Data Collection SMART Advanced

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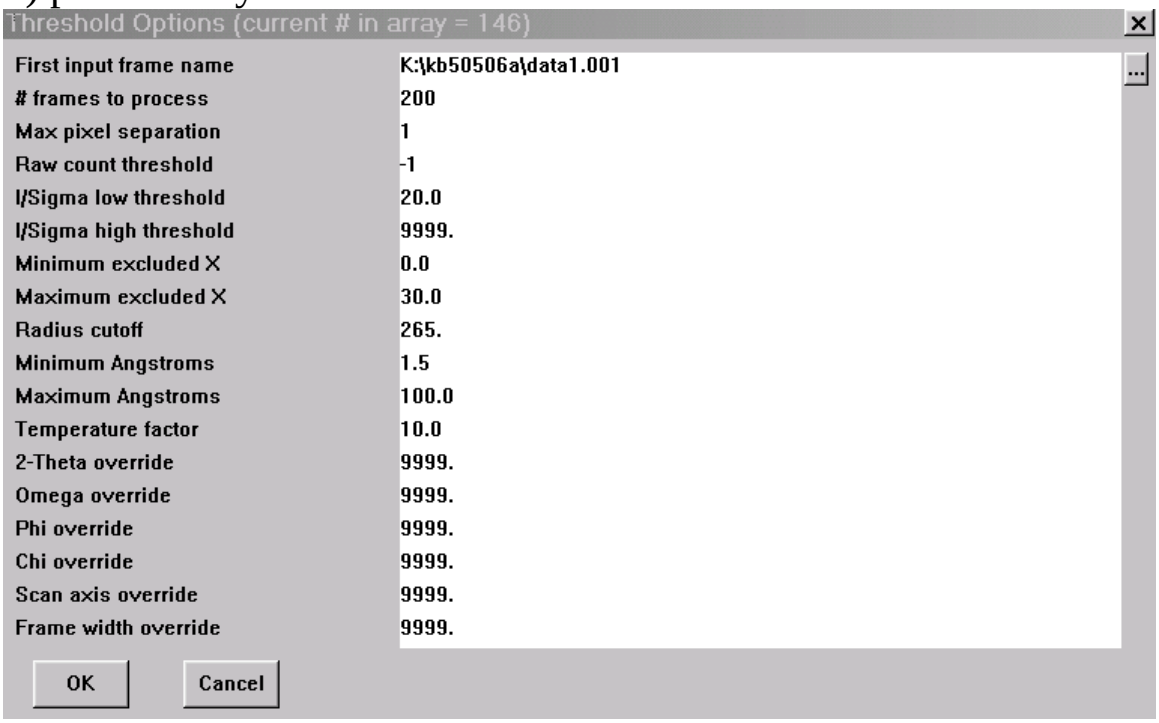
Version 1.0.0

