

**University of Minnesota
College of Science and Engineering Characterization Facility**

Ganesha SAXSLAB

User manual



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1. Planning a SAXS experiment

Before getting access to the instrument you should think about your measurement.

- 1) What q-range are you interested in?
- 2) How strongly does your sample scatter?
- 3) What are you looking for?
 - a. Peak Positions and peak-width?
 - b. Particle sizes?
 - c. Data that can be accurately modeled over a large q-range.

Select among the following configurations available

Aperture	Configuration	SD (mm)	q-range (Å)	d-space (Å)	I (Mph/s)
2 Apertures	21 WAXS	100	0.07-2.7	2.3-90	55
	22 MAXS	450	0.02-0.7	9-315	31
	23 SAXS	1050	0.007-0.25	25-900	4.5
	24 ESXAS	1500	0.003-0.2	30-2000	2.2
3 Apertures	1 WAXS	100	0.07-2.7	2.3-90	25
	2 MAXS	450	0.02-0.7	9-315	6.5
	3 SAXS	1050	0.007-0.25	25-900	1.3
	4 ESAXS	1500	0.003-0.2	30-2000	0.28

It is important to realize that in routine running of the instrument, there is a unique chance to trade off resolution for intensity. The intensity available in WAXS configuration is a factor of 4-5 times higher than in MAXS- configuration and again a factor 4-5 higher than in SAXS-configuration, and once again a factor 4-5 more than in extreme SAXS regime.

2. Initial Setup

1. Make sure that the yellow lamp on the tower on instrument tube is on, indicating that the X-ray source is on. Ramp up the X-ray source voltage and current using the following command in SPEC window **>x_ramp**

The X-ray source should ramp upto normal operating conditions 50 kV and 0.3 mA in 3 minutes.

2. On the desktop, you will find an icon for "GICC" (Ganesha System Control Panel).

Right-click this to "open". This action will open a service control panel and start a whole array of programs and windows needed to run the SAXS system. While this is happening, the user should allow the computer to process.



When the process is finished, the panel may look like this. **BLUE** indicates that a service is running. **RED** indicates that a crucial service is NOT running. **YELLOW** indicates that an optional service is NOT running.

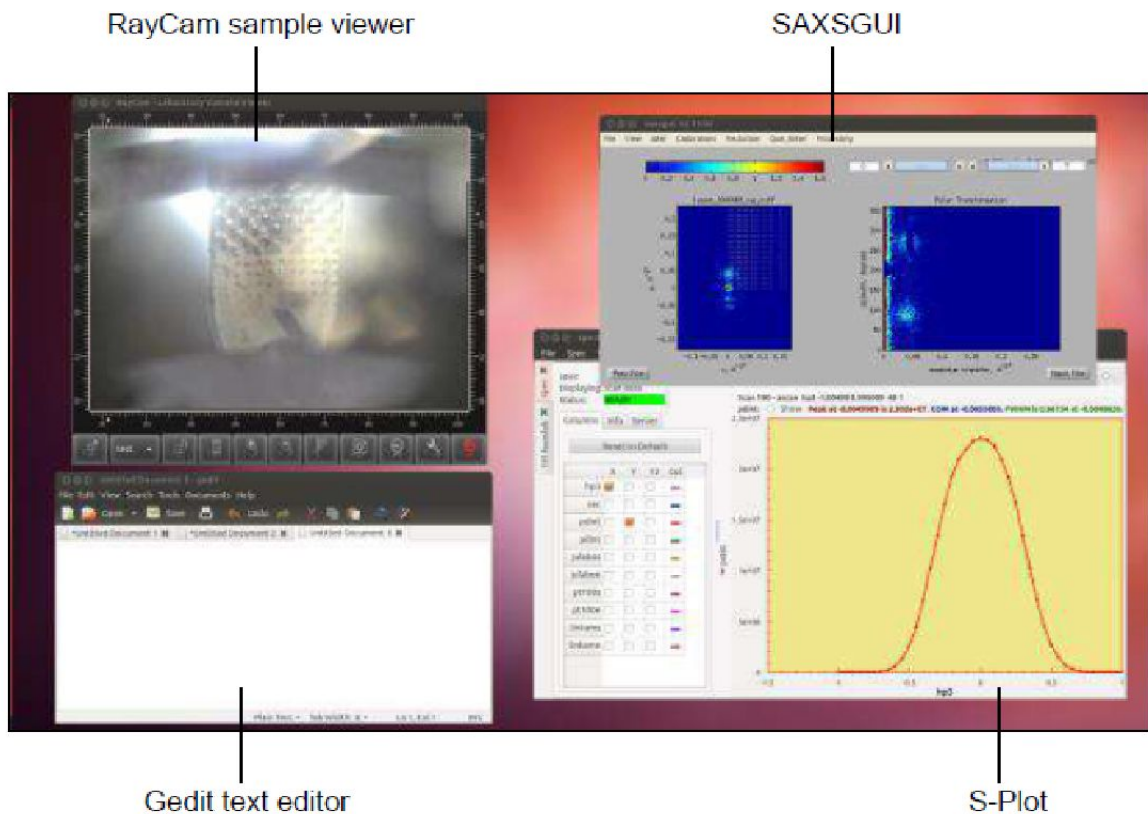
Once started, the two screens show the desktop with the programs that are needed to control the instrument.

