

Data Collection SMART Advanced

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Version 1.0.0

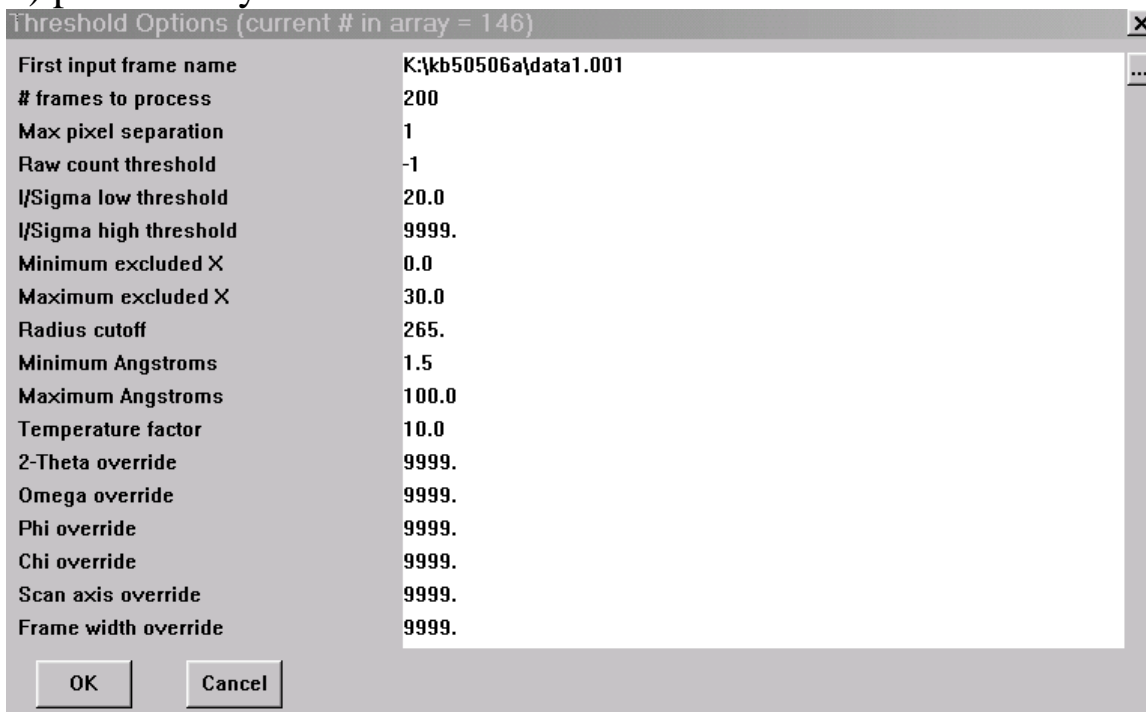
Unit Cell Determination

Peak Position file : P4P

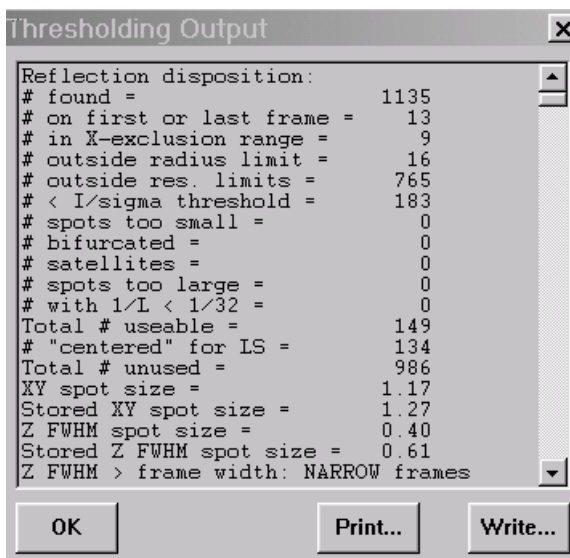
You can use the current peak list or create a new one to create a new list

1) point to Crystal/-Clear and point to OK

2) point to Crystal/-Threshold



use the ... to find the frame file you want to start at. Set the number of frames (here it is set at 200). Set the raw count and



I/sigma low threshold (-1 and 20 are ok for the SMART, 20 and 20 for the GADDS). Point to OK

The output information is displayed. The total # useable is 149. If you want more

reflections then increase the number of frames in the first step. For less decrease the number of frames. Point to ok repeat this for two more sets of frames.

