Pseudo Merohedral Twinning Example with Platon/TwinRotMat

Merohedral Twins are seen for high symmetry space groups (orthorhombic and above) and are not seen for monoclinic and triclinic structures. Given this fact the lower symmetry groups may "mimic" the merohedral twin if one or more of their lattice angles are close to 90 or 120 degrees. These "twins" are called pseudo merohedral twins (see Regine Herbst-Irmer section of **Crystal Structure Refinement: A Crystallographer's Guide to SHELXL (International Union of Crystallography Texts on Crystallography)** by Peter Muller editor, Regine Herbst-Irmer, Anthony Spek, Thomas Schneider, Michael Sawaya). An example is given below on how to use the program PLATON to resolve the problem.

Example :

The cell reported for this structure was

```
CELL 1.54178   8.6543  22.7909  16.7066  90.000  90.204  90.000
ZERR   4.00    0.0019   0.0054   0.0037   0.000   0.012   0.000
```

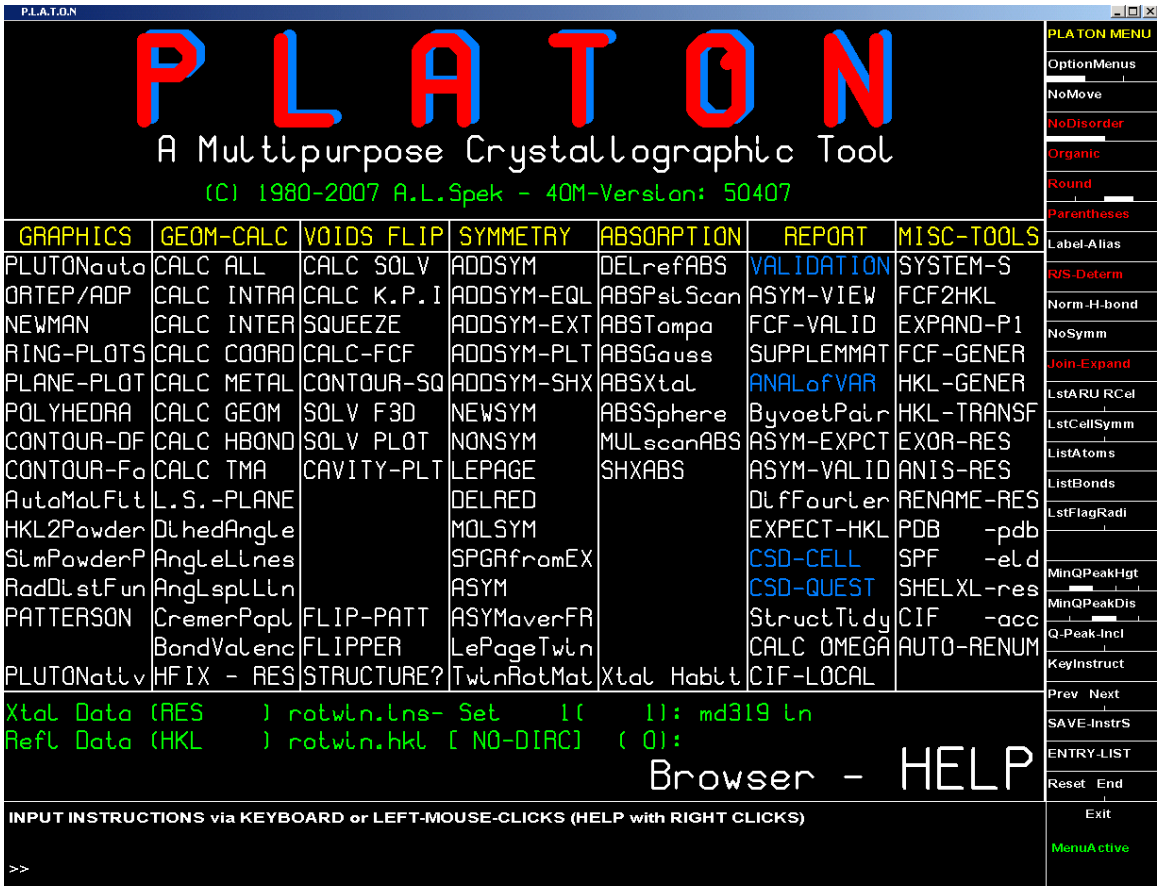
Note that the structure is monoclinic and that the beta angle is near 90^{o} (90.204^{o}). This may lead to a pseudo merohedral twin.

The structure as is refines poorly ….

