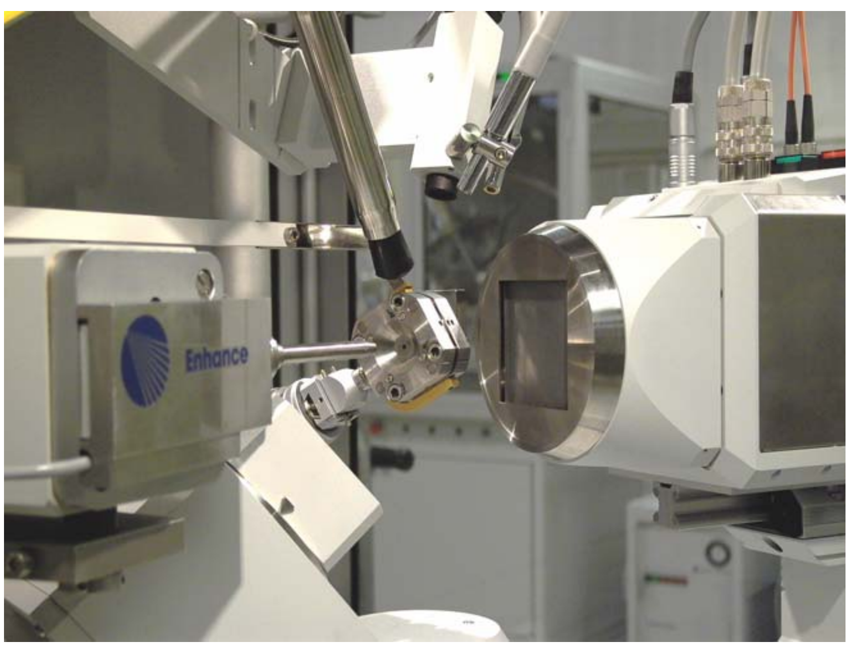
DAC Data Collection, Xcalibur-CCD

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



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 CCD detector operation	GUI should have CCD image display Correct HP parameter file should be loaded
If the software is in PD mode, change to CCD mode.	Select Tools|Setup file Select the HP-CCD.par file Exit from Tools|Setup Exit from program: **en** Restart Crysalis CCD from desktop
Check that the correct short collimator and the correct detector limit flag for the long beam stop.	Change if necessary
Remove beam stop	

Step 2: Physical Alignment of DAC

Operation	**Command/Action**
Switch to HP mode	**sw s 2**
Set max Ψ angle	**sw a 40**
Drive the diffractometer to zero	**gt a 0 0 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.	**gt e 0 0 90 90** Rotate the DAC until the face of the DAC is exactly horizontal, as measured by a spirit level. Gently tighten the height locking screw on the goniometer head.
Set focus of video microscope, and cell translation along beam	**F12** View image of cell.

	Loosen locking screw of video camera and move it to focus. **Spin cell by 180 on phi** 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 translation of DAC	**Spin cell by 180 on phi** And compare positions of image of gasket hole. Move the cell and repeat until the center of the gasket hole is in the same place before and after rotation by 180 deg.
Set height of DAC	**Lower position** Observe position of gasket hole centre on video screen. **Upper position** 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.
Use transmitted beam to align cell along beam as follows	
Drive goniometer to zero	**gt a 0 0 0 0**
Reduce generator power to 28 KV and 1mA	
Expose CCD with rotated cell	**gt o –30** **card raw 0.1 on**
Repeat with opposite omega	**gt o 30** **card raw 0.1 on**
Adjust cell along beam until the transmitted spot is in the same position for both $\omega = +30$ and $\omega = -30$.	
Install the long beam stop and align it with the direct beam transmitted through the DAC.	**gt a 0 0 0 0** **card raw 1 on** Repeat with higher generator power up to operating conditions.
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: Pre-designed run files

We have designed run files for data collection with the Sapphire-3 CCD set at dd=80mm and a DAC with a half-opening angle of 40 degrees:

DAC_psi40_dd80_tth60_full_sapphire3.run
DAC_psi40_dd80_tth80_full_sapphire3.run

Both run files attempt 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 information about designing run files for the DAC, see the Appendix.