To save your results point to Crystal/--LS

input the output P4P file name (here I have uncl.p4p). NOW POINT TO CANCEL (NOT OK). Point to Edit/--Save P4P. This will write the output file to the disk. Minimize SMART

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Directory of F:\structures\kb\kb54
05/08/2006 09:52a      138,564 dataa.p4p
05/08/2006 09:52a      138,564 datau.p4p
05/08/2006 09:52a      138,564 kb54.p4p
05/08/2006 09:52a      138,564 uncl.p4p
05/05/2006 11:42a      111,956 Unsorted.p4p
5 File(s)              666,212 bytes
0 Dir(s)               71,696,789,504 bytes free

F:\structures\kb\kb54>cell_now

CELL_NOW analyses a list of reflections to find a cell and orientation matrix despite the presence of several twin domains or other junk. In initial search mode the program tries to find sets of reciprocal lattice planes that pass close to as many reflections as possible. The corresponding real space vectors are sorted on a figure of merit (1.0 would be a perfect fit). In the output these are followed by the percentages of reflections that fit within 0.1, 0.2 and 0.3 times the interplanar separation, the components a1, a2 and a3 of the vector, the angles to previous vectors and a cross figure of merit to previous vectors. The latter should be larger for reflections belonging to the same twin component. Cosines of angles between vectors a and b can also be calculated from the components by (a1*b1+a2*b2+a3*b3)/(a*b).

After the vector list has been output, CELL_NOW attempts to suggest a suitable cell. This will not necessarily be the conventional cell, so it should be checked using XPREP (without an .hkl file) taking the lattice type found via CELL_NOW into account. If necessary this conventional cell may be reinput in 'specified cell' search mode to find the orientation matrix. If CELL_NOW fails to suggest a sensible cell, either something is seriously wrong with the reflection list (e.g. a wrong detector distance) or a cell axis is longer than the given search range.

In specified cell search mode the program tries to find the best cell within the specified ranges. The reflections that fit this cell within a specified fraction of all three interplanar spacings may be flagged as indexed, and a new .p4p or .spin file written in which they have the 'H' flag so that they can be displayed in a different color with MATT. Then the cell may be rotated to locate further twin domains iteratively using only the reflections that have not yet been indexed.

** WARNING: the exhaustive search employed in this program is VERY SLOW **
** so a CPU clock frequency of AT LEAST 3GHz is strongly recommended **

Full name of .p4p, .spin or .dxf file to read: uncl.p4p
278 reflections read in


Listing file funcl.cnl:

Initial search <<Enter>> or specified cell search <S>:

Superlattice threshold: an axis will be rejected if less than this percentage of reflections has indices not equal to 2n or 3n resp. [10]: 10

Minimum and maximum allowed values for cell edge [5 40]: 5 40

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Start a command prompt (point START/RUN  and type cmd). Navigate to your data directory and type cell_now

Input the P4P file (here it is uncl.p4p) and use the defaults for Initial Search, Threshold 10 and

cell edge 5 ,40. If you think you may have a larger cell then change the cell edge setting to 3, 80. Remember this will slow the program down!

