TWINNED/SPLIT CRYSTALS

CELL_NOW
SAINT
TWINABS

Instructor J. Reibenspies, Ph. D.
Nattamai Bhuvanesh, Ph.D.

Version 1.0.1

Start a command prompt (point START/RUN 

and type cmd). Navigate to your data directory and type cell_now

CELL_NOW - Find unit-cell and matrices for twins and other problem crystals

240 reflections read from file: dpcl.p4p

read in a good P4P file. The file should have a minimum of 100 reflections and no more than 500 reflections.

Searching for vectors with 5.00 < d < 40.00, superlattice threshold = 10.0%

Length, fom, #hits, %(0.1), %(0.2), %(0.3), components. Followed by angle/correlation to previous vectors:

....

Lots of vectors here. Ignore them unless you're insane!

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	72.1	7.881	8.293	11.030	112.34	107.23	115.58	501.6	P
2	0.851	72.1	7.881	8.631	11.496	105.55	113.67	119.81	502.4	P
3	0.779	99.2	16.143	7.881	28.021	90.29	97.63	89.35	3533.0	P
4	0.686	99.2	16.143	7.881	30.424	89.92	114.10	90.65	3532.9	P
5	0.634	63.8	7.881	8.701	16.561	94.59	114.74	98.11	1008.9	P
6	0.607	71.2	7.881	12.493	16.143	75.91	89.35	79.65	1515.6	P
7	0.529	71.7	7.881	8.293	26.141	94.37	98.22	115.58	1507.0	P
8	0.520	71.2	7.881	8.293	26.200	78.12	80.91	64.42	1506.8	P
9	0.518	71.2	7.881	13.525	16.143	102.63	90.65	114.63	1516.0	P
10	0.504	99.2	7.881	16.143	29.144	82.48	74.02	89.35	3532.7	P
11	0.502	98.8	7.881	16.143	29.072	97.16	105.43	90.65	3533.5	P

Ignore solutions with FOM less than .5

1 1.000 72.1 7.881 8.293 11.030 112.34 107.23 115.58 501.6 P
2 0.851 72.1 7.881 8.631 11.496 105.55 113.67 119.81 502.4 P
3 0.779 99.2 16.143 7.881 28.021 90.29 97.63 89.35 3533.0 P

These three solutions stand out. #1 is the smallest cell, but only 72% of the data fits this cell. Number 3 is a much bigger cell (about 7 times) but almost all of the data fits. Solution # 2 is a perturbation of solution #1. Since I know that the Volume of the unit cell should be about 500 I am going to choose this cell. Solution # 3 is a supercell of number 1. Therefore I have chosen cell #1 with a FOM of 1.0.

Cell for domain 1: 7.881 8.293 11.030 112.34 107.23 115.58

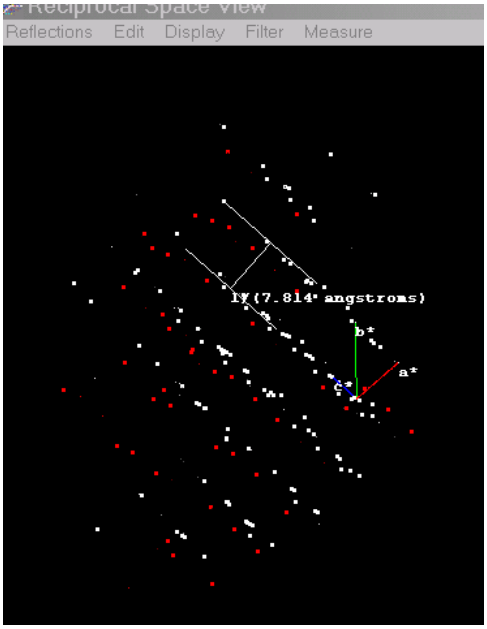
Figure of merit: 0.593 %(0.1): 55.4 %(0.2): 72.1 %(0.3): 78.8

