How-to-do Twin/Platon
Twins: My Doppelgänger
Pusedo-merohedral twin

- twin operator belongs to a higher crystal system than the structure
- seen with monoclinic and triclinic cells with angles near 90^o (beta angle for monoclinic) or with cells where two (or more) cell axis are almost equal.

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Example.
This is an example of a triclinic cell with two of the cell angles are near 90^o
This example is for the Mn(L)N_3 molecule. The data was collected on a BRUKER APEXII CCD. The program Cell_Now found a cell that looks monoclinic but in fact it is triclinic.

```
CELL_NOW - Find unit-cell and matrices for twins and other problem crystals
  1051 reflections read from file: ddm.p4p
 Searching for vectors with  5.00 < d <  40.00, superlattice threshold = 10.0%

  1 1.000  87.9   6.478  18.645  14.373   90.59  104.75   90.10   1678.9  P
  2 0.942  87.9   6.478  18.645  14.175   89.32  101.47   90.10   1677.9  P
  3 0.757  87.3   6.478  18.645  17.212   90.47  126.09   90.10   1680.0  P
  4 0.695  87.5   6.478  18.645  16.720   89.35  123.79   90.10   1678.3  P
  5 0.579  87.7   6.478  14.175  19.738   85.59   70.73   78.53   1676.7  P
  6 0.565  86.7   6.478  14.175  19.755   86.84   70.93   78.53   1680.3  P
  7 0.563  87.7   6.478  14.373  19.738   85.78   70.73   75.25   1677.7  P
  8 0.550  86.7   6.478  14.373  19.755   84.71   70.93   75.25   1681.2  P
  9 0.424  86.4   6.478  14.175  22.724   82.88   55.08   78.53   1676.9  P
 ...
 Cell for domain  1:   6.478   18.645   14.373   90.59   104.75   90.10

 Figure of merit: 0.587   %(0.1):  63.2   %(0.2):  87.9   %(0.3):  94.5

 Orientation matrix:  0.11784112  0.01607645 -0.02841868
                      0.05287077 -0.05043570  0.00141103
                     -0.09378863 -0.00863764 -0.06608317

 Percentages of reflections in this domain not consistent with lattice types:
 A: 49.5,  B: 48.6,  C: 49.2,  I: 49.1,  F: 73.7,  O: 67.5  and  R: 67.1%

 Percentages of reflections in this domain that do not have:
 h=2n: 47.3,  k=2n: 49.6,  l=2n: 49.6,  h=3n: 61.3,  k=3n: 69.9,  l=3n: 66.0%

  969 reflections within 0.250 of an integer index assigned to domain  1,
  969 of them exclusively;    82 reflections not yet assigned to a domain
```

~92% indexed!
Chain of events …
Collect Data → Reduce Data → Scale Data (sadabs) → XPREP

The first sign that something is wrong! High R(sym) for monoclinic cell!
(But not outrageous!)

```
Search for higher METRIC symmetry
Identical indices and Friedel opposites combined before calculating R(sym)

------------------------------------------------------------------------------
Option A: FOM = 0.196 deg.   MONOCLINIC   P-lattice   R(sym) = 0.168 [   3484]
Cell:    6.369  18.468  14.048   90.11  101.26   89.82    Volume:      1620.61
Matrix: 1.0000  0.0000  0.0000  0.0000 -1.0000  0.0000 -1.0000  0.0000 -1.0000
------------------------------------------------------------------------------
Option B: FOM = 0.000 deg.   TRICLINIC    P-lattice   R(sym) = 0.000 [      0]
Cell:    6.369  14.048  18.468   89.89   89.82   78.74    Volume:      1620.61
Matrix: 1.0000  0.0000  0.0000  1.0000  0.0000  1.0000  0.0000 -1.0000  0.0000
------------------------------------------------------------------------------
Option C retains original cell

Select option [B]:
```

