

Grazing incidence SAXS/WAXS using the Pilatus3R 300K detector on the SAXSLAB instrument



By Charles M. Settens, Ph.D.
Center for Materials Science and Engineering at MIT

For assistance in the CMSE X-ray facility contact settens@mit.edu

This SOP and others are available at <http://prism.mit.edu/xray/education/downloads.html>

The SAXSLAB instrument has the option to be setup to perform grazing incidence small or wide angle X-ray scattering on samples with nanostructured surfaces. Standard training shows how to perform specific alignment for GI-SAXS measurements with uniformly flat thin film samples by using the specularly reflected X-ray beam. After alignment, the sample to detector distance can be changed to probe the length scale of interest; for SAXS (30-2300 Å) for WAXS (3-70 Å).

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The instrument computer operating system is Ubuntu version 14.04 LTS.

Appendix B. Additional instrument info pg 11-12

This section provides additional information about the controlling the drives on the instrument via SPEC commands and Pilatus3R 300K detector information.

SAXSLAB Operation Checklist

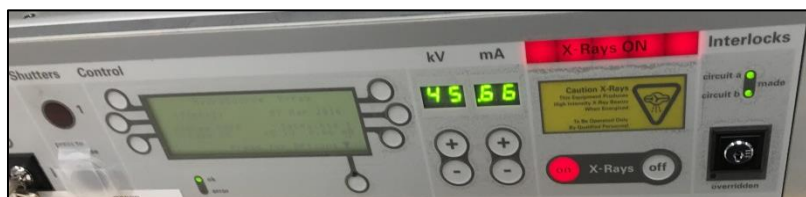
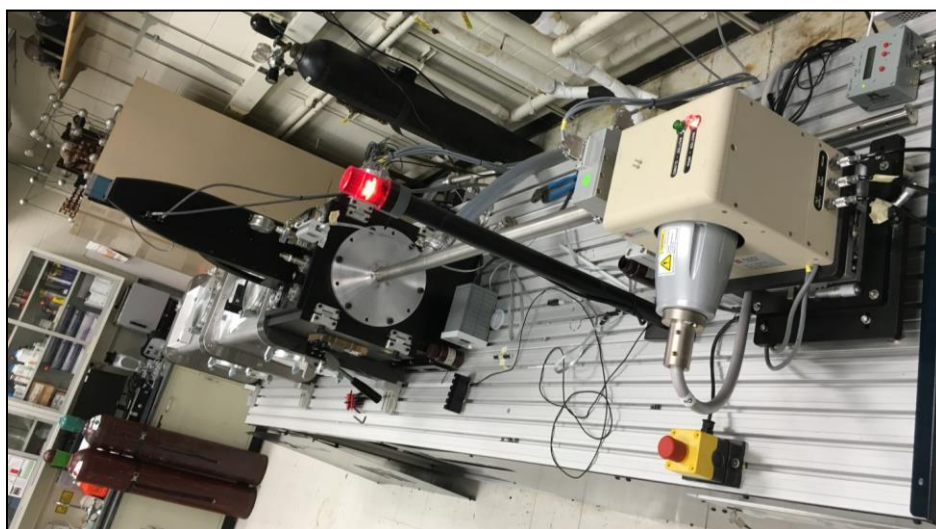
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c. Retrieve your sample	Ch VII
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e. Clean the sample stage and sample holders	
f. Copy your data to a secure location	
g. Disengage the SAXS in Coral	

The Chapter number and section number on the right-hand side of the page indicate where you can find more detailed instructions in the SAXS SOP.

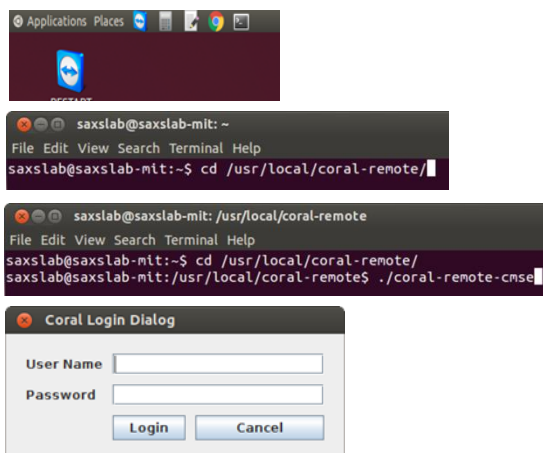
I. Startup instrument

This section walks through starting the SAXS instrument and logging into Coral. The instructions below give some descriptions on how to use the Linux system. If you are not familiar with Linux, read Appendix A.

