SAXS2 Operating Procedure
GI-SAXS, GI-WAXS & GI-MAXS Measurements
# SAXS Labs Ganesha

Ensure System Ready and Load Sample:

1. Make sure detector is on (APC power-strip at bottom-right of instrument)
2. Ensure SAXSLAB Program and Windows are open (check workspaces 1-4)
   a. Otherwise, double click the icon to reopen all of them
   b. If asked by Spec for pin diode gain setting, enter **>100**
   c. For SAXSGUI Prompt **->Data->Latest** , then scroll to last image, select it and click **Open**
3. Ensure X-ray tube and generator are on by checking that the yellow light at top of instrument is lit. If off, x-ray needs conditioning:
   a. In Spec **>x_start**, generator will then ramp x-ray tube's kV and uA to operating conditions
4. If needed, turn chamber light on via remote on top of chamber or **>light_on**
5. **>saxsconnect** to initiate detector computer and ready system for measurements
6. System should be ready for use, continue onto "Load Sample" below

Loading Sample

7. In Spec **>vent_system**
   a. Detector moves back & system will vent in ~5mins
8. Prior to mounting samples and placing them into the chamber, double check SMIF's protocol for SAXS samples and ensure your samples' properties in vacuum
   a. **Samples must be safe and secure down to 10^{-2}mbar**
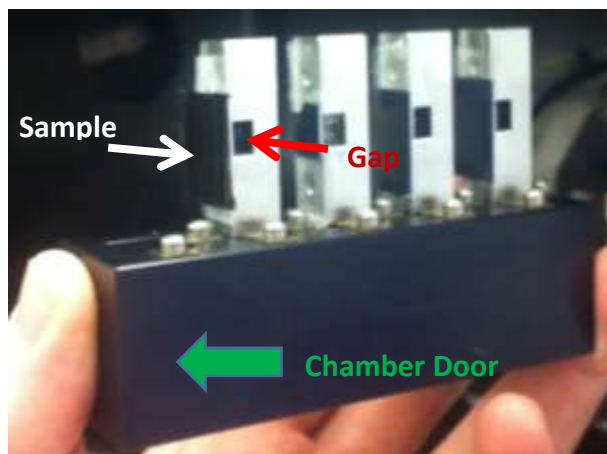
9. Mount samples on GI-SAXS stage, configuring samples as seen in above image
   a. Make sure that upon inserting stage into chamber, the intended surface of incidence is facing the chamber door
10. Close & lock chamber door (securing all 3 levers appropriately)
11. Give audible warning to fellow users, then **>evacuate_system**

12. Chamber will evacuate and a vacuum of 10^{-2}mbar will be reached in ~5mins
13. System is ready when ">**sleeping** t" in spec disappears and leaves the prompt, **####.Spec>**

Sample Measurement for GI-SAXS, GI-MAXS, GI-WAXS

1. >**mv thsam 0.2** to increase the area of incidence so cross hairs can land on sample's surface
2. Move to 1st sample of interest and place cross-hairs on surface using >**mvr ysam** y or >**mvr zsam** z commands
   a. Make sure you've placed the cross hairs on the middle of the sample face to your best approximation – slight discrepancies can lengthen or obstruct the following steps
3. >**qdo /usr/local/lib/spec.d/gisaxs_lineup_macro.mac**
   a. System will then scan through thsam and ysam values until it locates both the critical angle and position of grazing incidence
   b. This can take 10-15 minutes
   c. For manual alignment, please reference Appendix A
4. After the macro finishes, you'll want to **plot the 4 resultant images** by clicking **Next File** in SAXSGUI. **How to**:
   a. Find 0.2 theta scatter image, it's labeled in the scatter image header
   b. Click **Prev File**
   c. Close plot window
   d. Click **Next File** until 0.8 theta appears
   e. Plot window should have 4 resultant profiles
5. Double **check system's theta coordinate** (thsam) to make sure it coincides with measured theta. **How to**:
   a. Hover cursor on 0.2 peak and record "2theta" readout at top right of plot onto paper (see image)
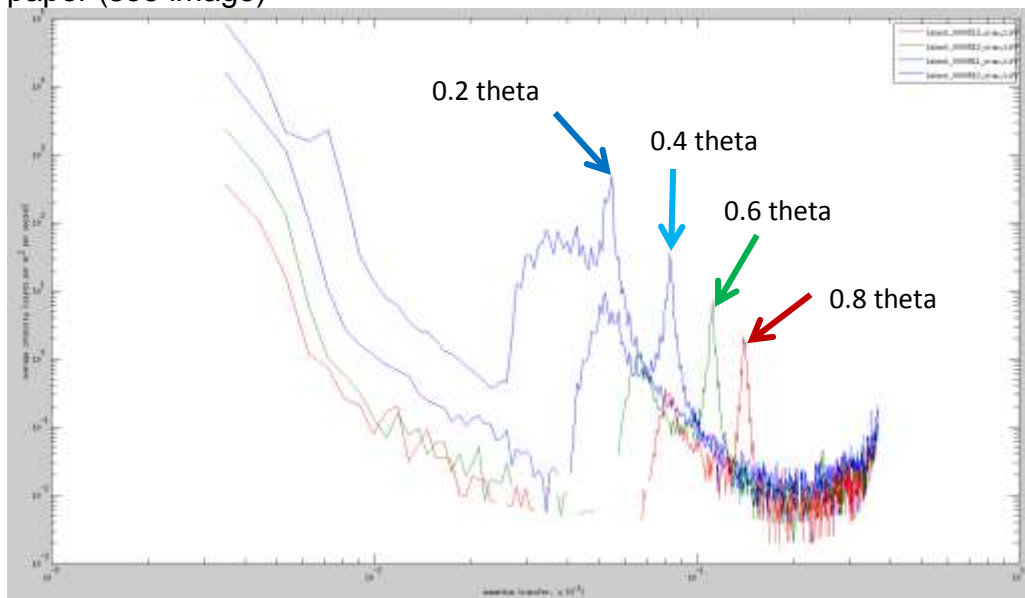
   b. Record 2thetas for 0.4, 0.6 and 0.8 max peaks in same way
   c. Divide 2thetas by 2 and compare result to respective thsam coordinate

