

# Data Collection FRAMO/GADDS Advanced

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Version 1.0.0



Data Collection on the GADDS system with FRAMBO or GADDS software.

start VIDEO

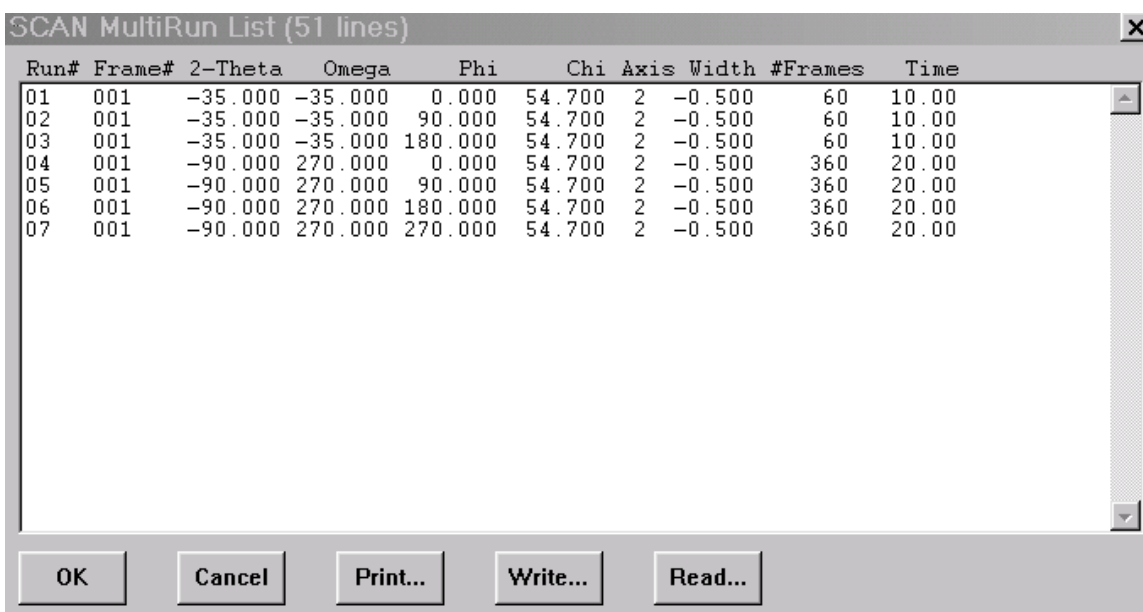
start FRAMBO (or GADDS).

Point to Collect/Goiniometer/Optical

Point to OK

Use the 1,2,3 and 4 keys on the remote keypad (same as A,B,C and D on the SMART remote keypad) to align xtal.

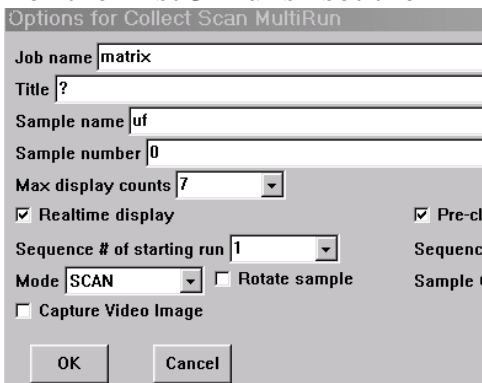
For a short matrix point to Collect/Scan/Editruns.



Run#	Frame#	2-Theta	Omega	Phi	Chi	Axis	Width	#Frames	Time
01	001	-35.000	-35.000	0.000	54.700	2	-0.500	60	10.00
02	001	-35.000	-35.000	90.000	54.700	2	-0.500	60	10.00
03	001	-35.000	-35.000	180.000	54.700	2	-0.500	60	10.00
04	001	-90.000	270.000	0.000	54.700	2	-0.500	360	20.00
05	001	-90.000	270.000	90.000	54.700	2	-0.500	360	20.00
06	001	-90.000	270.000	180.000	54.700	2	-0.500	360	20.00
07	001	-90.000	270.000	270.000	54.700	2	-0.500	360	20.00

Buttons: OK, Cancel, Print..., Write..., Read...

