DAC Data Collection, Xcalibur-1

VTX

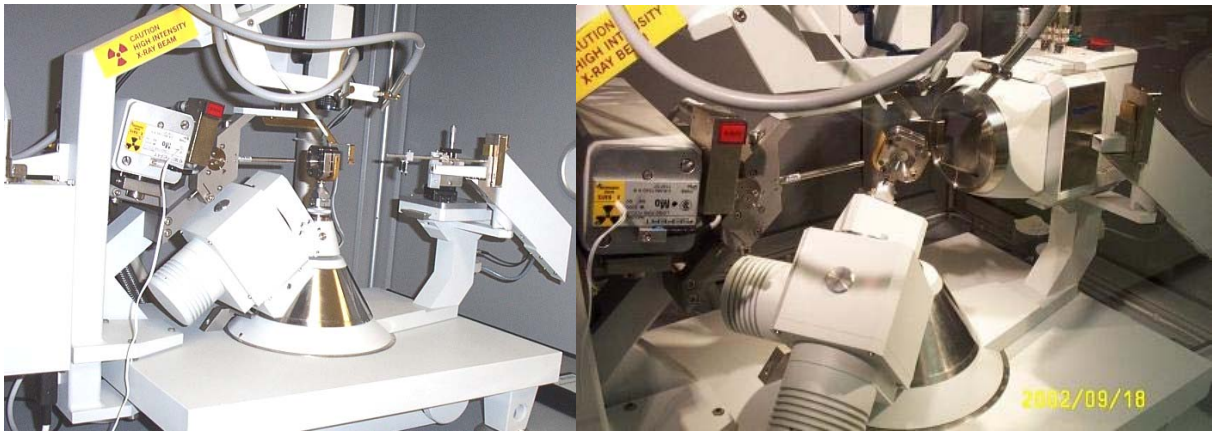
http://www.crystal.vt.edu/crystal/

This version: 22-July-2006

This is intended as a short guide to setting up a DAC for a data collection on an Xcalibur-1 diffractometer, equipped with a dual detector arm with point detector and CCD (see below).

- **If you do not have a CCD camera use these instructions for the point detector (old and new design Xcalibur instruments).**
- **If only you have a CCD detector, use the separate instructions for the DAC with CCD.**

The user is assumed to be familiar with the Crysalis software and commands and their use for data collections from crystals in air.



This diffractometer has a dovetail slide for mounting a CCD camera. When the point detector is used for DAC data collections this slide is used to hold a set of additional collimation slits as shown in the picture on the left.

In the following documentation, commands to be typed into the command line of the Crysalis GUI are indicated thus: **gt r 4 0 0**. Command line entries where numerical values should be substituted are indicated by italics, thus: **gt r *h k l***

Step 1: Preparation.

Operation	**Command/Action**
Create filespace for data collection	Open a file browser Create a new directory for this data (e.g. \P1)
Start program	Double-click on desktop icon for Crysalis CCD
Check that software is set for point detector operation	GUI should have angles and scan display
If the software is in CCD mode, change to PD mode.	Select Tools|Setup file Select Xcalibur1PD.par Exit from Tools|Setup Exit from program: **en** Restart Crysalis CCD from desktop
Switch to DAC mode	**sw s 2**
Set DAC opening angle (normally 40deg)	**sw a *angle***
On the diffractometer, remove the slit assembly from the sled on the dovetail by unscrewing three screws.	

Step 2: Physical Alignment of DAC

Operation	**Command/Action**
Drive the diffractometer to alignment position	**gt a 0 21.75 0 0**
Load DAC onto diffractometer. Tighten the base screw firmly.	
Align the DAC by eye, perpendicular to the beam	Loosen the locking screw for the height adjustment on the goniometer head and rotate the cell until it looks perpendicular to the beam direction.
Accurately align the DAC perpendicular to the beam.	Slide the sled on the dovetail to the back of the dovetail. Mount the aluminium alignment tool on the sled. Carefully slide the tool in to touch the DAC. Rotate the DAC until the face of the DAC is exactly parallel to the end of the alignment tool. Gently tighten the height locking screw on the goniometer head.
Remove the alignment tool.	Slide the sled back on the dovetail and lift off the alignment tool, taking care not to hit the beam stop.
Set focus of video microscope, and cell	**F12**