Orientation matrix: -0.11126408 -0.16391023 -0.08167753
0.09265278 -0.02068751 0.06747562
-0.08652867 -0.01022068 0.05087350

Percentages of reflections in this domain not consistent with lattice types:
A: 55.2, B: 54.6, C: 46.6, I: 46.6, F: 78.2, O: 67.2 and R: 67.2%

Percentages of reflections in this domain that do not have:
h=2n: 59.2, k=2n: 59.8, l=2n: 43.7, h=3n: 75.9, k=3n: 66.7, l=3n: 65.5%

RLATT color-coding employed in file: dpcn1.p4p White: indexed for first domain
Red: not yet indexed



RLATT is a program supplied by BRUKER to view the Reciprocal Lattice. The white reflections are indexed and the red are not. Notice that the red reflections are between the white layers, but NOT symmetrically placed (i.e. not 1/2 of the way between the white reflections). The distance between the white lines is 7.8Å which is the same distance between the red lines.

174 reflections within 0.250 of an integer index assigned to domain 1,
174 of them exclusively; 66 reflections not yet assigned to a domain

This line tells you how many of the original 240 reflections were indexed. In this case 174 fit the given cell and 66 do not.

In the program I have written the first cell to dpcn1.p4p

```
Cell for domain  1:    7.881    8.293   11.030   112.34   107.23   115.58

Figure of merit: 0.593   %(0.1):  55.4   %(0.2):  72.1   %(0.3):  78.8

Orientation matrix: -0.11126408 -0.16391023 -0.08167753
                     0.09265278 -0.02068751  0.06747562
                    -0.08652867 -0.01022068  0.05087350

Maximum deviation from integer index [0.25]:

Percentages of reflections in this domain not consistent with lattice types:
A: 55.2,  B: 54.6,  C: 46.6,  I: 46.6,  F: 78.2,  O: 67.2  and  R: 67.2%

Percentages of reflections in this domain that do not have:
h=2n: 59.2,  k=2n: 59.8,  l=2n: 43.7,  h=3n: 75.9,  k=3n: 66.7,  l=3n: 65.5%

New cell from list (number), reorientate (R), accept (A) or quit (Q) [A]:

.p4p or .spin file to write domain to: dpcn1.p4p

RLATT color-coding employed in file: dpcn1.p4p
White: indexed for first domain
Red: not yet indexed
```

Next I Search for another cell that has 7.88 8.293 11.030 112.34 107.23 and 115.58 cell dimensions. I find it.

Cell for domain 2: 7.881 8.293 11.030 112.34 107.23 115.58

Figure of merit: 0.886 %(0.1): 100.0 %(0.2): 100.0 %(0.3): 100.0

Orientation matrix: 0.10375278 0.16385944 0.08160987
0.05416423 0.02197985 -0.06778725
-0.12146208 0.00812149 -0.05056686

Rotated from first domain by 179.9 degrees about
reciprocal axis 1.000 -0.444 -0.425 and real axis 1.000 0.010 -0.002

Twin law to convert hkl from first to this domain (SHELXL TWIN matrix):

1.007 0.019 -0.003
-0.893 -1.009 0.001
-0.851 -0.007 -0.998

The two cells are related by a rotation of 180^{o} around the a axis (7.88Å axis)

RLATT color-coding employed in file: dpcn2.p4p
White: indexed for first domain
Green: current domain (but not in a previous domain)
Red: not yet indexed

164 reflections within 0.250 of an integer index assigned to domain 2,
66 of them exclusively; 0 reflections not yet assigned to a domain

Of the 66 remaining reflections I can fit all 66 to this new cell!

I now create a file called dpcn2.p4p which I will use in the SAINT data reduction program.