1. Assess Instrument Status and Safety
 - a. Is the X-ray generator on?
 - i. Look for indicator light on pole near X-ray source.
 - b. What is the X-ray source power?
 - i. The X-ray source is at full power (**45kV and .66 mA**), and the shutter is remotely controlled. Shutter 1 will not open when pressed on the source controller.
 - ii. If the source controller reads, (**00kV and .00 mA**), please contact the X-ray lab staff.
 - iii.



2. Engage the SAXS system in Coral.
 - a. Navigate to the Terminal
 - b. Type: `cd /usr/local/coral-remote`
 - c. Type: `./coral-remote-cmse`
 - d. Login to Coral with your username and password





3. Start the programs Ganesha System Control

- a. There are is a toolbar displayed along the bottom of Workspace 1, with the programs that are used to control of the SAXSLAB system labeled SPEC, SL, DET, TVX, GEN, CAM1, GUI, and ED.
- b.
 - SPEC: software for instrument control and data acquisition - <http://www.certif.com/>
 - SL: server runs on the detector computer and handles processing of the data (cumulative files, includes headers, advanced noise-reduction).
 - DET: server talks directly to the Pilatus3R 300K detector
 - TVX: server for low level communication to DET server
 - GEN: server for communication from SPEC to the source generator for opening closing shutter, modifying voltage and current settings
 - CAM1: software to view samples in chamber via off axis camera
 - GUI: software for data visualization, image analysis operations and transformations, including batch mode data reduction and well as basic fitting- <http://www.saxsgui.com/>
 - ED: text editor
- c. **Blue** indicates the service is active, **Yellow** indicates an optional service has been closed and can be reopened by clicking on the toolbar icon, and **Red** indicates that a critical service is not running.



- d. SAXSGUI will indicate to open an initial image file.
 - i. Open the file: /home/saxslab/SAXSShared/data/latest/ **latest_XXXXXXX_craw.tiff** where XXXXXXX is the largest number possible in the directory.
 - ii. Navigate to **File > Open latest**
 - iii. Select the file: /home/saxslab/SAXSShare/data/latest/**latest_XXXXXXX_craw.tiff**
 - iv. SAXSGUI now continuously updates as data is acquired. The indicator of this condition is the red Stop Monitoring button will be enabled at the bottom of the SAXSGUI data visualization screen.



4. Start the Ganesha GUI software (GICC = Ganesha Interactive Control Center).

- a. The GICC software has 3 panels, left to right as can be seen below.
 - Left: sample stage graphic control (opened by < button on left of main panel)**
 - Center: main control panel (always open)**
 - Right: macro editor panel (open by > button on right of main panel)**

**** Note: If the macro editor panel is OPEN, mouse click the main control and sample stage graphics panel will populate a list of instructions to run in a macro. Close if not intended to make a macro.**

1 Vent the system, place samples and evacuate

2 Locate the samples and a blank position

3 Set the blank position

4 Optional: Optimize the sample position (best done in MAXS)

5 Choose the configuration and beamstop, and fine-tune the beamstop

6 Fill in the measurement details

7 Start the measurement

Optional: graphic based sample positioning

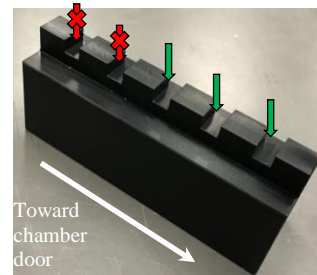
Optional: programming multiple measurements

II. Sample loading procedure

This section describes the procedure for venting, loading the sample (freestanding or capillary) on the Ambient 8x8 stage and evacuating the system.

