SAXS2 Operating Procedure
SAXS, WAXS & 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 tube & generator need conditioning:
   a. In Spec **>x_start**, generator will then ramp x-ray tube & generator'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
   b. Unlatch two left door latches so chamber doesn't build back pressure
8. Prior to placing samples into chamber, double check safety of all materials in chamber & their reactions to vacuum via SMIF's protocol for SAXS samples
   a. Volatile samples must be encased in container that is sealed to pressures below 10^{-2} mbar
   b. Powder samples must be tightly sealed
   c. Ask SMIF staff if at all unsure of sample's reactions to vacuum
9. Open chamber door and place sample in according to prior instruction from SMIF staff
   a. Note stage and sample orientation – chamber cam is disorienting
      i. Best practice is to place a portion of sticky note as a landmark
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 **####.Spec>** prompt

Sample Measurement for SAXS, WAXS or MAXS

14. In Spec **>conf_ugo** *N* , where "N" is the configuration number you'd like to use
    a. See GANESHA Configuration Overview spreadsheet on desktop or wall
    b. This will decide if you do SAXS, WAXS or MAXS
15. Detector and Slits will move into position

16. Move sample into position with reference to cross-hairs in RayCam by using **mvr** command in Spec.
    a. example: **>mvr ysam 10** will move stage relative to current position horizontally in the positive direction 10mm
    b. example2: **>mvr zsam 10** will move the stage relative to current position vertically in the positive direction 10mm
17. Ensure that the only object in the beam path (in cross-hairs) between the source and detector is your sample i.e. no tape
    a. Stay away from sample edges and potential adhesives such as tape on your sample – all are potential scatter prone materials which will skew results
18. For capillaries or other samples requiring precise alignment, please follow these sub-steps:
    a. Make sure sample is coarsely positioned under cross-hairs
    b. **>capalign zsam 2 40** which scans the capillary across 2mm in 40 intervals each lasting 1 second and moves to middle of capillary automatically
       OR
       >dscan ysam -3 3 12 1 which scans from -3mm to 3mm relative to your current position in 12 segments with 1 second at each segment. The "spec 1" plot that appears yields intensity vs. ysam position. Move to desired position accordingly
19. **>wu** and record positions for **ysam** and **zsam** from table listing
    a. Check that **thsam** is set at **0**, if it's not **>mv thsam 0**
20. Move to a blank position
    a. **>mvr ysam** or **zsam** *RelativePosition* where nothing lies under cross-hairs
    b. **>blankpos_def**
    c. **>mv_beam2bstop** twice
    d. **>wu** record coordinates of blank position (**ysam** & **zsam**)
21. Move back to sample
    a. **>mv zsam** or **ysam** *AbsoluteCoordinate* of what you measured and recorded during capalign, dscan or other methods
22. **>transmission_measure**, which then measures I_0 and the transmission which are stored as the Spec variables "I0" and "SAMPLE_TRANS" respectively
23. **>saxsmeasure 60** and analyze image in SAXSGUI after the 60 second collection to make sure that the beam spot is covering the 0th mode and positioned appropriately (see image(s) below for examples of good and bad beam stop alignment)
    a. Good Alignment
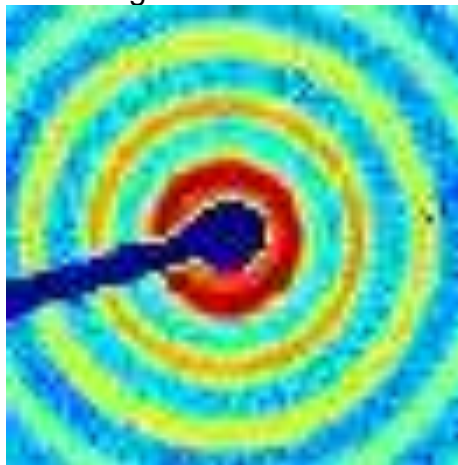
b. Bad Alignment
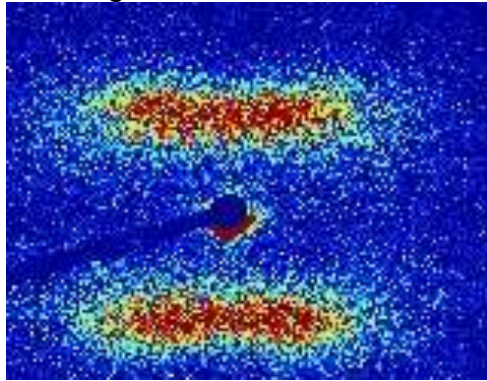
24. If the image and position of the beam stop look good – then continue to next step for measurement collection, otherwise
    a. Move back to blank position
        i. **>mv_blankpos**
    b. **>mv_beam2bstop** twice more
    c. Repeat from step 22
25. **>transmission_measure**
26. **>SAMPLE_DESCRIPTION="**_list name and information regarding measurement within apostrophes_**"** and this information will appear in the image header in SAXSGUI
27. **>SAMPLE_THICKNESS=**#.# in cm if known or needed for use as a reduction parameter later
    a. Standard capillary sample thickness is 0.1cm or **0.098cm** if wall thickness of capillary is subtracted
28. **>use_bsmask** to load configuration specific beam-stop mask
29. **Steps 25-28 are reset after each measurement, you must reenter them if you run a measurement more than once**
30. Double check that steps 1-27 are complete, then proceed to next step
31. **Note Image number** of collection, example: "im_**0000685_**craw.tiff" or "im_000068_caz.tiff"
32. **>saxsmeasure** _t_ where "_t_" is the time in seconds you would like to measure your sample
    OR
    >saxsmeasure _-c_ where "_c_" is the number of counts you would like to collect
    a. Experiment should be monitored in beginning to ensure measurement is not run for longer or shorter than reasonable
33. Instrument will begin measurement
34. Results can be monitored in SAXSGUI by clicking **Next File** or under the **File** menu by selecting **Continuous Monitoring**
35. When measurement finishes, another measurement can be done by simply repeating from **step 24** or if another sample or region need to be analyzed, by repeating from **step 16**
    a. If a new measurement needs to be made in another instrument configuration (SAXS, WAXS, MAXS,etc) then you will need to repeat from **step 14**
36. 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, use_bsmask, etc. were used, their information in addition to other important properties is saved in the header of the tiff and is readable by most SAXS viewing software

37. **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: For automation of multiple measurements, please reference Appendix A for guidance on using macros.