For the first 3 "runs" set the # frames to 60.



Options for Collect Scan MultiRun

Job name: matrix

Title: ?

Sample name: uf

Sample number: 0

Max display counts: 7

Realtime display  Pre-cl

Sequence # of starting run: 1

Mode: SCAN  Rotate sample

Capture Video Image

Buttons: OK, Cancel

Collect a short data set. Point to Collect/Scan/Multirun. For the Job name input matrix. Make sure that the Rotate sample box (next to the Mode box) is not checked. Point to OK

Threshold Options (current # in array = 0)

First input frame name	martix1.001
# frames to process	60
Max pixel separation	1
Raw count threshold	20
I/Sigma threshold	20.0
Minimum excluded X	0.0
Maximum excluded X	30.0
Radius cutoff	265.
Minimum Angstroms	1.5
Maximum Angstroms	100.
Temperature factor	10.
2-Theta override	9999.
Omega override	9999.
Phi override	9999.
Chi override	9999.
Scan axis override	9999.
Frame width override	9999.

OK Cancel

This will take about 30 mins. Stop after the third run. Point to Peaks/Refl.array/Clear and OK. Point to Peaks/Refl.array/Threshold. The First Input Frame Name is matrix1.001. There are 60 frames. Leave the raw count and I/Sigma threshold at 20. Point to OK. You should see around 10 to 100 usable

Thresholding Output

```

Reflection disposition:
# found = 127
# on first or last frame = 5
# in X-exclusion range = 0
# outside radius limit = 2
# outside res. limits = 16
# < I/sigma threshold = 25
# orphan pixels = 27
# bifurcated = 20
Total # useable = 57
# "centered" for LS = 46
Total # unused = 70

  X      Y      Rot  Inorm  #Sig  D Code
244.7 395.9 318.4 232178 197  2.3  C
149.7 362.3 310.0 396108 257  3.7  C
 99.8 269.4 309.3 281806 217  8.7  C
167.2 282.4 303.9 217222 191  4.3  C
257.0 396.8 313.2  98529 128  2.2  C
166.4 362.0 303.7 149629 158  3.4  C
  
```

OK Print... Write...

reflections. If not you may need to collect more frames. Point to OK. Repeat for martix2.001, matrix3.001 etc. You need to save the reflections to a file. Point to Peaks/Refl.array/LeastSQ. Near the bottom of the window you will

Frame halfwidth	0.125
Constraint mask	0
Output parameter filename	uncl.p4p

OK Cancel

see. Change the output file name to fit your needs. Always add the p4p extension. Point to Cancel (not OK). Now point to Point to Peaks/Refl.array/Save and the save

the file to disk. Start a command prompt (point START/RUN  and type cmd ). Navigate to your data directory and type cell\_now. Input the

```

Directory of F:\structures\khh54:
85-08-2006 09:52a      138,564 data_n.p4p
85-08-2006 09:52a      138,564 data_u.p4p
85-08-2006 09:52a      138,564 data_v.p4p
85-08-2006 09:52a      138,564 uncl.p4p
85-08-2006 11:42a      111,956 uncorrected.p4p
5 File(s)           666,212 bytes
0 Dir(s)           71,696,789,584 bytes free

F:\structures\khh54>cell_now

CELL_NOW analyzes a list of reflections to find a cell and orientation matrix despite the presence of several twin domains or other junk. In initial search 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 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 in CELL_NOW into account. If necessary this conventional cell may be output 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 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 'I' flag so that they can be displayed in a different color with RATT. Then the cell may be used 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 **
** to a CPU clock frequency of 01 LEOP3 30Hz is strongly recommended **
Full name of .p4p, .spin or .dpx file to read: uncl.p4p
278 reflections read in

Listing file uncl.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
  
```

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.

```

27.120 0.737 29 98.921 99.640 100.000 4.945 26.462 3.298
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 0.989 0.893 0.898 0.900 0.889 0.882 0.892 0.876 0.880 0.883 0.877

```

The following cells would appear to be plausible, but should be checked using XPREP because they are not necessarily the conventional cells.