1. Vent chamber to load samples.
 - a. In GICC, click the “Vent” button on the bottom right of the main control panel.
 - i. Dialogue box: *Please make sure the sample door is partially unlashed to prevent overpressure. Start venting?* Click **Start**.
 - b. After 120 seconds of venting the chamber door should be ajar. If this is not the case, click “Vent” button a second time and wait to see that the door opens.
 - c. Click “Isolate” button to close the valves.

2. Choose the GI-SAXS holder.
 - a. Place samples across the spaces in the GI-SAXS holder.
 - i. Only the first 3 spaces are available. **Spaces are 19mm center to center.**
 - b. Be sure to push holder in to back reference plate. Tighten thumbscrews.



****Only certain spaces in the GISAXS sample holder within the y motor limits (see above).**

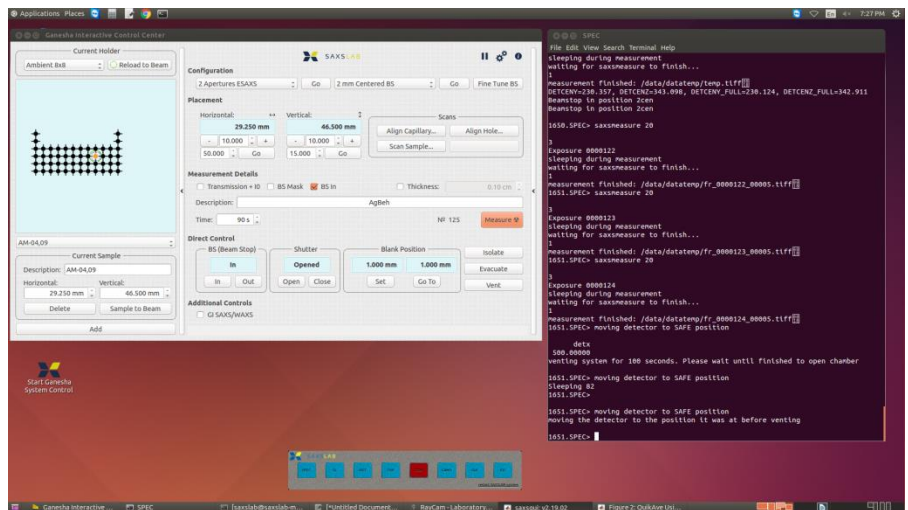
- c. Close chamber door and make sure that the O-ring seal is compressed by pulling up the handle.

3. Record the positions of your samples on the GI-SAXS holder.
4. Evacuate SAXS system
 - a. In GICC, click “Evacuate” button on the bottom right of the main control panel.
 - i. Expect a loud noise as the pump starts.
 - b. The vacuum gauge should read 0.08 mbar after 5 minutes of the chamber evacuating.

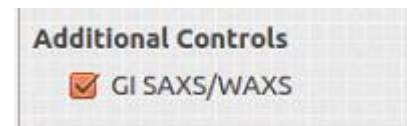
III. Alignment of sample to X-ray beam

This section describes how to position the sample into the beam and collect alignment measurements.

1. In the SPEC window, type the command
 - a. **SPEC> calib_gi**
 - b. This command moves the stage to the limit switch in Y and Z and center at the center position 03.



2. In GICC, open Current Holder panel with “>” button on the left of the Main Control panel.
 - a. In the dropdown menu, choose “**GISAXS 19x**” holder.
 - b. Loading “GISAXS 19x” holder with a reference point: Middle-03. Click **Go**.
 - c. Current Holder graphic is now accurately portraying the actual sample positions.
 - d. Verify that position 03 is Horizontal: 29.590 mm and Vertical 38.700 mm.
3. Use the graphic to move to the desired sample position.
4. Click the “**Sample to Beam**” button to move the sample into the beam path.
 - c. At the bottom of the Current Holder panel, click the “**Sample to Beam**” button to move the sample into the beam path.
 - d. In the Current Holder graphic, the green **O** should move to the desired position confirming the translation of the stage.
5. Change the Vertical position of the beam from 38.700 mm to 37.700 mm to ensure that the X-ray beam starts over top of the sample before the automated alignment procedure.
6. At the bottom of the main control panel in Additional Controls, **check** the “GI SAXS/WAXS box.



