

About the SAXS/WAXS Diffractometer

Small angle X-ray scattering (SAXS) is a premier technique for determining structural characteristics on the nanometer scale. The utilization of SAXS for high throughput sample characterization in a laboratory environment however is typically inhibited by a variety of factors, such as low x-ray brilliance for in-house setups, lack of synchrotron availability, and general and complexity in data analysis, among other factors. The BioPACIFIC Materials Innovation Platform (www.biopacificmip.org) attempts to overcome this bottleneck through the development of a high brilliance, high efficiency laboratory based SAXS-WAXS beamline.

The BioPACIFIC SAXS/WAXS instrument contains both the most advanced in-house x-ray source—a 70 keV liquid metal jet—as well as a large, 4 mega-pixel hybrid photon counting detector. This, in conjunction with in-house developed improvements to scatterless slit beam collimation, allows for rapid characterization of samples at length scales below $\sim 300\text{nm}$. SAXS experiments on bio-derived polymers have demonstrated a near 100-fold increase in performance compared to conventional in-house SAXS setups. With a flux comparable to a 2nd generation synchrotron beamline, the BioPACIFIC SAXS machine alleviates the need of obtaining synchrotron time for most groups.

The diffractometer is capable of running samples in both transmission and grazing incidence geometries as well as various in situ environments such as temperature, microfluidics, and stop flow.

To optimize the workflow, a custom-developed graphical user interface (GUI) has been developed to provide rapid measurement turn around, boasting automated sample alignment, real-time viewing of 2D SAXS data alongside on the fly 1D data reduction, as well as AI driven automated data analysis.

How to run a sample

Step 1: Load samples into the desired sample holder and slide the sample holder onto the base of magnetic base of the sample stage.



Step 2: Close the hutch and secure it by fastening the top and bottom clamps of the SAXS hutch.



Step 3: On the SAXS computer check to see that the GUI is open. If not, or if the computer has to be

restarted open the file named "TO REOPEN GUI" located on the computer's desktop. Follow the directions on that text file, then proceed to step 4 when the GUI is operational.

Step 4: Check to see that the system at Full Power if at Low Power Mode, click on the switch to power up the source. If you are the first user of the day, or if you changed the beam size, click on the 'align optics' button. The optics alignment step takes around 4 minutes to complete.



Step 5: Go to the 'Manual Data Collection Tab' and click within the live camera feed pane to move the sample stage. The sample stage will move so that the x-ray beam will hit where you clicked. Thus click where on the sample you would like to run the experiment. At the end of this step the red dot on the camera feed pane should overlap with your sample.



Step 6: If your sample is smaller than 2 mm, you will likely need to use the x-ray beam to accurately align your sample. To do so, fill in the necessary boxes in the 'Scan Parameters' section and click on the 'Begin Scan' button. If you don't know what to put in for the start and end values, -2 and 2 are generally good guesses.



Step 7: When the scan is complete look at the updated graph in the middle of the GUI and click on the point where your sample is. For capillary samples, you will want to click on the center of the "valley" (see image above).

Step 8: Fill in the desired scan range (q_{min} and q_{max}), exposure time, and file name for the experiment. Note: To avoid errors, keep the length of the file name below 28 characters (you can change the file name after it is done running).



Step 8b: If you are running a temperature experiment, drop down the temperature experiment panel, and click on the switch. Fill in the targeted temperature for the experiment, and how long to for the computer to wait before running the exposure upon reaching the targeted temperature.

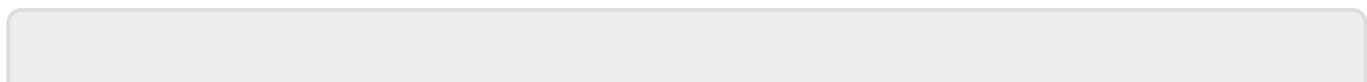


Step 8c: If you are running a sample in the GI geometry, drop down the grazing incidence experiment panel, and click on the switch. In the 'Omega' box, fill in the motor position for omega at which the sample is parallel to the beam. In the 'Omega Offset' box, enter the desired omega offset.



Step 8: When all relevant information for the experiment have been entered click on the 'Accept Alignment and Experimental Parameters' button to add it to the queue.

Step 9: Repeat Steps 5-8 for all samples that you would like to run. Once complete click on the 'Run Samples' button run all of the experiments in the queue.



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