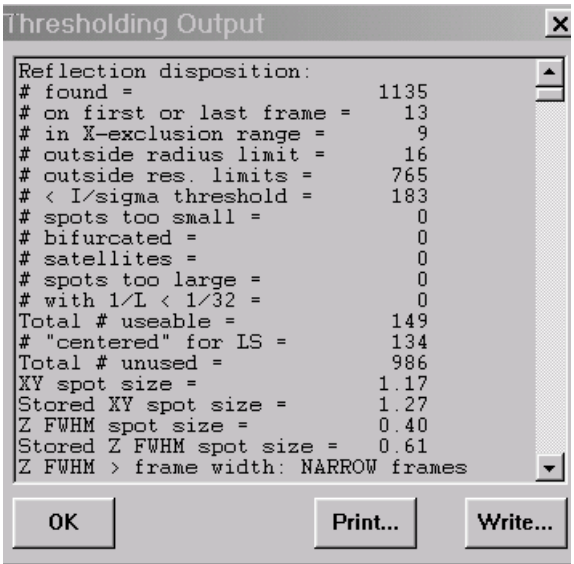
Unit Cell Determination

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



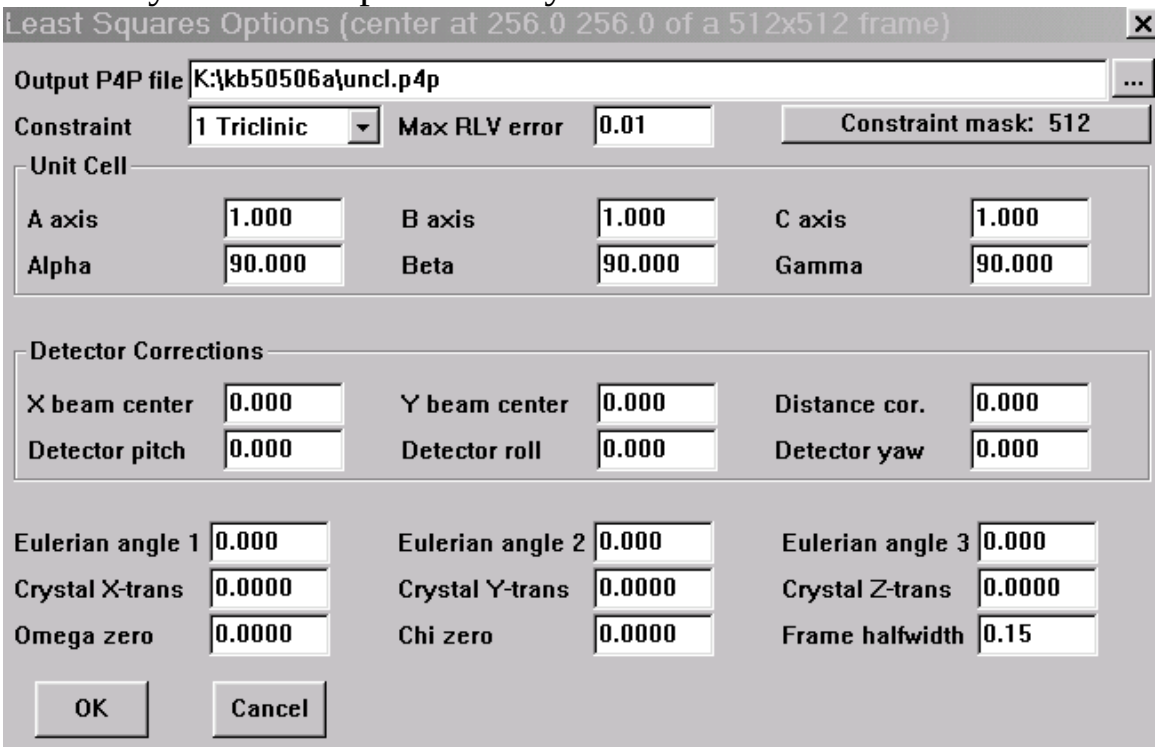
use the ... to find the frame file you want to start at. Set the number of frames (here it is set at 200). Set the raw count and I/sigma low threshold (-1 and 20 are ok for the SMART, 20 and 20 for the GADDS). Point to OK



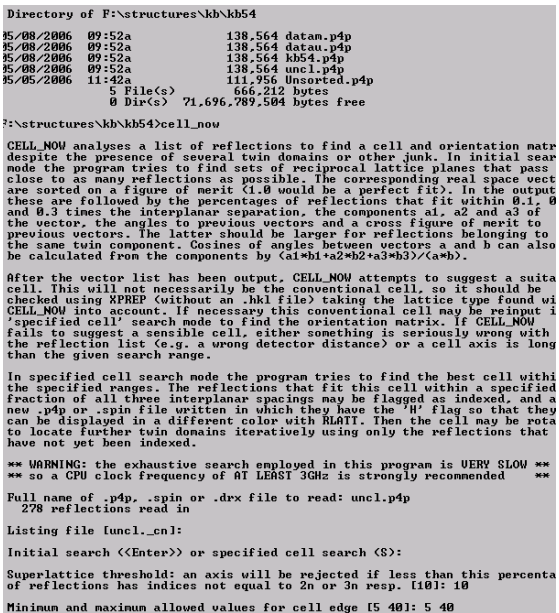
The output information is displayes. The total # useable is 149. If you want more