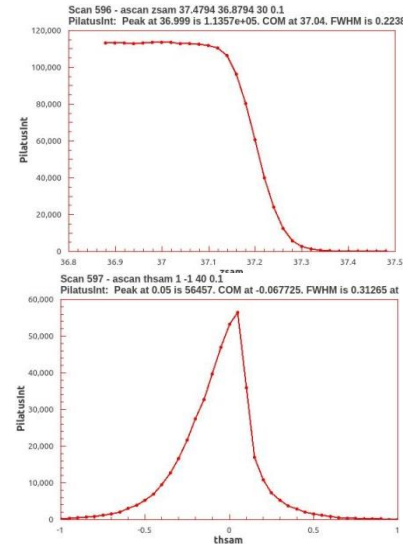
7. In Sample Alignment, choose Hybrid Lineup.



a. This is the automated direct beam half cut alignment procedure that performs a series of successive scans of zsam and thsam in order to align the sample surface to the X-ray beam.

8. A window for SPECplot will automatically open and the entire active area of the Pilatus detector is summed to a single intensity value as a function zsam, the sample height with respect to the X-ray beam.

9. Zsam is repositioned to the center of mass (COM) of the sample surface. Then, thsam will tilt the sample to find the most parallel position over top of the sample. This will happen 2x more times until a well-defined triangular shaped thsam plot is produced about the zero position.



10. In SAXSGUI, navigate to **File > Open latest**

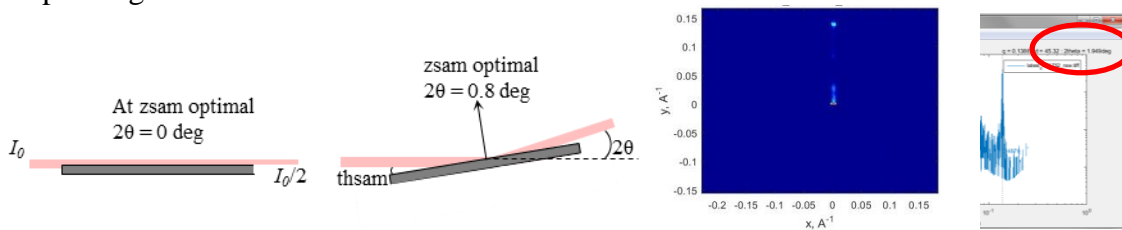
a. Select the file:

/home/saxslab/SAXSShare/data/latest/latest_XXXXXXX_craw.tiff where XXXXXXX is the largest number possible in the directory.

- i. craw files are not corrected for cosmic background radiation.
- ii. caz files are corrected for cosmic background radiation (in corrected folder).

b. SAXSGUI now continuously updates as data is acquired. The indicator is the Stop Monitoring button will be enabled.

11. The Hybrid Lineup procedure finishes with 4 detector images at thsam of 0.2, 0.4, 0.6, 0.8 degree tilt angles. The images will display a specular reflection that must be interpreted by the user in order to redefine the zero position of thsam. The scheme is shown below with a sample diagram.

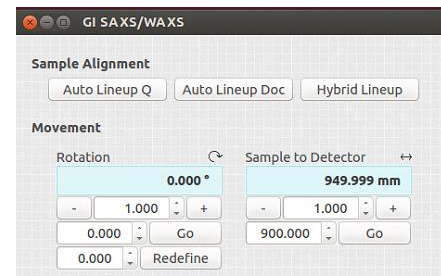


12. In the profile plot in SAXSGUI, hover the cursor over the intensity maxima from the specular reflection. It reads $2\theta = 1.949$ degrees, however it should read 1.6 degrees.

13. To redefine the zero position of thsam, divide 2θ by 2. That is, $1.949/2 = 0.9745$ degrees.

14. In the GI SAXS/SAXS panel

- a. Redefine = 0.9745 deg (3 decimal places, 0.975 deg).
- b. Go = 0.8 deg



15. To check the alignment of the specular reflection has indeed been redefined to $2\theta = 1.6$ degrees.

- a. In the Main GICC control panel, in Measurement Details section, take a test measurement
 - i. Description: “GI-SAXS alignment 0.8 deg 30sec”
 - ii. Time: 30 sec
 - iii. Note down Image number and click **Measure** button.