- The instrument is controlled by **SPEC**, which gives full control over virtually all the aspects of the instrument.
- **GICC** (Ganesha Interactive Control Center) is the interface, which can be used for data acquisition. the interface sends commands to SPEC based on User input. Clicking on the **left bar with arrow** on the mail screen opens **sample position editor**. Clicking on the **right bar** opens the **macro menu**.
- **S-plot** window is not normally displayed when SPEC is started. When you scan over a part of your sample and record transmitted intensity, for example during hole alignment, S-plot window appears showing counts vs distance.
- **RayCam** sample viewer facilitates sample positioning.
- **SAXSGUI** is used for data processing. All the data is set to be stored in **Computer>**

disc 2> data > latest. The data is stored in two formats. *craw* files are the raw data, *caz* files are data from which cosmic background has been corrected. Open any of the file in the folder to load SAXSGUI.

- **Text editor** can be used for manual macro writing and note keeping.

The master log file can be accessed at **Home>master.dat** (a link available on the desktop). A unique file number is generated for all the scans. The details along with sample name, sample stage used and comments can be accessed in the master log file. Image data is saved in tiff format and should be readable by most data-handling packages.



3. Sample mounting and loading sample holder

A. Sample mounting

1. In the SAXSLAB Ganesha, X-ray scattering is measured in a vacuum chamber in order to reduce the background intensity. Consequently, samples may need to be enclosed appropriately,

Powders must be contained in order to prevent them from blowing away when the vacuum chamber is either pumped down or vented. Powder samples may be mounted in capillaries or between two sides of tape.

Non viscous liquid samples can be inserted in capillary, which should be sealed either by wax or by flame sealing.

Volatile samples must be contained in an enclosure, which is sealed to pressures < 0.01 mbar.

Tape may be used in the vacuum chamber to attach samples or sample enclosures to a sample holder; however, it is important to choose a tape which has weak x-ray scattering in order to reduce interference with the scattering from your sample. Suitable tapes include 3M Scotch tape or Kapton tape

2. The ambient plate sample holder has a set of 4 mm diameter holes, which are 8 mm centre to centre, arrayed in ten columns of four holes in the centre and one column of six holes at each end. However, the column at each end is not accessible for measurements because the sample stage has insufficient range. In addition, there are three larger rectangular holes across the top, which are accessible.
3. Mount the samples on the ambient stage sample holder or sandwich sample holder (while looking at the SAXSLAB logo in front, samples are to be mounted on left side). While mounting, make sure that you **leave**
0306 position blank in the ambient sample holder/
0101 position blank in the sandwich sample holder.

This position is to be used for fine-tuning beam stop. Make a note of the blank and sample positions loaded on the stage matrix (clicking a picture always helps). Try to mount **samples close to 0306** position on the sample stage.

B. Loading the sample stage

1. Click **Vent** in the interface. An over pressure warning sign is displayed “Please make sure the sample door is partially unleashed to prevent overpressure. Start venting”. Click **Start**. The detector moves to a safe position and system draws in dry air through pipe at the back.