==========

Content of dpcn2.p2p

```
FILEID FRAMBO-WNT   V             4.00        08/01/05 08:53:09 dpcl
SITEID Texas A&M University                Bruker Analytical X-Ray Sys
TITLE dpcl
CHEM  ?
CELL      7.8811    8.2935   11.0304  112.3441  107.2251  115.5824    501.623
CELLSD    0.0016    0.0017    0.0022    0.0300    0.0300    0.0300     0.251
ORT1       -0.111264080    -0.163910225    -0.081677526
ORT2        0.092652783    -0.020687509     0.067475617
ORT3       -0.086528674    -0.010220677     0.050873496
ZEROS   0.0000000  0.0000000  0.0000000    0.0000    0.0000    0.0000
ADCOR       -3.0386     -1.1271      0.0630      0.0000     -0.1217      0.3552
```

Cell number 1 with orientation matrix and zeros and corrections

```
CELL2     7.8811    8.2935   11.0304  112.3441  107.2251  115.5824    501.623
CELLSD2   0.0016    0.0017    0.0022    0.0300    0.0300    0.0300     0.251
ORT12       0.103752784     0.163859442     0.081609868
ORT22       0.054164235     0.021979848    -0.067787252
ORT32      -0.121462077     0.008121490    -0.050566856
ZEROS2  0.0000000  0.0000000  0.0000000    0.0000    0.0000    0.0000
ADCOR2      -3.0386     -1.1271      0.0630      0.0000     -0.1217      0.3552
```

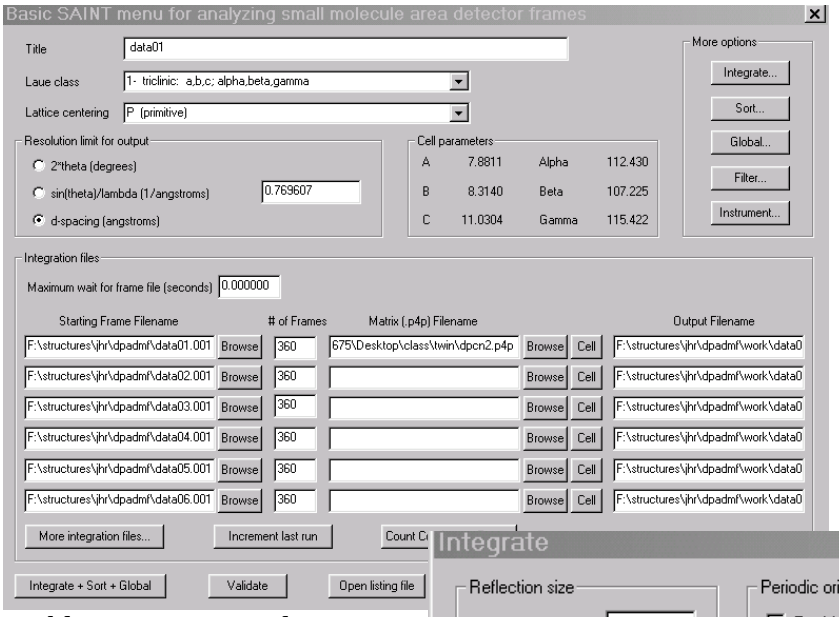
Cell number 2 with orientation matrix and zeros and corrections