translation along beam	View image of cell. Loosen locking screw of video camera and move it to focus. **Spin cell by 180 on phi (keypad)** If image not in focus, adjust half way to focus with goniometer head slide, and half with camera adjustment. Repeat until cell is in focus at both of these two positions .
Set height of DAC	**Lower position (keypad)** Observe position of gasket hole centre on video screen. **Upper position (keypad)** Compare position of gasket hole and adjust height. Repeat until image of gasket hole does not move vertically between these two positions. Tighten height locking screw
Set cell translation across beam (x direction)	**gt e 0 0 90 –90** Observe position of centre of gasket hole. **gt e 0 0 90 90** Compare position and adjust with slide on goniometer head. Repeat until image of gasket hole does not move between these two positions. Tighten slide locking screw
Check that DAC is still perpendicular to beam at zero	**gt a 0 21.75 0 0** See instructions above for using alignment tool on sled. Correct alignment if necessary.
Obtaining an image of the crystal	
Start video utility	**abs grab**
Position goniometer so that the DAC is perpendicular to the video camera and the crystal is visible	Select **goniometer** Hit **override remote control** *twice.* Go to **lower** position Rotate **phi position** until crystal is visible (normally phi = 0). **Record which face of the DAC is facing the camera!** Close the goniometer control window.
Capture the image	Check the focus and illumination. Select **clipboard** once
Save the image	Open a graphics program (e.g. MS Paint) Select **paste from clipboard** Save the image as a jpeg.

Step 3: Determine Initial Orientation Matrix.

There are several possibilities, depending on the stage of the high-pressure experiment:

1. The UB from a measurement on the Xcalibur diffractometer at a previous pressure is known
2. The UB from a measurement on the Huber diffractometer is known.
3. The UB is not known.

Step 3.1: UB known from previous measurement on Xcalibur.

Important: If you read in an old par file, you will overwrite the current peak table in the software, and you will have the peak table associated with the par file that you read in!!

Operation	**Command/Action**
Copy the peak table from the previous measurement	Use the Windows file browser to copy the *.tab file to the current working directory. **rd t**
Or input values at command line	**um s *u11 u12 u13 u21 u22 u23 u31 u32 u33***
Check table is correct by calculating lattice parameters from UB	**ty l**
Go to step 3.4	

Step 3.2: UB known from previous measurement on Huber.

Operation	**Command/Action**
Convert the UB matrix from the Huber (Single software)	$UB(Crysalis) = 0.71 \times \begin{pmatrix} 0 & -1 & 0 \\ 1 & 0 & 0 \\ 0 & 0 & 1 \end{pmatrix} UB(Huber)$
Input values at command line	**um s *u11 u12 u13 u21 u22 u23 u31 u32 u33***
Check table is correct by calculating lattice parameters from UB	**ty l**
Save UB to disk	**wd t**
Go to step 3.4	

Step 3.3: UB unknown

Operation	**Command/Action**
Switch to CCD	
Perform short data collection	Use a DAC run file but cut down the runs to those at kappa=0
Find peaks, index, save peak table	
Switch back to point detector	
Go to step 3.4	

