7ID-C Users Manual

Version 0.9

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Abstract

This is a user manual for doing diffraction experiments in the 7ID-C hutch of the XOR-7 insertion device beamline of the Advanced Photon Source. This hutch specializes in ultrafast pump-probe diffraction experiments. The equipment is briefly described and SPEC-based operation of the diffractometer is discussed.

1 Introduction

This document describes some of the aspects of using the 7ID-C station of the Advanced Photon Source. This hutch is dedicated toward time-resolved scattering, most often pump-probe diffraction using the 7ID Titanium:Sapphire laser, which is housed in the laser lab downstream of 7ID-D. This beamline has as its source an Undulator A and has a water-cooled diamond 111 double-crystal monochromator in the 7ID-A hutch.

The layout of the 7ID-C hutch, upstream to downstream along the beam, are three optical tables followed by a large Huber diffractometer, with a small breadboard on the downstream wall. The first, fixed optical table holds several x-ray-conditioning instruments. The second, motorized, optical table houses the 7ID streak camera, which is beyond the scope of this manual. The third optical table usually holds the KB mirrors and laser-delivery optics. The breadboard on the downstream wall often holds an ion chamber which is often used to find the ideal sample position (by adjusting the sample height to cut the beam in half).

Most operations at 7ID-C, such as motor motion, are controlled by EPICS. Diffractometer calculations are performed by SPEC, either with four circles in the "fourc" geometry or all six circles in the "psic" geometry; SPEC communicates with EPICS for diffractometer control. This brief manual will describe the important 7ID-C equipment, the psi-circle diffractometer, and control of the diffractometer via SPEC [1]. Information common to all SPEC geometries (such as setting up an orientation matrix) is generally not included. The psic and fourc help files in SPEC may provide additional help.

1.1 Safety

Safety is of utmost importance in everything we do at 7ID-C and all of APS. Observe and respect all safety rules and procedures, whether the hazard is x-ray, laser, chemical, electrical, or whatever.

1.2   Patch Panels

There are plenty of patch panels connecting the various optical tables and also traveling to the rack on the downstream wall and to the racks outside the hutch. If at all possible, *please* use these rather than run cables all over the place. If you do run extra cables, make sure they are not trip hazards; run them out of the way or use TredTrak.

1.3   Beampipe vacuum

2" aluminum or steel pipes with KF-50 flanges in a variety of lengths are used as flightpaths for the x-ray beam. "Permanent" pipes are set up for the incident beam and for the detector arm, and there is a third hose that can be used for other pipes. There is a manifold on the upstream wall, near the floor, that has the valves (and the vents) for each of these hoses. Pumping is done by a diaphragm pump outside of the hutch. The upstream beampipe should always be under vacuum when x rays are present, to protect the Be window at the upstream end of the hutch. Flanges with Kapton windows are available to cap the other end of the pipe.

1.4   Hutch coordinates

In general, we use the right-hand-rule with positive X pointing outboard (away from the accelerator), positive Y pointing up, and positive Z pointing downstream. (Of course, we don't use these rules for the sample on the Huber, since they rotate with the diffractometer circles.)

2   7ID-C Equipment

2.1   Detectors

A variety of detectors is used at 7ID-C, including scintillator detectors (not for time-resolved experiments), avalanche photodiodes (APDs), Vortex fluorescence detectors, and PILATUS pixelated area detectors. All of these have been mounted on the diffractometer at one point or another. The detector pool website has information on the various detectors.

2.2   Beam Position Monitor

First on the upstream optical table, under beamline vacuum, is a Beam Position Monitor (BPM). This homemade instrument consists of a thin titanium foil that fluoresces under x-ray exposure. Four diodes behind the foil absorb the fluorescence and produce three signals: the total voltage (a useful monitor of the beam intensity) and the horizontal and vertical positions, normalized to micrometers. The box that generates these signals (next to the BPM) should be at its second highest amplification for x rays below about 11-12 keV, and at the highest amplification above that. It's not very useful at higher energies.

The vertical position can be used to stabilize the beam by feeding it back into the second mono crystal's piezo through a PID loop. Usually this is pretty stable if the PID parameters are set appropriately.

