

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 beam 20-80%: 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 10^6 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 position (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
 - a. Spec> nextsam [advances from sample n to sample n+1]
 - b. Spec> prevsam [returns from sample n to sample n-1]
 - c. Spec> gosam *n* [move to sample *n*]
 - d. 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 [$\theta_c(\text{Si}, 11 \text{ keV} = 0.166^\circ)$]
- 21) Update Filename and File# on Pilatus interface
- 22) TAKE A 1 sec EXPOSURE 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)