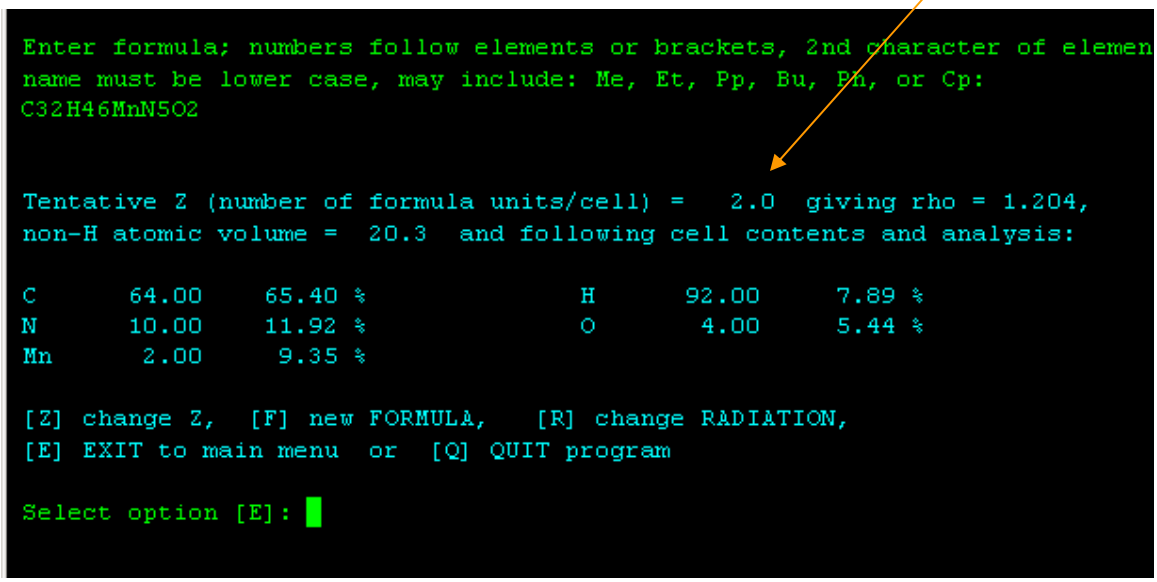
Choosing the monoclinic cell will eventually lead to an unusual space group (P2) and an unsolvable structure. You may need to try this first just to convince yourself it is not correct. In this example the Triclinic cell is chosen.

The next indication that something is wrong is the low E^2-1 value of 0.691

```
Lattice exceptions: P      A      B      C      I      F      Obv    Rev    All

N (total) =          0   8654   8651   8659   8646  12982  11534  11545  17330
N (int>3sigma) =     0   3668   3643   3675   3623   5493   4887   4887   7362
Mean intensity =   0.0   14.1   12.9   13.4   14.0   13.5   13.8   13.8   13.8
Mean int/sigma =   0.0    4.3    4.2    4.3    4.3    4.3    4.3    4.3    4.3

Lattice type [P, A, B, C, I, F, O(obv.), R(rev. rhomb. on hex. axes)]

Select option [P]:


Mean |E*E-1| = 0.691 [expected .968 centrosym and .736 non-centrosym]

Systematic absences not required for triclinic

Identical indices and Friedel opposites combined before calculating R(sym)

Option  Space Group  No.  Type  Axes  CSD  R(sym) N(eq)  Syst. Abs.   CFOM

[A] P-1           #  2  centro   1  8646  0.000      0  0.0 /  4.3   9.99
[B] P1            #  1  chiral   1   700  0.000      0  0.0 /  4.3   2.67

Select option [B]:
```

This low E^2-1 value indicates a possible twin. You may wish to choose the P -1 space group, but experience has shown that the lower P1 space group will normally return a solution which can be converted back to P -1 at a later time.

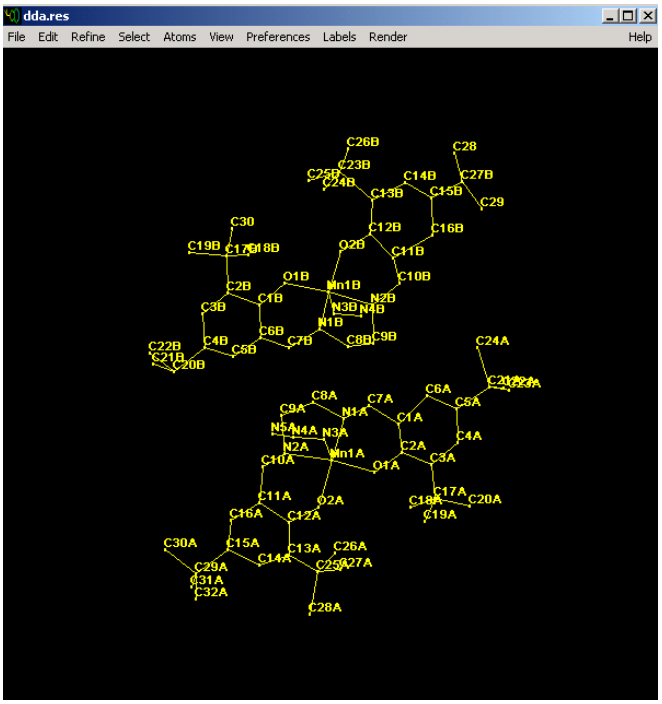
Another indication that P -1 is the true space group is the Z value. A value of 2 is normal for P-1 space groups. A value of one (for a molecule without a center of symmetry) would be normal for P1 space groups.