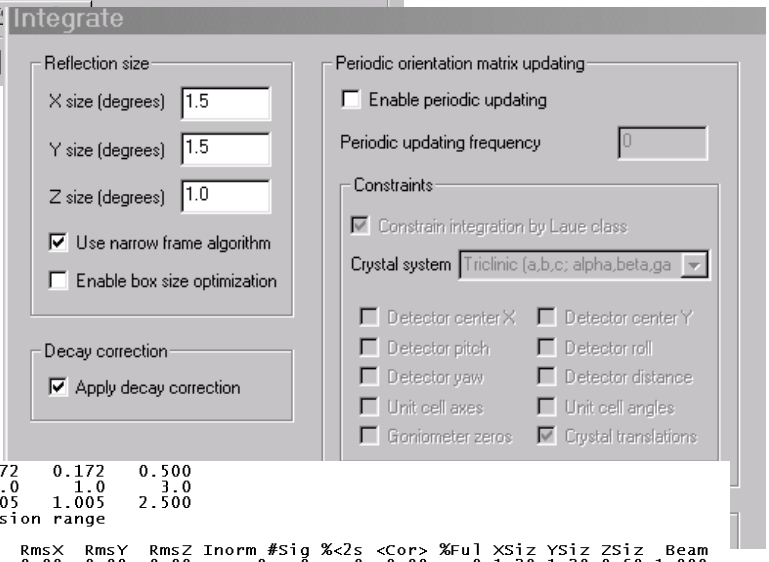
```
SOURCE CU     1.54184   1.54056   1.54439   2.00000   40.00   40.00
LIMITS    0.00    0.00    0.00    0.00    0.00    0.00    0.00    0.00    0.00
0.00
MORPH   ?
DNSMET  ?
CCOLOR  ?
CSIZE  ?           ?           ?           ?           ?
ADPAR      255.6000    255.7000      5.0000     512
REF05 ACHS    0   0   4 325.000 292.850   0.000  54.736 293.25 309.65  214861
189.4    0.326837 -0.269797 -0.204985

... etc ...
```

SAINT will read this file and know automatically to refine BOTH orientations. SAINT will create data files named *.mul, which we will input to TWINABS.

Start SAINT and initialize program. Now BROWSE the matrix file name and find dpcn2.p4p. Check the cell by pointing to Cell button. If ok then delete any names in the remaining Matrix (.p4p) Filename boxes. Be sure to check the more integration files button to check ALL of the raw data sets are covered. Point to the Integrate button. Make sure that the box size is reasonable and that the enable orientation updating is unchecked. Now point to Integrate+Sort+Global and start the integration. You will see a new set of lines in the output, the ALL, 1 and 2. These have the same meaning as those in the normal SAINT output only formatted to show both "twin" components.






```
Profile X,Y,Z spacing (deg):      0.172   0.172   0.500
Profile convolver halfwidth:        1.0     1.0     3.0
Profile convolver expansion:      1.005   1.005   2.500
Rescan threshold is top of A/D conversion range
Background pixels updated =  81.14%
Comp    # File #Ref   ErrX  ErrY  ErrZ  RmsX  RmsY  RmsZ Inorm #Sig %<2s <Cor> %Ful XSiz YSiz ZSiz  Beam
 All    0 .001    0  0.00  0.00  0.00  0.00  0.00  0.00       0    0    0  0.00    0 1.20 1.20 0.60 1.000
   1    0 .001    0  0.00  0.00  0.00  0.00  0.00  0.00       0    0    0  0.00    0 1.20 1.20 0.60 1.000
   2    0 .001    0  0.00  0.00  0.00  0.00  0.00  0.00       0    0    0  0.00    0 1.20 1.20 0.60 1.000

 All    1 .002    1 -3.07 -2.32 -0.21  3.07  2.32  0.21 991.44    5    0  0.11   40 1.20 1.20 0.60 1.000
   1    1 .002    0  0.00  0.00  0.00  0.00  0.00  0.00       0    0    0  0.00    1 1.20 1.20 0.60 1.000
   2    1 .002    1 -3.07 -2.32 -0.21  3.07  2.32  0.21 991.44    5    0  0.11   20 1.20 1.20 0.60 1.000

 All    2 .003    1  0.11 -0.08  0.04  0.11  0.08  0.04 21.738    1  100  0.02    9 1.20 1.20 0.60 1.000
   1    2 .003    1  0.11 -0.08  0.04  0.11  0.08  0.04 21.738    1  100  0.02   27 1.20 1.20 0.60 1.000
   2    2 .003    0  0.00  0.00  0.00  0.00  0.00  0.00       0    0    0  0.00    0 1.20 1.20 0.60 1.000

 All    3 .004    3 -2.29  0.64  0.10  2.61  1.20  0.38 1760.6    5   33  0.11   13 1.20 1.20 0.60 1.000
   1    3 .004    2 -1.48  1.24  0.32  1.60  1.41  0.39 216.09    2   50  0.10   17 1.20 1.20 0.60 1.000
   2    3 .004    1 -3.92 -0.57 -0.34  3.92  0.57  0.34 4849.6   13    0  0.11   10 1.20 1.20 0.60 1.000

 All    4 .005    1 -1.23 -1.82 -0.38  1.23  1.82  0.38 408.82    3    0  0.26   20 1.20 1.20 0.60 1.000
   1    4 .005    0  0.00  0.00  0.00  0.00  0.00  0.00       0    0    0  0.00    0 1.20 1.20 0.60 1.000
   2    4 .005    1 -1.23 -1.82 -0.38  1.23  1.82  0.38 408.82    3    0  0.26   20 1.20 1.20 0.60 1.000

 All    5 .006    2 -2.59  0.66 -0.05  2.60  0.93  0.06  32427   48    0  0.39    7 1.20 1.20 0.60 1.000
   1    5 .006    1 -2.37  1.31 -0.01  2.37  1.31  0.01  30041   46    0  0.02    7 1.20 1.20 0.60 1.000
   2    5 .006    1 -2.82  0.01 -0.08  2.82  0.01  0.08  34812   50    0  0.54    7 1.20 1.20 0.60 1.000
```