2.3   JJ slits

The JJ-brand slits are fixed on the first optical table, immediately downstream of the BPM. These are high-quality slits used for defining the beam. The motion convention is that a positive motion moves each blade in the positive direction (as defined in Section 1.4), and are compatible with the normal slit medm screen.

2.4   Normalization detector

Immediately downstream of the JJ slits is an incident-beam monitor. Inside a KF cross is a Kapton foil at 45°, which scatters x rays into an APD. The signal from this APD can be used to normalize the incident beam intensity without being subject to loss of signal by attenuation (since it is upstream of the XIA filter box). The APD output can be gated to individual bunches, useful in timing modes. This is known as the "A" APD to distinguish it from the "B" APD on the diffractometer detector arm.

2.5   XIA filter boxes

XIA PF-4 filter boxes are available on the upstream optical table and on the detector arm for attenuating the x-ray beam. They are controlled by EPICS and there are SPEC macros available to operate them. Typically Cu or Mo foils are used, depending on the x-ray energy.

2.6   ADC Blue slits

A pair of blue slits from ADC are usually used on the diffractometer's detector arm to define the detector resolution, and another set is sometimes used to define the incident beam. Unfortunately, the motion convention is different than for the JJ slits: a positive blade motion opens the blade.

2.7   Chopper

The x-ray chopper can be used to isolate single x-ray bunches. It sits on the farthest upstream optical table. A full description requires far more detail than there is room for here.

2.8   Kirkpatrick-Baez mirrors

The Kirkpatrick-Baez (KB) system for focusing x rays involves two glancing-angle x-ray mirrors, each bent to reflect and focus in one direction. With an appropriately selected glancing angle, the KBs also serve as harmonic rejection.

The KB system at 7ID-C was manufactured by University of Chicago shops. The mirrors are dynamically bent into an elliptical shape. Each mirror is 200 mm long, with an actively bent length of 180 mm. Often, for pump-probe diffraction measurements we use only the horizontal mirror, because increased divergence from vertical focusing would obscure the tiny laser-induced shifts of Bragg peaks.

3   Laser and Timing

3.1   Laser specifications

The 7ID laser system is a standard kilohertz Ti:Sapphire amplified system. The oscillator is a Coherent Micra with a maxiumum bandwidth of 100 nm and an 88 MHz repetition rate. It is designed to be synchronized to the accelerator RF within 250 fs rms. The chirped-pulsed amplification (CPA) laser amplifier is pumped by a 30 W diode-pumped Nd:YLF (Coherent Evolution 30). The 800-nm Ti:Sapphire CPA regenerative amplifier (Coherent Legend) delivers a maximum 2.5 W of average power compressed to 50-60 fs. Pulse energies of 2.5 mJ/pulse at 1 kHz or 0.5 mJ/pulse at 5 kHz are possible, as well as conversion to higher harmonics using nonlinear crystals. The essential parameters of laser control and beam delivery are now controlled via an EPICSinterface. Fig. 1 shows a schematic timing diagram.
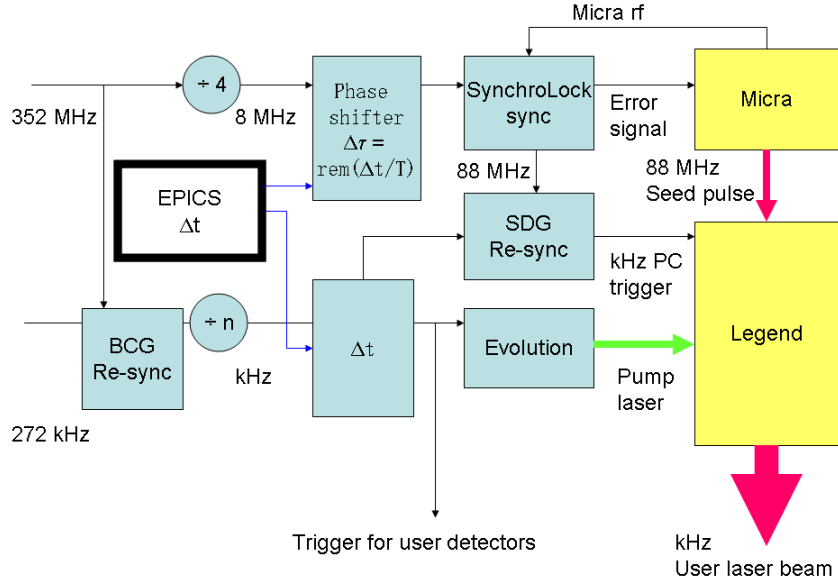
Figure 1: Timing diagram that summarizes the timing schematics of the 7ID laser system. The laser is phase-locked to the input RF signals from the APS accelerator. The laser/x-ray delay Δt is set by the user.