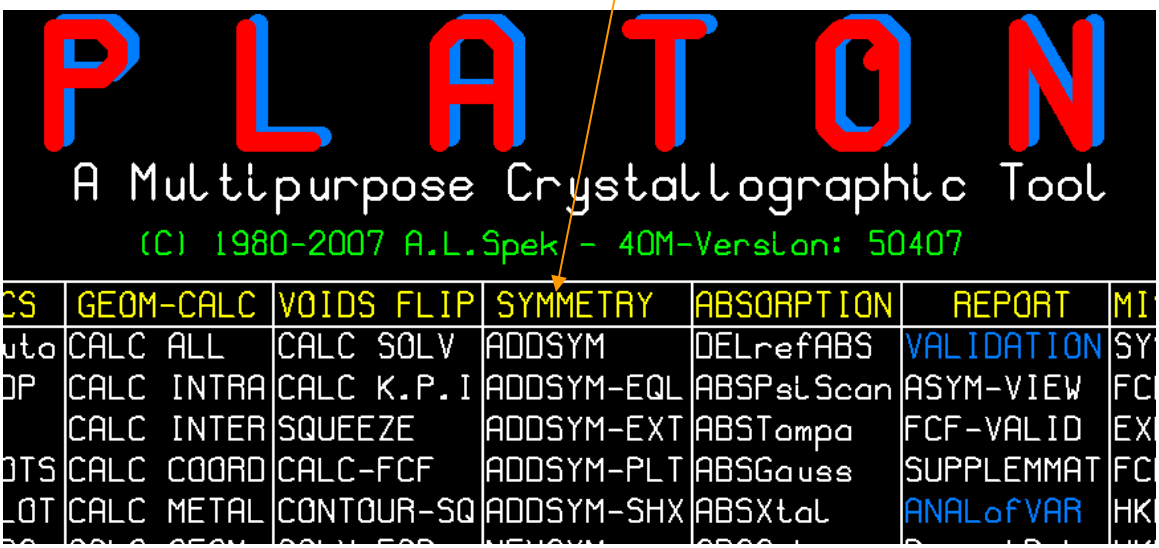
Generate the INS file (P1 space group) and run SHELXS.
The solution looks like this

```
TITL dda in P1
CELL 0.71073  6.3691  14.0483  18.4678  89.886  89.821  78.742
ZERR   2.00   0.0060   0.0148   0.0173   0.017   0.012   0.011
LATT -1
SFAC C  H  N  O  Mn
UNIT 64 92 10 4 2
L.S. 2
BOND
FMAP 2
PLAN 90
WGHT  0.200000  0.000000
FVAR  0.203070
TEMP 0
MOLE 1
Mn1A   5   0.195442  0.742356  0.097330 11.000000  0.035680
O1A    4   0.116358  0.640325  0.049509 11.000000  0.042550
O2A    4   0.147533  0.824006  0.007321 11.000000  0.034190
N1A    3   0.343656  0.646129  0.172088 11.000000  0.026450
...
```

SHELXS solution has two Mn(L)N_3 molecules. One of the two is finished while the 2nd is missing a few terminal atoms. Name all atoms (as many as you can find) and save as a RES file.



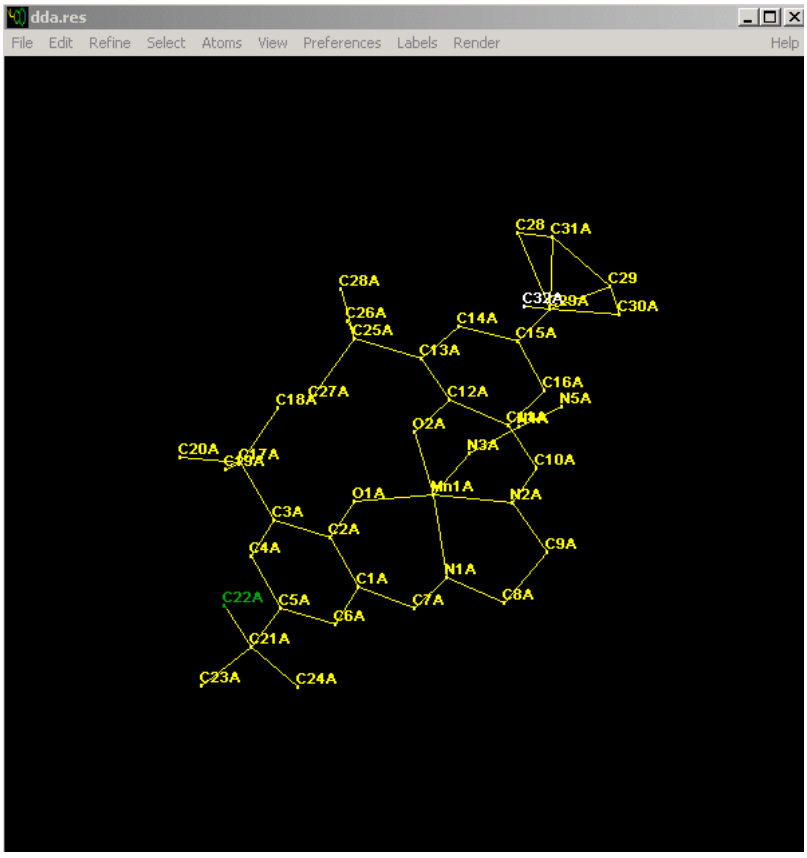
Start the program PLATON (Speck, 2001) and point to the ADDSYM (Le Page, 1988) command.



