

Sample Geometry and Size

Alfred Baron, 2023

For those fortunate enough to be able to choose the sample size and geometry, the following may be of interest. Please also read the advice for single crystals in any case (if you are measuring a single crystal).

Transmission/Laue Geometry:

A transmission or Laue geometry is usually the most flexible allowing access to the largest range of accessible momentum transfers. It can be the best geometry if you want very low momentum transfers. It is also generally less sensitive to surface preparation/quality. The ideal thickness for transmission is 1 attenuation length. Practically between 1 and 2 is usually OK. Much less than 1 or much more than 2 essentially wastes beam time. It is usually easiest if the transverse dimensions of the sample are ~1-5 mm. See also the note about the cryostat below.

Reflection/Bragg Geometry:

A reflection or Bragg geometry (for thick samples) generally can provide higher rates than a transmission geometry, but limits one to total momentum transfers, Q , that are not too far from the surface normal (n.b., little q , q , is *not* limited in the same way). If you know where (what Q) you want to measure it can be a very good choice. Note, however, that spectra can be sample surface sensitive and you need a larger flat surface - large enough to comfortably intercept the entire transverse beam size in all Bragg geometries of interest. Flat surfaces of ~2-5 mm length scale are easiest. Also, lightly etching the surface can remove strain and reduce elastic scattering in the phonon spectra. For samples with small lattice constants also consider how to access two alignment reflections (primary and secondary).

Chi & Sample Stability when using a Cryostat/Cryofurnace.

In general the spectrometer has a sphere of confusion of a few 10's of microns - sample positions when mounted in the center of the circles will tend to stay stable at that level. **However, if the cryostat or cryofurnace is mounted, these have a center of mass that is far outside the center of circles and when chi is changed by more than (say, roughly) ± 20 degrees, it is very easy for the sample position to shift by 0.1 mm (even 0.5 mm for chi ~ 90 degrees).** The position is motorized, and we can correct it, but the correction needs to be done (usually by hand) and can slow an experiment. And, of course, if for some reason your sample has a small (< 0.1 mm scale) transverse size, this can slow the experiment a lot if you need to work at larger chi-values.

Determining grazing angles onto the sample surface.

Diffraction codes usually can provide the x-ray beam grazing angles onto and off of the sample surface. For spec and related codes, set the sample normal vector (in h,k,l) using the "setaz" command and then alpha is the angle onto the sample surface and beta is the angle out of the sample surface into analyzer 6 (at BL43LXU). If alpha and beta are the same sign then you have a Bragg/reflection geometry, if they are different signs, a Laue/transmission geometry. Note that having alpha small (say $< \sim 3$ deg) can make alignment to the incident beam tricky while having beta small ($< \sim 3$ deg) can mean some analyzers may be blocked by the sample surface.