in the data0m._ls listing you will see that 6546 *reflections* belonged to only 1 component and 835 *reflections* belonged to both components (i.e. overlapped). In this case 9 files

```
Number of spots with 1 component(s) =         6546
Number of spots with 2 component(s) =          835 (excluded from histograms)
Number of spots with 3 component(s) =            0 (excluded from histograms)
Number of spots with 4 component(s) =            0 (excluded from histograms)
```

ending in *.mul were created. These files will be read by the next program TWINABS to generate the *hkl* file(s) we will use in the twin deconvolution.

TWINABS.

Start a command prompt (point START/RUN 

and type cmd). Navigate to your data directory and type TWINABS.

```
 Directory of C:\Documents and Settings\jhr6675\Desktop\class\twin\work

05/22/2006  10:49a             164,223 data01.mul
05/22/2006  10:52a             163,709 data02.mul
05/22/2006  10:54a             160,368 data03.mul
05/22/2006  10:57a             278,331 data04.mul
05/22/2006  11:00a             255,972 data05.mul
05/22/2006  11:03a             264,967 data06.mul
05/22/2006  11:06a             267,280 data07.mul
05/22/2006  11:10a             284,242 data08.mul
05/22/2006  11:14a             272,420 data09.mul
05/22/2006  11:15a           2,111,512 data0m.mul
              10 File(s)      4,223,024 bytes
               0 Dir(s)   3,300,950,016 bytes free

C:\Documents and Settings\jhr6675\Desktop\class\twin\work>twinabs

 TWINABS - Bruker Nonius scaling and corrections for twinned crystals - V1.05
 ----------------------------------------------------------------------------

 Maximum number of reflections allowed (500000):
 Enter listing filename [twin.abs]:

 Laue group numbers:

 [1] -1                          [8] -3m (rhombohedral axes)
 [2] 2/m (Y unique)              [9] -31m (Z unique)
 [3] mmm                         [10] -3m1 (Z unique)
 [4] 4/m (Z unique)              [11] 6/m (Z unique)
 [5] 4/mmm (Z unique)            [12] 6/mmm (Z unique)
 [6] -3 (rhombohedral axes)      [13] m3
 [7] -3 (Z unique)               [14] m3m

 [0] to write list of equivalent indices for Laue/point groups to listing file

 Enter Laue group number [2]: 1

 Treat Friedel opposites as equivalent for parameter refinement (Y or N) ?
 Answering "N" halves the data to parameter ratio and is not recommended
 unless you have a high redundancy and know what you are doing [Y]:

 Read reflection files written by EVALCCD with extension .sam specified) or by
 SAINT (extension .mul, default if no extension).  Either individual files for
 each scan or a single merged file may be read.  It is important that all files
 are from the same crystal and that reflections have been indexed consistently,
 i.e. that the orientation matrices are similar (no rows with signs reversed)!
```

Choose the correct Laue group and answer Y to the next question. Enter the file name for the first data set, in this case it is data01.mul. Now enter the rest one by one and end with a /.