Let the program think. It may take a while depending on your computer etc.

```

27.120 0.737 27 98.921 99.640 100.000 4.945 26.462 3.270
145.2 76.7 137.9 65.7 34.8 114.4 99.5 58.4 19.5 155.8 146.9 55.4 124.8
0.913 0.912 0.909 0.893 0.898 0.900 0.889 0.882 0.892 0.876 0.880 0.883 0.877

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The following cells would appear to be plausible, but should be checked using
XPREP because they are not necessarily the conventional cells.
POM. % within 0.2, a..gamma, volume and lattice type for potential unit-cells:
  1 1.000 99.3 11.326 11.157 15.000 90.07 109.51 90.15 1786.5 P
  2 0.912 98.9 11.326 11.157 15.486 90.20 114.07 89.85 1786.7 P
  3 0.606 88.5 11.326 11.157 15.202 90.63 108.14 89.85 1825.4 P
-----
Cell for domain 1: 11.326 11.157 15.000 90.07 109.51 90.15
Figure of merit: 0.947 %(<0.1): 98.9 %(<0.2): 99.3 %(<0.3): 99.3
Orientation matrix: -0.04940999 0.06342901 -0.04357016
                   0.03834816 0.06235370 0.04918709
                   0.06973705 0.01104426 -0.02617857
Maximum deviation from integer index [0.25]: 0.25
Percentages of reflections in this domain not consistent with lattice types:
A: 51.4, B: 48.9, C: 48.2, I: 51.1, F: 74.3, O: 68.1 and R: 67.4%
Percentages of reflections in this domain that do not have:
h=2n: 49.6, k=2n: 51.4, l=2n: 50.0, h=3n: 70.7, k=3n: 69.2, l=3n: 65.9%
New cell from list (number), reorientate (R), accept (A) or quit (Q) [A]:
.p4p or .spin file to write domain to: uncn1.p4p
RLATT color-coding employed in file: uncn1.p4p
White: indexed for first domain
Red: not yet indexed
    276 reflections within 0.250 of an integer index assigned to domain 1,
    276 of them exclusively; 2 reflections not yet assigned to a domain
Re-refine initial cell (R), search for next domain (S), quit (Q) or choose
new cell from list (enter number) [Q]: q

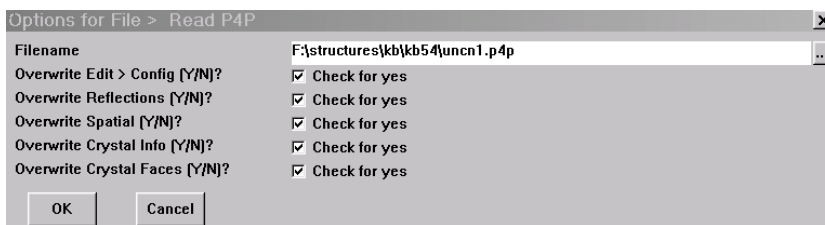
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Cell_now found three solutions. All are very similar. Solution number 1 is the best with 99.3 % coverage and a Figure of Merit of one.

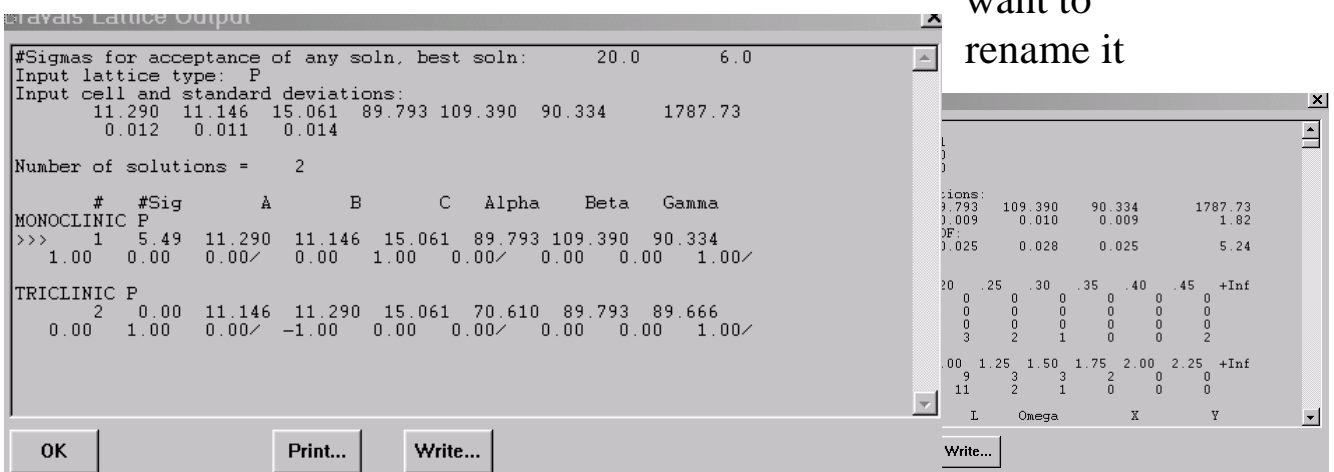
Accept this solution (A) and write a new P4P file in this case

uncn1.p4p. Notice that of the 278 reflections the cell "fits" 276.

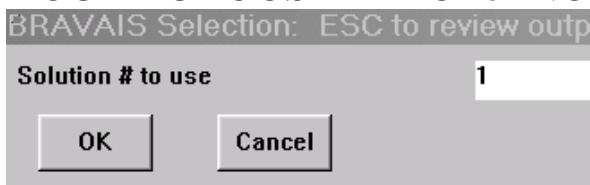
Return to SMART and read in the new file. Point to FILE/READ .P4P. Find the new file.



Point to Crystals/--LS and do a LS refinement on the cell (you may want to rename it



to something else). Check the output it should fit in the good, bad and ugly cell definition. If too bad or too ugly then redo cell_now and find a new cell. Now point to CRYSTAL/--BRAVAIS point to OK and you will see something like this. Your choices are 1 for monoclinic, 2 for triclinic and 0 for no action. WHEN IN DOUBT CHOOSE ZERO for NO ACTION.



