

**Steps for PXRD measurements:**

**Ctrl + C** command will stop whatever SPEC is doing. Use this command if you notice something wrong.

- umv tth 4 (move 2theta arm to 4 degrees).
- mname \_\_\_\_\_ (enter the file name. No spaces or special characters).
- newfile \_\_\_\_\_ (enter the file name again).
- opsh (open the shutter).
- ascan tth 3 80 154 2 (starts the scan).

When the scan is done

- spin end (stop capillary spinner. "spin 3" will start it spinning).
- umv tth 4 (start 2theta moving back to 4 degrees).
- review the merged file in PyMCA.
- load and align a new sample.

Some extra tips:

Take care not to bump the camera when loading the sample.

Align the sample: Watch the sample on the camera screen while rotating the spinner by hand. Center the sample by shifting the stub on the magnetic base. If necessary, remove the sample stub, reposition the capillary in the stub, and add it back on the magnetic base.

The spinning sample must be aligned so that the wobble is less than 0.5x the capillary diameter (Bergamaschi et al., *JSR*, **17**, 653 2010), or else the peak shape will change significantly. Spinner power supply voltage should be set to 3 V.

You can use the command mvr z 1 or -1 to position the sample inboard-outboard.

Open the individual frames in PyMCA as the scan is progressing. Check the signal level for your sample. The count rate per second should be less than 700,000 Hz. If it is greater, abort the scan (ctrl-C), and adjust the filter wheel/beam attenuation (filter1 X) to keep count rates in the linear range. Then restart the scan.

When the scan is complete, ensure that the data appears as a merged file, and review in PyMCA. To see the file. Open → file: 001\_mgd.xye

- Auto replace, choose the first file.
- Column 0 → X
- Column 1 → Y

A good Bragg Brentano scan is to start at th 1.5 tth 3. Th2th 0 67 134 2