      d. i.e. (thsam) – (2theta/2) = difference in set vs. measured

      e. Find average deviation of these comparisons and add/subtract deviation from set coordinate

- i. **>mv thsam 0.2**
- ii. add/subtract average deviation to/from 0.2 = real thsam
- iii. **>set thsam *real thsam***
- iv. **>mv thsam n** , "n" is the degree of reflection you want to measure

6. **>mv detx x** ,"x" is an appropriate coordinate for the detector representing whether you want to do GI-SAXS, GI-WAXS, GI-MAXS or something in between these options

    a. See spread sheet above monitor on control computer desktop for common *detx* values

7. Adjust slits to set appropriate beam footprint on sample:

    a. For a large beam, try:

- i. **>mv hg1 0.7** , **>mv vg1 0.7** , **>mv hg2 2** , **>mv vg2 2** , **>mv hg3 0.1** , **>mv vg3 0.9**

    b. For a small beam, try:

- i. **>mv hg1 0.3** , **>mv vg1 0.5** , **>mv hg2 2** , **>mv vg2 2** , **>mv hg3 0.1** , **>mv vg3 0.3**

    c. Any of the slit spacings can be varied to optimize resolution vs. intensity

- i. The 1st slit is commonly set to "shape" the beam footprint while the 2nd and 3rd slits control scatter off of the previous slits
- ii. Note, in the above examples, slit 2 (hg2 and vg2 are set to 2 which is wide open)
- iii. Note also, the above examples produce a rectangular/asymmetric beam where the horizontal dimension of the beam is narrow and the vertical dimension is long. For symmetry, just set the vg components equal to the hg components above (you want to keep *hg* narrow, otherwise you will produce a lot of smearing or blurred reflection patterns

8. **>mvr ysam 2** to move sample out of beam path

9. **>blankpos_def** to define the position as a blank position

    a. if at any time you want to go back to the blank position, **>mv_blankpos**

10. Load appropriate beam-stop configuration (if known or needed)

    a. **>change_bstop_conf** *3* for example

    b. Not a commonly needed step

11. When prompted again, **>mv_beam2bstop**, 3-4 times
    a. **>saxsmeasure 30** and see if 0th mode of beam is covered by beam stop such as below image. If not, then repeat step 11 until beam stop is placed correctly and evenly over the 0th mode
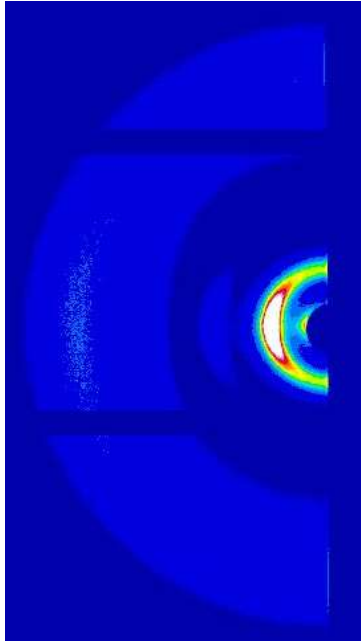
12. **>mvr ysam -2** to move sample back into beam path
13. **>saxsmeasure 30** and check resultant image to ensure beam stop is in correct place and forecast needed collection time for measurement
    a. If the image does not look good, consult a SMIF staff member or repeat procedure from step 2
14. **>SAMPLE_DESCRIPTION="** *relevant information pertaining to sample & run*"
15. **>saxsmeasure** *t*, "t" is the time in seconds needed to make an accurate measurement
16. **Note image number to be able to find data later**
    a. i.e. "im_**0000685**_craw.tiff" or "im_000068_caz.tiff"
17. In SAXSGUI, data can be monitored by selecting **Next File** or by going to **File -> Open Latest (cont)**
18. When measurement finishes, another measurement can be done by repeating from step 2 and moving to next sample. The critical angle will have to be found again for the new sample and therefore thsam will have to be recalibrated
19. Data is saved in the saxsgui directory in the folder "**Data**" which can be reached via the desktop folder labeled, "**Link to Data**"
    a. Images from measurement are saved in **3 directories** w/ 2 ("images" & "latest") having raw and corrected sub-directories. Sub-directories contain "Corrected data" where the Dark current/Dark counts of the images is/are removed
        i. **Latest** – real-time *live* sum of all short measurements, equals Images equivalent when measurement finishes
        ii. **Images** – end product after measurement finishes

        iii. **Frames** – zipped folder of all 15 second frames collected during measurement associated with specific image number

    b. If SAMPLE_DESCPTION was used, its information is saved in the header of the tiff and is readable by most SAXS viewing software in addition to other important properties