. Point to OK. Point to CRYSTAL/--LS and OK to save the Bravais results.

For Twin or Split crystals repeat the procedure. In this case the percent coverage will be lower for each "solution". Pick the "best" solution first and let the program search for a second solution. Save the second solution in a P4P file.

Data Collection Strategies

If your unit cell is greater than 40 Å (Mo Radiation) you will need to move the detector from the normal 5.0 or 6.0 cm distance. If so you will need to collect more than the "normal" set of frames.

Detector Distance (for SMART1000)

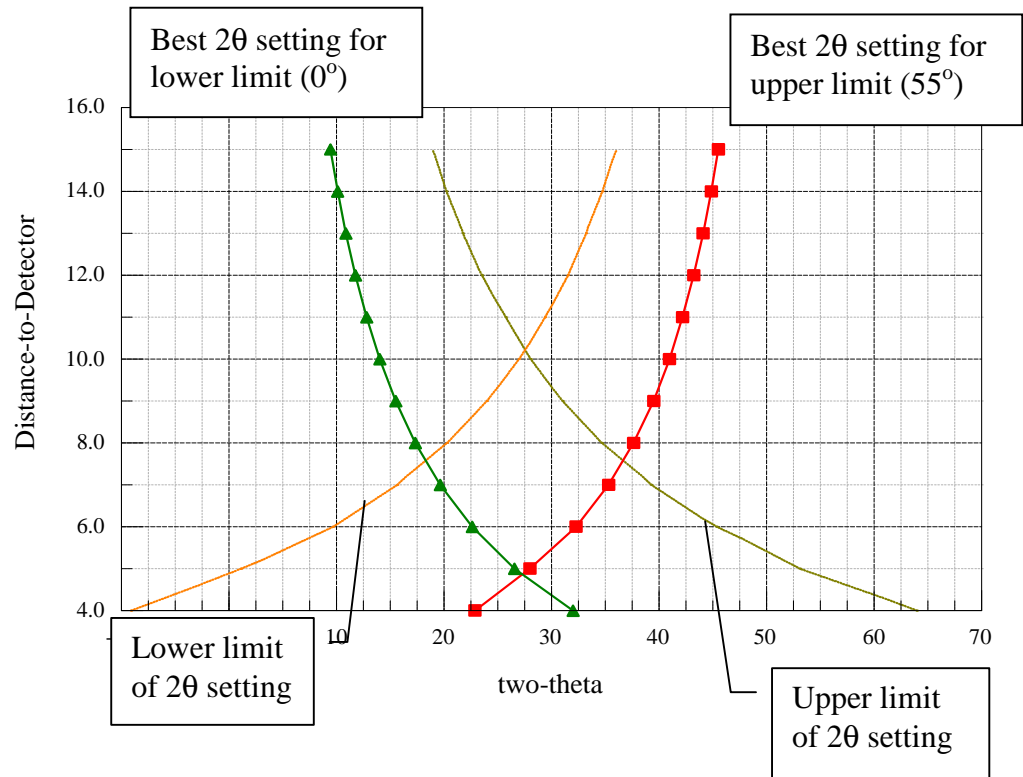
Detector Distance (mm) > 1.5 * Max Cell Length (Å)

Minimum and Maximum 2θ settings for Data Collection to 55° : $2\theta_{\max}$

How to use.

With the distance-to-detector distance from your experiment find the min and max 2θ setting angles. Collect one set of frames at the min 2θ setting and one set at the max 2θ setting. If the difference in the max and min value is large, then you may need to collect an extra set of frames at an intermediate value of 2θ .

For resolution to 55° 2θ (0.770Å)



Suggested 2θ settings for Smart 1000 to resolution to 55° (Mo)

Distance(cm)	Sets of Frames	Lower setting	Mid. Setting	Upper Setting
4	1	-32°	NA	NA
5	1	-28°	NA	NA
6	2	-23	NA	-32°
7	2	-20°	NA	-35°
8	2	-18°	NA	-38°
9	2	-16°	NA	-40°
10	2	-14°	NA	-41°
11	3	-13°	-28°	-42°
12	3	-12°	-28°	-43°
13	3	-11°	-28°	-44°
14	3	-10°	-28°	-45°
15	3	-9.5°	-28°	-45.5°

Cosmo for GADDs

Point to File/Load Configuration. Find cosmo_5.ini (normally in F:\frames\) for the 5 cm detector distance.

Point to File/Import Crystal Data import your file

Point to Refine Strategy and let the program refine a few seconds

Source	HVPC	ZTheta	Omega	Phi	Chi	Axis	Width	#Frames	Sweep
Custom	5.00	-35.00	-35.00	+0.00	+54.70	Omega	-0.50	360	180.00
Custom	5.00	-35.00	-35.00	+90.00	+54.70	Omega	-0.50	360	180.00
Custom	5.00	-35.00	-35.00	+180.00	+54.70	Omega	-0.50	360	180.00
Custom	5.00	-90.00	+270.00	+0.00	+54.70	Omega	-0.50	360	180.00
Custom	5.00	-90.00	+270.00	+90.00	+54.70	Omega	-0.50	360	180.00
Custom	5.00	-90.00	+270.00	+180.00	+54.70	Omega	-0.50	360	180.00
Custom	5.00	-90.00	+270.00	+270.00	+54.70	Omega	-0.50	360	180.00
Intern	5.00	-85.00	-165.00	+359.50	+54.74	Phi	-0.50	719	359.50
Intern	5.00	-25.00	-85.00	+359.50	+54.74	Phi	-0.50	719	359.50
Intern	5.00	-40.00	-160.00	+174.13	+54.74	Phi	-0.50	348	174.00
Intern	5.00	-70.00	-110.00	-0.50	+54.74	Phi	-0.50	213	106.50
Intern	5.00	-20.00	-120.00	+359.50	+54.74	Phi	-0.50	719	359.50