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



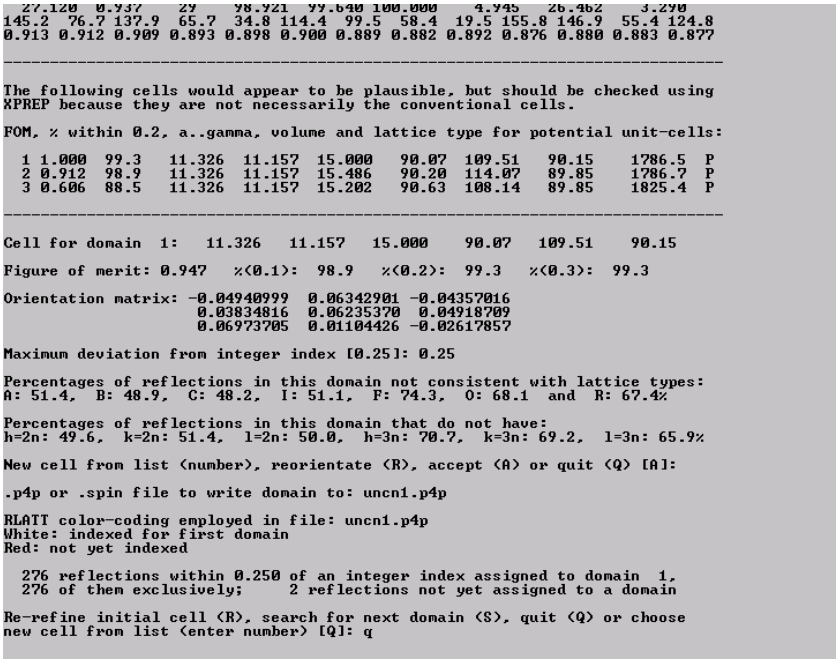
Start a command prompt (point START/RUN 

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

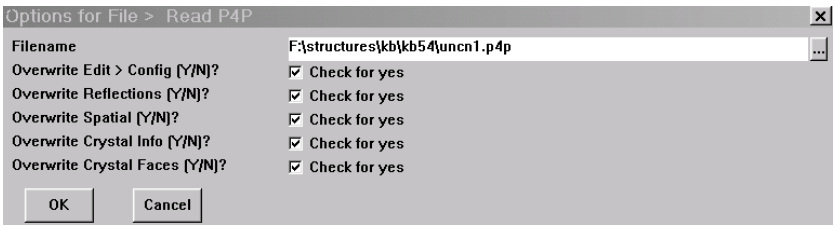
cell edge 5 ,40. If you think you may have a larger cell then change the cell edge setting to 3, 80. Remember this will slow the program down!