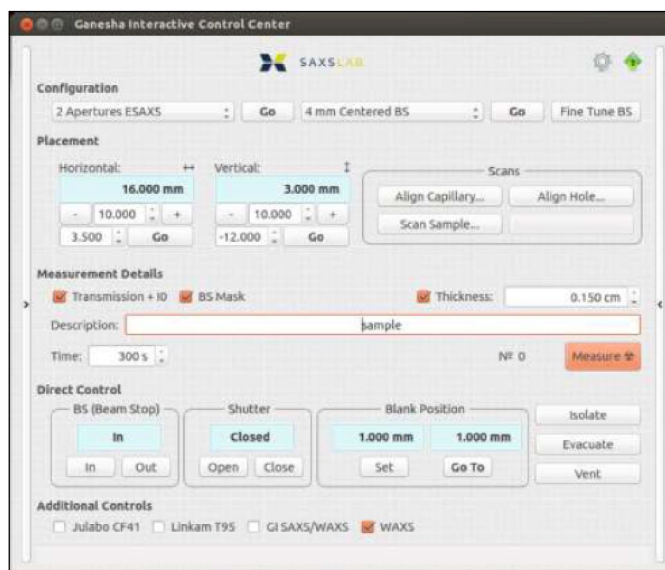
2. Load the sample holder by sliding it on the block in sample stage, pushing it all the way back. You know that the stage is loaded correct, if you are able to see the SAXSlab logo on the front side of the loaded sample holder.
3. Tighten 2 screws by hand on the left side of sample holder.
4. Close the door, and lock the door making sure to leave the handle in the horizontal position.
5. Click **Evacuate** in the interface. You will hear a loud hissing sound and pressure in the vacuum chamber drops slowly. It will take roughly 4 minutes to reach the operating pressure of $2E-1$ mbar. When the pressure decreases, the handle of chamber door will drop to the safe lock position.

The door must be kept closed at all times, other than loading and unloading the sample holder.

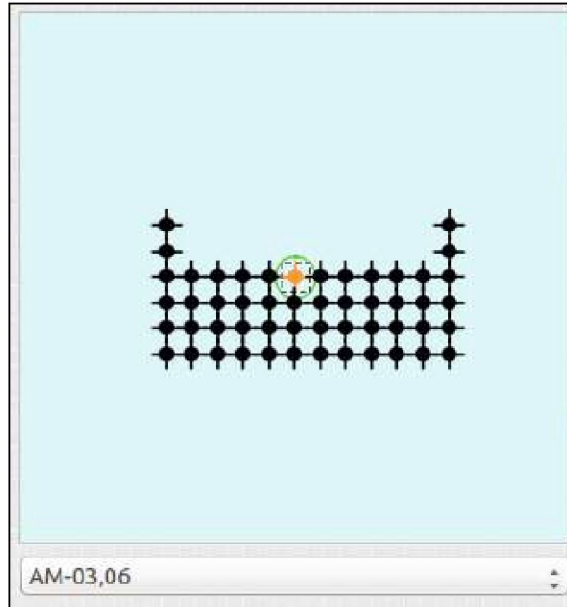
4. Sample alignment

A. Alignment of sample stage

1. It is important to first bring the sample stage to the home position in the view of Raycam viewer (make sure that crosshair for 1707_1 is selected in the window) . **The blank hole is the home position for the particular sample holder (0306 for ambient sample holder and 0101 for sandwich sample holder)**. So, depending upon the sample position viewed in the RayCam immediately after loading the sample holder, use '+' and '-' buttons to move the sample holder up/ down and left/right (vertically "+" moves sample stage up and horizontal "+" moves sample stage right).



2. In case X-ray generator is at low power (30 kV/0.3 mA), power it up to full (50 kV/0.6mA) using command '>x_ramp' or '>x_start' in the SPEC window.
3. Once 0306 position is close to red small cross- hair in the view of Raycam, select **align hole**. The position of the hole is optimized by measuring the intensity of the beam on the pin diode while scanning through the hole. Align the hole *horizontally* and then *vertically*. The pin diode data is displayed in S-plot window.
4. Now select **Load sample holder** (the left panel of GICC) and select ambient sample stage/sandwich sample holder. The green circle in the left window should move to the home position in the simulated sample stage.