It is better to use the unmerged data sets at this point. You will need the unmerged data later. Fit the data to a single component (in this case component 2) In this case use the defaults and go to the refinement step

```
 Enter filename (/ if no more) [ ]: data01
 Enter filename (/ if no more) [data02.mul]:
 Enter filename (/ if no more) [data03.mul]:
 Enter filename (/ if no more) [data04.mul]:
 Enter filename (/ if no more) [data05.mul]:
 Enter filename (/ if no more) [data06.mul]:
 Enter filename (/ if no more) [data07.mul]:
 Enter filename (/ if no more) [data08.mul]:
 Enter filename (/ if no more) [data09.mul]:
 Enter filename (/ if no more) [data010.mul]: /

  2 twin components present

 Mean and maximum errors in direction cosine check function =   0.001   0.005
 The mean error should not exceed 0.008, and is usually caused by matrix
 changes during data processing.

 Maximum 2-theta = 124.89 deg.   Approximate wavelength = 1.54178 Angstroms

 ============================================================================

 PART 1 - Refinement of parameters to model systematic errors

     3273 data (   1201 unique ) involve component  1 only,  mean I/sigma  22.1
     3273 data (   1205 unique ) involve component  2 only,  mean I/sigma  21.2
      835 data (    400 unique ) involve   2 components,  mean I/sigma  27.8

 Enter N to fit component N and apply to all data, e.g. when one component
 dominates;  -N for one  parameter set to fit equivalent singles and composites
 that contain at least one contribution from domains 1..N;  or 0 to fit each
 component separately [-2]: -2
```

The refinement will model the absorption surface. The refinement went well so accept the results. Go to the outlier rejection step

```
Refinement of a single parameter set to fit all single and composite
reflections that contain at least one contribution from domains 1.. 2
------------------------------------------------------------------------------
   4711 single and    550 overlapped reflections used for parameter fitting
Effective data to parameter ratio =    0.96

R(int) =  0.0585 (selected reflections only, before parameter refinement)

Cycle  R(incid)  R(diffr)  Mean wt.
   1    0.0536    0.0512    0.9379
   2    0.0504    0.0495    0.9414
   3    0.0497    0.0495    0.9420
   4    0.0497    0.0495    0.9422
   5    0.0496    0.0495    0.9423
   6    0.0497    0.0495    0.9422
   7    0.0497    0.0496    0.9422
   8    0.0497    0.0496    0.9422
   9    0.0497    0.0496    0.9421
  10    0.0498    0.0497    0.9421
  11    0.0498    0.0497    0.9421
  12    0.0498    0.0497    0.9420
  13    0.0499    0.0498    0.9420
  14    0.0499    0.0498    0.9419
  15    0.0499    0.0498    0.9419

R(int) =  0.0498 (selected reflections only, after parameter refinement)

Repeat parameter refinement (R) or accept (A) [A]: a
```