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



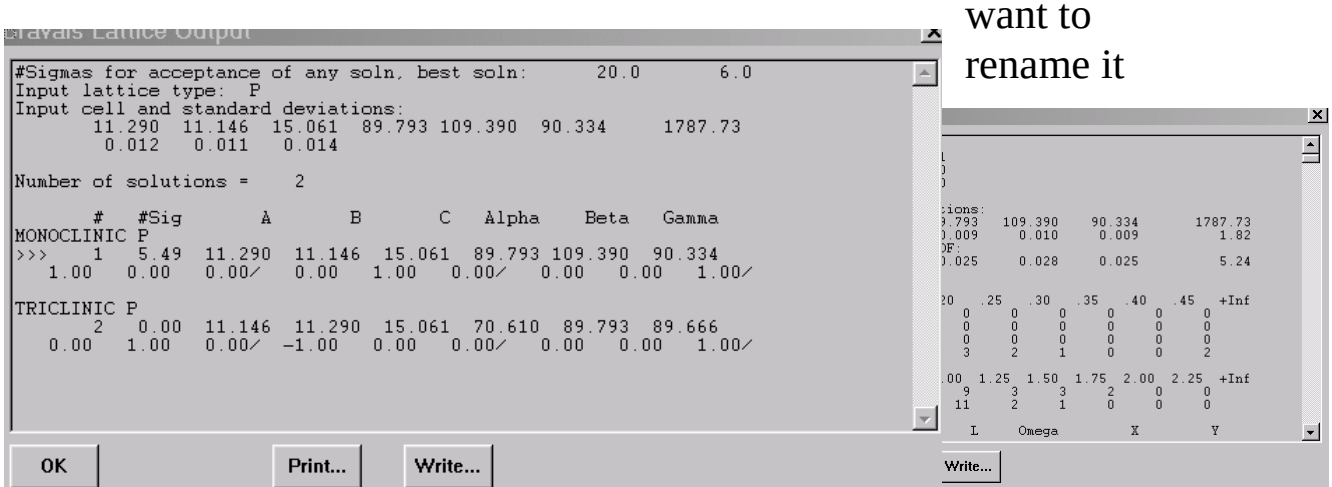
Cell_now found three solutions. All are very similar. Solution number 1 is the best with 99.3 % coverage and a Figure of Merit of one.

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.

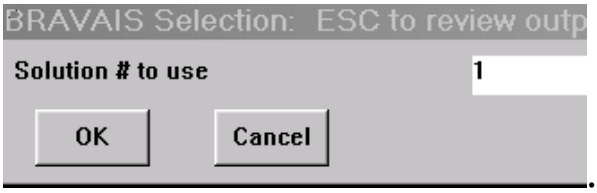


Return to SMART and read in the new file. Point to FILE/READ .P4P. Find the new file.

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 to 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)

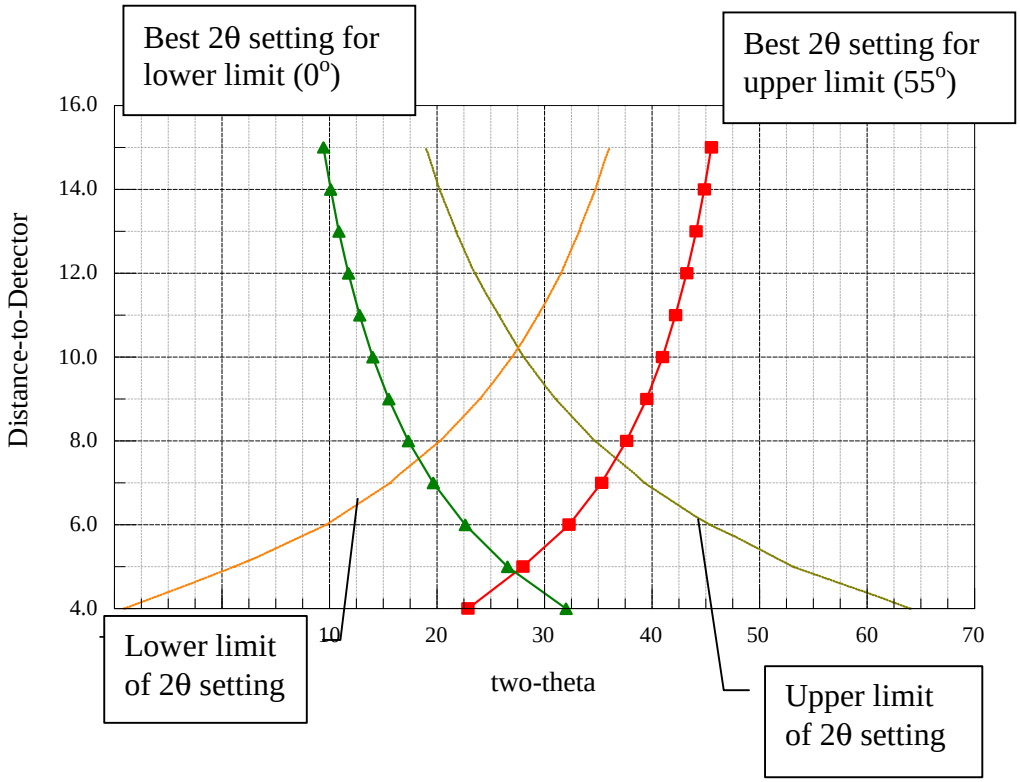
Detector Distance (mm) > 1.5 * Max Cell Length (Å)