Step 3.4: Look for reflections

Operation	Command/Action
Insert detector slits 3.3 2.0	Insert pair of slits labeled 3.3 (horizontal) and 2.0 (vertical) with notches towards the back of the cabinet.
Set slit values in software	**da 3.3 2.0**
Drive to a strong reflection	**gt r *h k l***
Scan the position....set the scan width	**sc w 2.0 0.0**
Do the omega scan	**sm s 40 0.1**
If the maximum is in the scan, check two more reflections. If all ok, go to step 4	
If maximum is not in the scan, drive around in omega and rescan until you find it.	**gt o *omega*** (*omega* should be shifted by 1 deg) **sm s 40 0.1**
When peak is found, drive to position of maximum	**gt o *omega***
Centre the reflection *Note: it is often better to do the centering with slits 2 and 2*	**ce 0**
Add reflection to peak table	**pt a**
Look this reflection up in the peak list select it, edit it, insert the correct hkl values, exit the list Save the table	**pt e** **wd t**
Repeat for another reflection	
Select the two reflections in the table	**pt e** **Select each reflection in turn, select Edit, click "select". Make sure indices are correct.**
Do two-reflection calculation of UB	**um f2 *a b c* $\alpha\beta\gamma$**, where ***abc*** and $\alpha\beta\gamma$ are the estimated cell parameters.
Check the result: If the lattice parameters change a lot, then your indexing was incorrect; change the indexing and try again.	**ty l** **pt e** **um f2 *a b c* $\alpha\beta\gamma$**
When you have a valid UB, save it	**wd t**
Check the UB finds other reflections: Drive to a strong reflection See if it is in the detector window	 **gt r *h k l*** **sm i 1** (or *F7*) and observe counts
If all Ok, proceed to step 4. If not fix it.	

Step 4: Refine UB

Operation	Command/Action
Edit the peak list - make a note of the *hkl* - delete all reflections (see note at end of this table) - insert the *hkl* of one of each symmetry-equivalent set - exit from the peak list	**pt e**
Expand the peak list by Laue symmetry	**pt l *n*** (if you do not know ***n*** for your Laue group then type **pt l** to obtain a list)
Edit the peak list to remove reflections with low kappa angles. At this stage you need 20-30 strong reflections.	**pt e** Select "angles" at bottom of display Click on "kappa" column header to order reflections Delete reflections with $-15^o < \kappa < 15^o$. Exit the peak list editor
Save the peak list	**wd t**
Start reflection centering	**um u**
Note: If a reflection in the list cannot be centered it will be skipped and deleted from the list. If many reflections are skipped then either the UB matrix and the cell parameters are wrong, or the software has the wrong values loaded (with **da**) for the detector slits.	To recover from this problem: Read the original table back into software, **rd t** Enter the correct slit values **da *hslit vslit*** Repeat centering **um u**
At the end of centering, the UB is determined. Save it and the peak positions	**wd t**
Switch to smaller slits, h=2.0, v=1.0	Exchange brass slits on detector Update values in software: **da 2.0 1.0**
Check the UB finds reflections with smaller slits: Drive to a strong reflection See if it is in the detector window	**gt r *h k l*** **sm i 1** (or *F7*) and observe counts
If ok, repeat centering	**um u**
At the end of centering, the UB is determined. Save it and the peak positions	**wd t**

Note on the peak table: The Crysalis centering procedure um u works by first driving to the angular positions given in the peak table. This is different from the Single software in which the starting position for centering is calculated from the *hkl* in the peak table, and the current UB matrix. This means that in Crysalis when the UB is changed significantly, the peak table must be cleared and the indices of reflections be reloaded into the table; this procedure ensures that the peak positions are calculated from the current UB.

Step 5: Determine crystal offsets

At this stage the gasket hole of the DAC has been well-centered optically across the beam, but the positioning along the beam has relied on focusing the video microscope on the sample. The centering along the beam can be improved by "diffracted beam centering". There are two ways to achieve this:

1. By 8-position centering of a single reflection with a Eulerian-chi value between 80^o and 90^o.
2. By collecting data scans of 30 or more low-angle reflections and refining the crystal offsets by the method of Dera and Katrusiak (1999, Journal of Applied Crystallography 32:510-515).