3.2 Timing scheme

For most pump/probe measurements at 7ID, gatable detectors are used to count only x rays from the desired bunches (sometimes the chopper is used to block all other bunches). When an APD is used as the detector in photon-counting mode, several gates can be set, as shown in Fig. 2. This allows counting from several bunches simultaneously. In 24-bunch mode, these bunches might be called "on" and "late." For the "on" channel, the gate is delayed (typically with an SRS delay generator) to observe the bunch that is supposed to coincide with the laser pump pulse. The "late" channel is delayed a further 153 ns to gate the next bunch in the accelerator. Further gates are set 3.682 μs earlier (i.e., one round trip of the accelerator) to measure the laser-off signals from the same bunches as was used for the "on" and "late" channels; these channels can be called "off" and "off-late." It's important to measure the off signal from each bunch, since the charge per bunch in the ring will vary (especially considering top-up). The gates for the B APD (on the detector
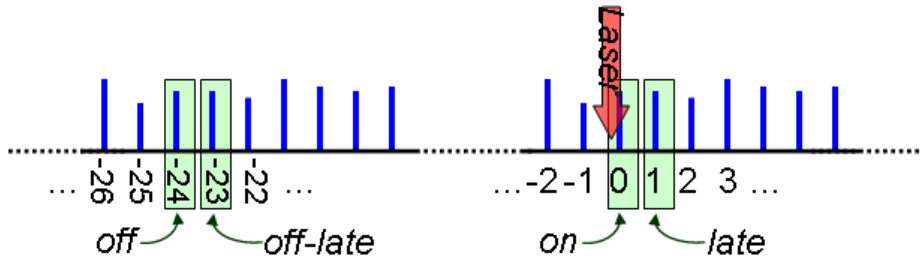

Figure 2: Sample gating scheme typically used for APDs at 7ID-C. Gates are set on several bunches to increase collection efficiency and to collect laser-off signals for normalization.

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arm) are triggered at the laser repetition rate, i.e., 1 or 5 kHz. The gates for the A APS (the normalization detector) can be triggered by P0 to count each bunch every time, for a significant improvement in statistics. The Pilatus can only accumulate one set of counts, so effectively it can only be gated to the "on" bunch. To observe the laser-off signal one has to change the laser delay. For APDs in current mode, one can measure the output signal on a scope trace and thus collect data from many bunches at once.