5. As this is blank position (no sample on the hole), **Set this as blank position.**

B. Fine tuning beam stop

It is important to fine-tune the beam stop position to ensure it perfectly blocks the incoming beam. Fine-tuning process works by measuring the position of the direct beam on the detector and subsequently making the beam stop cover this point. Thus, fine tuning is done in a blank position, where the beam is not blocked by the sample stage or a sample.

Select the appropriate configuration (click Go) and beam stop (click Go) for your experiment. Click *fine tune BS*.

Whenever the configuration is changed, it is necessary to fine tune the beam stop position so that it blocks the direct beam as well as possible at the detector.

C. Sample alignment

As you know the position of your sample, you can **directly type** it, in the bottom field and press **Enter or click Go**. Alternatively the sample can be moved stepwise using the Raycam viewer. You can move sample stage in any increments by entering the particular number.

After a rough sample placement with the help of RayCam viewer, the sample position can be optimized by selecting **Align hole or Align capillary** and choose to align horizontally

and/or vertically. The data count from pin diode can be observed in S-plot. Once the alignment is complete, set this position as sample.



The cross indicates where the beam is. And one can then use a transmission measurement combined with a scan (as immediately above) to do a fine alignment

5. Sample measurement

Before starting measurement, ensure that

- Vacuum is close to 2E-1 mbar.
- The correct configuration and beam stop is defined.
- Blank position has been defined.
- Beamstop has been fine tuned with respect to the particular configuration.
- The position on sample stage where the sample is loaded is known.

For collecting each measurement, you have the option to:

- 1) Measure the sample transmission and incident x-ray beam intensity.
- 2) Block the direct beam at the detector with the beam stop.
- 3) Place a mask over the beam stop position when the 2D X-ray scattering image is displayed.
- 4) Include the sample thickness.

In general, it is advisable to check all of these options (the last option, if you know thickness of sample).

Once a blank and sample position has been set, **you must define sample name** under description as per the following code

PPPP_QQQ_RRRRR_SSSSSSSSSS

where PPPP is your **four digit charfac registration number**.

QQQ is the code for **sample holder description**. Use one of the following codes

ASH	Ambient sample holder
SCH	Sandwich sample holder
GIV	GISAXS vertical
GIH	GISAXS horizontal
TST	Tensile tester

SHC shear cell
LVT Linkam variable temperature
CVT Capillary variable temperature
IVT Instec variable temperature
LVT Linkam variable temperature

RRRR is the specific configuration used for measurement, which may be one of the followings

2SAXS, 2WAXS, 2MAXS, 2ESAXS, 3SAXS, 3WAXS, 3MAXS or 3ESAXS

SSSSSSSSSS is the **sample description**.

Enter the measurement time in the *Time* box. Now click *Measure*.

Record the sample description and the measurement number, immediately to the left of the measure button. The data filename will include this number.

The measurement may be aborted at any time by pressing *Abort* button. After aborting, check whether the shutter is open or closed. If the shutter remains open, close it by pressing the Close button in the shutter section of direct control or the command *c_shut* in SPEC.

You can view the data in *computer>disc2>data>latest*.

All the processed files like macros, ID curves or integrated data, must be stored in the corresponding yearly subfolder in the Ganesh User folder.

Home> saxslab> Ganेशha users > year > charfac code_ name.

A link to *Ganेशha users* folder is available on desktop.