Method 1 takes less time, but method 2 is often more reliable. Both alternatives are described below:

Step 5.1: 8-position centering

Operation	**Command/Action**
Find a strong reflection with Eulerian chi > 80^o	**gt r *h k l*** **sm i 1**
Do 8-position centering	**ce HP**
Adjust goniometer position according to offsets from **ce HP** procedure	
Repeat until offsets are small or zero	

Step 5.2: Crystal offsets from data collection

*Important Note: If you set the parameters for this step by opening an old par file, then you will overwrite the current UB matrix and peak table with the one from the par file. Recover from this problem by reading in the peak table (**rd t**) that you saved in step 4.*

Operation	**Command/Action**
Set up parameters for a short low-angle data collection , as follows.	
Set detector slits to h=2.0, v=0.5	Exchange brass slits on detector Update values in software: **da 2.0 0.5**
Set scan parameters to stop rescanning	**mo s 1 60 10 0 0.005**
Set fast scan speed	**sc s 0.05**
Set background calc	**mo b 0.5**
Set scan width	**sc w 1.200 0.000 1.00300**
Set omega scan	**sc t 0.0 0.0**
Set index limits to cover all reciprocal space *Note:* this sets the maximum values of indices to be tested against 2theta limits etc. Just make them sufficiently large.	**il –10 10 –10 10 –10 10**
Clear ma limits	**ma b 0 0 0 0 0 0 0 0 0**
Set 2theta limits	**tr 2 25**
Set absence conditions	**Rc**
Clear reference reflections	**rr 0**

Check all values are correct	**ty p**
Check number of reflections that will be collected	**dc t**
Adjust 2theta limits and/or index limits until you have 30-50 reflections to be collected	**tr *tthmin tthmax*** **il *hmin hmax kmin kmax lmin lmax***
Save parameters (it is useful to call this something like *orient.par*)	**wd p**
Start data collection (use a distinct filename such as orient*n*) where *n* indicates the iteration through this process	**dc s**
When data collection is complete, export the data to a *dca* file	Open the Crysalis Reduce software type **dc redpd** on its command line Select your data file Select Convert to Ascii
Open the WinIntegrStp program	Double-click on desktop icon
Select the *dca* file you just created	
Select Xcalibur.par as the instrument parameter file.	
Run preprocessing option to obtain the peak positions	Run Preprocess in WinIntegrStp. Set I/sigma to 10.0 Set Intensity, peak width, position to be refined Set background to not refined, with default value “D” Set eta and Iratio to not refined Start preprocess with “Go”
If insufficient (<20) reflections are stored after Preprocess, reduce I/sigma or adjust the test limits on the parameters.	
Once you have >20 reflections stored from Preprocess, calculate the UB	Utilities|Calc UB Select refine crystal offsets Run
Record the crystal offsets reported (in mm). The X and Z offsets should already be small (<50micron). The Y offset is along the beam. If it is less than 30 micron go to step 6.	
If Y offset >30 micron proceed as follows	
Drive goniometer to zero	**gt a 0 0 0 0**
Place the dial gauge in contact with the downstream face of the cell.	
Adjust the cell position along the beam	One division on the dial gauge is 25 micron. If the Y offset is positive, move the DAC towards the X-ray tube. If the Y offset is negative, move the DAC away from the X-ray tube.
Repeat step 5.2 until Y offset is <30 micron.	

Step 6: Data Collection with point detector

Note: See subsequent sections for data collection with CCD

Operation	**Command/Action**
Install additional slits on detector arm	Screw down the slits onto the carrier on the dovetail Set the slide to 9.55
Install Be cones into DAC	**gt e 0 0 90 90** install one cone into top side of cell **gt e 0 0 90 –90** install the other cone Make sure they do not fall out!
Set detector slits to h=2.0, v=0.5	Exchange brass slits on detector Update values in software: **da 2.0 0.5**
Set up parameters for the data collection as follows:	
Set scan parameters	**mo s 1 60 1 10 0.005**
Set fast scan speed	**sc s 0.05**
Set background calc	**mo b 0.5**
Set scan width	**sc w 1.200 0.000 1.00300**
Set omega scan	**sc t 0.0 0.0**
Set index limits to cover required portion of reciprocal space *Note:* this sets the maximum values of indices to be tested against 2theta limits etc. Just make them sufficiently large.	**il *hmin hmax kmin kmax lmin lmax***
Set ma limits if required	**ma b *n***
Set 2theta limits	**tr *thmin thmax***
Set absence conditions	**rc**
Set reference reflections	**rr 3 200 0.15 15.0 *h k l 0 h k l 0 h k l 0***
Check all values are correct	**ty p**
Check number of reflections that will be collected	**dc t**
Check that parameters and UB are ok by scanning several reflections	**sm r *h k l***
Save parameters	**wd p**
Start data collection	**dc s**
Record details of data collection in log book.	
When data collection is complete, check that the data looks ok as follows:	Open the Crysalis Reduce software type **dc redpd** on its command line Select your data file Select Convert to Ascii
Open the WinIntegrStp program	Double-click on desktop icon
Select the *dca* file you just created	

