QSG – HYSPEC Crystal Alignment Guide

Cheat sheet for aligning a crystal using a monochromatic neutron beam

1. If you're starting from scratch at HYSPEC, you're already too late!
    a. CG1 and x-ray Laue alignment stations available.
        i. X-ray Laue limitation: surface sensitive (vs. neutrons bulk sensitive)
        ii. CG1 at HFIR is only available when reactor is on
    b. Mounting sample
        i. Wrap in Al foil before applying any wax. Wax is hard to remove and scatters many neutrons.
        ii. We have a set of alignment jigs and tools that can be helpful aligning a single crystal.
        iii. Know dimensions and mounting interfaces for your sample environment
        iv. Mark faces with shiny sharpie pens
2. Anticipating Bragg reflections
    a. Know basics of crystal before you arrive
    b. Lower incident energy Ei restricts Bragg peaks to low HKL indices, reducing confusion
    c. Location in scattering angle
        i. ICSD website
        ii. HYSPEC has some planning tools (Crystal class and WhereIsBragg)
        iii. May need to move detector vessel to see desired scatter angle
    d. Expected intensity
        i. ICSD
        ii. Others are available.
3. Finding the Bragg peak
    a. Slow down sample rotation and start a run
    b. Hopefully you can eyeball the right angle to within a few degrees to start
        i. If needed, rotate ~180 degrees and stop when you observe desired peak
        ii. If not visible, tilt sample by ~6 deg on one tilt axis and repeat, because the Bragg peak may be way out of plane
        iii. Still not visible? Tilt even more or with other tilt axis
    c. Once you are close, set a Region of Interest, and scan rotation angle s1. Plot is generated with a ~Gaussian peak. Center of peak is where desired Bragg peak is.
        i. If the peak countrate is over ~300 c/s, you run the risk of saturating a detector tube. You may need an attenuator.

    d. Confirm another peak about 90 degrees away.
4. Orienting a crystal plane in the horizontal plane
    a. Set ‘mirror’ plane on tilt axis to avoid crosstalk
    b. Cubic crystals: easy to find 90 deg separation between Bragg peaks (e.g. (100) and (010)); good for orthogonal tilt axes
    c. Other crystals: look for roughly 90 deg Bragg peaks
5. Horizontal translation
    a. Not needed if sample environment well mounted and sample well mounted
    b. Increases flux.
    c. Like tilt, translation x and y are better done on orthogonal reflections
6. Reducing background
    a. Inelastic scattering: ~1E-4 of elastic scattering, so background can be very bad
    b. ToF background: elastic scatter away from the sample can look like energy gain or energy loss
    c. Tighten up the upstream apertures using scans!