ADDSYM will find the P -1 space group, return to the main menu and point to the ADDSYM-SHX to write the SHELX RES file. (You can skip the ADDSYM command and go directly to the ADDSYM-SHX if you are sure of the results)

```
                    PLATON/ADDSYM for dda in P1
ADDSYM Search on ALL NON-H Chemical Types [Max NonFit 20 Perc]
Criteria:  1.00 Deg (Metric), 0.25 Ang (Rot.), 0.45 Ang (Inv), 0.45 Ang (Transl)
Symm.  Input  Reduced  (Ang)        (Deg) ( )   (Ang)                 Input Cell
Elem Cell Row Cell Row   d  Typ Dot Angle Fit  MaxDev.              x     y     z
-1 * ==================================   93   0.406  at      0.187 0.188 0.281
                                          C31A -C28

  Reduced-to-Convent       Input-to-Reduced     T = Input-to-Convent:   a' = T a

(    1    0    0 )   (    1    0    0 )   (    1    0    0 )      Det(T)
(    0    1    0 ) X (    0    1    0 ) = (    0    1    0 )        =
(    0    0    1 )   (    0    0    1 )   (    0    0    1 )      1.000

Cell Lattice  a       b       c       Alpha   Beta    Gamma   Volume CrystalSystem Laue
Input    aP  6.369  14.048  18.468  89.89   89.82   78.74   1621    Triclinic    -1
Reduced  P   6.369  14.048  18.468  89.89   89.82   78.74   1621
Convent  aP  6.369  14.048  18.468  89.89   89.82   78.74   1621    Triclinic    -1
Origin shifted to: 0.187, 0.188, 0.281 after transformation
Missed/Additional Symmetry (Ignore Nonfit): Suggested SPGR = P-1          (No   2)
 *** PLEASE COMPARE with 'CALC ADDSYM EXACT'
```

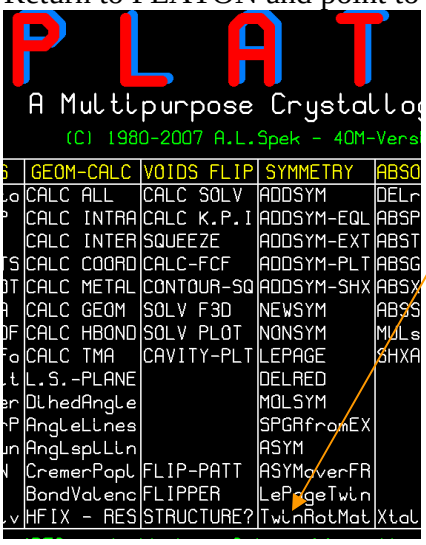
The RES file generated from PLATON can be input to XSHEL or XSEED. Notice a few atoms such as C28 and C29 are out of place. Clean up the structure save as an INS file and run a refinement



The refinement is still very poor (R1 ~27%) so it is now time to look for twins. The structure is now in the P-1 space group.