Once delays are set for gating the detector, they should be left alone. The pump-probe time difference is adjusted by delaying the laser relative to the x rays. This is done with electronics in the laser lab, by adjusting Δt in Fig. 1. Due to the physical motion that happens in delaying the RF signal to the laser, you shouldn't change the delay too fast (that is, give the delay line time to move before changing the delay again). Time zero, when the laser and x ray are coincident, is typically found with a fast pin diode or a sample that has large, fast response (e.g., InSb).
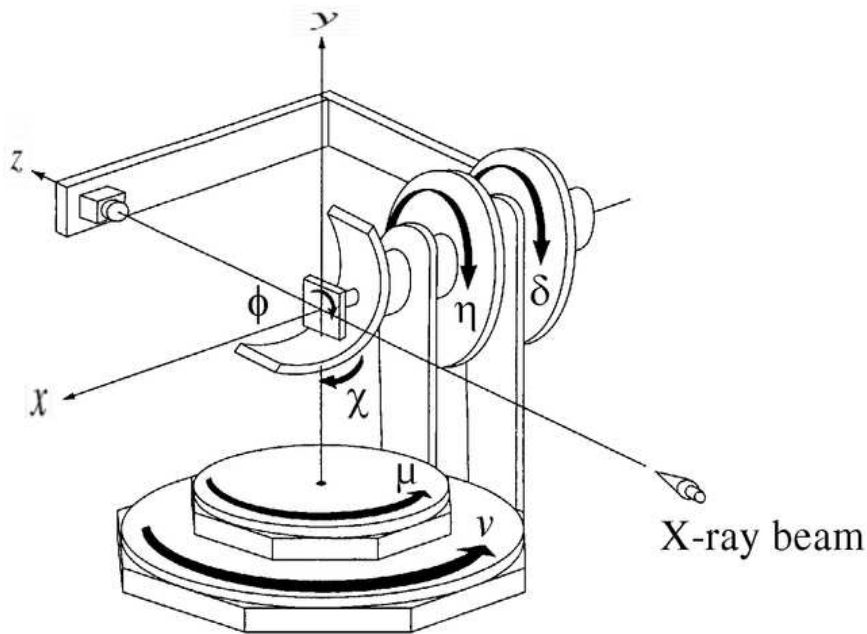

Figure 3: Basic definition of (Eulerian) psi-circle diffractometer angles, adapted from Ref. [2].

4   Huber diffractometer

The Huber "psi" diffractometer in 7ID-C has six motors for sample and detector orientation. A psi-circle diffractometer is also known as "4S+2D," after the four sample-orientation axes and two detector-orientation axes; the sample and detector move independently. (Such is not the case for the SPEC six-circle geometry.) The angle calculations for this diffractometer were worked out by Hoydoo You of MSD, Argonne [2]. Two SPEC geometry codes are available for controlling the diffractometer: fourc, to operate four circles (not moving the vertical axes), and psic, to operate all six circles. (There are actually several different geometry names, which are intended to make it easier to do either laser- or non-laser-based experiments without renaming or redefining a bunch of detectors and macros.)
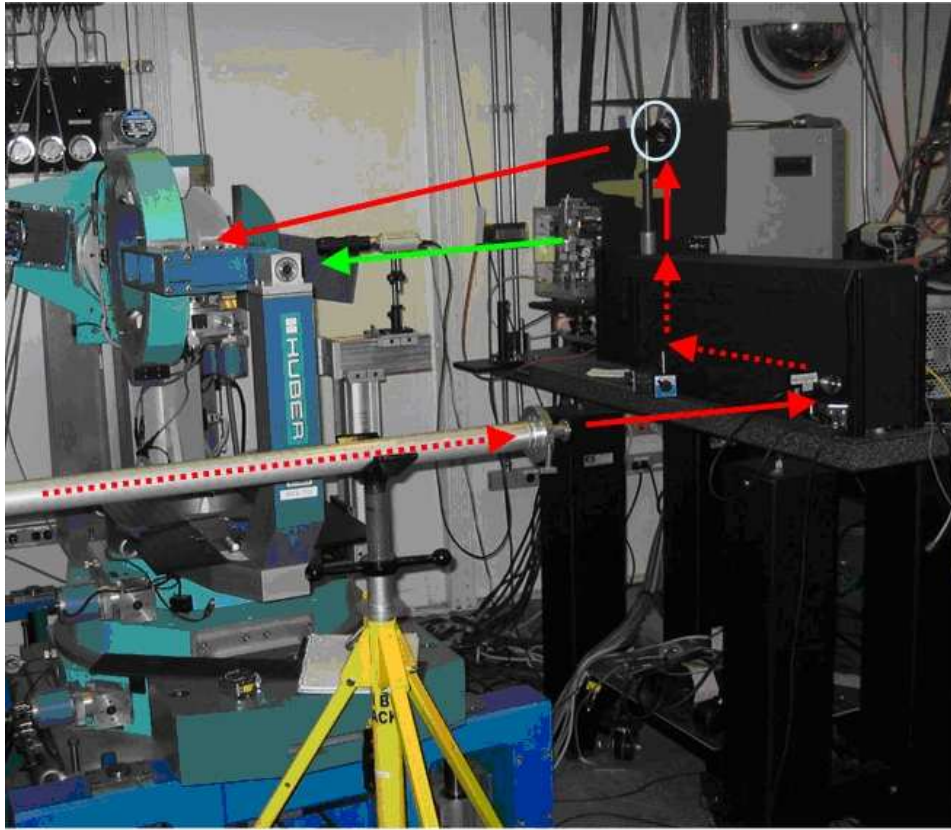
Figure 4: Photo of diffractometer showing x-ray and (typical) laser paths.

