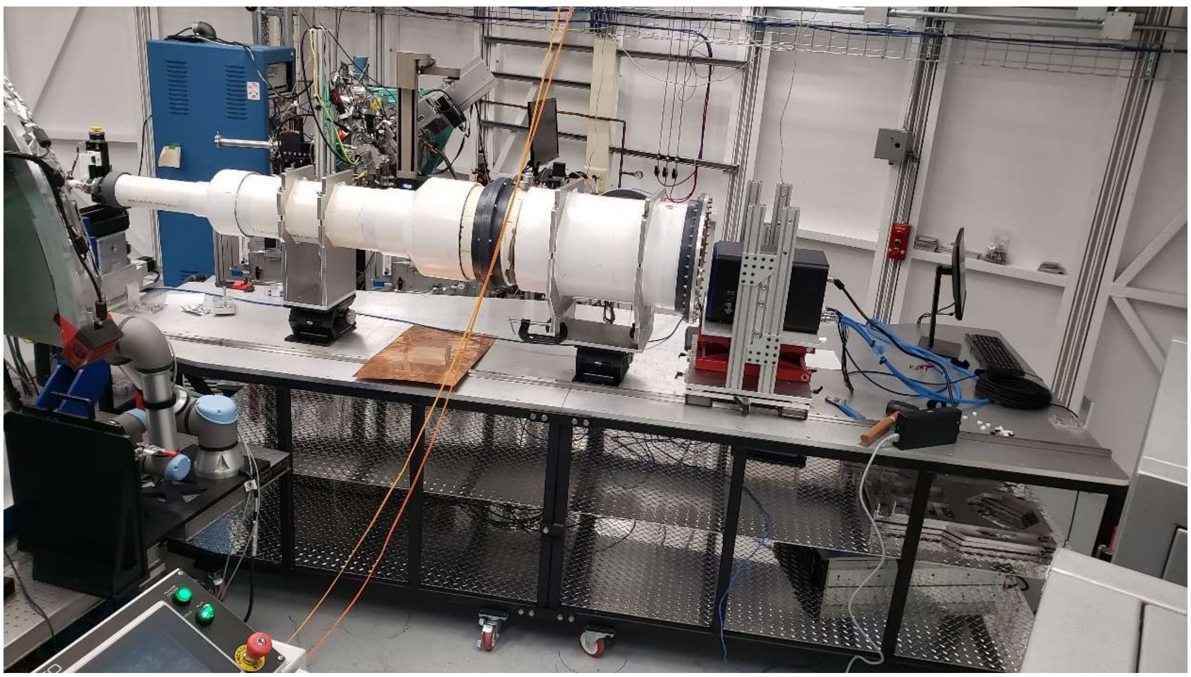
Small and Wide Angle X-ray Scattering

(SAXS/WAXS) Endstation

Standard Operating Procedures



Adam Leontowich, Karim Louca
December 10, 2024
Revision 1

Brockhouse Sector (BXDS) Contact List

Note: First dial “9” to access an *external* line from a CLS phone. For CLS internal numbers, you only need to dial the 4-digit extension number.

Serious emergency (Fire/Ambulance):	**911**	
U of S Security:	9-306-966-5555	

Beamline and CLS staff:	**Extension**	**Personal cell**
Beatriz Moreno (beamline responsible)	3868	306-241-1999
Adam Leontowich	3555	306-850-0408
Graham King	3760	639-998-1886
Narayana Appathurai	3648	306-514-1384
Karim Louca	3583	226-504-1169
Al Rahemtulla	3530	519-993-9137
Joel Reid	3854	
Garth Steel (controls lead)	3730	
Deborah Nguyen (mech. eng. lead)	3656	
Brian Schneider (elec. eng. lead)	3841	

Call the beamlines:

Wiggler Low Energy beamline (WLE, SOE-1)	657-3821
Wiggler High Energy beamline (WHE, SOE-2)	657-3830
In-Vacuum Undulator beamline (IVU, SOE-3)	657-3832

Other important contacts:

Floor coordinator	657-3639
Control room	657-3570
User services office (USO)	657-3700
Health and safety (HSE)	657-3663
Front desk	657-3500

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Introduction

Some expectations and responsibilities for staff and users

Users are responsible for:

- Requesting the SAXS/WAXS endstation in their proposals.
- Clarifying the photon energy required, in advance of the beamtime. Choices are 9.8 keV with Si(111) or 18.8 keV with Si(311). These are the only options.
- Their samples, including preparation, proper labelling, mounting them on the endstation, storage, return and/or disposal. *We highly recommended discussing sample mounting with beamline staff before arriving for beamtime.*
- Operating the data collection software SPEC during their experiments.
- Leaving a beamline unattended note if they leave the user area for more than 15 minutes.
- Cleaning up the area after the beamtime has ended.
- Reporting any perceived issues to beamline staff, before attempting to fix them.