Select Xcalibur.par as the instrument parameter file.	
Check the scans are ok and centered	Use Integrate | Manual Profile Fit to review the dataset
If all is ok: - drive diffractometer to zero - remove DAC from diffractometer - remove Be cones from DAC - Record details in log book - Inform the next user that the diffractometer is available - Integrate the data and refine the structure	**gt a 0 0 0 0** **WinIntegrstp, Absorb, Average**

Note: the following sections are for data collection with the CCD

Step 7: Switching over to CCD detector

Operation	**Command/Action**
Switch the par file	**Select Tools|Setup** **Select Xcalibur1CCD.par**
Shutdown CCD program	**En**
Start CCD program	**Verify that Xcalibur1CCD.par is loaded**
Check dd = 70	**Tools|Options|Instrument Model I**
Load CCD camera to dovetail	**gt t 90** **Remove slits from dovetail** **Mount CCD detector from rear of dovetail** **Slide CCD to 70mm** **gt t 0**

Step 8: Pre-designed run files

We have designed a run file for data collection with the Sapphire CCD set at dd=70mm and a DAC with a half-opening angle of 40 degrees:

DAC_psi40_dd70_tth60_full_sapphire1.run

This run file attempts to cover all of accessible reciprocal space. If only one-half of that space is required, then the runs at negative values of 2theta can be deleted.

For more details about the design of DAC run files, see the Appendix to this manual.

Step 9: Data collection

1. Enter **ccd skipremeasure 1** to prevent remeasuring on diamond reflection overflow.
2. Check that the correct flood field file is loaded (Tools|Correction files).
3. Check the correct detector distance is set in Tools|Options
4. Enter **dc s**. In the notes section make a note of the χ values you are using as these are hard to figure out afterwards.
5. Say "OK" to the warning about skipping the remeasuring. If this warning does not appear, interrupt and go back and do step 2 again!