4.1 Angle nomenclature

There are plenty of ways to name a diffractometer motor. Unfortunately, some motors have different names in the different SPEC geometries. Table 1 sorts out which motion goes does what, for which geometry. Table 2 lists some other angles which are important in SPEC. Fig. 3 displays the psi-circle angles in Eulerian geometry.

4.2 Setting motor positions

Sometimes one can set a motor position in SPEC with the `set` command, such as

`set tth 5`

However, don't try that in psic; it won't work! There is no motor to set a value to! It is also a bad idea even to set real motors to arbitrary values, whether using SPEC or EPICS; it will only lead to confusion and problems when calculating positions of reflections. For example, if you find a symmetric Bragg reflection at tth = 20 and th = 10.5, you may be tempted to set th to 10 since you "know" that is where the reflection should be. Please don't do it!

All of the motors (except phi) have well-defined zero positions. A careful survey was done with an autocollimator to find these positions. As shown in Fig. 5, the dials of the motors have been aligned such that zero lines up with the real "dial zero." Note, however, that there is often an offset between "dial zero" and "user zero." This could, for example, be due to the KB mirrors deflecting the incident beam. For mu and nu, there is an offset due to the diffractometer not being set down on the floor exactly square to the incident beam. As of 9/16/2009, the offsets for these motors, for the *direct beam*, should be -0.52895 for mu and -0.32235 for nu (these are the values you would

| EPICS name | SPEC name | | Comments |
	fourc	psic	
Real Motors			
2-theta	tth	del	horizontal axis for detector arm
Theta	th	eta	horizontal axis for sample
Chi	chi	chi	sample motor
Phi	phi	phi	sample motor
Nu	N/A	nu	vertical axis for detector, only for psic
Psi	N/A	mu	vertical axis for sample, only for psic
Sample Motors			
Huber X	xs		one lateral motion of sample (the lower one)
Huber Y	ys		the other lateral motion of sample (the top one)
Huber Z	zs		vertical motion (along phi axis)
Table Motors			
Xtab Huber	Xtab		Lateral motion of diffractometer
Ytab Huber	Ytab		Vertical motion of diffractometer

Table 1: Names of the six-circle Huber diffractometer's motors.

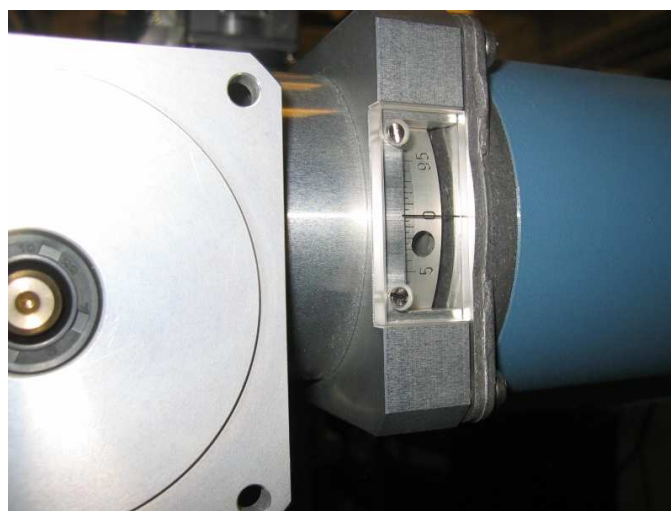enter into the "offset" field of the motor's EPICS screen).

Figure 5: Photo of a dial showing the zero position of a motor.

