

## 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° (90.204°). 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 ]

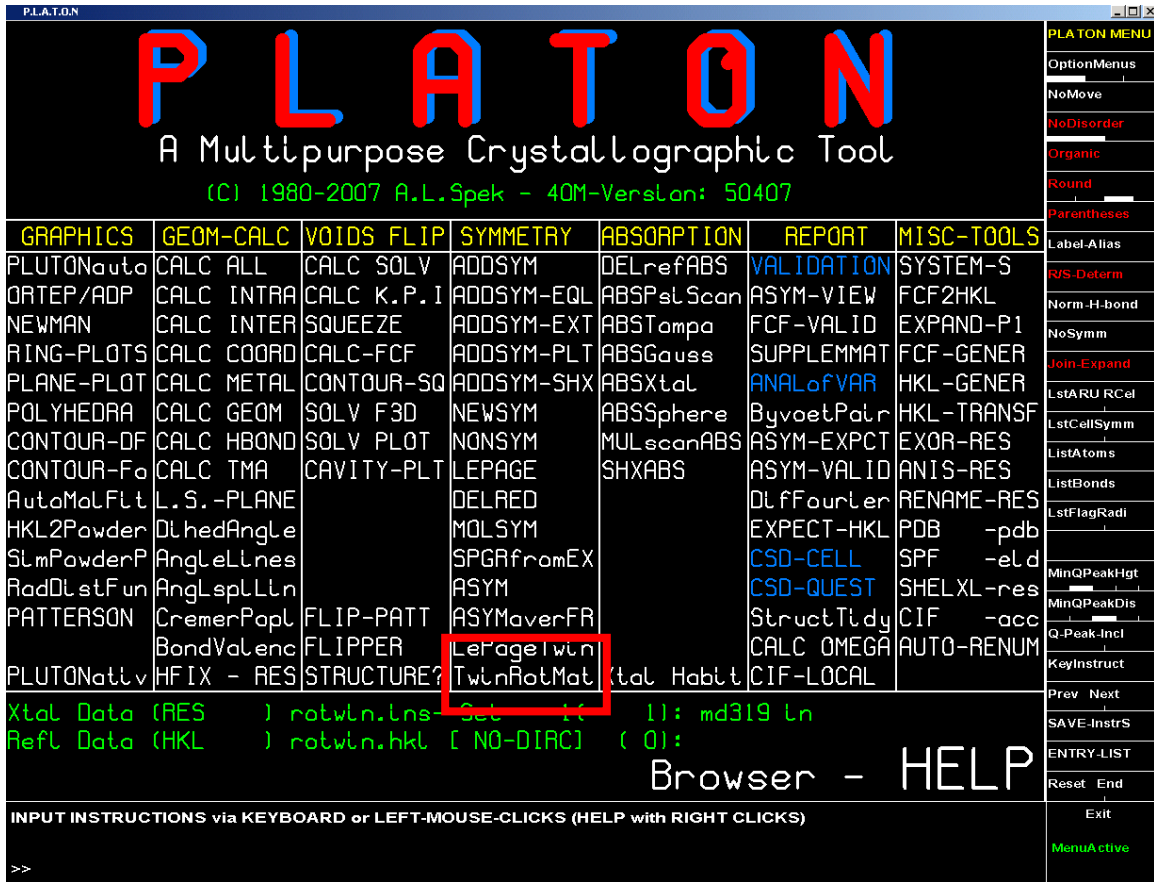
*****
+ rotwin finished at 10:59:38 Total CPU time: 11.8 secs +
*****
```

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

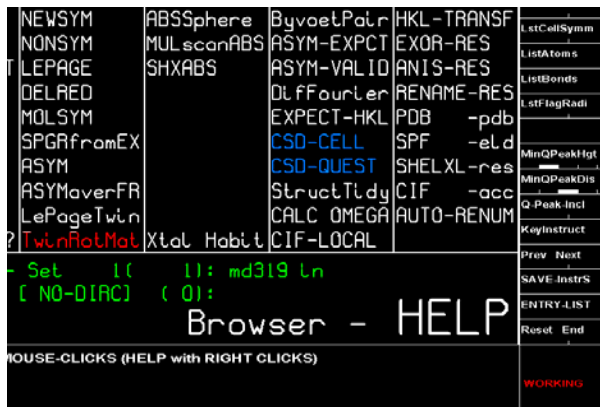
To refine this structure better, first you must find the twin law.

Use the ACTA in XL to write the \*.cif

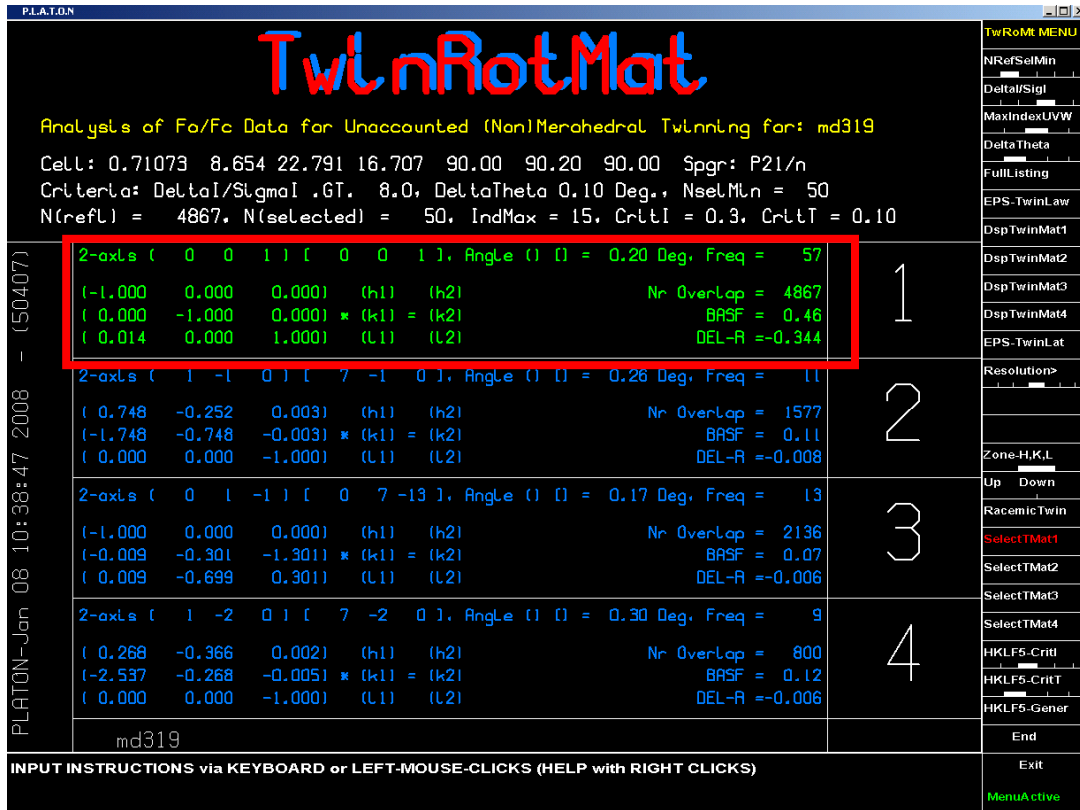
Start PLATON by the command `platon *.cif` (replace \* with your project)



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 to write the twin law down for use in SHELXL. In this case the twin law we choose is the 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.