

XRD Quick Reference Guide

- 1 Mounting your sample
 - a. **If your sample is a powder** – use the 2-piece powder sample holders. Metal inserts can be used to fill the window if you do not have enough material. Compact and level the powder as best as possible to create a flat sample surface.
 - i. If you have a very small amount of powder mount your sample with double sided tape to a glass slide and follow (c).
 - b. **If you have a pellet or other solid sample** – use the clay sample holders. Mount your sample using modeling clay and press till flush with the sample holder surface. Glass disks can be used to keep your sample separate from the clay if necessary.
- 2 Running a scan
 - a. Check that the system is not in use – the data collector software is closed and the instrument is set to 45 kV and 20 mA.
 - b. Check the shutter indicator – if safe, open the doors and place your sample on top position of the sample changer.
 - c. Close the doors and make sure the shutter indicator returns to ‘safe.’
 - d. Log in to the data collector software with your name, email, PTA, and software password.
 - e. In the software, select “Instrument” > “Connect.” Make sure “New Spinner Stage (2013)” and “Diffracted beam path2” are selected.
 - f. All notices should be yellow – if there is a red warning, contact the manager.
 - g. Set X-ray tube current by double clicking “Current = 20 mA” > change 20 to 40 mA > “OK”
 - h. Load the sample by selecting “Status = There is no sample loaded” > “Load from magazine position: 1” > “OK”
 - i. If you are making a new measurement program, go to “File” > “Open Program”
 - i. Modify an existing program to your needs – suggest using “_Basic Scan_”
 - ii. Save your modified program – “File” > “Save As...”
 - j. To run the program, select “Measure” > “Program”
 - i. Find and select your preferred scan program
 - ii. Make sure your data is saved to the appropriate directory with a useful file name
 - k. When done, unload your sample if necessary (if not automatically unloaded by the program) and change tube current back to 20mA.
 - l. Exit the data collector.
- 3 Running a batch scan
 - a. Load all samples into magazine in appropriate locations – edit a batch program (suggest editing “_Basic Scan with Changer_” with correct scan programs and file names, saving as new batch program) – run batch program.
- 4 Accessing your data
 - a. Use HighScore to analyze your data and to export your scan as an .ASC file. Email to yourself as an attachment or use the ftp server at <ftp://xrd.caltech.edu> or via the “XRD Data” link at the top of xrd.caltech.edu.
 - i. Username: XRDuser
 - ii. Password: xrd

Note: This instrument is for polycrystalline samples *only*.

There is no mechanical latch holding the doors closed – avoid contact with the doors while the shutter is open.