4.3   Miscellaneous hints

A Thorlabs XT-95 rail is usually mounted on the detector arm. Newport rail carriers (e.g., CX95A or CX95AS) are often mounted on that rail. The distance from the top of that rail carrier to the height of the beam is 2.5 inches $\pm$ 1/16".

The physical circle of the chi motion is offset from the center of the diffractometer; it can be upstream or downstream of the center. Often we have it downstream to free up space for laser delivery, but it can also be upstream to allow a wider range of two-theta (i.e., to not block the diffracted beam). To switch between the two configurations, (A) rotate theta by 180° and change

Special Angles in SPEC	
Common to fourc and psic	
F_*XYZ*	frozen value of angle *XYZ* for fixed-*XYZ* modes (*XYZ* = ALPHA, DEL, etc.)
CUT_*XYZ*	cut point of motor *XYZ* (see sections 5.1.3 and 5.2.3)
alpha	incident angle (if reference vector is surface normal)
beta	exit angle
Specific to fourc	
omega	th-tth/2
azimuth	rotation angle of reference vector about Q
Specific to psic	
tth	total scattering angle, a function of del *and* nu
psi	azimuthal angle of the reference vector with respect to Q and the scattering plane
tau	longitudinal angle of the reference vector with respect to Q and the scattering plane
Qaz	azimuthal angle of scattering vector Q
Naz	azimuthal angle of the reference vector
omega	angle between Q and the chi-circle
sigma_az	the chi part of the azimuthal angle specification (see Sect. 5.2.3)
tau_az	the phi part of the azimuthal angle specification (see Sect. 5.2.3)

Table 2: Various angular parameters for SPEC geometries. Q is the diffraction vector, with $|Q| = 2\sin(2\theta/2)/\lambda$. See Sects. 5.1.2 and 5.2.2 on how to define the reference vector.

its offset; (B) switch chi to the negative of its position, and switch its sense of direction in EPICS; (C) rotate phi by 180 and change its offset (which is usually not necessary since the zero of phi is arbitrary).

When the displex is used, the chi motor/gear reducer needs to be rotated $90°$ to be out of the way. Therefore, the chi offset needs to be adjusted by $0.25°$. This orientation makes it easier for a collision to occur with the detector arm so be careful about the relative positions of theta and two-theta.

5 SPEC Geometries

To reach a given $hk\ell$ point in reciprocal space, for example, using the `br` command, the diffractometer's angles must be moved to certain positions. But there are, in general, ambiguities which must be addressed in order to choose those positions. There are three sources to those ambiguities, which SPEC resolves via the user's selection of *modes*, *sectors*, and *cut points*. Since there are some differences between the fourc and psic geometries in how these selections are made, they are addressed separately. The current state of all the settings described in this section can be checked with the `pa` command.

5.1 fourc

5.1.1 Modes

The general issue in transforming from reciprocal space to diffractometer angles in fourc geometry is that there are three constraints (the Miller indices h, k, and ℓ) but four variables (the angles tth, th, chi, and phi). Thus one additional constraint is needed to lift this degeneracy and uniquely determine the diffractometer angles; SPEC calls this constraint a *mode*. A mode is selected with the `setmode` command; type

`setmode` *value*

to select mode *value*, or just type

`setmode`

to see the available modes. Table 3 lists the seven modes in four-circle geometry.

Each mode requires an angle (or two) to be fixed. The fixed value(s) can be chosen with the `freeze` command. In a given mode, typing

`freeze` *value*

will freeze whatever angle is to be fixed, at *value* (two values are needed for mode 2). Leaving off *value* will freeze the appropriate angle at its current position (and leave the angle fixed there); you won't be prompted for a value. In frozen mode, the constraint will be used regardless of the current diffractometer position. The command

`unfreeze`

will calculate modes based on the current position of the appropriate angle. If the relevant angle is moved (by the command `umv`, for instance), then the calculation will yield a different result (unlike frozen mode).