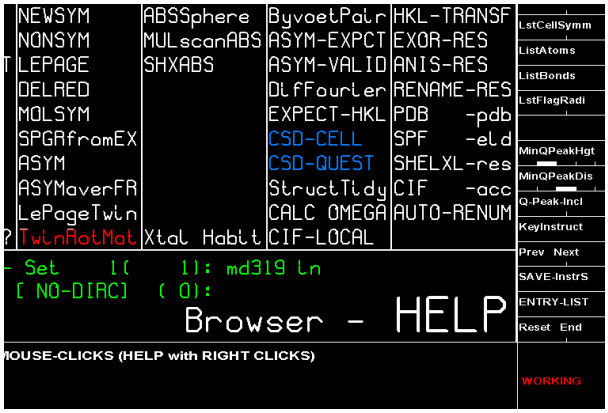
```
+++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++
+  XL  - CRYSTAL STRUCTURE REFINEMENT - SHELXTL Ver. 6.12 W95/98/NT/2000/ME  +
+  Copyright(c) 2001 Bruker Analytical X-ray Solutions  All Rights Reserved  +
+  rotwin                                 started at 10:59:25 on 08-Jan-2008  +
+++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++

Read instructions and data
Data:    4217 unique,      0 suppressed   R(int) = 0.1642   R(sigma) = 0.1598
Systematic absence violations:    0    Bad equivalents:    0
wR2 =  0.5734 before cycle   1 for   4217 data and   380 /   380 parameters
GooF = S =     4.029;     Restrained GooF =      3.977  for    192 restraints
Mean shift/esd =   0.587    Maximum =  -6.927 for  OSF           at 10:59:29
Max. shift = 0.098 A for C13      Max. dU = 0.011 for C4
wR2 =  0.5324 before cycle   2 for   4217 data and   380 /   380 parameters
GooF = S =     3.264;     Restrained GooF =      3.192  for    192 restraints
Mean shift/esd =   0.334    Maximum =  -1.914 for  U13 Fe2       at 10:59:32
Max. shift = 0.080 A for C13      Max. dU = 0.007 for C2
wR2 =  0.5251 before cycle   3 for   4217 data and   380 /   380 parameters
GooF = S =     3.177;     Restrained GooF =      3.106  for    192 restraints
Mean shift/esd =   0.125    Maximum =   0.944 for   x  C13       at 10:59:35
Max. shift = 0.038 A for C13      Max. dU = 0.004 for C2
wR2 =  0.5237 before cycle   4 for   4217 data and   380 /   380 parameters
GooF = S =     3.160;     Restrained GooF =      3.089  for    192 restraints
Mean shift/esd =   0.071    Maximum =   0.503 for   x  C13       at 10:59:37
Max. shift = 0.020 A for C13      Max. dU = 0.002 for O2
wR2 =  0.5231 before cycle   5 for   4217 data and     0 /   380 parameters
GooF = S =     3.154;     Restrained GooF =      3.084  for    192 restraints
R1 =  0.2395 for   2460 Fo > 4sig(Fo)  and  0.2912 for all   4217 data
wR2 =  0.5231,  GooF = S =   3.154,  Restrained GooF =    3.084  for all data
R1 =  0.2769 for   4217 unique reflections after merging for Fourier
Highest peak    2.06  at  0.4364  0.1422  0.0805  [  1.32 A from H5C ]
Deepest hole   -1.02  at  0.0708  0.0696  0.0235  [  1.65 A from S2 ]

+++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++
+  rotwin            finished at 10:59:38   Total CPU time:      11.8 secs  +
+++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++
```

The structure refines to 0.2395 R1 (4sigF) and 0.2769 for all data.

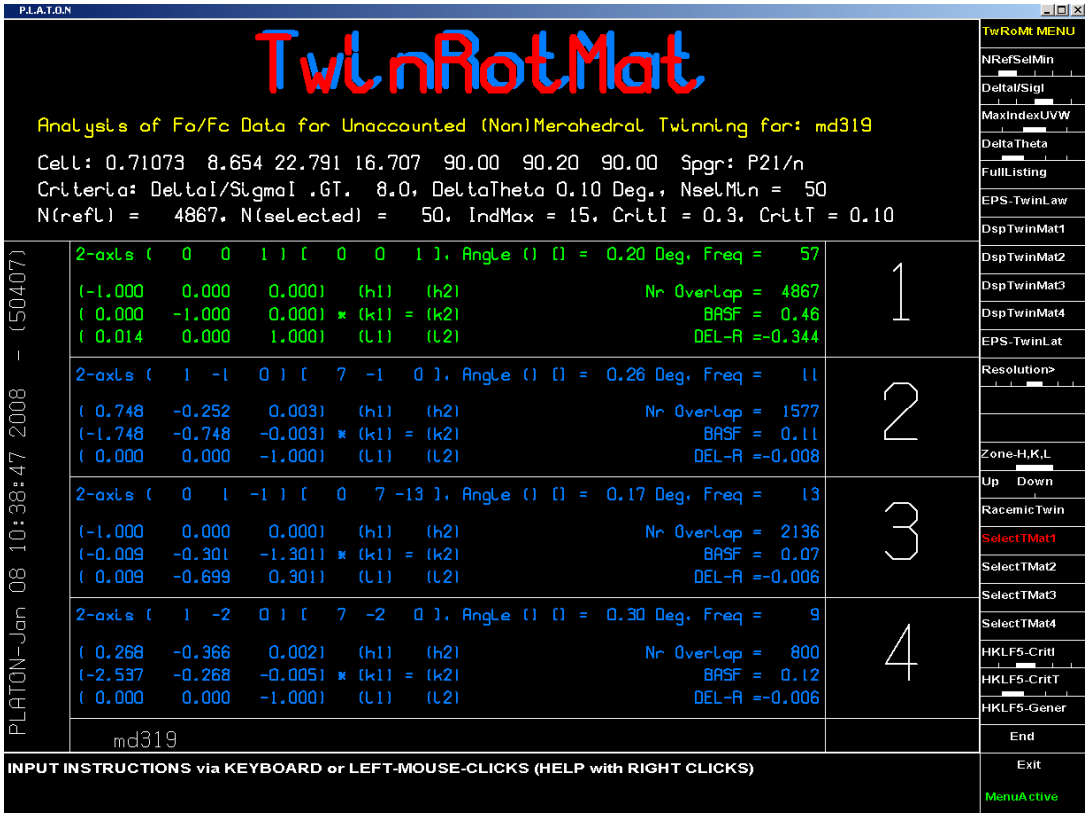
To refine this structure better, first you must find the twin law.
Start PLATON by the command platon rotwin (replace rotwin with your project)