Step 4: 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 5: Data Integration

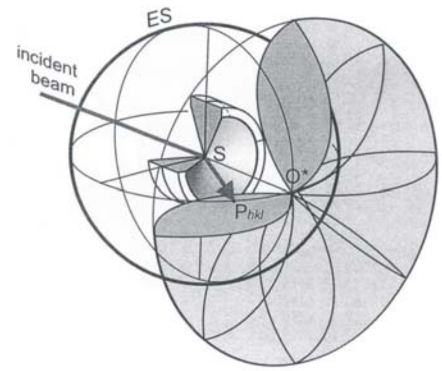
1. Start the Reduce software.
2. Check that the correct high-pressure parameter file is loaded (Tools|Setup File).
3. Use Setup|Options|Instrument model 1 to set dd = 80mm (or whatever you used).
4. Turn on DAC mode: **sw s 2** and **sw a** Ψ_{max}. This prevents the software from attempting to search 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** and **Average** to correct the intensities for the effects of the DAC, and refine the structure!

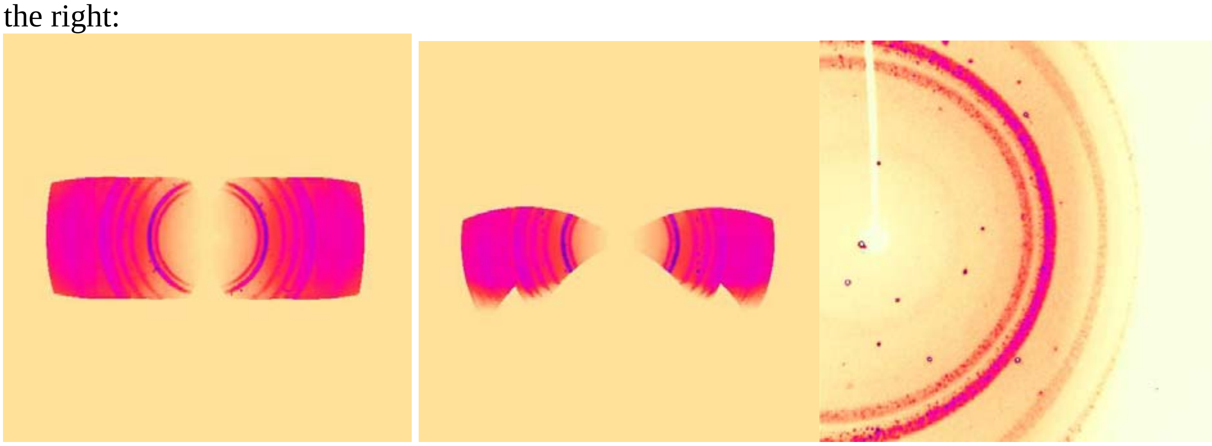
Appendix: Designing a run list

Principles

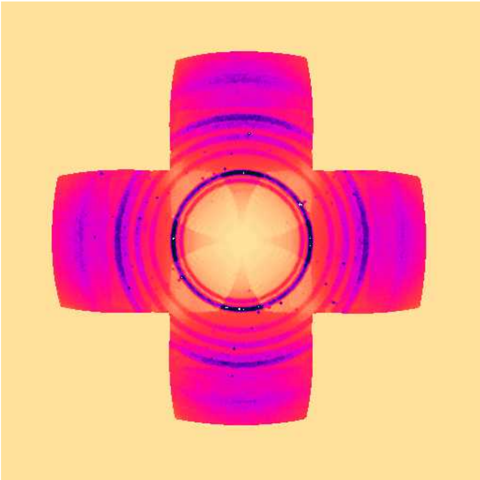
The accessible region of reciprocal space of a crystal mounted in a DAC is toroidal in form, as shown in the diagram (from R. Miletich). The exact shape depends on the opening angles of the cell (see Miletich et al. in MSA Reviews in Mineralogy volume 41, available at www.minsocam.org). The challenge 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 χ = 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-2, Δ = 20^o at dd=80mm, 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: $-|\psi_{D\max}| \leq 2\theta - \omega \leq |\psi_{D\max}|$

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

The incident beam: $|\omega| \leq \psi_{\mathrm{Im}ax}$

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 ϕ_{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).