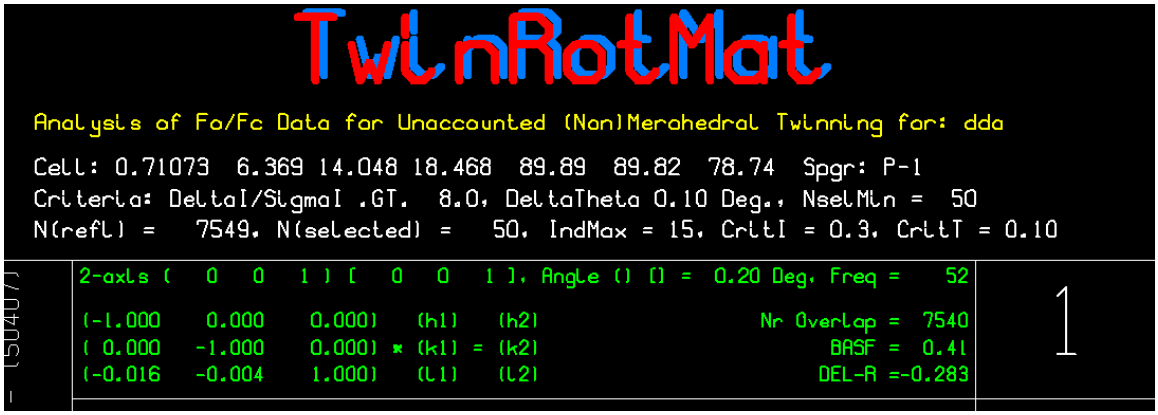
```
+  dda           finished at 08:48:04   Total elapsed time:      27.7 secs  +

C:\Documents and Settings\jhr6675.CHXRDPC6\DESKTOP\DDMN1>xl dda

+  XL  - CRYSTAL STRUCTURE REFINEMENT - SHELXTL Ver. 6.12 W95/98/NT/2000/ME  +
+  Copyright(c) 2001 Bruker Analytical X-ray Solutions  All Rights Reserved  +
+  dda                                  started at 09:05:42 on 04-May-2007  +

Read instructions and data
Data:    7549 unique,      0 suppressed   R(int) = 0.0965   R(sigma) = 0.1595
Systematic absence violations:    0    Bad equivalents:    2
  ** Cell contents from UNIT instruction and atom list do not agree **
wR2 =  0.7200 before cycle   1 for   7549 data and   161 /   161 parameters
GooF = S =     6.016;     Restrained GooF =     6.016  for      0 restraints
Mean shift/esd =   1.256    Maximum = -29.217 for  OSF              at 09:05:43
Max. shift = 0.147 A for C29A      Max. dU =-0.035 for C29A
wR2 =  0.6368 before cycle   2 for   7549 data and   161 /   161 parameters
GooF = S =     3.473;     Restrained GooF =     3.473  for      0 restraints
Mean shift/esd =   0.805    Maximum =   4.849 for  U11 C31A       at 09:05:43
Max. shift = 0.056 A for C11A      Max. dU = 0.023 for C30A
wR2 =  0.6195 before cycle   3 for   7549 data and   161 /   161 parameters
GooF = S =     3.198;     Restrained GooF =     3.198  for      0 restraints
Mean shift/esd =   0.312    Maximum =   1.652 for  U11 C31A       at 09:05:44
Max. shift = 0.032 A for C30A      Max. dU = 0.011 for C32A
wR2 =  0.6169 before cycle   4 for   7549 data and   161 /   161 parameters
GooF = S =     3.156;     Restrained GooF =     3.156  for      0 restraints
Mean shift/esd =   0.141    Maximum =  -0.661 for   x  C30A       at 09:05:45
Max. shift = 0.023 A for C30A      Max. dU = 0.004 for C32A
wR2 =  0.6164 before cycle   5 for   7549 data and   161 /   161 parameters
GooF = S =     3.149;     Restrained GooF =     3.149  for      0 restraints
Mean shift/esd =   0.070    Maximum =  -0.384 for   x  C30A       at 09:05:46
Max. shift = 0.013 A for C30A      Max. dU = 0.001 for C32A
wR2 =  0.6162 before cycle   6 for   7549 data and     0 /   161 parameters
GooF = S =     3.148;     Restrained GooF =     3.148  for      0 restraints
R1 =  0.2721 for   4132 Fo > 4sig(Fo)  and  0.3168 for all   7549 data
wR2 =  0.6162,  GooF = S =   3.148,  Restrained GooF =    3.148  for all data
R1 =  0.3117 for   7549 unique reflections after merging for Fourier
Highest peak    1.74  at  0.9888  0.4568  0.4496  [  0.86 A from C27A ]
Deepest hole   -1.52  at  0.0279  0.6568  0.4508  [  2.08 A from C19A ]

+  dda              finished at 09:05:46   Total CPU time:       4.5 secs  +
```

Return to PLATON and point to the TwinRotMat button (Cooper et al., 2002)



After a few seconds (minutes) you should see a new screen. PLATON will suggest a few TWIN matrices. The matrices with positive BASF numbers are the solutions. If the BASF number is small (below .04) or is zero then the matrix is not useable.

In this case the matrix -1 0 0 0 -1 0 0 0 1 has a BASF number of 0.41. Write this matrix down and add it as a TWIN command in the INS file.




```
TITL dda in P1                New: P-1
CELL  0.71073   6.3690  14.0480  18.4680   89.886   89.821   78.742
ZERR 1        0.0060   0.0150   0.0170    0.017    0.012    0.011
LATT 1
SFAC C  H  Mn N  O
UNIT 68 0 2 10 4
L.S. 5
ACTA
BOND $H
FMAP 2
PLAN 5
FVAR  1.000000
TWIN -1 0 0 0 -1 0 0 0 1
BASF  0.41
```

Add the TWIN and BASF instruction.

(It is possible to generate a HKLF 5 file that can be employed in SHELXL. This mode is suggested only if you know what you know what you are doing!)