20. **To export data:** in saxsgui image viewer **->Processing->AutoProcess->AP From MetaData** and then select ALL images you want auto processed - **Open**

    a. In next window, assign a proper folder for data to export to, i.e. create a "data" folder within your user folder

    b. Select it

    c. Click **OK**

    d. Data should be processed and sent to the folder you assigned

Note: Multiple angles of reflection can be automated for measurement by using the "gisaxs_example.mac" macro located on the desktop or below in Appendix B. Make sure to "Save As" into your folder and rename with a date so the original template on the desktop is not corrupted. Unfortunately, there is no way to automate multiple samples for grazing incidence (GI) studies at this time.

System Stand-By and Removal of Samples

1. In Spec, **>mv thsam 0**
2. **>x_off**
3. **>saxsdisconnect**
4. **>vent_system**, and unlatch the two left latches
5. After waiting for chamber to come to atmosphere, carefully remove samples from instrument
6. Close chamber door securing all three latches
7. **>light_off**
8. Give audible warning, **>evacuate_system** and also ensure that the chamber door is closed tight

Appendix A: Manual Alignment Tools

In the event that you wish to have more hands on during the experiment, the following commands may be useful:

- **>change_bstop_conf** *3*
    - o Chooses the beam-stop configuration to your selection next time you use conf_ugo or mv_beam2bstop
- **>pilatus_narrowbeam_dscan** *motor start stop steps time*
    - o This is like a normal dscan, except that the intensity on the Pilatus detector is used instead of the pin-diode by only using counts found in a small region around the central portion of the beam found on the detector face
- **>p get_halfpoint(pilint**)

- o Prints the motor value for which the intensity is half the maximum for from doing an edge scan, where the counts are taken from the Pilatus detector image. Edge scans are essential for aligning the sample in the beam.
- **>p get_peak(pilint)**
  - o Prints the motor value for which the scan has a peak value with the counts taken from the Pilatus detector.

Appendix B: GI-SAXS Multi-Angle &/or Multi-Configuration Macro

```
# to run >qdo /home/saxslab/Users/yourname/macros/gisaxstemplatename.mac
# in order to make the macro work it must have ".mac" at the end of its filename
# also make sure to reflect its directory listing appropriate when entering the above command

# this macro assumes the sample has been aligned (sitting at half-beam intensity with
# thsam=0 being parallel to the beam) or rather, the gisaxs_lineup.mac macro has been run

# Choose setting of hg1, and hg3 that give you a desirable beamsize and angular resolution in the
# horizontal plane

# Choose settings of vg1 and vg3 that give you a desirable beamsize in the angular resolution in the
# vertical plane

# Feel free to comment out or add sections as needed for GISAXS, GIWAXS &/or GIWAXS
# measurements


change_bstop_conf 3   #set desired beam stop configuration

#GIWAXS

mv detx 0     # detector is all the way forward usually for GI-WAXS, can be set differently if needed
mvr ysam 2   # move sample out of way for beam stop alignment
mv_beam2bstop  # beam stop alignment
mv_beam2bstop
mvr ysam -2  # move sample back into position

sampledesc="test_sample"   #whatever the name of your sample is or other pertinent information

# loops through angles from 0.1deg to 0.5deg in 0.1deg increments for 900s measurements on each
# increment

for (icnt=0;icnt<5;icnt++) {
        myangle=icnt*0.1+0.1
        mv thsam myangle
        mytime=900
        SAMPLE_DESCRIPTION=sprintf("%s in GIWAXS at thsam=%5.3f,
for %i s",sampledesc,myangle,mytime)
        saxsmeasure mytime
}
```

#GISAXS

```
mv detx 950  # detector is almost all the way back for GI-SAXS, can be set differently if needed
mvr ysam 2   # move sample out of way for beam stop alignment
mv_beam2bstop  # beam stop alignment
mv_beam2bstop
sampledesc="test_sample"  #whatever the name of your sample is or other pertinent information
mvr ysam -2  # move sample back into position

# loops through angles from 0.1deg to .5deg in 0.1deg increments for 3600s measurements on each
# increment

for (icnt=0;icnt<5;icnt++) {
        myangle=icnt*0.1+0.1
        mv thsam myangle
        mytime=3600
        SAMPLE_DESCRIPTION=sprintf("%s in GISAXS at thsam=%5.3f,
for %i s",sampledesc,myangle,mytime)
        saxsmeasure mytime
}
```