Point to the TwinRotMat button



The program will begin. Be patient it may take a few minutes to find the twin law (if any).



After a few minutes the program will return the following screen. In this case look at the BASF and DEL-R lines. The BASF is the parameter you will refine in SHELXL. The larger the value of the BASF instruction the greater the overlap in the twinned components and the larger the drop in the final R value you will see. In this case the DEL-R predicts a drop in the R factor by 34%.

The easiest way to proceed is the write the twin law down for use in SHELXL.
In this case the twin law we choose is first one given (the 2-fold)
TWIN -1 0 0 0 -1 0 0.014 0 1
You may want to experiment with the HKLF5 refinement at a later date.

Add the twin law and the BASF statement to the INS file

```
TWIN -1 0 0  0 -1 0  0.014 0 1
BASF   0.344
```

Enter the BASF that is given in TwinRotMat as a starting value for the refinement

```
+++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++
+  XL  - CRYSTAL STRUCTURE REFINEMENT - SHELXTL Ver. 6.12 W95/98/NT/2000/ME  +
+  Copyright(c) 2001 Bruker Analytical X-ray Solutions  All Rights Reserved  +
+  rotwin                              started at 11:13:22 on 08-Jan-2008  +
+++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++

Read instructions and data
Data:    4219 unique,      0 suppressed   R(int) = 0.1660   R(sigma) = 0.1599
Systematic absence violations:    0    Bad equivalents:    0
wR2 =  0.1882 before cycle   1 for   4219 data and   381 /   381 parameters
GooF = S =     1.060;     Restrained GooF =      1.046  for    192 restraints
Mean shift/esd =   0.076    Maximum =  11.093 for BASF  1          at 11:13:27
Max. shift = 0.002 A for C2       Max. dU =-0.001 for C29
wR2 =  0.1829 before cycle   2 for   4219 data and   381 /   381 parameters
GooF = S =     1.026;     Restrained GooF =      1.012  for    192 restraints
Mean shift/esd =   0.027    Maximum =   3.568 for BASF  1          at 11:13:30
Max. shift = 0.001 A for C2       Max. dU = 0.000 for C29
wR2 =  0.1825 before cycle   3 for   4219 data and   381 /   381 parameters
GooF = S =     1.024;     Restrained GooF =      1.011  for    192 restraints
Mean shift/esd =   0.030    Maximum =   0.356 for  U13 Fe2         at 11:13:34
Max. shift = 0.001 A for C2       Max. dU = 0.000 for C29
wR2 =  0.1824 before cycle   4 for   4219 data and   381 /   381 parameters
GooF = S =     1.024;     Restrained GooF =      1.010  for    192 restraints
Mean shift/esd =   0.003    Maximum =   0.066 for BASF  1          at 11:13:37
Max. shift = 0.000 A for C30      Max. dU = 0.000 for C4
wR2 =  0.1824 before cycle   5 for   4219 data and     0 /   381 parameters
GooF = S =     1.024;     Restrained GooF =      1.010  for    192 restraints
R1 =  0.0789 for   2460 Fo > 4sig(Fo)  and  0.1424 for all   4219 data
wR2 =  0.1824,  GooF = S =   1.024,  Restrained GooF =    1.010  for all data
R1 =  0.1298 for   4217 unique reflections after merging for Fourier
Highest peak    0.56  at  0.1231  0.1495 -0.0013  [  1.18 A from S2 ]
Deepest hole   -0.53  at  0.1303  0.1947  0.9512  [  0.82 A from FE2 ]

+++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++
+  rotwin           finished at 11:13:38   Total CPU time:      15.0 secs  +
+++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++
```

The structure now refines to 0.0789 R1 (4sigF) and 0.1298 for all data.

The BASF refined to 0.43791

Complete the refinement in the normal fashion.