Intern	5.00	-10.00	-75.00	+359.50	+54.74	Phi	-0.50	719	359.50
Intern	5.00	-85.00	-115.00	-0.50	+54.74	Phi	-0.50	124	62.00
Intern	5.00	-85.00	-110.00	-0.50	+54.74	Phi	-0.50	584	292.00
Intern	5.00	-80.00	-180.00	+44.93	+54.74	Phi	-0.50	90	45.00
Intern	5.00	-80.00	-155.00	+213.45	+54.74	Phi	-0.50	427	213.50
Intern	5.00	-80.00	-130.00	+73.02	+54.74	Phi	-0.50	146	73.00
Intern	5.00	-80.00	-120.00	+359.50	+54.74	Phi	-0.50	719	359.50
Intern	5.00	-80.00	-110.00	+359.50	+54.74	Phi	-0.50	719	359.50
Intern	5.00	-80.00	-105.00	+95.49	+54.74	Phi	-0.50	191	95.50
Intern	5.00	-80.00	-95.00	+202.22	+54.74	Phi	-0.50	404	202.00
Intern	5.00	-80.00	-85.00	+213.45	+54.74	Phi	-0.50	427	213.50
Intern	5.00	-75.00	-150.00	-0.50	+54.74	Phi	-0.50	292	146.00
Intern	5.00	-70.00	-140.00	+331.41	+54.74	Phi	-0.50	663	331.50
Intern	5.00	-70.00	-130.00	+254.01	+54.74	Phi	-0.50	528	254.00
Intern	5.00	-90.00	-105.00	+359.50	+54.74	Phi	-0.50	719	359.50
Intern	5.00	-70.00	-100.00	-0.50	+54.74	Phi	-0.50	506	253.00
Intern	5.00	-70.00	-80.00	-0.50	+54.74	Phi	-0.50	67	33.50
Intern	5.00	-70.00	-75.00	+359.50	+54.74	Phi	-0.50	719	359.50

Point and hold (left mouse button)

Refine Strategy and choose the sort runs option let the program sort the runs.

Point to View/Detailed Strategy. The black is the permanent strategy from cosmo_5.ini. The blue is the extra frames from cosmo. Select all of the blue and then right click. Disable the

	Current	Target	Priority
Completeness [%]:	96.47	100.00	100
Redundancy:	2.54	4.00	20
Time [h]:	7.00	24.00	5
Strategy:	Custom		

Sort Runs for Completeness

sets. Return to main menu screen. Take note of the Completeness. In this case the default

Bijvoet Pairs:	merged
Laue Class:	-1
Lattice Type:	P

strategy is 96% complete without any new data sets. This is for a triclinic data set in the space group P-1. I would like to collect ALL of the data for a P1

Bijvoet Pairs:	not merged
Laue Class:	-1
Lattice Type:	P

space group. Therefore change the Bijvoet Pairs to not-merged by pointing to merged and

	Current	Target	Priority
Completeness [%]:	78.06	100.00	100
Redundancy:	1.57	4.00	20
Time [h]:	7.00	24.00	5
Strategy:	Custom		

Sort Runs for Completeness

left clicking. Now check the Completeness.

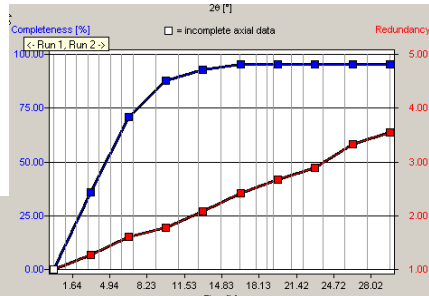
We are not at 78% which is unacceptable (should be near 90%). Run the refine strategy by pointing at the sort runs button and left clicking. Let the program run until at least 90% completeness is seen (higher is better). Note the

results. After run 12 the blue line (% completeness line) levels off. We can stop the data collection after the 12 set of frames and still have a complete data set. Point to View/Detailed Strategy and select all frame sets after # 12, right click them and disable them.

