



Rev 2006.02.13

Define Region

1. [Shutter closed] "Regions/File"; select appropriate region filename from list

Tune Mono

2. [Shutter closed] Determine energy at end of scan region ("Mono/Move/Region Summary")
3. If necessary, move to this energy ("Mono/Move/Move Absolute")
4. Check that the monochromator is fully tuned (XASUTILS | MONO Tuning)

Change Sample (also see RAS Patented Sample Change Procedure)

5. Open He valve on black box; assure He is exiting relief valve at cryostat
6. Open sample space valve (on top of cryostat)
7. Remove old sample carefully and replace sample rod with dummy rod
8. Replace sample with new one, chill, put sample rod back into cryostat, and install clamp
9. PUT OLD SAMPLE BACK IN STORAGE DEWAR!!
10. Pump/flush with He for 5 cycles; leave He flowing out relief valve
11. Close sample space valve (on top of cryostat)
12. Close He valve on black box

Align Sample

13. [Al attenuator in; shutter open] Align sample vertically ("Motors/Scan Motor"): Scan motor CRYOVERT and scan detector I1 from (absolute) _____ to _____ mm (_____ pts). "Accept Position" in center and record on data log form. Align sample horizontally: Scan motor CRYOHOR and scan detector I1 from (absolute) _____ to _____ mm (_____ pts). Record range of sample area on data log form and set CRYOVERT and CRYOHOR to initial spot. [Shutter closed; Al attenuator out]

Set Up Collection

14. Replace calibration foil between I₁ and I₂ ionization chambers. Replace fluorescent filter
15. [Al attenuator in; shutter open] Reset SCAs on fluor. detector for new K_α using GE30 program
16. Recollect dark currents (beam off) as offsets for all counters ("Detectors/Current/Offsets")

Adjust Detector

17. [Al attenuator out; shutter open] Adjust horizontal position of detector (in/out) using "Motors/Move Motor", move DETHOR, until highest ICR (in GE30, use COUNTS button) is ca 110 kcps (0.125 μs shaping time). Sometimes this is not possible; choose optimal position [Shutter closed]

Start Collection

18. "Run/Start Run"
19. Fill in Output File (5-character filename), No. Sweeps, Comments
20. [Shutter open; Al attenuator out] "Apply/Start Run"...at end of run, shutter closed

Realign Beamline

21. [Shutter closed] Open hutch slits ("Motors/Move Motor", move S1VGAP to 10 mm)
22. Tune monochromator (XASUTILS | MONO Tuning). Scan M1VERT -2 to +2 (0.1-mm steps); optimize I₀.
23. Close hutch slits (S1VGAP to 1 mm) and center experiment on beam: TABLEVERT -2 to +2 (0.1-mm steps; "Scan against backlash" OK); optimize I₀; TABLEHORZ +3 to -3 (0.1-mm steps), optimize I₀. In each case, center cursor in plateau, then "Accept Position"