuss 3.0 system.

1. The 15 slots powder sample holder is made of 3 racks of 5 holes each, all with diameter 2.5 mm. Kapton windows (12.5 µm thickness) and O-rings are also provided to prepare powder samples.

2. The various size multi-slots solid sample holder is a plate with 16 holes of diameter 5 mm, 5 holes of diameter 10 mm, 4 holes of diameter 20 mm and a wide hole to hold very large samples and enable measurements at various positions on the sample (e.g. for sample mapping).

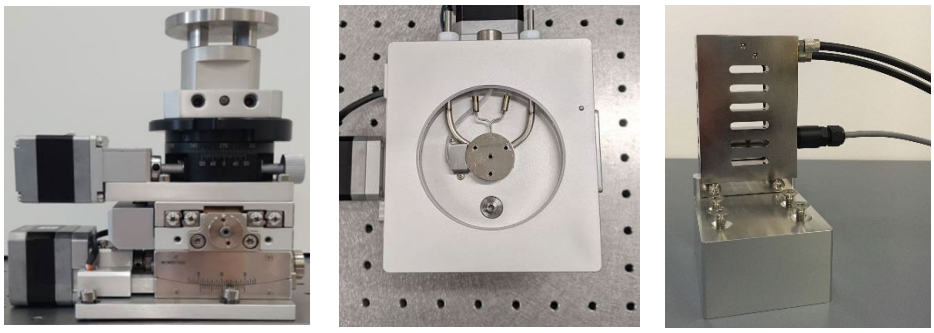
3. The multi-capillary sample holder can hold up to 27 capillary tubes. The holder has been designed to accept capillaries with an external diameter of 2 mm in the holes. Larger capillaries can be mounted on the external face of the sample holder (then the sample – detector distance has to be adjusted).

4. The gel and powder sample holder can hold eight capsules. The capsules have been designed to maintain the samples isolated from the vacuum so that the gels do not evaporate. The holder can be assembled in two configurations giving sample thicknesses of 0.5 mm or 1 mm by selecting a proper spacer. Three O-rings and two 12.5 µm thick Kapton windows isolate the samples from the vacuum.

Each sample holder is equipped with calibration sample slots (four bottom holes of 3 mm diameter).

Standard sample holders for: powders, solids, capillaries, and gel/powder samples (left to right)

In addition, we have a Grazing Incidence (GISAXS) stage, variable-temperature Linkam HFSX stage for solid samples, and variable-temperature JSP capillary stage for liquid/gel samples.



Advanced stages: GISAXS sample stage, Linkam stage, and JSP capillary stage (from left to right)

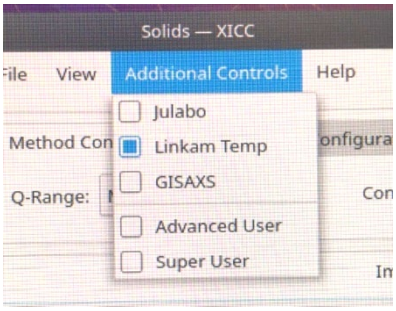
There are 3 options for the top sample holding section of GISAXS stage. The standard section consists just of a solid block. In this configuration only four degrees of freedom are available (x, y, z, and incidence angle ω). The solid block can be replaced by the Linkam HFSX stage, thus offering the

temperature control capability. Advanced GISAXS stage configuration has two additional drives for the stage rocking angle (ry) and stage rotation (φ).

The Linkam HFSX stage can be used in transmission mode as well as a part of GISAXS stage. It allows heating and liquid nitrogen cooling in the -150 to 350 ^{o}C range. The stage is connected by electrical and fluid feedthroughs to two external controllers and a LN_2 Dewar. The temperature of Linkam stage can be programmed using XICC software.

JSP capillary stage allows temperature-controlled SAXS measurements in transmission mode with liquid or gel samples that are loaded into a reusable sealed glass capillary, equipped with a thermo-couple for precise temperature reading. JSP stage is connected to a dedicated Julabo cooling/heating circulator providing computer-controlled temperature variability from -30 to 200 ^{o}C.

Installation of advanced stages require making electrical and fluid connections with external devices. Besides venting the system, mounting the stage, and evacuating the system (as with standard sample holders), one needs to take additional steps. It includes stopping X-controller by pressing 'Stop' in SPEC window and turning off 'Motion controller' switch on the front panel before mounting the stage. After the stage has been installed and all the required cables and tubes have been connected, the 'Motion controller' switch should be turned on again. Then one needs to start both SPEC and the dedicated server for the selected stage in X-Controller window (see the example in section 2). Then in XICC window one needs to open 'Additional Controls' and enable the additional control window for that stage:



More details on the specifics of the non-standard sample stages have been provided in the full Xeuss 3.0 manual, available as PDF file on the Xeuss computer.

Emergency Information:

Medical Emergencies: Contact 911 and Public Safety (609) 258-1000
Room / facility emergencies: Contact Public Safety (609) 258-1000
Issues related to the instrument:

1. Contact IAC Staff.
2. If unsure, leave system as is.
3. Try to turn off X-rays and shut down system.

Audible/Siren Emergency Alerts:

Follow previous steps 1, 2 and leave the building.

Emergency Contact Information:

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