Mode	Description	Frozen variable	Comments
0	Omega equals zero	—	simplest case; special case of mode 1
1	Omega fixed	F_OMEGA	
2	Zone	F_CHI_Z, F_PHI_Z	chi and phi fixed; can only reach a plane of reciprocal space (use `cz`, `pl`, `mz`, and `sz` commands)
3	Phi fixed	F_PHI	three-circle geometry
4	Azimuth fixed	F_AZIMUTH	fix azimuth at ± 90 for alpha = beta
5	Alpha fixed	F_ALPHA	typically the incidence angle
6	Beta fixed	F_BETA	typically the exit angle

Table 3: Modes in fourc geometry.

5.1.2 Azimuth

Selecting the azimuth essentially means picking a reference direction on the sample. This direction could be the surface normal, magnetic field direction, etc. In fourc geometry it is only chosen by Miller indices. Type
`setaz` h k ℓ
to give the Miller indices (the vector's magnitude, of course, does not matter), or just
`setaz`
to be prompted for its indices one at a time.

5.1.3 Sectors and cut points

In addition to the constraint provided by the choice of mode, there are further ambiguities in the selection of angles to reach a given $hk\ell$. As a somewhat trivial example, the set of angles (tth,th,chi,phi) of (40,20,0,0) has the exact same diffraction condition as does (-40,160,0,0), but usually one wants to diffract upwards, not downwards. SPEC is told how to resolve this type of ambiguity through *sectors*.

There are eight symmetry transformations which lead to equivalent diffraction conditions; these are labeled as sectors 0 to 7. I will not reprint them here, but they are listed in SPEC's online help file (under "fourc"). To choose a sector, use the command
`setsector` *value*
to choose sector *value*, or leave off *value* to be prompted for it. More usefully, you can see all the possible sets of angles for a given reciprocal lattice point using the command
`sectors` h k ℓ.
This command will list alpha, beta, azimuth, omega, and the Eulerian angles for all possible sectors (only sectors 0 through 3 are available to modes 4, 5, and 6).

The final ambiguity is due to the 360° periodicity of the circle: the angle of $-10°$ is equivalent to 350°, but if you are currently at $+5°$, you probably want to move down by 15° instead of up by 345°. *Cut points* are used to tell SPEC where to divide the circle. An angle with a cut point of x will move between x and $360+x$. Cut points of the motors are set with the command `cuts`. Typing
`cuts`
will prompt the user for the cut points of the four motors. Alternately, one can type
`cuts` *angle value*
to set the cut point of a particular *angle*. The SPEC helpfile is not quite up-to-date on cut points. Typically, cut points at -180 are fine for most motors, but sometimes it's seemed better to use -170 for theta/eta.

5.2 psic

5.2.1 Modes

Selecting a mode in psic geometry is considerably more complicated than in fourc geometry, since three constraints must be added to the three Miller indices to uniquely determine all six angles. Unlike SPEC's sixc code, which "features" an ever-lengthening list of modes to choose from, psic's mode selection is based on a table formalism. That is, up to five variables (g_mode1, g_mode2, ...g_mode5) select the mode according to Table 4. Although this formalism may be confusing, it allows for, in principle, a very general selection of angular constraints. But beware that some sets of constraints have not had geometry code written or fully debugged.

To select a mode in psic geometry, the general format is
`setmode` *g_mode1 g_mode2 g_mode3 g_mode4 g_mode5*

psic	g_mode1	g_mode2	g_mode3	g_mode4	g_mode5
0	·	·	··· omega-fixed ···		X
1	delta-fixed	alpha=beta	······ eta-fixed ······		
2	nu-fixed	alpha-fixed	······ mu-fixed ······		
3	qaz-fixed	beta-fixed	······ chi-fixed ······		
4	naz-fixed	psi-fixed	······ phi-fixed ······		
5	Zone	X	eta=del/2	X	X
6	X	X	mu=nu/2	X	X

Table 4: Table to select modes in psic geometry, from SPEC's psic helpfile and Ref. [2]. Entries with X are not used.

where *g_modeN* = 0 to 6, although you often don't need to specify all five numbers each time. Generally, g_mode1 refers to a detector angle constraint, g_mode2 to an azimuthal angle constraint, and g_mode3 thru g_mode5 to sample angle constraints. Here are several examples of mode selection.