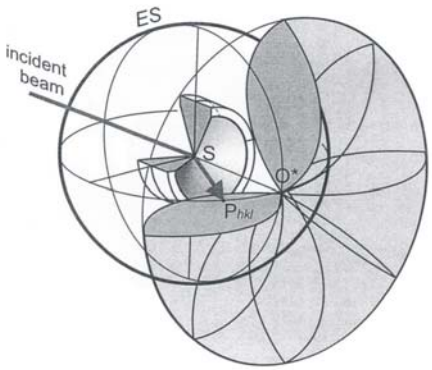
Step 10: Data Integration

1. Start the Reduce software.
2. Check that the correct high-pressure parameter file is loaded (Tools|Setup File).
3. Use Setup|Options|Instrument model 1 to set dd = 70mm (or whatever you used).
4. Turn on DAC mode: **sw s 2** and **sw a Ψ_{max}**. This prevents the software from attempting to search or integrate at peak positions that are obscured by the DAC.
5. Limits to the areas to be searched for peaks with **ph s** can be controlled with the **um skip** commands:
    a. **um skipd dmax dmin** prevents peak searching between dmax and dmin
    b. **um showskipd** lists the forbidden regions
    c. **um clearskipd** clears the restrictions.
6. Read the necessary d-spacings off some images.Always set a skip region for 999.0 down to slightly longer than your unit-cell.
7. Run **ph s**. Use background subtraction with 5,5.
8. Use **pt e** to delete the strongest reflections (usually diamonds). Use **pt ewald** to inspect the peak list. Remove obvious Be rings etc.
9. Attempt indexing. Better still, use a known UB matrix to index the reflections.
10. Before doing the data reduction, clear the skip list with **um clearskipd** because the skip list also applies to data integration.
11. Run **dc red**:
    a. In step 4, set the background evaluation to 10,5.
    b. In step 5, set the DAC opening angle (in skip filters), the 2theta limit, and set *use background LS plane* (in peak finding).
    c. In step 6, switch off outlier rejection.
    d. In step 7, select the option to produce Shelx direction cosines on the output file.
12. Use **Absorb, Average** to correct the intensities for the effects of the DAC, and refine the structure!

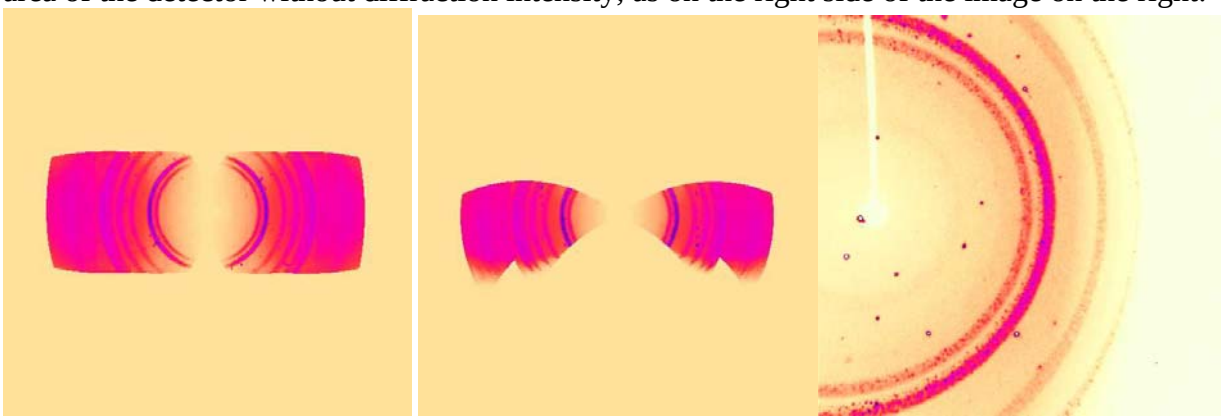
Appendix: Designing a run list

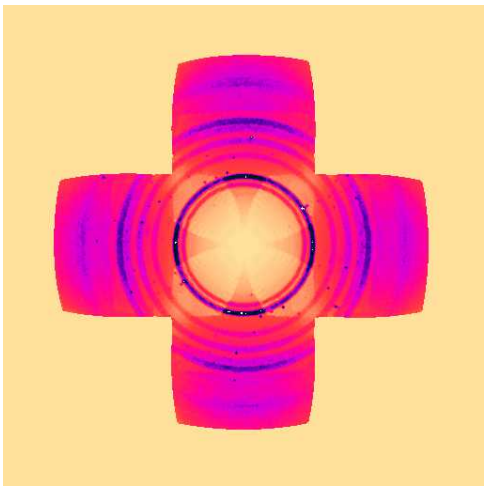
Principles

The accessible region of reciprocal space of a crystal mounted in a DAC is toroidal in form, as shown in the diagram (from R. Miletich). The exact shape depends on the opening angles of the cell (see Miletich et al. in MSA Reviews in Mineralogy volume 41, available at www.minsocam.org). The challenge for a CCD data collection is to collect this volume of reciprocal space efficiently, without too much obscured (and thus unused) area of the detector, and without too much overlap of frames from different runs.