Repeat the refinement.

```
C:\Documents and Settings\jhr6675.CHXRDPC6\DESKTOP\DDMN1>XL DDA

 ++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++
 +  XL  - CRYSTAL STRUCTURE REFINEMENT - SHELXTL Ver. 6.12 W95/98/NT/2000/ME  +
 +  Copyright(c) 2001 Bruker Analytical X-ray Solutions  All Rights Reserved  +
 +  dda                              started at 09:10:40 on 04-May-2007  +
 ++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++

 Read instructions and data
 Data:    7549 unique,      0 suppressed   R(int) = 0.0965   R(sigma) = 0.1595
 Systematic absence violations:    0    Bad equivalents:    2
   ** Cell contents from UNIT instruction and atom list do not agree **
 wR2 =  0.4636 before cycle   1 for   7549 data and   162 /   162 parameters
 GooF = S =     2.677;      Restrained GooF =      2.677  for      0 restraints
 Mean shift/esd =   1.325    Maximum = -26.423 for  OSF             at 09:10:42
 Max. shift = 0.089 A for C29A       Max. dU =-0.031 for C12A
 wR2 =  0.3789 before cycle   2 for   7549 data and   162 /   162 parameters
 GooF = S =     1.785;      Restrained GooF =      1.785  for      0 restraints
 Mean shift/esd =   1.453    Maximum = -15.000 for BASF  1          at 09:10:43
 Max. shift = 0.094 A for C27A       Max. dU =-0.023 for C29A
 wR2 =  0.3269 before cycle   3 for   7549 data and   162 /   162 parameters
 GooF = S =     1.518;      Restrained GooF =      1.518  for      0 restraints
 Mean shift/esd =   1.115    Maximum = -10.692 for BASF  1          at 09:10:44
 Max. shift = 0.074 A for C30A       Max. dU = 0.017 for C31A
 wR2 =  0.3152 before cycle   4 for   7549 data and   162 /   162 parameters
 GooF = S =     1.456;      Restrained GooF =      1.456  for      0 restraints
 Mean shift/esd =   0.262    Maximum =  -2.601 for   x  C30A        at 09:10:45
 Max. shift = 0.038 A for C30A       Max. dU = 0.004 for C31A
 wR2 =  0.3147 before cycle   5 for   7549 data and   162 /   162 parameters
 GooF = S =     1.454;      Restrained GooF =      1.454  for      0 restraints
 Mean shift/esd =   0.084    Maximum =  -0.804 for   x  C30A        at 09:10:46
 Max. shift = 0.012 A for C30A       Max. dU =-0.001 for C11A
 wR2 =  0.3146 before cycle   6 for   7549 data and     0 /   162 parameters
 GooF = S =     1.452;      Restrained GooF =      1.452  for      0 restraints
 R1 =  0.1199 for   4132 Fo > 4sig(Fo)  and  0.1772 for all   7549 data
 wR2 =  0.3146,  GooF = S =   1.452,  Restrained GooF =    1.452  for all data
 R1 =  0.1642 for   7549 unique reflections after merging for Fourier
 Highest peak    0.69  at  0.9438  0.4361  0.1975  [  0.43 A from MN1A ]
 Deepest hole   -1.63  at  0.9605  0.4875  0.1977  [  0.64 A from MN1A ]

 ++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++
 +  dda                 finished at 09:10:47   Total CPU time:       7.2 secs  +
 ++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++

C:\Documents and Settings\jhr6675.CHXRDPC6\DESKTOP\DDMN1>
```