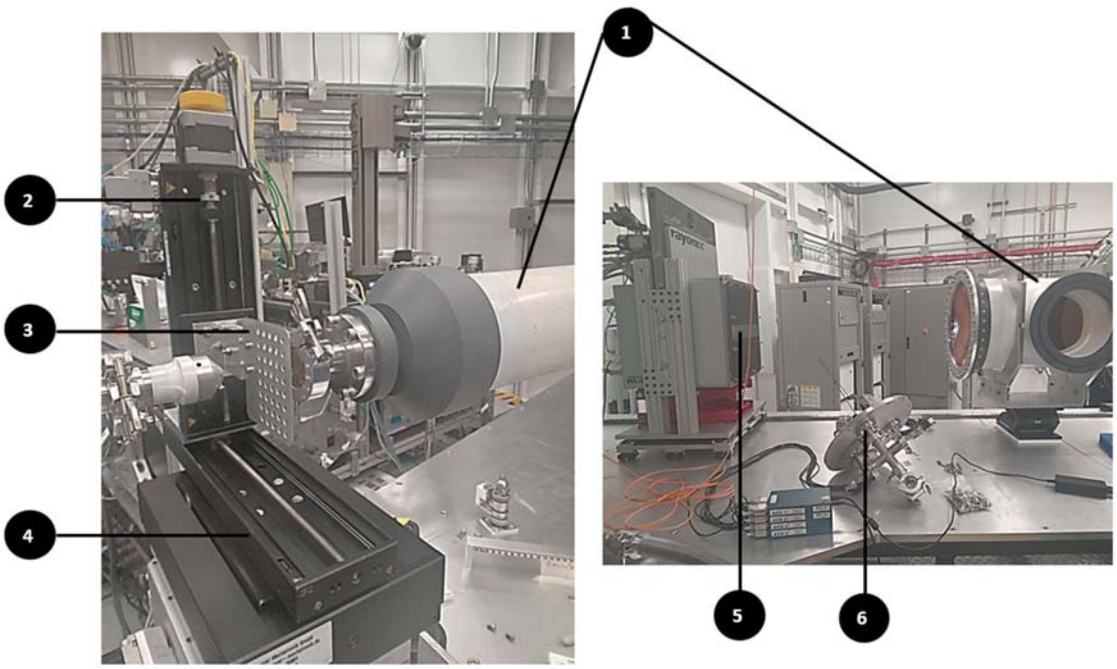
BXDS staff are responsible for:

- Instructing the users on safe operation of the Sector (BSO training), endstation and software.
- Assisting with signing onto the experiment permit at the start of the run.
- Setting up, aligning and calibrating the beamline and endstation to enable the users' experiments.
- Prompt troubleshooting of any issues reported by users.
- Moving the users' data over to CLS long term data storage, after the run is complete.
- Updating and maintaining the online logbook in Confluence.
- Periodic maintenance and upgrades of the Huber endstation to support safe, trouble free operation.

Beamline staff are here to help – please ask us questions! Tell us your comments and suggestions!

The SAXS/WAXS Endstation and Subsystems

First SAXS/WAXS data were collected at WLE in Fall 2019. The major equipment pieces were the Huber diffractometer for sample positioning, a donated 1.9 m steel flight tube and beamstop from HXMA, and a donated Rayonix detector from CMCF. The setup has evolved over time into its own dedicated endstation.



1. SAXS/WAXS Vacuum Tube
2. Vertical Motor (saxsz)
3. Sample Holder
4. Horizontal Motor (saxsx)
5. Rayonix MX300 Detector
6. Beamstop

Control System

The SAXS/WAXS endstation and the beamline are controlled using programs on the CLS desktop computer OPI1603-002, which runs a version of Scientific Linux. Upon starting or restarting the computer, log in with the following details:

User name: Password:

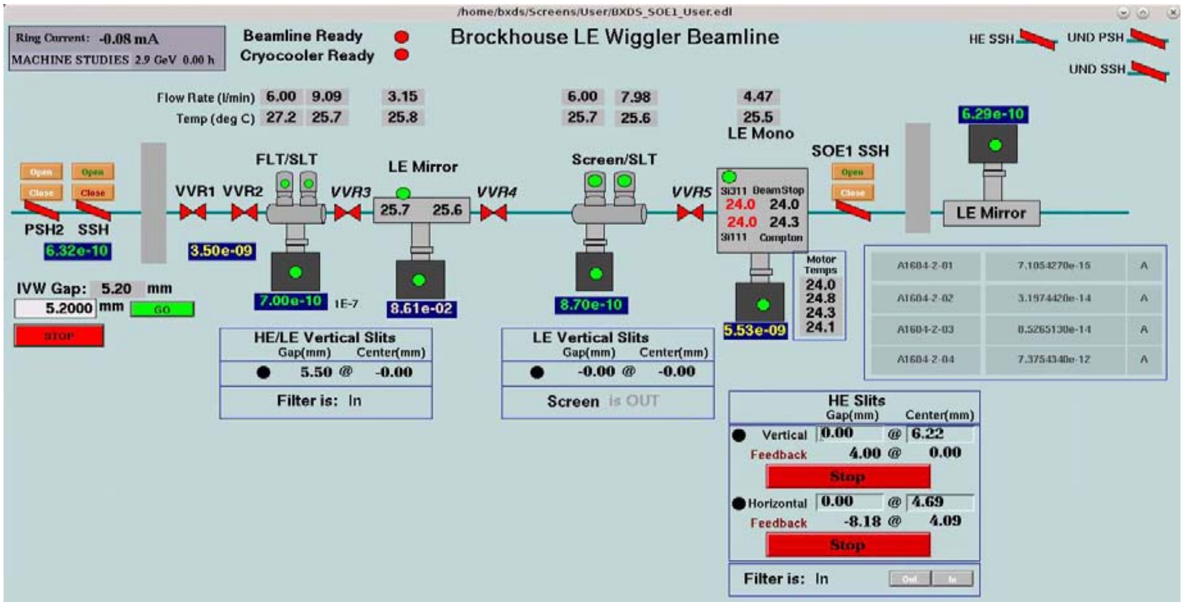
This will bring you to the main screen:

From this screen one can access the five most important programs for running experiments:

- BXDS User
- BXDS Picoammeters
- SPEC
- ImageJ
- Rayonix viewer
- Jiggler

There are also beamline cameras accessible on a separate computer, WKS-W004716 (monitors are located on top of the OPI1603-002 monitors and labelled). The beamline cameras are described in the WLE Beamline Manual.