Our solution (also that of the Poznan group) is to do a series of runs at a fixed chi value. This collects a swathe of reciprocal space across the toroid, as shown below. On the left is the section of reciprocal space perpendicular to the beam, in the middle is a section including the beam direction running vertically. On the lower edge of this section you can see the shadowing (pale yellow areas) caused by the DAC. This shows up on the individual CCD images as an area of the detector without diffraction intensity, as on the right side of the image on the right:





Subsequent sets of runs are designed to fill in the gaps. Our run files do a second full scan at $\chi = 90^{o}$. Together with the first scan, the coverage of reciprocal space in the plane of the diamond culets now looks like this.

The remaining sets of runs fill in the "corners" of this section. They are run at χ values of $+/-45^{o}$, but only at the higher values of 2θ, as runs at lower 2θ values would only duplicate what is already collected.

Practical

The parameters controlling the data collection procedure are:

1. The detector distance and thus the 2θ aperture of the detector. We use Δ to specify the half-width of the detector in degrees
2. The maximum opening angles of the DAC, Ψ_{Imax} and Ψ_{Dmax}
3. The minimum proportion of the CCD you want illuminated.

Data collection proceeds as a series of scans in omega at fixed 2θ, ϕ, and χ or κ.

The aperture of the CCD controls only the choice of 2θ steps. For Xcalibur-1, $\Delta = 20^o$ at dd=70mm, and we therefore step in 20^o increments in 2θ. The maximum in 2θ is usually set at the step previous to the maximum possible (see below).

The limits on absolute omega are given by two sets of conditions.

The diffracted beam: $-|\psi_{D\max}| \le 2\theta - \omega \le |\psi_{D\max}|$

or: $\omega \ge 2\theta - |\psi_{D\max}|$ and $\omega \le 2\theta + |\psi_{D\max}|$

The incident beam: $|\omega| \le \psi_{Im\,ax}$

To calculate the scan limits, proceed as follows:

1. Decide on the 2θ values to be used. The maximum value of 2θ should be equal to twice Ψ_{Dmax} (but you will not use this value).
2. For each 2θ value calculate the minimum and maximum values of ω consistent with Ψ_{Dmax}.
3. Cut down the values of ω to those consistent with Ψ_{Imax}.

Example for $\Psi_{Imax} = \Psi_{Dmax} = 30^o$

2θ	**ω from step 2**	**ω from step 3**	
-60	-90 to –30	-30 to -30	No scan!!
-40	-70 to –10	-30 to -10	
-20	-50 to +10	-30 to +10	
0	-30 to +30	-30 to +30	
20	-10 to +50	-10 to +30	
40	+10 to +70	+10 to +30	
60	+30 to +90	+30 to +30	No scan!!

Example for $\Psi_{Imax} = \Psi_{Dmax}=40^o$

2θ	**ω from step 2**	**ω from step 3**	
-80	-120 to -40	-40 to -40	No scan!!
-60	-100 to -20	-40 to -20	
-40	-80 to 0	-40 to 0	
-20	-60 to +20	-40 to +20	
0	-40 to +40	-40 to +40	
20	-20 to +60	-20 to +40	
40	0 to +80	0 to +40	
60	+20 to +100	+20 to +40	
80	+40 to +120	+40 to +40	No scan!!

These values of ω apply to $\phi = \chi = 0$. For other values of χ, proceed as follows:

1. For each value of χ use **gt e 0. 0. χ 0**. to calculate the kappa angles required to set the cell perpendicular to the beam (i.e. so Eulerian $\phi = 0$).
2. Note the kappa goniometer angles at this position.
3. Use **dc editruns** to create a run at the noted ϕ_{Kappa} and κ values
4. Add the limits calculated above to the noted value of ω_{Kappa} to get the limits for ω at this goniometer setting.

Repeat for as many values of χ as required. Here are some commonly-used settings:

χ	ω_{Kappa}	κ	ϕ_{Kappa}
89.	-56.	133.	-56.
45.	-20.0	60.0	-20.

One can duplicate the coverage of reciprocal space by doing further runs but at $\phi_{Kappa}+180^o$.

Test the coverage and duplication by unwarping a dataset with the default UB matrix in Crysalis (with x along the beam, z vertical).