The R1 has dropped fro 27% to 12% ! You are on the right course. Complete the structure refinement in the usual manner (add H, aniso etc).

```
 ++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++
 +  XL  - CRYSTAL STRUCTURE REFINEMENT - SHELXTL Ver. 6.12 W95/98/NT/2000/ME  +
 +  Copyright(c) 2001 Bruker Analytical X-ray Solutions  All Rights Reserved  +
 +  dda     started at 12:00:15 on 04-May-2007  +
 ++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++

 Read instructions and data
 Data:    7549 unique,      0 suppressed   R(int) = 0.0965   R(sigma) = 0.1595
 Systematic absence violations:    0    Bad equivalents:    2
 wR2 =  0.2545 before cycle   1 for   7549 data and   374 /   374 parameters
 GooF = S =     1.203;      Restrained GooF =      1.203  for      0 restraints
 Mean shift/esd =   0.632    Maximum =  -9.371 for BASF  1          at 12:00:21
 Max. shift = 0.108 A for H28C       Max. dU = 0.004 for C19A
 wR2 =  0.2452 before cycle   2 for   7549 data and   374 /   374 parameters
 GooF = S =     1.135;      Restrained GooF =      1.135  for      0 restraints
 Mean shift/esd =   0.266    Maximum =  -2.927 for BASF  1          at 12:00:26
 Max. shift = 0.049 A for H28C       Max. dU = 0.002 for N3A
 wR2 =  0.2454 before cycle   3 for   7549 data and   374 /   374 parameters
 GooF = S =     1.134;      Restrained GooF =      1.134  for      0 restraints
 Mean shift/esd =   0.043    Maximum =  -0.230 for   x  C2A         at 12:00:31
 Max. shift = 0.004 A for H28C       Max. dU = 0.000 for N3A
 wR2 =  0.2454 before cycle   4 for   7549 data and   374 /   374 parameters
 GooF = S =     1.134;      Restrained GooF =      1.134  for      0 restraints
 Mean shift/esd =   0.015    Maximum =   0.119 for  U13 Mn1A        at 12:00:36
 Max. shift = 0.002 A for H27B       Max. dU = 0.000 for C7A
 wR2 =  0.2454 before cycle   5 for   7549 data and   374 /   374 parameters
 GooF = S =     1.134;      Restrained GooF =      1.134  for      0 restraints
 Mean shift/esd =   0.004    Maximum =   0.018 for  U12 C10A        at 12:00:41
 Max. shift = 0.000 A for H22B       Max. dU = 0.000 for C10A
 wR2 =  0.2454 before cycle   6 for   7549 data and     0 /   374 parameters
 GooF = S =     1.134;      Restrained GooF =      1.134  for      0 restraints
 R1 =  0.0919 for   4132 Fo > 4sig(Fo)  and  0.1519 for all   7549 data
 wR2 =  0.2454,  GooF = S =   1.134,  Restrained GooF =    1.134  for all data

 ** Extinction (EXTI) or solvent water (SWAT) correction may be required **

 R1 =  0.1411 for   7549 unique reflections after merging for Fourier
 Highest peak    0.67  at  0.9755  0.4569  0.1204  [  1.19 A from MN1A ]
 Deepest hole   -1.36  at  0.9166  0.4198  0.2034  [  0.74 A from MN1A ]

 ++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++
 +  dda              finished at 12:00:42   Total CPU time:      26.3 secs  +
 ++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++
```

After refinement the R1 is 9% and the wR2 is near 24%. I still have a few bad reflections, these can be taken care of with the OMIT command and addition of the OMIT -4 50 the refinement is finished.

```
+  XL  - CRYSTAL STRUCTURE REFINEMENT - SHELXTL Ver. 6.12 W95/98/NT/2000/ME  +
+  Copyright(c) 2001 Bruker Analytical X-ray Solutions  All Rights Reserved  +
+  dda                                  started at 09:17:04 on 04-May-2007  +

Read instructions and data
Data:    5535 unique,      0 suppressed   R(int) = 0.0871   R(sigma) = 0.1197
Systematic absence violations:    0    Bad equivalents:    2
wR2 =  0.1699 before cycle   1 for   5535 data and   374 /   374 parameters
GooF = S =     1.023;     Restrained GooF =      1.023  for      0 restraints
Mean shift/esd =   0.051    Maximum =   0.672 for BASF  1          at 09:17:08
Max. shift = 0.005 A for H32B      Max. dU = 0.000 for C7A
wR2 =  0.1698 before cycle   2 for   5535 data and   374 /   374 parameters
GooF = S =     1.022;     Restrained GooF =      1.022  for      0 restraints
Mean shift/esd =   0.016    Maximum =   0.198 for BASF  1          at 09:17:12
Max. shift = 0.001 A for H24B      Max. dU = 0.000 for C1A
wR2 =  0.1698 before cycle   3 for   5535 data and   374 /   374 parameters
GooF = S =     1.022;     Restrained GooF =      1.022  for      0 restraints
Mean shift/esd =   0.002    Maximum =   0.012 for  U11 C5A         at 09:17:15
Max. shift = 0.000 A for H26C      Max. dU = 0.000 for C19A
wR2 =  0.1698 before cycle   4 for   5535 data and   374 /   374 parameters
GooF = S =     1.022;     Restrained GooF =      1.022  for      0 restraints
Mean shift/esd =   0.001    Maximum =   0.005 for  OSF             at 09:17:19
Max. shift = 0.000 A for H22A      Max. dU = 0.000 for C7A
wR2 =  0.1698 before cycle   5 for   5535 data and   374 /   374 parameters
GooF = S =     1.022;     Restrained GooF =      1.022  for      0 restraints
Mean shift/esd =   0.000    Maximum =   0.001 for BASF  1          at 09:17:23
Max. shift = 0.000 A for H30B      Max. dU = 0.000 for N4A
wR2 =  0.1698 before cycle   6 for   5535 data and     0 /   374 parameters
GooF = S =     1.022;     Restrained GooF =      1.022  for      0 restraints
R1 =  0.0714 for   3813 Fo > 4sig(Fo)  and  0.1036 for all   5535 data
wR2 =  0.1698,  GooF = S =   1.022,  Restrained GooF =    1.022  for all data
R1 =  0.0972 for   5535 unique reflections after merging for Fourier
Highest peak    0.48  at  0.8034  0.4119  0.2333  [  1.58 A from O2A ]
Deepest hole   -0.88  at  0.9033  0.4248  0.2074  [  0.80 A from MN1A ]

+  dda               finished at 09:17:23   Total CPU time:      19.4 secs  +
```