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

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^{o} 2θ (0.770Å)


Best 2θ setting for lower limit (0°)
Best 2θ setting for upper limit (55°)
Distance-to-Detector
16.0
14.0
12.0
10.0
8.0
6.0
4.0
10
20
30
40
50
60
70
two-theta
Lower limit of 2θ setting
Upper limit of 2θ setting

Suggested 2θ settings for Smart 1000 to resolution to 55^{o} (Mo)

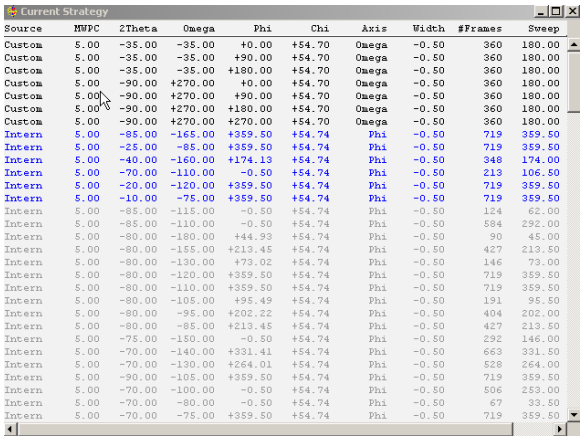
Distance(cm)	Sets of Frames	Lower setting	Mid. Setting	Upper Setting
4	1	-32^{o}	NA	NA
5	1	-28^{o}	NA	NA
6	2	-23	NA	-32^{o}
7	2	-20^{o}	NA	-35^{o}
8	2	-18^{o}	NA	-38^{o}
9	2	-16^{o}	NA	-40^{o}
10	2	-14^{o}	NA	-41^{o}
11	3	-13^{o}	-28^{o}	-42^{o}
12	3	-12^{o}	-28^{o}	-43^{o}
13	3	-11^{o}	-28^{o}	-44^{o}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

Cosmo for GADDS

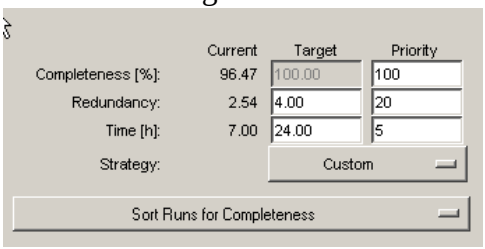
Point to File/Load Configuration. Find cosmo_5.ini (normally in F:\frames\) for the 5 cm detector distance.

Point to File/Import Crystal Data import your file

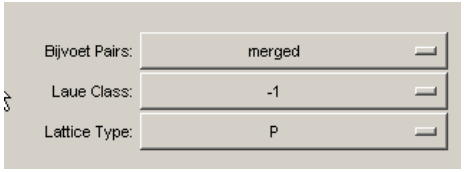
Point to Refine Strategy and let the program refine a few seconds