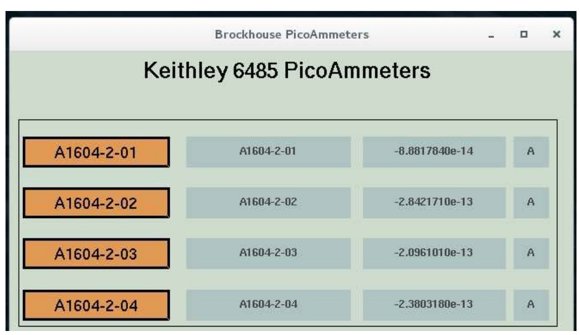
BXDS SOE1 User



This EPICS application provides an overview of the WLE beamline, and presents the status and values of beamline components including vacuum, temperature, valves and shutters. **Users should be aware of how to open/close shutters using this screen.** This program can be opened and closed at will.

BXDS Picoammeters

This EPICS application displays the current readings of several Keithley 6485 picoammeters of the Sector. The top four readings are the picoammeters inside WLE. Typically, these picoammeters are connected to the four ion chambers. Sometimes they are temporarily set up to read diodes or active beamstops.

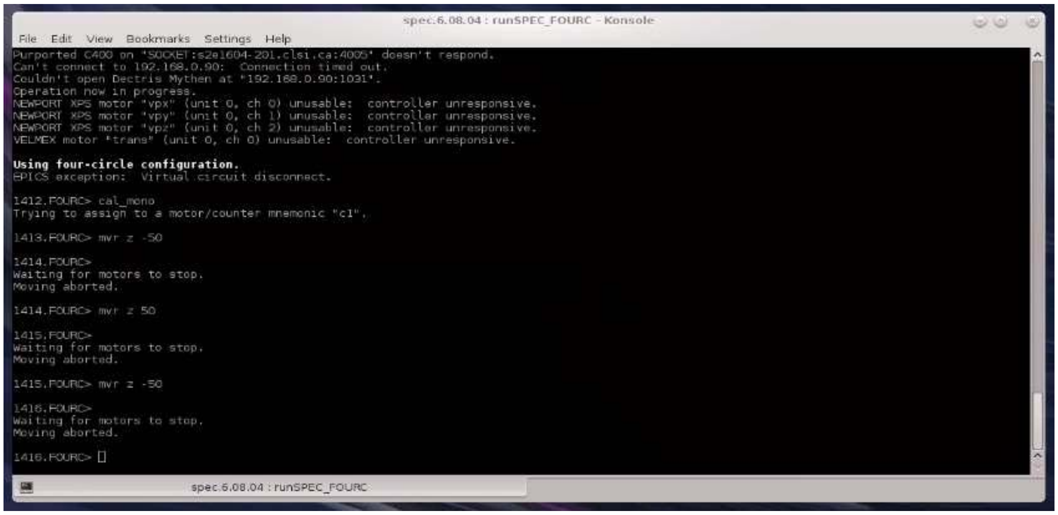


01 – Downstream of the Slit 1 and the filter wheel but upstream of the M2 mirror. Useful for setting Slit 1 and the filter wheel.
02 – Optional. Just downstream of the M2 mirror. Useful for setting the M2 mirror angle.
03 – At the end of the LE translation table, and downstream of Slit 2. Useful for setting slit 2 and determining the flux on the sample.
04 – Optional. On or downstream of the endstation.

Values of 10^{-13} A or less are the baseline noise level. This program can be opened and closed at will.

SPEC

SPEC is the main piece of software used to run experiments. SPEC features a command line interface, and controls all of the motors, takes data from all of the detectors, runs scans, shutters, saves the data, etc.



Use the command “ctrl-C” to stop a motor move or any operation in progress!!

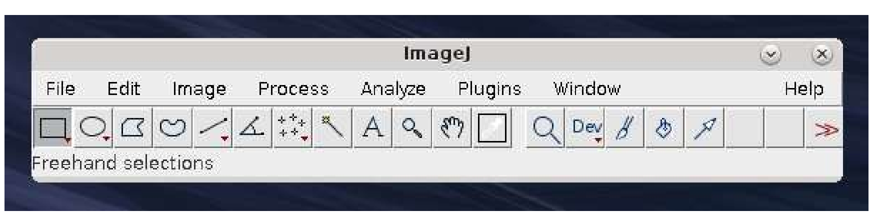
Users will need to learn a handful of commands to run their experiments independently. The SPEC manual and tutorials can be found on the company’s website, https://www.certif.com/spec_manual/idx.html , and a printed copy is available at the beamline.

To run SPEC:

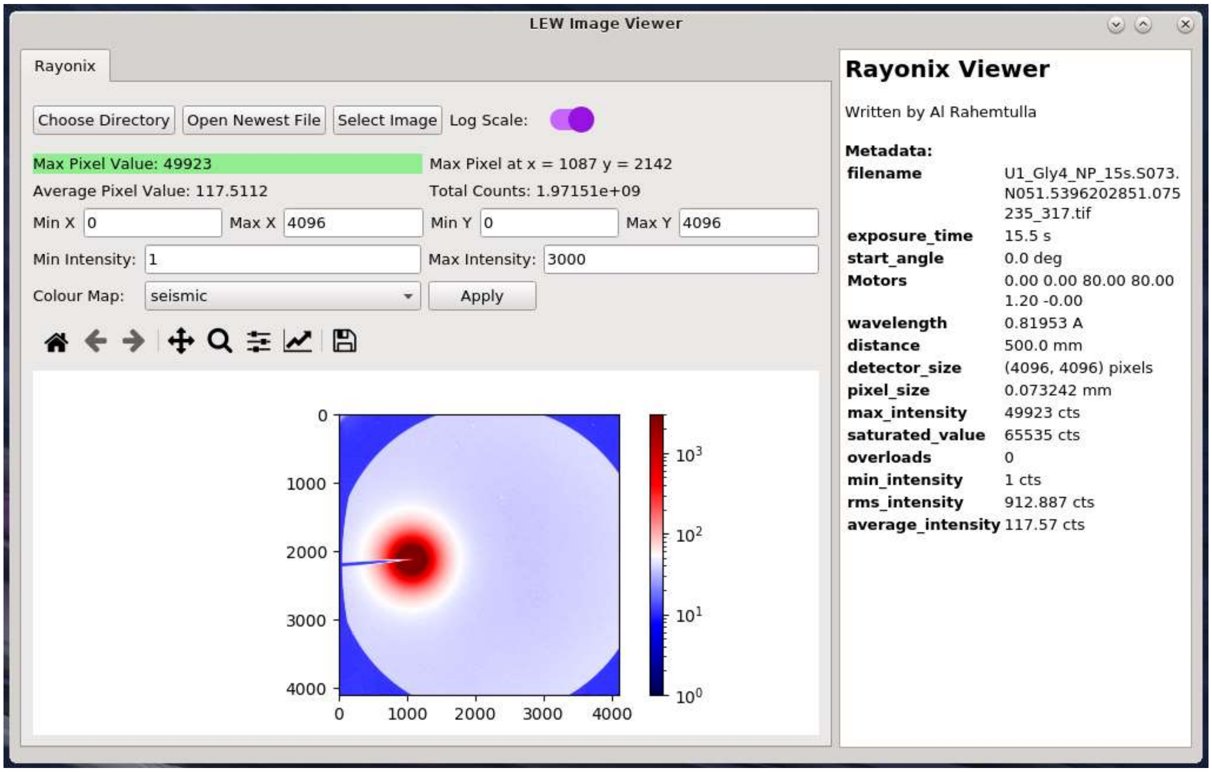
Click on the SPEC folder on the desktop, and then click the ***FOURC.desktop*** version. SPEC can be closed by typing “quit” in the command line. When SPEC is closed, most information such as the motor positions and soft limits will be retained. However, some information such as the Mythen calibration setup details will be lost. Therefore, **users should avoid closing and re-opening SPEC.**

ImageJ

ImageJ is a free program for working with all types of image data, particularly the .tif files from the Rayonix detector. After loading a CCD image, select Image → Adjust → Brightness/Contrast.
It is very useful for viewing a time/temperature series of CCD images using File → Import → Image Sequence, select the data folder and then click “use virtual stack”. This program can be opened and closed at will.



Rayonix Viewer



This program was written in-house to enable a quick review of Rayonix images as they are being collected. You will have to “Choose Directory”, and then “Select Image” to see an image you’ve collected. Then min and max intensity, log scale and colour map can be adjusted to get an optimal view of some features of interest.

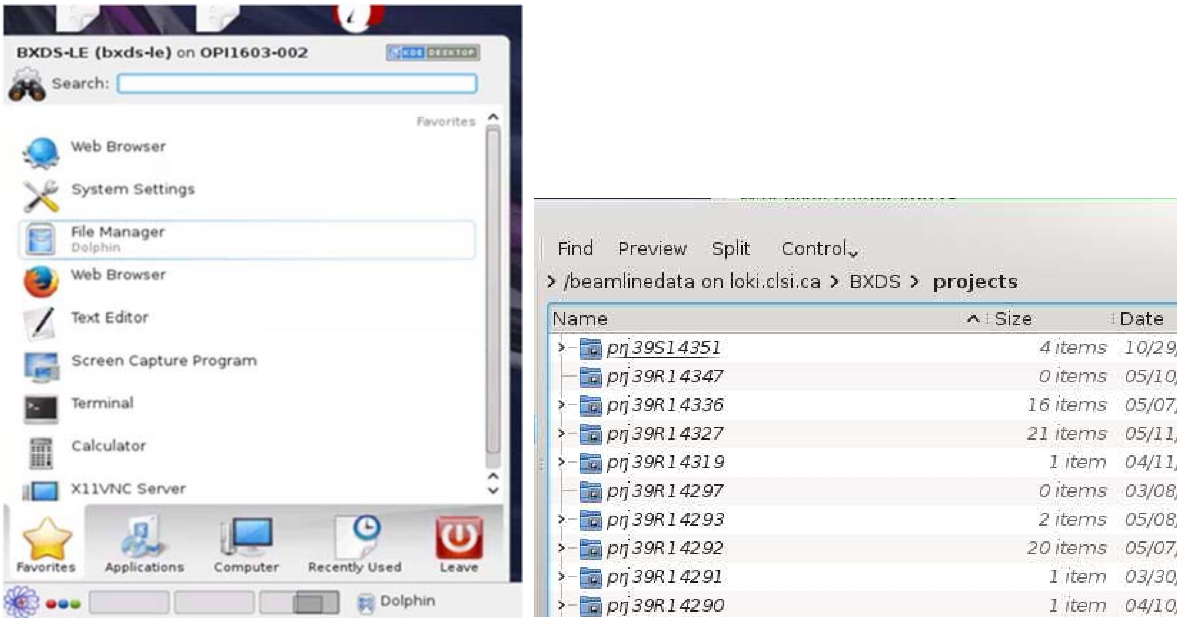
On the right side, metadata embedded in the .tif image is presented in an easy-to-access way. Note that the exposure time presented contains an addition 0.5 s for shutter delays. Wavelength, distance, and the 6 motors to display are set in SPEC.