The final refinement now has a R1=7% and a wR2 = 17%. The BASF for the twin matrix has refined to 0.39 (very close to the 0.41 predicted by PLATON)

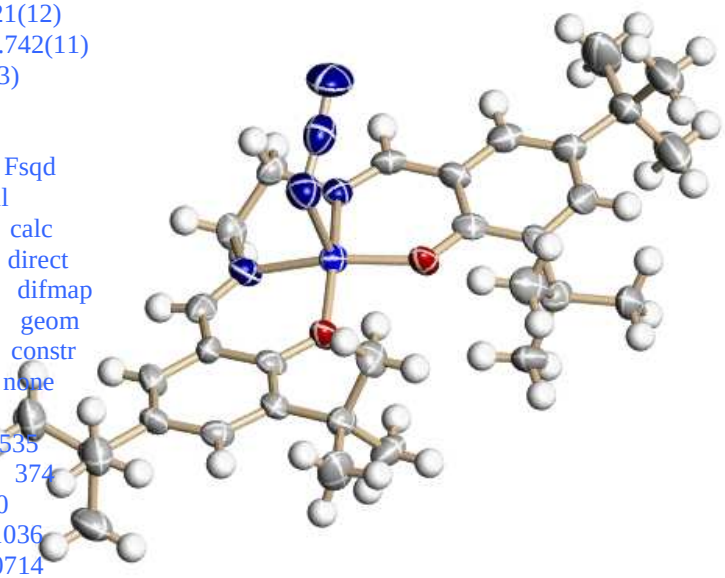
TWIN -1 0 0 0 -1 0 0 0 1
BASF 0.39869

```
TwinRotMat

Analysis of Fo/Fc Data for Unaccounted (Non)Merohedral Twinning for: dda

Cell: 0.71073  6.369 14.048 18.468  89.89  89.82  78.74  Spgr: P-1
Criteria: DeltaI/SigmaI .GT.  8.0, DeltaTheta 0.10 Deg., NselMin =  50
N(refl) =   7549, N(selected) =   50, IndMax = 15, CritI = 0.3, CritT = 0.10

2-axis (   0   0   1 ) [   0   0   1 ], Angle () [] =  0.20 Deg, Freq =   52

(-1.000    0.000    0.000)   (h1)   (h2)          Nr Overlap =  7540
( 0.000   -1.000    0.000) x (k1) = (k2)               BASF =  0.41
(-0.016   -0.004    1.000)   (l1)   (l2)           DEL-R =-0.283

                                                        1
```

The final CIF is checked and the structure is finished.

_cell_length_a	6.369(6)
_cell_length_b	14.048(15)
_cell_length_c	18.468(17)
_cell_angle_alpha	89.886(17)
_cell_angle_beta	89.821(12)
_cell_angle_gamma	78.742(11)
_cell_volume	1621(3)
_cell_formula_units_Z	2

_refine_ls_structure_factor_coef	Fsqd
_refine_ls_matrix_type	full
_refine_ls_weighting_scheme	calc
_atom_sites_solution_primary	direct
_atom_sites_solution_secondary	difmap
_atom_sites_solution_hydrogens	geom
_refine_ls_hydrogen_treatment	constr
_refine_ls_extinction_method	none
_refine_ls_extinction_coef	?
_refine_ls_number_reflns	5535
_refine_ls_number_parameters	374
_refine_ls_number_restraints	0
_refine_ls_R_factor_all	0.1036
_refine_ls_R_factor_gt	0.0714
_refine_ls_wR_factor_ref	0.1698
_refine_ls_wR_factor_gt	0.1516
_refine_ls_goodness_of_fit_ref	1.022
_refine_ls_restrained_S_all	1.022
_refine_ls_shift/su_max	0.001
_refine_ls_shift/su_mean	0.000



COOPER, R. I., GOULD, R. O., PARSONS, S. & WATKIN, D. J. (2002) The derivation of non-merohedral twin laws during refinement by analysis of poorly fitting intensity data and the refinement of non-merohedrally twinned crystal structures in the program CRYSTALS. *Journal of Applied Crystallography*, 35**,** 168-174.

LE PAGE, Y. (1988) MISSYM1.1 - a flexible new release. *Journal of Applied Crystallography*, 21**,** 983-984.

SPECK, A. L. (2001) PLATON, a multipurpose crystallographic tool. The Netherlands, Utrecht University, Utrecht.