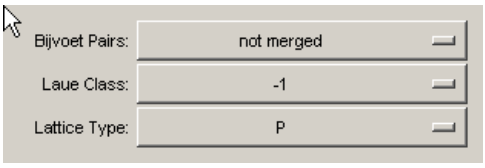


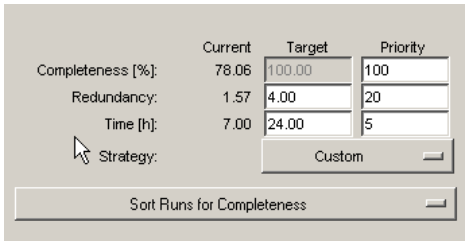
Point and hold (left mouse button) Refine Strategy and choose the sort runs option let the program sort the runs. Point to View/Detailed Strategy. The black is the permanent strategy from cosmo_5.ini. The blue is the extra frames from cosmo. Select all of the blue and then right click. Disable the

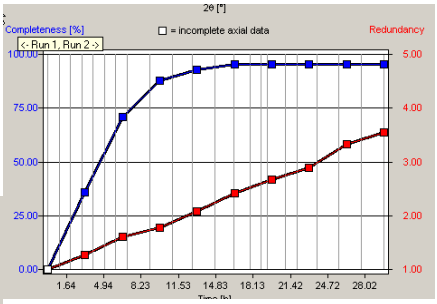


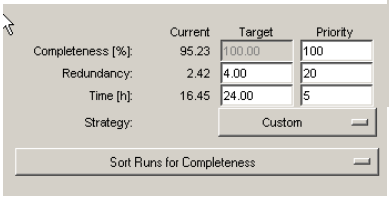
sets. Return to main menu screen. Take note of the Completeness. In this case the default strategy is 96% complete without any new data sets. This is for a triclinic data set in the space group P –1. I would like to collect ALL of the data for a P1 space group. Therefore change the Bijvoet Pairs to not-merged by pointing to merged and 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,





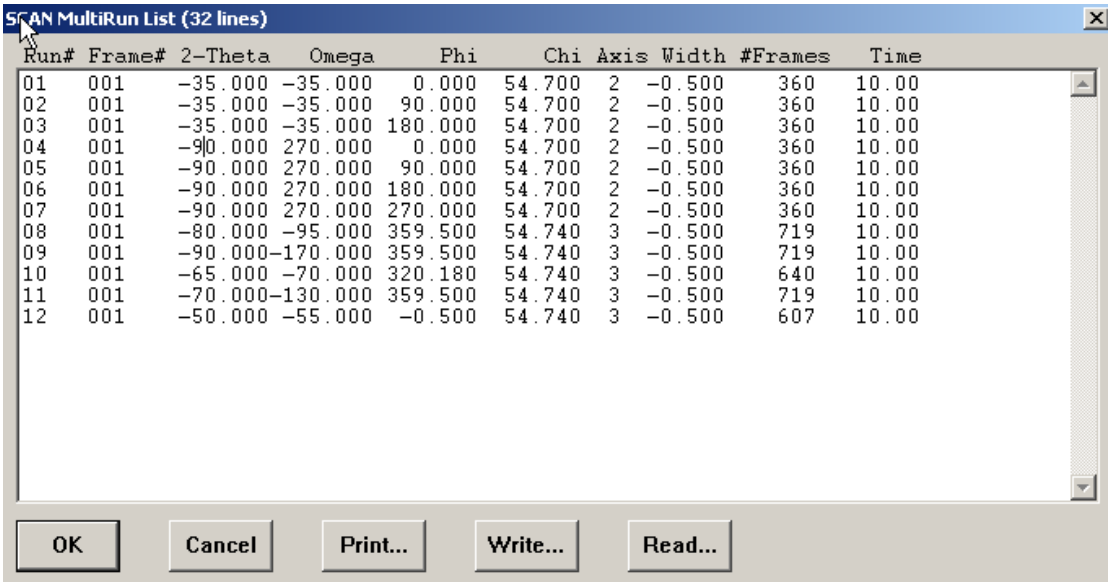






right click them and make them permanent. Return to main menu and note the completeness (in this case ~95%) with ok redundancy 2.4. Point to File/Export strategy and write a text file with the results.

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.



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 +2θ, +θ and -2θ, -θ frames will work. An example is shown below.