IV. Plan and collect GI-SAXS measurement

This section describes how to plan GI-SAXS measurements, change configurations and collect GI-SAXS data prior to automated data acquisition. Specific details on how to create and run an automated batch program are covered in sections IV and V.

EXAMPLE IS FOR A POLYETHYLENE POLYMER THIN FILM ON A FLAT SUBSTRATE.

1. What is the intended size you would like to probe?
 - a. SAXS/WAXS measurements are taken as a function on scattering angle, 2θ , from the forward direction of the radiation. A plot of relative intensity as a function of scattering angle, 2θ assumes the radiation wavelength is known ($\text{CuK}_{\alpha 1} = 1.5409\text{\AA}$, 8.06 KeV). To normalize the scattering profile to a wavelength independent scale the conversion to, \mathbf{q} , the momentum transfer is


$$\mathbf{q} = 4\pi \sin\theta / \lambda, \quad \text{where } \theta \text{ is } \frac{1}{2} \text{ the scattering angle and is the } \lambda \text{ wavelength in \AA ngstroms.}$$

$$\mathbf{q} = 2\pi/d, \quad \text{where } \mathbf{d} \text{ is real space length in units of } \text{\AA} \text{ and } \mathbf{q} \text{ is in units of } \text{\AA}^{-1}.$$

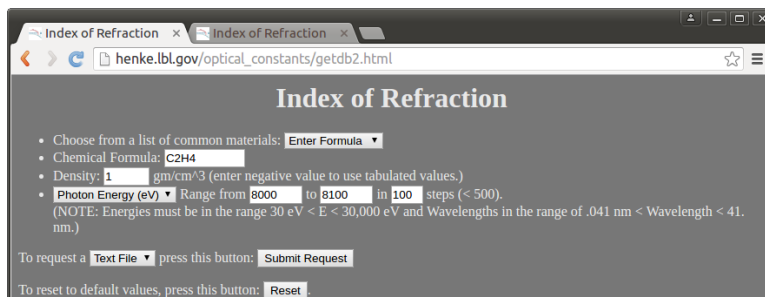
2. In GICC main control panel, select an appropriate **Configuration** to observe a specific length scale. Options are 2 Apertures WAXS, MAXS, SAXS and ESAXS. Click **Go** button.

Configuration	Name	Description	detx value	Sample-detector distance	Aperture Sizes Slit1-Slit2	q_{\min}	q_{\max}	d_{\max}	d_{\min}
0	Open	Open Apertures	mm	mm	mm	1/\AA	1/\AA	\AA	\AA
1	WAXS	Wide Angle 2mm beamstop	0	109.1	0.9-0.9	0.037	2.06	168	3.05
2	MAXS	Medium Angle 2mm beamstop	350	459.1	0.9-0.4	0.009	0.50	706	12.5
3	SAXS	Small Angle 2 mm beamstop	950	1059.1	0.7-0.3	0.0039	0.22	1629	28.7
4	ESAXS	Extreme Small Angle 2 mm bstop	1400	1509.1	0.45-0.2	0.0027	0.18	2321	41

Calculated with help from <http://www.bio.aps.anl.gov/xraytools.html>, assume 172 μm pixel size, 8.06keV, Image radius of 330 pixels.

3. Find the Google Chrome Browser,  navigate to http://henke.lbl.gov/optical_constants/getdb2.html

4. Enter the chemical formula and mass density for a photon energy of 8000 to 8100 eV ($\text{CuK}_{\alpha 1}$ is 8060 eV).



- a. To request a Text File and Click “Submit Request.”