Jiggler

This is a small application written in-house to jiggle the sample during an acquisition, to minimize radiation damage.

Data Handling

SAXS/WAXS data is primarily in the form of .tif images, captured by our Rayonix detector. At the start of a user run, beamline staff will direct the .tif image to be saved into a folder on Loki corresponding to the project number for that specific user group. Sub-directories can be made inside this folder, for manual transferring and filing of the data.



Check that data files are saving as the data is coming in. Press F5 to refresh the file folder. Delete useless images e.g., from sample alignments. Each image is 33 MB and it adds up quick.

Users can transfer their data to a removable USB drive during the experiment or when the experiment is complete. The transfer of hundreds of .tif images produced by the Rayonix detector can take hours, so please, **don't wait until the very end!!!**

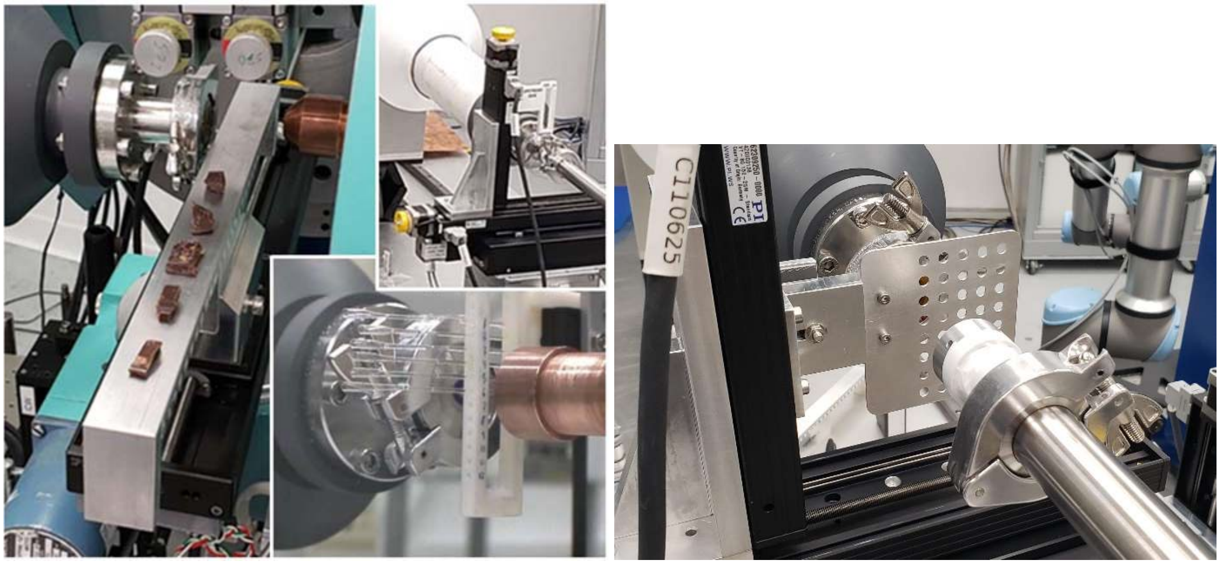
We will endeavor to store user data as long as possible, at least one year. We also use globus.org to transfer large data sets to remote users.

Sample Preparation and Energy Selection Guidelines

We highly recommended discussing any questions about sample prep and mounting before arriving for beamtime. Help keep our workspace safe by preparing all samples in the Life Science Lab, not in the user area.

We see a wide variety of samples for SAXS/WAXS!! We have two photon energies, 9.85 and 18.8 keV, and there are two main sample formats: transmission, or grazing incidence.