and use the defaults. The g is predicted to be 0.085 which is good and should be accepted. Do not accept anything over 0.2.

```
Statistics for singles for twin component  1
-------------------------------------------
 Run 2-theta  R(int)  Incid. factors  Diffr. factors    K     Total I>2sig(I>
   1  -35.0  0.0504  2.805 - 3.063  0.897 - 1.063  0.752     249      221
   2  -35.0  0.0473  2.463 - 2.856  0.868 - 1.038  0.751     262      234
   3  -35.0  0.0399  2.903 - 3.234  0.880 - 1.113  0.765     273      244
   4  -90.0  0.0359  0.366 - 0.417  0.897 - 1.202  0.604     391      334
   5  -90.0  0.0316  0.351 - 0.416  0.863 - 1.042  0.613     380      324
   6  -90.0  0.0353  0.311 - 0.383  0.880 - 1.111  0.580     385      322
   7  -90.0  0.0267  0.296 - 0.358  0.863 - 1.054  0.518     368      316
   8  -60.0  0.0407  0.875 - 1.058  0.864 - 1.427  0.627     449      409
   9  -60.0  0.0452  0.802 - 0.947  0.879 - 1.002  0.622     445      409

Statistics for singles for twin component  2
-------------------------------------------
 Run 2-theta  R(int)  Incid. factors  Diffr. factors    K     Total I>2sig(I>
   1  -35.0  0.0466  2.798 - 3.053  0.898 - 1.062  0.856     245      209
   2  -35.0  0.0399  2.463 - 2.855  0.867 - 1.043  0.766     264      231
   3  -35.0  0.0475  2.901 - 3.243  0.880 - 1.184  0.784     274      242
   4  -90.0  0.0305  0.365 - 0.417  0.898 - 1.326  0.581     402      342
   5  -90.0  0.0294  0.351 - 0.416  0.865 - 1.056  0.583     371      312
   6  -90.0  0.0374  0.311 - 0.382  0.880 - 1.104  0.615     382      314
   7  -90.0  0.0335  0.298 - 0.358  0.865 - 1.059  0.572     380      307
   8  -60.0  0.0471  0.878 - 1.056  0.862 - 1.417  0.654     438      389
   9  -60.0  0.0424  0.792 - 0.947  0.882 - 1.008  0.691     443      393

Statistics for all composite reflections
----------------------------------------
 Run 2-theta  R(int)  Incid. factors  Diffr. factors    K     Total I>2sig(I>
   1  -35.0  0.0368  2.800 - 3.063  0.907 - 0.968  0.856      63       52
   2  -35.0  0.0234  2.540 - 2.846  0.888 - 1.059  0.559      50       47
   3  -35.0  0.0201  3.133 - 3.245  0.880 - 1.058  0.485      31       29
   4  -90.0  0.0331  0.367 - 0.414  0.907 - 1.016  0.554     132      118
   5  -90.0  0.0524  0.351 - 0.416  0.880 - 1.059  0.645     118      106
   6  -90.0  0.0313  0.317 - 0.382  0.884 - 1.036  0.550     120      108
   7  -90.0  0.0428  0.299 - 0.358  0.863 - 1.060  0.651     139      120
   8  -60.0  0.0425  0.900 - 1.058  0.863 - 1.207  0.730      92       87
   9  -60.0  0.0371  0.794 - 0.947  0.909 - 0.970  0.637      77       74

Statistics for all single and composite reflections
---------------------------------------------------
 Run 2-theta  R(int)  Incid. factors  Diffr. factors    K     Total I>2sig(I>
   1  -35.0  0.0463  2.798 - 3.063  0.897 - 1.063  0.811     557      484
   2  -35.0  0.0400  2.463 - 2.856  0.867 - 1.059  0.745     576      512
   3  -35.0  0.0403  2.901 - 3.245  0.880 - 1.184  0.761     578      514
   4  -90.0  0.0331  0.365 - 0.417  0.897 - 1.326  0.588     925      795
   5  -90.0  0.0347  0.351 - 0.416  0.863 - 1.059  0.604     869      744
   6  -90.0  0.0356  0.311 - 0.383  0.880 - 1.111  0.592     887      744
   7  -90.0  0.0324  0.296 - 0.358  0.863 - 1.060  0.558     887      746
   8  -60.0  0.0436  0.875 - 1.058  0.862 - 1.427  0.650     979      885
   9  -60.0  0.0427  0.792 - 0.947  0.879 - 1.008  0.656     965      876

su = K * Sqrt[ sigma^2(I) + (g(I>)^2 ]  where sigma(I) is estimated by SAINT
```

The statistics for the data set are good. Note the low R(int) and the similar K values. This indicates an acceptable correction. Keep it and continue.