Energy	Delta	Beta
8056.84766	3.65661253E-06	4.56079663E-09
8057.84912	3.65570145E-06	4.55848825E-09
8058.84912	3.65479264E-06	4.55618432E-09
8059.85059	3.65388223E-06	4.55388038E-09
8060.85205	3.65297228E-06	4.55156979E-09
8061.85352	3.65206301E-06	4.54926719E-09

5. A text file will be generated with information of the refractive index in 3 columns.

6. Delta is the real part of the refractive index.

7. To calculate the critical angle for total external reflection, $\theta_c = \sqrt{2\delta}$.

- a. $2 \times 0.0000036529 = 0.0000073058$
- b. $\sqrt{0.0000073058} = 0.0027029$ radians.
- c. Change radians to degrees, $0.0027029 \times (180/\pi) = 0.15486$ degrees
- d. $\theta_c = 0.15486$ degrees

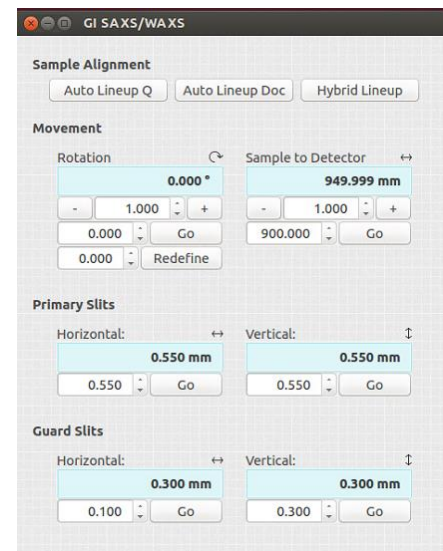
8. To ensure an evanescent wave of X-rays passes along the film surface, choose an angle above the critical angle.
 - a. In the GI SAXS/SAXS panel, Go = 0.12 deg.

9. In the Main GICC control panel, in Measurement Details section, take a test measurement

- a. Description: “GI-SAXS Sample name 0.12 deg 120sec”
- b. Time: 120 sec
- c. Note down Image number and click **Measure** button

10. For well resolved GI-SAXS measurements, collimate the main beam by changing the slit sizes.

- a. In the GI SAXS/SAXS panel,
 - i. Primary - Horizontal: 0.3 mm Vertical: 0.3 mm
 - ii. Guard – Horizontal: 0.1 mm Vertical: 0.1 mm



11. In the Main GICC control panel, in Measurement Details section, collect a GI-SAXS measurement.

- a. Description: “GI-SAXS Sample name 0.12 deg 600sec”
- b. Time: 600 sec
- c. Note down Image number and click **Measure** button

IV. Writing and Running an automated measurement macro

This section covers making a macro to automate the data collection at multiple tilt conditions of the sample, detector configurations.

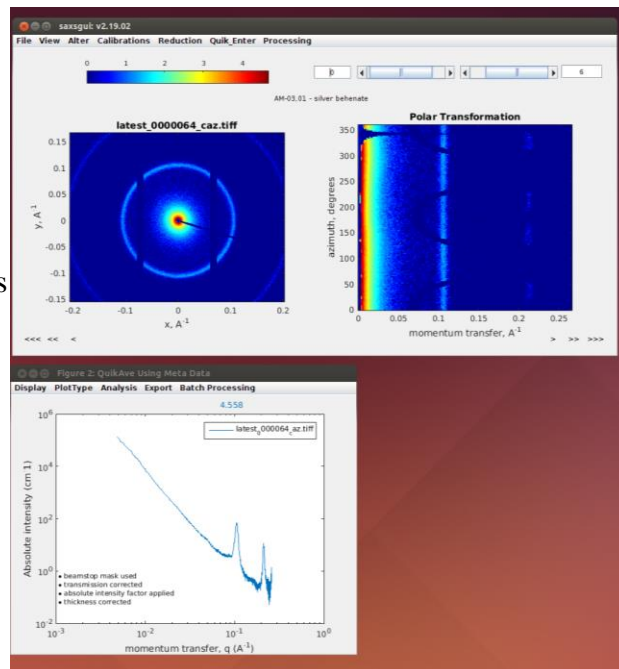
1. Open the right edge of the Main GICC panel for the macro editor with the “>” button.
Note: When clicking on any button in the GICC, the action is not performed but instead, written into the macro list.