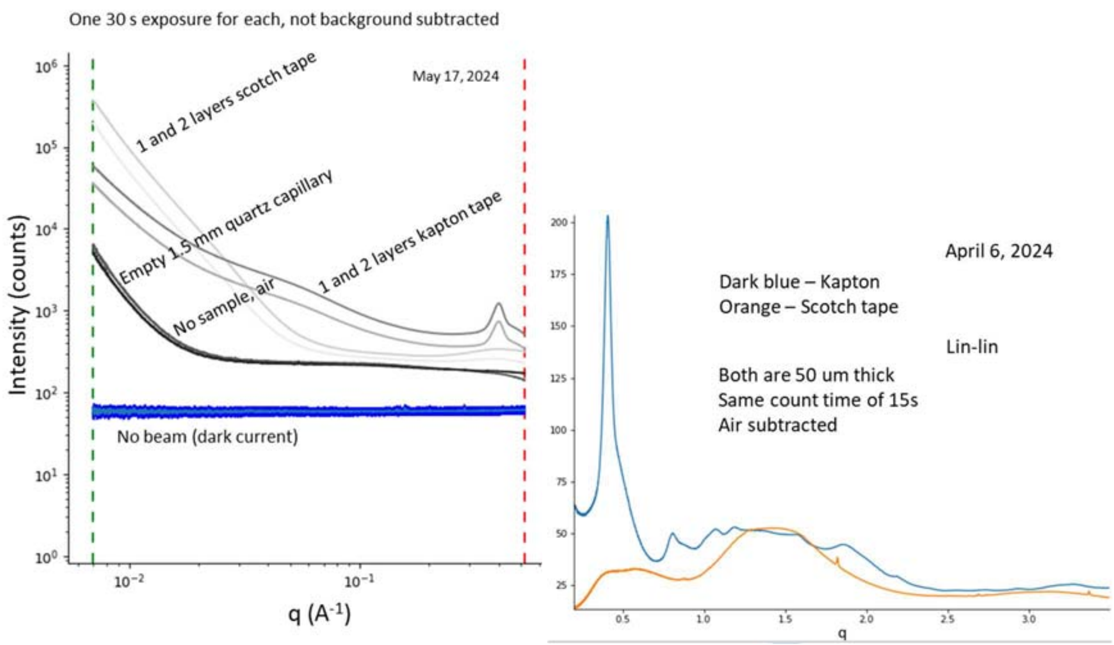
Transmission



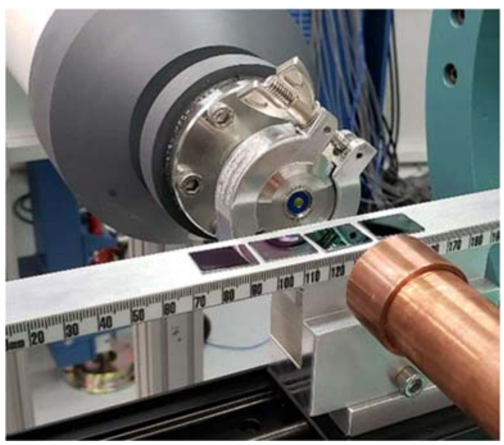
For a transmission measurement, the sample has to be transmissive: some X-rays need to be transmitted through your sample to reach the detector. Ideally, *sample transmission is about 37%*, (or 1/e, or an Absorption = 1.0). Calculate the transmission of your sample at the wavelengths we use. Samples for transmission should in general be around 1 to 2 mm thick… usually not more than 5 mm.

Solid samples can be measured freestanding. For a series of such samples, *ensure that all samples in the series are the same thickness*. This great aids with data analysis. Samples that can't be measured freestanding (powders, gels, liquids) must be placed in some kind of containment to hold them in the beam. The best containers are thin wall quartz capillaries. These are almost invisible to the beam, but they are fragile and expensive. Users must buy their own capillaries!! Scotch Magic tape (cellulose acetate tape) is also good. Kapton tape or capillaries are not ideal as Kapton has features in the WAXS and SAXS region.

For liquid samples, we generally need minimum 40 uL for the measurement. SAXS of liquids requires concentrations of 10 mmol, or about 1 to 10 mg/mL, if you are interested in the form factor.



Grazing incidence



9.8 keV is generally better for measuring in grazing incidence geometry. Typically, the object of interest in a grazing incidence experiment is a thin film on a substrate. 9.8 keV is less penetrating than 18.8 keV, so more signal comes from the thin film rather than the substrate. Also, powder samples are not suitable for grazing incidence.

Samples and substrates measured in grazing incidence geometry need to be FLAT (better than 1 mm) to achieve a good alignment. Ideal substrates are Si wafers, or glass microscope slides. Grazing incidence samples don't need to be more than 10 mm long in the beam direction. Min θ step with our stage stack is 0.01°.

We also can do a wide variety of in-situ and operando measurements in transmission or grazing incidence… just ask the beamline staff.

And remember, bring blanks, bring a range of concentrations, Know what your data should look like. Find published examples, and bring a known good sample!

Detector Distance and Tilt Calibration

Detector distance and photon energy together set the q range that can be observed during the measurement. Users must let staff know the q range required (ideally this information is included in the proposal). We will then tune the endstation to your problem. Q ranges are posted on the beamline website, https://brockhouse.lightsource.ca/about/low-energy-wiggler-beamline/

LaB6 (NIST 660b) and silver behenate calibration standards are available at the beamline.

Ag behenate standard (gisaxs.com)		
Ring	**q (Å^{-1})**	**d (nm)**
1	0.1076	5.839
2	0.2152	2.920
3	0.3228	1.946
4	0.4304	1.460
5	0.5380	1.168
6	0.6456	0.9732
7	0.7532	0.8342
8	0.8608	0.7299

Lanthanum hexaboride (LaB_6) standard (NIST SRM 660b certificate)		
Ring	**q (Å^{-1})**	**d (nm)**
1 (100)	1.5115	0.41569
2 (110)	2.1376	0.29393
3 (111)	2.6180	0.24000
4 (200)	3.0230	0.20784
5 (210)	3.3799	0.18590
6 (211)	3.7024	0.16970
7 (220)	4.2752	0.14697
8 (300)	4.5345	0.13856
9 (310)	4.7798	0.13145

Sample Measurement

A few tips and some reminders for staff before getting started,

- Set the soft limits on scz, sth, schi, sfz, saxsx and saxsz to prevent crashes.
- DO NOT STAND ON THE HUBER OR DETECTOR TABLES!!
- Remind users not to close the EPICS GUI, or any program really.
- Set up the plotted detector to be the beamstop diode.
- Check the signal level from users samples. RED pixels on the MarCCD screen are saturations (65536 counts). Adjust the dwell time to keep count rates in the linear range.
- Disable unnecessary detectors to simplify the user screen information (“disable c1 c2 c4 mon…”)
- Update the detector background image daily.

Typical transmission measurement

Ctrl + C   command will stop whatever SPEC is doing. Use this command if you notice something wrong.

