



XEUSS STANDARD SAXS STAGES S.O.P

Instrument:

Xeuss 3.0 SAXS/WAXS/GISAXS/USAXS/Imaging Beamline

Software:

X-CONTROLLER, RAYCAM, XICC, XSACT

XSACT is available for purchase from Xenocs

Purpose:

For the elucidation of atomic and molecular structures using scattering ranges from ultra-small (USAXS) to wide (WAXS) x-ray scattering. Suitable for interrogating solid and powder samples at variable temperatures and in transmission and grazing incidence modes. Liquid samples can be tested with select attachments.

To Begin:

➤ **Know the specifications of the instrument.**

Specifications	ViewSizer 3000
Detector	Eiger 2R 1M
Source	GeniX μ -source, Cu K α source, 1.54184 Å
Auxillary Source	Cu K α source, 1.54184 Å
SDD Ranges	42.5 mm to 900 mm
Sample Types	Solid, Powders, Gels, Thin Films, Liquids (with JSP and Biocube stages only)
Temperature Range	-150°C to 350°C with Linkam Stage
Heating Rate	30°C /min
Cooling Rate	30°C /min when above -100°C

➤ **System Start-up**

- Log In: Log into your session using FOM to turn on the control computer monitors. Log in to the computer using the password listed on the FOM.
- Check Control Software: If the control softwares are closed, open “X-controller,” “Raycam cameras,” and “XICC.”
- In XICC, Ensure the controllers for Genix 3D CU, Com, XDetector, and SPEC are turned on.
- For the Cameras, use "Overview" for positioning the stage and detector, and "Stage View" for aligning samples with the x-ray beam.

➤ **Sample Loading**



- Solids stage: Attach the sample with Kapton tape to the specimen window.
- Powders stage: Load powder into a washer with Kapton windows and mount it on the rack.
- Capillary stage: Load 2mm capillaries into the capillary stage. Load larger capillaries onto the front of the stage with Kapton tap (need to recalibrate SDD for these). Ensure capillaries can withstand vacuum pressures before evacuating chamber.
- Gels/Powders stage: Use capsules with 3O-rings, 2 Kapton windows, 1 spacer and 1 cap. Load your sample between the 2 kapton windows.
- **Stage Loading**
 - Vent Chamber: Use XICC to vent the sample chamber.
 - Load Stage: If using a standard SAXS stage, align the base of the stage with the magnetic insert on the standard SAXS stage mount to install. If using an advanced stage, consult the Xeuss User Manual for more details on setup and installation.
 - Evacuate Chamber: After loading the stage, close the chamber and select "Evacuate" in XICC. The process will take ~10 minutes to reach ~0.03 mbar.
- **Setting Up an Acquisition**
 - Power Up Source: After evacuation, set the source from standby to full power.
 - Stage Calibration: Use the refresh button to identify the stage and reset the stage position to (0,0). Manual calibration to (0,0) may be necessary.
 - Align Sample: Use the "Go To" function for sample positioning on specific points and "Scan/Align" to fine-tune beam alignment.
 - Set Configuration: Choose the sample-to-detector distance (SDD) and slit size based on your required q-range.
 - File Location: Set your file save location to your data folder and name the scans.
 - Measure: Set exposure time and hit "Measure" to take the scan. Select advanced scan options as desired.
- **Viewing Data (XSACT)**
 - Home: Choose the relevant analysis module based on the sample type.
 - Analyze: Drag and drop data for processing and analysis.
 - View: Use "View" for quick evaluation of 2D and 1D data. Right-click to auto-scale if necessary.
 - Export Data: Right-click images, graphs and data sets to export files. Use a flash drive to take your data.
- **Ending the Session**
 - Standby Mode: Set the source back to standby mode.
 - Return Standard Stage: Vent the chamber, remove the sample, and replace it with a standard SAXS stage. Uninstall any advanced stages used.
 - Chamber Evacuation: Follow the evacuation procedure and ensure the chamber returns to vacuum before logging off of the FOM.