

Notes on use of spec “sixc” at ID-34-C

1. Motor and variable names for six-circle angle calcs.

| | |
|------------|-------------|
| spec sixc | Super frz=6 |
| del | Tth |
| th | th |
| chi | chi |
| phi | phi |
| mu (=0) | alp (=0) |
| gam | gmm |
| Sigma | -fchi |
| Tau | -fphi |
| ALPHA | B (bet) |
| BETA | B2 (bet2) |
| setmode 10 | bem=1 alm=3 |
| setmode 11 | bem=2 alm=3 |
| setmode 9 | bem=3 alm=3 |

2. Setting the mode of sixc

Startup the first time with “sixc -f” to erase local modes of earlier users, such as no_hkl.

“setconfig” offers two options: z-inward should be YES; sigma/tau fundamental YES. The latter is probably the same as g_sigttau=1. If this is not set, the values of sigma and tau get overwritten by the “azimuth” vector, which is otherwise a derived quantity.

We don’t have a mu motor. Alm=3 mode of super corresponds to setmodes 9-11. The alpha=beta mode (setmode 9) requires the azimuth angle to be set to 90 (not tested)

“Sectors” are not yet implemented. Use “cuts” to determine the range of the motors calculated.

3. Setting the orientation matrix

LAMDBA=value sets the wavelength.

“setlat” allows the crystal lattice parameters (real space) to be entered.

“sigtau” allows the (negatives of) flat angles to be entered.

“or0” and “or1” enters the current angles.

“setor0” and “setor1” allow the angles to be entered as well.

4. Using sixc

“ca” and “cal” are not the same. “cal”, which updates the A[] array, appears to be the better choice.

ALPHA and BETA variables are used for both input and output, so they can get overwritten easily, for example when a “cal” is executed. The separation of B and BET, B2 and BET2 used in super has not been adopted. The correct way is to use the “freeze” command with ALPHA and BETA. There is mention of F_ALPHA in the manual but it appears to be hidden from the user.

In setmode 9, use “freeze azimuth mu”, entering the desired target values (mu=0 always).
In setmode 10, use “freeze alpha mu”, where alpha is the target for ALPHA
In setmode 11, use “freeze beta mu”, where beta is the target for BETA

The “azimuth” angle is the rotation about the surface normal vector, which is derived from the “flat” angles of the sample (sigma and tau). It corresponds roughly to the target value of theta. Mode 9 is good for finding reflections close to specular.

5. Crystal alignment on 34-ID-C using sixc.

The Microcontrole diffractometer with the Huber tower is designed for thin film and surface studies, and can be used for general crystallographic samples if the required tilt is not too much (± 10 on chi, ± 20 on phi). Usually the surface normal direction is known and some in-plane direction. It is assumed there is a flat face mounted roughly perpendicular to the main (theta) axis, which is the reference surface for ALPHA and BETA.

Step 0. Start the program.

Log in to hia as epix and go to the directory where the scan datafile is needed. Type “sixc”. The previous user’s setup will be recovered, including his/her orientation matrix. For a fresh start, type “sixc -f” and use “newfile” to open a datafile. It is no longer necessary to “look” after moving motors externally. “wa” tells you the motor positions. “config” edits the selection of motors, but motor parameters, including limits, are all managed by epix. “counters” edits the scaler selection.

Step 1. Level the sample and set the height.

This is done with the Tischler-cam viewing the beam grazing across the surface. Phi, chi and z-sample are adjusted until the reflected beam merges with the horizon at all theta’s. Type “sigtau” and accept the (negatives) of the current values for phi and chi as the “flat” angles.

Step 2. Enter a dummy orientation matrix.

This can be viewed by typing “pa”. With the 6-circle instrument there are two simple settings, with vertical and horizontal scattering planes. Conveniently, these correspond to the known crystallographic directions in the sample.

“setlat a b c al be ga” sets the real-space lattice parameters.
LAMBDA=value sets the wavelength.

“setor0 del th chi phi mu gam h k l” enters all parameters for the primary reference reflection; “setor1” does the second. Only the direction matters, so dummy values can be assumed in the 2:1 ratio for gam/phi and del/th:

for the surface normal: “setor0 0 180 90 10 0 20 h k l” using the normal h k l
Sixc requires that a pure phi rotation tilts the sample **away** from the beam at th=180 and the zero of th has to be set correctly for the angle calculations to work.

for the in-plane direction: “setor1 20 10 90 0 0 0 h k l” using the in-plane h k l
This assumes the in-plane direction (edge of the sample) was placed perpendicular to the beam at th=0. If another th was used, enter th+10 in place of the 10.

Step 3. Find some reflections.

Within a few degrees, it should be possible to find some reflections. The surface normal can be used, but it can be a problem because of the large tilt. It is usually better to find general reflections, neither in-plane (problem of refraction), nor specular (large tilt). General reflections near grazing incidence (“setmode 10”, “freeze 1 0”, so ALPHA=1) are easiest because the misalignment angle will usually be just th. Always calculate the position first with “ca h k l”, check it is reasonable, then use “ubr h k l”. Use the widest possible detector slits and search a few degrees in th. If this doesn’t work try a different reflection and come back later.

Three angles are enough to define the position in reciprocal space of a reflection. The usual choice is (th, gam, del). They can be peaked with “lup” and centered afterwards with “mv angle CEN”, since sixc sets CEN to the center of mass of the last scan. Spec does not do this automatically. When the first peak is found it should be entered as the secondary reflection by typing “or1 h k l”. This overwrites the less-well known of the dummy alignment reflections, so sixc doesn’t get lost finding the next one. The second reflection becomes “or0 h k l” and they can be swapped with “orswap”.