Output reflections, here we choose to output a standard *hkl* file with only one component, which we name DPA_HKLF4.hkl and a non-standard *hkl* file with both twin components (1 and 2) as

```
PART 3 - Output Postscript diagnostics and corrected data

Write Postscript diagnostic file (Y or N) [Y]: n
It is now possible to generate a HKLF 4 format file with crude intensities
for structure solution or a more accurate HKLF 5 format file for refinement.
Repeat (R), write .hkl file (4 or 5), or quit (Q) [4]: 4
Average equivalent reflections (Y or N) [Y]: Y
Enter name of output .hkl file [twin.hkl]: DPA_HKLF4.HKL
Mu*r of equivalent sphere for additional spherical absorption correction.
Enter (CR) if none:
Average Friedel opposites in output file (Y or N) [Y]: Y
Use component N only (recommended); 0 to use all [1]: 1

   1471 Corrected reflections written to file DPA_HKLF4.HKL

Reflections merged according to point-group -1
Ratio of minimum to maximum apparent transmission:  0.604371


It is now possible to generate a HKLF 4 format file with crude intensities
for structure solution or a more accurate HKLF 5 format file for refinement.
Repeat (R), write .hkl file (4 or 5), or quit (Q) [5]: 5
Average equivalent reflections (Y or N) [Y]: Y
Enter name of output .hkl file [twin.hkl]: DPA_HKLF5.HKL
Mu*r of equivalent sphere for additional spherical absorption correction.
Enter (CR) if none:
Average Friedel opposites in output file (Y or N) [Y]: Y
Output only reflections containing component N (0 to output all) [1]: 0

   3193 Corrected reflections written to file DPA_HKLF5.HKL

Reflections merged according to point-group -1
Ratio of minimum to maximum apparent transmission:  0.603891


It is now possible to generate a HKLF 4 format file with crude intensities
for structure solution or a more accurate HKLF 5 format file for refinement.
Repeat (R), write .hkl file (4 or 5), or quit (Q) [Q]:
```

DPA_HKL5.hkl. Notice for the first file I asked for component 1 when prompted and for the next file I asked for both. Also the first file is in HKLF 4 format and the 2nd file is in HKLF 5 format. We will use them both later.

You now have two *hkl* files. Use the HKLF4 file to solve the structure and refine it. If the solution and refinement look good that is all you may want to do.

I solved and refined the structure using the HKLF 4 data set in the normal way. The residuals refined to

_refine_ls_R_factor_all	0.0561
_refine_ls_R_factor_gt	0.0488
_refine_ls_wR_factor_ref	0.1832
_refine_ls_wR_factor_gt	0.1711
_refine_ls_goodness_of_fit_ref	1.076
_refine_ls_restrained_S_all	1.076
_diffrn_reflns_theta_full	62.12
_diffrn_measured_fraction_theta_full	0.899

This is perfectly acceptable, but we can do better if we refine the HKLF5 data. In this case insert the line **BASF 0.5** before the FVAR line and change the HKLF 4 line to HKLF 5. Now refine the file with the DPA_HKLF5.hkl data. The new residuals are

_refine_ls_R_factor_all	0.0459
_refine_ls_R_factor_gt	0.0397
_refine_ls_wR_factor_ref	0.1206
_refine_ls_wR_factor_gt	0.1130
_refine_ls_goodness_of_fit_ref	1.056
_refine_ls_restrained_S_all	1.056
_diffrn_reflns_theta_full	62.55
_diffrn_measured_fraction_theta_full	0.962

The R(all) decreased more than a percent while the wR(all) decreased more than 6 %. Also the percent coverage increased from ~90% to ~96%.

The BASF which is the refined component scale is ~ 0.49 or a 50/50 mixture of twin components.