### Alignment and Data Collection for GISAXS Users: Pilatus 1M-F and 11 keV

Change the sample, search and interlock the hutch

1. Make the name of the spec datafile appropriate for your sample: Spec> newfile *filename*
2. Spec> go\_apd
3. Vent the sample chamber
4. Change the sample wheel ,
5. Pump the sample chamber,
6. Search and interlock the hutch
7. Open the photon shutter
8. Adjust the sample height until the sample cuts the beam20-80%: Spec> umvr samz *[increment in mm]*
9. [If it hasn’t been done recently, run the tth alignment macro: Spec> pind3zlup ]
10. Select the desired sample (nextsam, prevsam, gosam *n* , reportsam )
11. Spec> zcen
12. Spec> thcen
13. If scan looks ok, reset the zero value: Spec> set th 0
14. Repeat steps (11), (12), (13) as needed
15. Look at a reflection: Spec> an 0.3 0.15
16. (Optional) May want to decrease attenuation: Spec> att *[att value]*
17. Spec> lup th -0.05 0.05 20 0.2
18. Spec> umv th CEN
19. Spec> set th 0.15
20. Spec> an 0.16 0.08
21. Adjust the attenuation to get APD readout to 50-80K: Spec> att *[14]*
22. Run reflectivity macro. Example:Spec> refqc *or*  Spec> th2th 0.08 0.24 64 0.5
23. Choose the incident angle you want for gisaxs (Spec: umv th 0.14)
24. Spec> go\_ccd
25. Update the Filename and File # fields on the Pilatus GUI
26. [Take a test image, note the maximum counts, Pilatus can count up to 106 counts] Spec> pilexp *[time in seconds]*
27. Run a gisaxs macro, if you are using one. For example: Spec> thseries *[time in seconds]*
28. Do it again with detector in down positon (Spec> pildown)
29. Change to next sample and do it again

GIWAXS with vacuum GIXS chamber

1. Spec> attin (put in attenuation)
2. [optional] Make the name of the spec datafile appropriate for your sample: Spec> newfile *filename.spec*
3. PUT ON HEARING PROTECTON BEFORE ENTERING HUTCH
4. Cover the Pilatus and then vent the sample chamber
5. Change the sample wheel ,
6. Pump the sample chamber, then uncover the Pilatus
7. search and interlock the hutch
8. Open the photon shutter
9. Spec> pilct (puts Pilatus in Alignment mode, starts taking 1 sec exposures, watch counts in Stats1:Total)
10. { *optional:* Spec> pilstop stops the Pilatus and takes it out of Alignment mode, if desired) }
11. Spec> bstopxout (take beamstop out)
12. Select the sample watching the view in the web camera. Useful commands are
    1. Spec> nextsam [advances from sample n to sample n+1]
    2. Spec> prevsam [returns from sample n to sample n-1 ]
    3. Spec> gosam *n* [move to sample *n* ]
    4. Spec> reportsam [Spec reports which sample is in position]
13. Spec> umv th 0
14. Change height of sample to cut 20-80% of beam: Spec> umvr samz *increment*
15. Spec> zpil (scans and moves samz to correct height to cut beam in half)
16. Spec> thpil (scant th in the direct beam, moves th to PEAK value.)
17. Inspect: if the scan captured the peak, Spec> set th 0
18. Iterate 15-17 as necessary
19. Spec> bstopxin (position the giwaxs beamstop – should go to zero)
20. Go to the desired incident angle: Spec> umv th 0.14 [ θc(Si, 11 keV = 0.166°]
21. Update Filename and File# on Pilatus interface
22. TAKE A 1 sec EXPOPSURE ON THE PILATUS WITH attin – verify that beamstop is in position: Spec> pilexp 1 (max counts should be in single digits. If not, remove attenuation gradually and take more test exposures: Spec> att 10; pilexp 1 )
23. Spec> att 0 (remove attenuators)
24. Spec> pilexp 1 (take 1 sec exposure, inspect max counts on STATS 2)
25. Take additional exposures at desired incident angles
26. [optional] If you want to take data on a fresh part of the sample, use the vsamx motor. Do NOT use the samx motor (moves sample AND beamstop!) Spec> umvr vsamx *increment*
27. Spec> pildown (move Pilatus to the ‘down’ position)
28. Repeat the exposures to have data to fill in gaps if desired
29. Spec> pilup
30. Go back to step 9 and repeat procedures for the other samples (macro nextgiwaxs does steps 9-13)