Move to load/unload position

umv saxsz 100	# position to safely remove the sample holder
Load a new sample plate, lock up	

Open shutter

open_SSH	# or press the button on the ACIS panel, or user screen

Move to sample position

umv saxsz 0, 5, 10, 15 …	# move to sample holder position 1, 2, 3, 4 …

Align the sample

ccd_off	# stops detector image acquisitions
dscan saxsz -2 2 50 0.1	# scan to find center of the capillary, find the value from plot
umv saxsz ____	# move to center of capillary

Acquire the data

ccd_on	# starts detector image acquisitions
ccdfile ______	# no spaces or special characters. Include dwell time
click jiggler “start”	# turns on jiggler
ct 1	# This will count and acquire an image for 1 second. If the signal # is weak, count longer. If the signal is saturated (red), count for # less time
click jiggler “stop”	# When ct is done

Move to load/unload position

umv saxsz 100

- Count until you see red (i.e., saturation, 65535 counts per pixel), then back off the count time.
- Collect blanks at same dwell time and conditions

Typical grazing incidence measurement

Ctrl + C command will stop whatever SPEC is doing. Use this command if you notice something wrong.

Move to load/unload position

umv saxsx 0 # position to safely remove the sample holder
Load a new sample tray, lock up

Open shutter

open_SSH # or press the button on the ACIS panel, or user screen

Move to sample position

umv saxsx 0, 5, 10, 15 … # move to sample holder position 1, 2, 3, 4 …

Align the sample

do lessard # performs the Lessard alignment scan, consisting of a coarse height scan, a coarse theta scan, a fine Z height scan, and a fine theta scan. After alignment, Z and theta are set to be 0, the sample is translated laterally by 1 mm, and the ccd_on command is issued

Acquire the data

ccdfile ______ # no spaces or special characters. Include angle of incidence and dwell time

umv sth X.X # move to a desired angle of incidence "sth", in degrees
ct 1 # This will count and acquire an image for 1 second. If the signal # is weak, count longer. If the signal is saturated (red), count for # less time

Move to load/unload position

umv saxsx 0

- Sample heights MUST be close or it will confuse the lessard alignment script.

Reviewing and Processing the Data

The most important files are the .tif files, which are the detector images. There are also _spec files with no extension that contain addition information from scans, including ion chamber and ring current data for normalization, and diode intensity values for mesh scans. Tif files open immediately with ImageJ. The _spec files can be opened with PyMCA.

To access your data in GSAS-II: Under the tab “import”, choose “image”, and then choose “From tif image file”. Now the data file should open. By refining the data for the calibration standard, you can create save and apply your own instrument parameter file to the data.

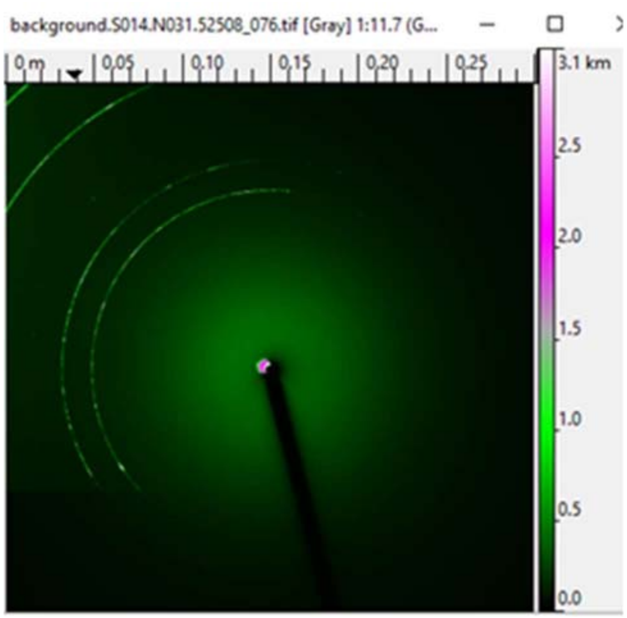
Further info on data processing is outside the scope of this manual. Talk with beamline staff, and check the help tab in GSAS-II. There are many excellent tutorials included with this program.

What Should I Check for While Reviewing the Data?

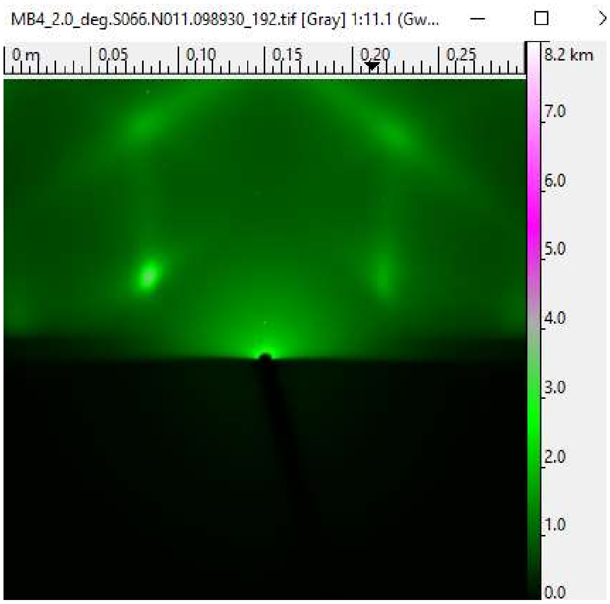
Are the files saving?
Check that data files are saving as the data is coming in. Press F5 to refresh the file folder.

Is the dynamic range being exceeded?
The data should be free of saturation. Adjust count times until you see red (i.e., saturation, 65535 counts per pixel), then back off the count time.

Extra peaks in the diffraction pattern
A pinhole in lead tape is the final optic element of the setup for WAXS. If the pinhole is misaligned, this will be visible as extra signal from the lead. Add wavelength, detector distance and q scale.