System Stand-By and Removal of Samples

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

Appendix A: Multiple Sample & Automated Experiment (Configure .txt Script)

When using a previous macro, make sure to open and "Save As" immediately to another file name and within your own directory so as not to mess up the original, working copy. Templates for macros can be found in the "Macros" folder located on the desktop.

For Capillaries

```
#capillary position holder list and rough alignment

ysampos2=24.4877
zsampos2=-4.8238
```

```
ysampos3=24.4877
zsampos3=8.1763


ysampos4=24.4877
zsampos4=21.1763


ysampos5=24.4877
zsampos5=34.1763


#ysampos6=24.3 #will not use position 6 b/c it is unreachable
#zsampos6=47.3002


ysamposblank=ysampos5 #position 5 will be our blank position
zsamposblank=zsampos5


#List sample names and associated slots
sample2="water 1"
sample3="water 2"
sample4="water 3"
sample5="blank"
#sample6="temp sensor"


#Fine tuning capillary holder positions using capalign macro
mv ysam ysampos2 #goes to the position
mv zsam zsampos2
capalign zsam 2 40 #calculates center of capillary
zsampos2=A[zsam] # reassigns position to found center


mv ysam ysampos3
mv zsam zsampos3
capalign zsam 2 40
zsampos3=A[zsam]


mv ysam ysampos4
mv zsam zsampos4
capalign zsam 2 40
zsampos4=A[zsam]


#mv ysam ysampos5 # don't need to capalign blanks
#mv zsam zsampos5
#capalign zsam 2 40
#zsampos5=A[zsam]


#mv ysam ysampos6
#mv zsam zsampos6
#capalign zsam 2 40
#zsampos6=A[zsam]



#moving to blank

mv ysam ysamposblank
mv zsam zsamposblank
blankpos_def #stores position as the blank position
```

```
#Configure use of Julabo Cool Stage
#julabo_pt100_control #setting external sensor control
#julabo_stabilize 10 3600 #setting temperature
#do_sleep(1800) # equilibration time after having reached temp

#automating measurement collection
#the "for" loop below is for going b/w multiple configurations
#(waxs,maxs,saxs)
#consult with SMIF staff if you would like to measure multiple
#positions instead of multiple configurations, or do both

for (mycnt=22;mycnt<23;mycnt++) {
      myconf=mycnt
      mysaxstime=10600 #number of seconds to collect each sample
      conf_ugo myconf #move to configuration
      mv_blankpos #move to blank position
      mv_beam2bstop #center beam on beam stop

      #water 1
      mv ysam ysampos2
      mv zsam zsampos2
      transmission_measure #record I0
      use_bsmask
      #SAMPLE_THICKNESS=0.098  #always in cm
      SAMPLE_DESCRIPTION=sprintf("%s in Conf %i, T=%5.3f,
I0=%i",sample2,myconf,SAMPLE_TRANS,I0)
      saxsmeasure mysaxstime

      #water 2
      mv ysam ysampos3
      mv zsam zsampos3
      transmission_measure
      use_bsmask
      #SAMPLE_THICKNESS=0.098  #always in cm
      SAMPLE_DESCRIPTION=sprintf("%s in Conf %i, T=%5.3f,
I0=%i",sample2,myconf,SAMPLE_TRANS,I0)
      saxsmeasure mysaxstime

      #water 1
      mv ysam ysampos4
      mv zsam zsampos4
      transmission_measure
      use_bsmask
      #SAMPLE_THICKNESS=0.098  #always in cm
      SAMPLE_DESCRIPTION=sprintf("%s in Conf %i, T=%5.3f,
I0=%i",sample2,myconf,SAMPLE_TRANS,I0)
      saxsmeasure mysaxstime
      }
```

Make sure to save your macro with ".mac" at the end of its name and also note
its directory location

To execute the above macro, in Spec

>qdo /home/saxslab/Users/*YourDirectory***/***YourMacroName***.mac**

For Stage Mounted Samples

First note all sample positions on stage, then use "For Capillaries" macro directions above and delete the section under "#Fine tuning capillary holder positions..." while also replacing values in "Capillary position list . . ." with your samples ysam, zsam and thsam positions.

Appendix B: Using Cool Stage in Experiments (Julabo Cool Stage)

1. Ensure Julabo Cool Bath controller is on and set to external control
2. In Spec, **>julabo_start**
   a. Controller should turn-on and stage should begin to cool
3. **>julabo_stabilize 10 3600** where "10" is the degrees in temperature and "3600" is the time in seconds it is allowed to take to equalize at that temperature before quitting
   a. Remember, stage temp may take slightly longer to stabilize after bath has reached assigned temperature
   b. Temp limits for bath are $-95^0C – 200^0C$ with temperatures below 0^0C and above 100^0C needing different fluid than currently loaded water (consult SMIF Staff)