- a. At the top of the macro editor, click the “New” button
 - b. In the GI SAXS/SAXS panel, Go = 0.12 deg.
 - c. In Measurement Details, select a measurement
 - ii. Add a description including: sample name, exposure time, hole position, sample thickness
 - iii. Choose a measurement time in seconds.
 - iv. Click **Measure** button.
 - d. Repeat for b and c and increment by 0.1 degrees to a final critical angle value of 0.25 degrees.
 - e. Save macros written in the GICC panel with the .gicc extension.
2. If you’d like to insert an instruction in a macro ahead of another without deleting all of the instructions, right click on the instruction and select “**Insert before**”. The instruction clicked has an anchor icon next to it.
 - a. Perform mouse clicks on GICC to insert intended action.
 - b. When done adding actions, right click and “**Disable insert before**”
 - c. Save macros written in the GICC panel with the .gicc extension.
 3. Check the sequence of the measurement macro.
 4. Click the “**Run**” button to watch automated collection of the SAXS/WAXS patterns for each sample specified in the macro.

V. Data reduction with SAXSGUI

This section walks you through the steps of data reduction with SAXSGUI software from 2D image data to 1D profiles that can then be loaded into standard SAXS data analysis software (SASview).

1. SAXSGUI Basics for output of data:
 - a. Processing > AP with Metadata
 - i. Select the cosmically corrected .caz files from **/home/saxslab/SAXSShared/data/latest**
 - ii. The shared folder to save files: **/home/saxslab/SAXSShared/Your folder**
 - b. Data will be outputted in the follow file extensions
 - i. .grad file (ASCII file with header details)
 1. 3 column format (q, I, \sqrt{I}) or
 - ii. .jpg of 1D data from azimuthally averaged 2D data
 - iii. .jpg of 2D image with colorbar
 - iv. .jpg screenshot of SAXSGUI screen
 - c. If the SAXSGUI program fails to Autoprocess, Close it and reopen via the SAXSLAB Toolbar.



IV. When you are done

This section describes the procedure for venting, loading the sample and evacuating the system.

1. Vent chamber to load samples.
 - a. In GICC, click the “**Vent**” button on the bottom right of the main control panel.
 - i. Dialogue box: *Please make sure the sample door is partially unlashd to prevent overpressure. Start venting?* Click **Start**.
 - b. After 120 seconds of venting the chamber door should be ajar. If this is not the case, click “**Vent**” button a second time and wait to see that the door opens
2. Close chamber door and make sure that the O-ring seal is compressed by pulling up the handle.
3. Evacuate SAXS system
 - a. In GICC, click “Evacuate” button on the bottom right of the main control panel.
 - i. Expect a loud noise as the pump starts.
 - b. The vacuum gauge should read 0.08 mbar after 5 minutes of the chamber evacuating.
4. Logout of Coral.

Appendix A. Linux info

The instrument computer operating system is Ubuntu version 14.04 LTS. Tutorials available at <http://linuxsurvival.com/>

Appendix B. Additional instrument info

This section provides additional information about the controlling the drives on the instrument via SPEC commands and Pilatus3R 300K detector information.

Detector info:

PILATUS3 R	300K
Number of detector modules	1 ×3
Sensitive area: width × height [mm ²]	83.8 ×109.5
Pixel size [μm ²]	172 ×172
Total number of pixels	487 ×619 = 301,463
Gap width: hor. / ver. [pixel]	- / 17
Inactive area [%]	6.5
Defective pixels	< 0.03%
Maximum frame rate [Hz]	20
Readout time [ms]	7
Point-spread function	1 pixel (FWHM)
Counter depth	20 bits (1,048,576 counts)
Threshold energy [keV]	2.7 - 18
Power consumption [W]	36
Dimensions (WHD) [mm ³]	198 ×193 ×262
Weight [kg]	7.5
Module cooling	Water-cooled
Electronics cooling	Water-cooled
External trigger / gate	5V TTL
Energy calibrations	res of Cr, Mn, Fe, Cu, Ga, Ni
Standard configuration	450 μm silicon sensor
Detector options	1000 μm silicon sensor
	vacuum compatibility