Now pick all the remaining blue sets,

	Current	Target	Priority
Completeness [%]:	95.23	100.00	100
Redundancy:	2.42	4.00	20
Time [h]:	16.45	24.00	5
Strategy:	Custom		

Sort Runs for Completeness



right click them and make them permanent. Return to main menu and note the completeness (in this case ~95%) with ok redundancy 2.4. Point to File/Export strategy and write a text file with the results.

In FRAMBO (or GADDS or SMART) point to COLLECT/SCANS EDITRUNS highlight all of the frame sets and delete them. Point to READ and import the text file you made with COSMO. Edit this file. Remember to always place a space between numbers. (e.g. when editing line 09 place a space between -90.000 and -170.000).

Run#	Frame#	2-Theta	Omega	Phi	Chi	Axis	Width	#Frames	Time
01	001	-35.000	-35.000	0.000	54.700	2	-0.500	360	10.00
02	001	-35.000	-35.000	90.000	54.700	2	-0.500	360	10.00
03	001	-35.000	-35.000	180.000	54.700	2	-0.500	360	10.00
04	001	-90.000	270.000	0.000	54.700	2	-0.500	360	10.00
05	001	-90.000	270.000	90.000	54.700	2	-0.500	360	10.00
06	001	-90.000	270.000	180.000	54.700	2	-0.500	360	10.00
07	001	-90.000	270.000	270.000	54.700	2	-0.500	360	10.00
08	001	-80.000	-95.000	359.500	54.740	3	-0.500	719	10.00
09	001	-90.000	-170.000	359.500	54.740	3	-0.500	719	10.00
10	001	-65.000	-70.000	320.180	54.740	3	-0.500	640	10.00
11	001	-70.000	-130.000	359.500	54.740	3	-0.500	719	10.00
12	001	-50.000	-55.000	-0.500	54.740	3	-0.500	607	10.00

Be sure to use the CURSOR KEYS to move between number NOT THE MOUSE

08	001	-80.000	-95.000	359.500	54.740	3	-0.500	719	10.00
09	001	-90.000	-170.000	359.500	54.740	3	-0.500	719	20.00
10	001	-65.000	-70.000	320.180	54.740	3	-0.500	640	10.00
11	001	-70.000	-130.000	359.500	54.740	3	-0.500	719	10.00

Change the time to fit your data collection etc.

Start the data collection normally.

Data Collection for Absolute Configuration.

For absolute configuration studies you need to collect many Bijvoet Pairs (F(h) and F(-h)). One way to do this is to use Cosmo to determine the data collection strategy. Or a generic data collection which includes both $+2\theta$, $+\theta$ and -2θ , $-\theta$ frames will work. An example is shown below.

Run#	Frame#	2-Theta	Omega	Phi	Chi	Axis Width	#Frames	Time	
1	001	-28.00	-28.00	0.00	54.70	2	-0.300	600	10.00
2	001	28.00	28.00	0.00	54.70	2	-0.300	600	10.00
3	001	-28.00	-28.00	90.00	54.70	2	-0.300	600	10.00
4	001	28.00	28.00	90.00	54.70	2	-0.300	600	10.00
5	001	-28.00	-28.00	180.00	54.70	2	-0.300	600	10.00
6	001	28.00	28.00	180.00	54.70	2	-0.300	600	10.00