



RSXS diffraction alignment

The goal is to align θ , χ , and ϕ at two orientations of structural Bragg peaks. The approach will be different from one sample crystallographic symmetry to the other. Here is just an example with a cubic system and the out-of-plane (L) direction is normal to a sample surface and one of the in-plane (H or K) direction is set to the scattering plane.

Use following steps to prepare the instrument for diffraction scans:

1. Perform the sample alignment as described in “RSXS sample alignment” procedure.
2. All detectors can be used to detect structural Bragg peaks. The detector height position on the scattering plane and its relative $t\theta$ distance can be viewed with **statDET**. For better visualization, the MCP 2D area detector is preferred (**umv detz 0** and $t\theta$ offset (**set tth xxx + tth_offset of MCP**)).
3. Input the lattice parameter info (**setlat**).
4. Normally we set the FOURC mode to χ and ϕ fixed (**setmode 2**).
5. Searching for the (00L) peak at higher energy:
 - a. Calculate the motor position for a given 00L and energy (**ca 0 0 L**).
 - b. Move to that calculated position (either **ubr** or **uan**).
 - c. Monitoring the analog MCP 2D image by moving θ about $\pm 5^\circ$ (**umvr θ**) at every χ step of 3° (**umvr χ**) until finding a potential peak.
 - d. Optimizing the peak by aligning θ and $t\theta$ (**lup θ , lup $t\theta$, and $\theta 2\theta$**).
 - e. Set the θ as half of $t\theta$ (**set θ**).
 - f. Checking the HKL value at the peak position (**wh**). If needed, adjust the c lattice parameter (**setlat**) in order to match with the intended 00L.
 - g. Input the θ , $t\theta$, χ values for the first orientation (**setor0**).
6. Searching for the (H0L) peak at higher energy:
 - a. Calculate the motor position for a given H0L and energy (**ca H 0 L**).
 - b. Move to that calculated position (either **ubr** or **uan**).
 - c. Monitoring the analog MCP 2D image by moving θ about $\pm 5^\circ$ (**umvr θ**) at every ϕ step of 3° (**umvr ϕ**) until finding a potential peak.
 - d. Optimizing the peak by aligning θ and $t\theta$ (**lup θ , lup $t\theta$, and $\theta 2\theta$**).
 - e. Checking the HKL value at the peak position (**wh**). If needed, adjust the a and b lattice parameter (**setlat**) in order to match with the intended H0L.
 - f. Input the θ , $t\theta$, χ , and ϕ values for the second orientation (**setor1**).
 - g. Update also the ϕ value in the first orientation (**setor0**).
7. If χ and ϕ position are quite different from the normal χ and ϕ position at 90° and 0° , respectively, then it is good to verify x, y, and z position again.