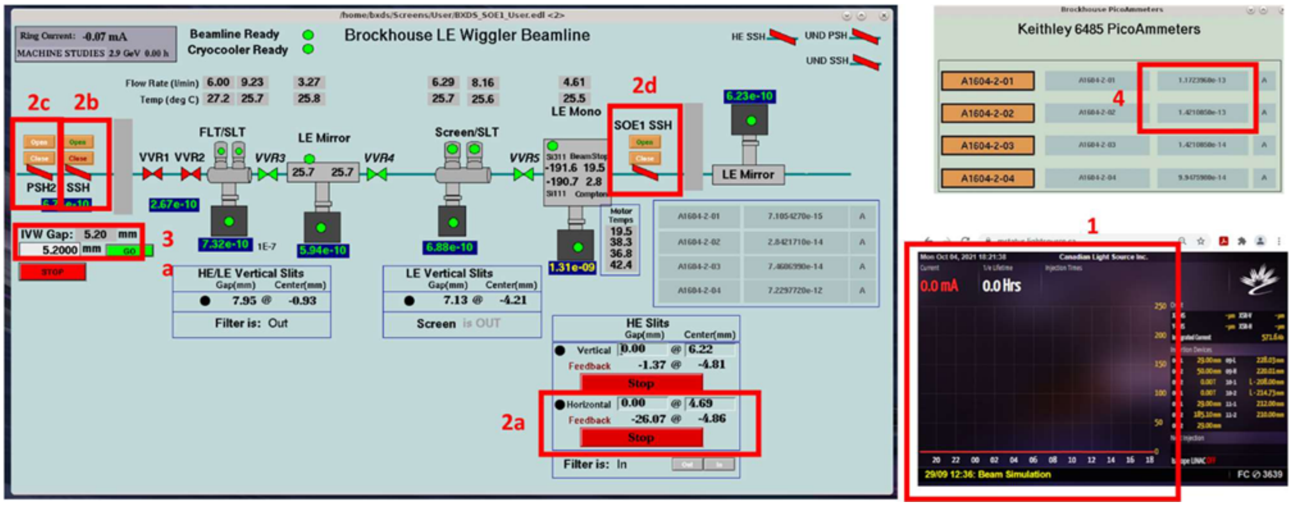
For a GIWAXS measurement with a silicon wafer substrate, the bare silicon wafer has its own set of peaks which can overlap with signals from the sample.



Aluminum bar sample holder?

Recovering From a Beam Dump

Hey, where'd my beam go?



1. Is beam available at CLS?

Check the beamline status screen (machine.lightsource.ca)

- If the screen is RED, there is no beam available, sorry!
- If the screen is YELLOW, and the ring current is 220 mA, then beam should be available for users. The status message at the bottom of the screen should say “Beam available”

There is often a delay of several minutes from the moment where the ring current recovers to 220 mA, to the point where the operator allows users to open shutters and play with insertion device gaps.

2. Are all the shutters open?

When the beam trips, the operators will typically close our front end shutters for the recovery process. They usually re-open them when beam is back. If they don’t do it, this must be done by users or staff.

2a – Check the SSH button (box 2b). If you see a message above the SSH button that says “Close HE horizontal slits”, then you must first close the horizontal slits behind the HE mono (these normally close automatically if the beam trips). Only do this step if the message “close HE horizontal slits” appears above the SSH button: Click on the box shown above (2a), type -20 in the gap box (even if it already says -20, you need to retype it), and hit enter. The gap feedback value should start changing… it takes a while for these slits to fully close.

2b – Open the SSH shutter by clicking the OPEN button. If it is GREEN it is open.

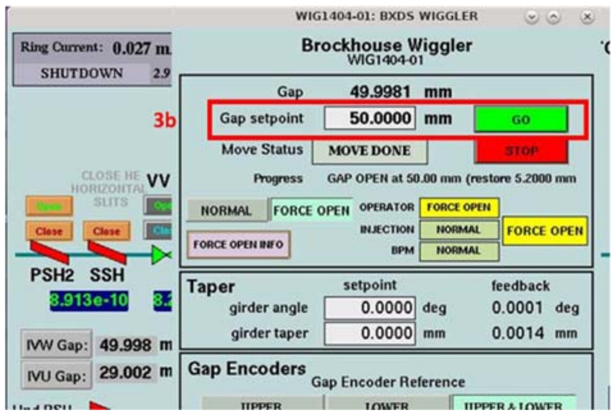
2c – Then open the PSH2 shutter by clicking the OPEN button. If it is GREEN it is open.
If you click OPEN but these two shutters do not open, then check to see if POE1 has been searched and locked up. The floor coordinator can help with this.

2d – Open the SOE-1 hutch shutter by clicking the OPEN button. If it is GREEN it is open. The shutter will only open if the hutch has been searched and locked up.

3. Is the wiggler gap closed to 5.2 mm?
When the beam trips, the operators will open our wiggler gap to 50 mm for the recovery process. They usually close the gap back to our normal operating value of 5.2 mm after recovery, but not always.

3a – Check if the gap is 5.2 mm. If it is, go to step 4.

3b – If the gap is not 5.2 mm, click on the “IVW Gap” box. The “Brockhouse Wiggler” window will pop up, as shown below. Click on the gap setpoint box, enter the value 5.2, then click the GO button.



If you are able to open the SSH and PSH2, but the gap will not close to 5.2 mm after a few attempts, wait a few minutes, try again, then call the operator. Sometimes they forget to hand over control of the gap.

4. Is there now beam in the hutch?
Check the ion chamber readings for A1604-2-01 and A1604-2-02. Both of these ion chambers typically read in the 10^{-6} A range when beam is on.