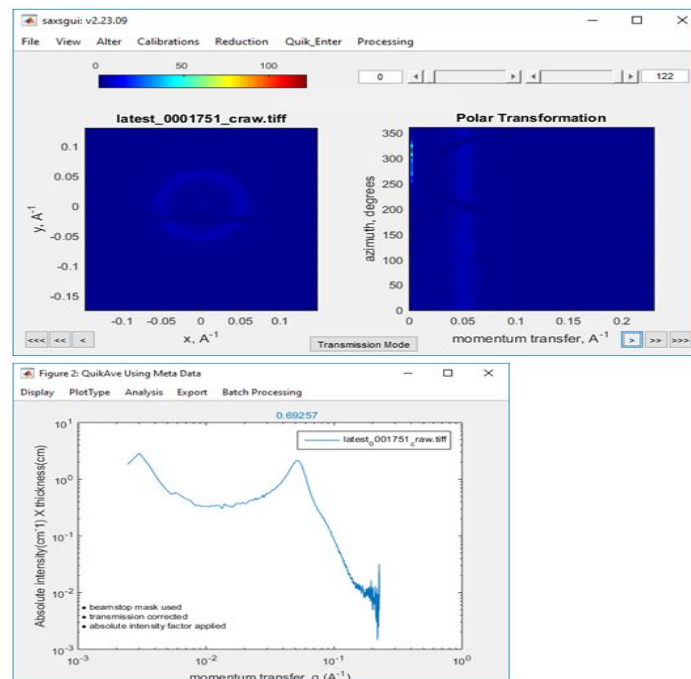
6. Data reduction with SAXSGUI

All the collected data is set to be stored in Computer> disc 2> data > latest. The data is stored in two formats.

.craw files are the raw data.

.caz files are data from which cosmic background has been corrected.

The master log file can be accessed at home>master.dat. A unique file number is generated for all the scans. The details along with sample name, sample stage used and comments can be accessed in the master log file.



Data can be output in the following file extensions

- .grad file (ASCII file with header details) as 3 column format (q, I, \sqrt{I}) or ($q, d\sigma/d\Omega, \sqrt{d\sigma/d\Omega}$)
- csv file of 1D data from azimuthally averaged 2D data
- image screenshot of SAXSGUI screen

7. Automated measurement macro

To automate data collection at multiple sample locations and/or different detector configurations, macro can be generated using following instructions (make sure you have already set the blank position after aligning the 0306 position on sample holder)

1. Click the right ">" on GICC panel to open macro editor. Once macro editor is open, clicking on GICC panel converts to command in the editor.
2. Click "New" on top bar in macro editor.
3. Select appropriate configuration, click Go, appropriate beam stop, click Go, click fine tune beamstop.
4. Select the correct position on the sample holder, click Go.
5. In the measurement detail, add sample description, measurement time in seconds, click Measure.
6. Repeat step 4 and 5 for all the positions on the sample holder.
7. Save the macro written in GICC panel in your personal Ganesha user folder.
8. Click "Run" to start automated data collection.
9. In case you want to collect data for different detector configuration, repeat from step 1 onwards.

8. Finishing experiment

It is your responsibility to retrieve your samples promptly after your measurements are completed. Unless you have made other arrangements with instrument incharge, you will be responsible for instrument charges as long as your samples remain in the vacuum chamber. Completely remove tape or other adhesives from the sample holder. Do not remove any calibration samples such as AgBeh.

The procedure for unloading samples is as follows.

1. In the GICC window, press Vent. Wait for venting to complete (a little over 1 minute).
2. Open the vacuum chamber door. With your left hand, loosen the two screws which hold the sample holder onto the sample stage by about $\frac{1}{2}$ turn each. With your left hand, slide the sample stage out and place it in the sample stage cabinet.
3. Close and latch the vacuum chamber door. In the GICC window, press Evacuate.
4. In SPEC window, power down X-ray generator to standby setting using command '**>x_standby**'
5. Return the clean sample holder to the cabinet.

9. Troubleshooting tips

- a. Detector does not respond

Connect the detector through internet. IP address 172.17.1.2.

Go to *Systems setting--- Eiger--- test---Image capture test.*

If still does not work, *DCU control--restart DAQ.*

If still does not work--*reboot.*

- b. Some useful passwords

Ubuntu computer:

Username: User

Password: saxsuminn

Eiger detector:

Username: eiger

Password: #EIGER_Detector#

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 and start measuring

Optional: graphic based sample positioning

Optional: additional controls for GISAS and heating stages

Optional: programming multiple measurements

The screenshot shows the 'Genie Interactive Control Center' software. It features a 'Current Holder' window with a grid of sample positions. The main interface includes sections for 'Configuration', 'Placement' (with horizontal and vertical coordinates), 'Measurement Details' (with transmission, mask, and thickness settings), and 'Direct Control' (with shutter and blank position controls). A 'Measure' button is visible. An inset graph shows a plot of 'Point 20.1' intensity versus '2θ (mm 2θ.1)' with a peak at approximately 0.1.

Remember CharFac is a user facility, and we all need to do our part to keep the work area clean!