FOM, % 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 x<0.1>: 98.9 x<0.2>: 99.3 x<0.3>: 99.3

```

Orientation matrix: -0.04940999 0.06342301 -0.04357016
                   0.03834816 0.06233370 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:  
 0: 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

27% reflections within 0.250 of an integer index assigned to domain 1,  
 27% 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

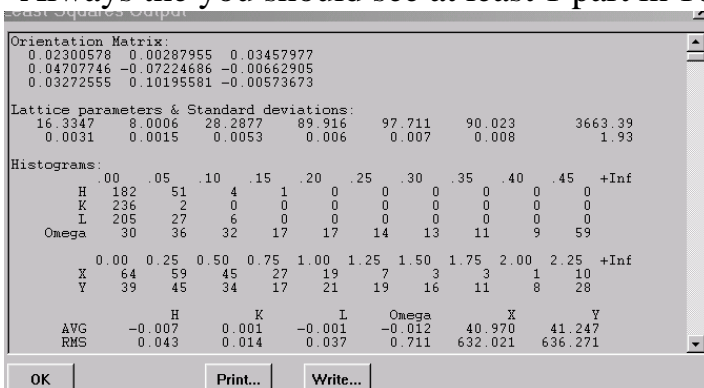
Accept this solution (A) and write a new P4P file in this case uncn1.p4p. Notice that of the 278 reflections the cell "fits" 276.

Return to FRAMBO and read in the new file.

Point to Peaks/Refl.array/Load. Find the new file and



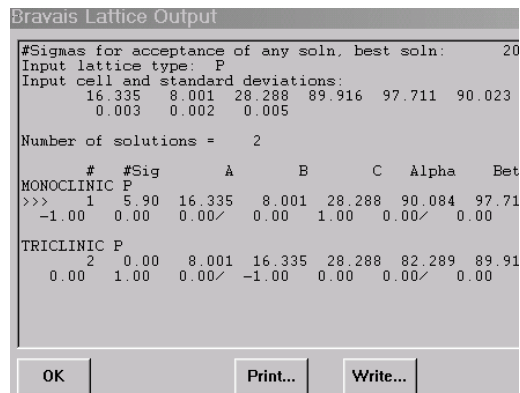
load it. Point to Peaks/Refl.array/LeastSQ and OK. You will see the cell parameters. As Always the you should see at least 1 part in 1000



monoclinic cell. Finally save the cell with the Peaks/Refl.array/Save or Peaks/Refl.array/LeastSQ command.

Now you are ready to collect data.

for the e.s.d. of the Volume for an acceptable cell. Point to Peaks/Refl.array/Bravais and do the reduction. In this case the program predicts a



Start a generic data collection. Point to Collect/Scan/Editruns and edit the data collection runs. Change the 60 back to 360. Change the time as necessary. A good rule of thumb is to collect the first run slow enough so that you will see some reflections near the left hand edge of the detector. For SMART sized crystals this will take only 3 to 10 secs. For very small crystals this may take 60 secs. Do not exceed 60 secs / frame, more time

SCAN MultiRun List (51 lines)

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	5.00
02	001	-35.000	-35.000	90.000	54.700	2	-0.500	360	5.00
03	001	-35.000	-35.000	180.000	54.700	2	-0.500	360	5.00
04	001	-90.000	-90.000	0.000	54.700	2	-0.500	360	15.00
05	001	-90.000	-90.000	90.000	54.700	2	-0.500	360	15.00
06	001	-90.000	-90.000	180.000	54.700	2	-0.500	360	15.00
07	001	-90.000	-90.000	270.000	54.700	2	-0.500	360	15.00
08	001	-60.000	-60.000	0.000	54.700	3	-0.500	719	15.00
09	001	-60.000	180.000	0.000	54.700	3	-0.500	719	15.00

OK Cancel Print... Write... Read...

will only result in more air scatter. If you do need to collect the frames much longer see the manager for a special beam stop apparatus that will reduce air scatter.

Now Point to Collect/Scan/Multirun. Give the data collection a job name. I prefer data and point to OK.

You may want to run COSMO to refine your data collection strategy. See the corresponding handout for details on how to run COSMO for GADDS.

Reduce the data as you would with the SMART data sets.