These three modes can imitate a four-circle diffractometer, if `F_NU=0` and `F_MU=0`:

    `setmode 2 1 2`   chooses alpha=beta

    `setmode 2 2 2`   choose alpha

    `setmode 2 3 2`   choose beta

Typical surface diffraction modes:

    `setmode 4 1 5`

    `setmode 4 2 5`   For these modes, `F_NAZ=0` keeps the surface normal horizontal, and rod scans are done by rocking phi (chi stays near zero). But it may be better to operate with `F_NAZ` in the range of 5 to 15 (12 is often used) to avoid cut-point type issues.

Horizontal or vertical scattering:

    `setmode 3 1 5`   `F_QAZ=0` keeps the diffraction vector Q horizontal.

    `setmode 3 1 6`   `F_QAZ=90` keeps the diffraction vector Q vertical.

Zone mode:

    `setmode 5`   chi and phi are chosen to keep Q in a chosen plane, using the macros `sz`, `cz`, and `mz`.

Other general modes:

    `setmode 0 0 `*s1 s2 s3*   Three sample angles fixed. May be useful with cryostats or other equipment which cannot rotate much. Obviously, *s1*, *s2*, and *s3* must all be different.

    `setmode `*d1 a1 s1*   *s1* can be any sample circle, or the special values 5 or 6 for eta=del/2 or mu=nu/2 respectively

    `setmode 0 `*a1 s1 s2*   selects one azimuthal and two sample constraints. Allegedly, the mu-fixed plus phi-fixed mode isn't working.

    `setmode `*d1*` 0 `*s1 s2*   selects one detector and two sample constraints. Allegedly, the eta-fixed plus phi-fixed, eta-fixed plus chi-fixed, and naz-fixed modes aren't working.

5.2.2 Azimuth

In psic, selection of the azimuth can be done with angles as well as with Miller indices. To specify by angles, the command is

`setaz` *sigma tau*

where *sigma* and *tau* are the chi and phi angles. To specify by Miller indices, the command is

setaz *h k ℓ*

The macro sigtau tells how to specify the azimuth:

sigtau 1

is for selection by angles, and

sigtau 0

is for selection by Miller indices.

If you are working with a sample whose surface normal is the azimuth, then it's convenient to use the `setaz2` macro. First, find (or know) the values of phi and chi that make the surface normal parallel to the eta axis; this is often done by reflecting a laser beam, if the surface is reflective. (Specifically, adjust phi and chi until the reflected beam does not move when eta is rotated). These values are called flat_phi and flat_chi, and are like tau and sigma except for a sign (or signs). Then, at the SPEC command line, type

setaz2

and you can use the current angular positions or enter your own.

5.2.3   Sectors and cut points

In psic geometry, the additional motors lead to additional sector transformations, 16 in all. (In most recent versions, there are even more, to deal with degeneracies when certain axes are at zero.) They are listed in SPEC's psic help file. In psic, use the command

sector *value*

(not `setsector`, as in fourc geometry) to choose which transformation to use (if you leave off *value*, you will be prompted for it). You could also set the variable g_sect to the number of the transformation you wish:

g_prefer = *value*.

Fortunately, there is a handy alternative in psic to keeping track of all the angular positions in the 16 different sectors. Sector 0, instead of selecting a certain transformation, ranks all the transformations according to convenience. The ranking schemes are chosen by the variable g_prefer, but so far, only two have been implemented: g_prefer = 1 is for a 'vertical' diffractometer ($0 \leq$ del < 180 and $-90 \leq$ nu ≤ 90), which is the appropriate choice for the six-circle in 7-ID-C. g_prefer = 2 is for a 'horizontal' diffractometer ($-90 \leq$ del < 90 and $0 \leq$ nu < 180). One could still use the `sectors` command, as described in Section 5.1.3.

Cut points in psic operate as they do in fourc, so see Sec. 5.1.3 for full details. The difference is that psic has nine cut points instead of seven, due to the addition of the nu and mu motors. Again, cut points of -180 are usually fine as default values, but eta sometimes operates a bit smoother with cut points of -170.

Acknowledgments

References

[1] G. Swislow. Certified Scientific Software, www.certif.com.

[2] H. You. *J. Appl. Cryst.*, 32:614, 1999.