Twinning

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http://shelx.uni-ac.gwdg.de/~rherbst/twin.html
“Twins are regular aggregates consisting of individual crystals of the same species joined together in some definite mutual orientation.”


Simple example for a two-dimensional twin:

Twin Law: \[
\begin{pmatrix}
1 & 0 \\
0 & -1
\end{pmatrix}
\]

fractional contribution \( k_1 \) for twin domain 1: 5/9
fractional contribution \( k_2 \) for twin domain 2: 4/9
Four Kinds of Twins (I)

1. Twinning by *merohedry*
   Twin operator: symmetry operator of the crystal system but not of the point group of the crystal
   1.1. racemic twin
   1.2. twin operator: not of the Laue group of the crystal
Reciprocal Space Plot \( l = 0 \)
Reciprocal Space Plot \( l = 0 \)
Reciprocal Space Plot $l = 0$
1. Twinning by **merohedry**
   Twin operator: symmetry operator of the crystal system but not of the point group of the crystal
   1.1. racemic twin
   1.2. twin operator: not of the Laue group of the crystal
   - only in tetragonal, trigonal, hexagonal and cubic space groups
   - exact overlap of the reciprocal lattices
   - often low value for $<|E^2-1|>$
   - Laue group and space group determination may be difficult
   - structure solution may be difficult

2. Twinning by **pseudo-merohedry**
   Twin operator: belongs to a higher crystal system than the structure
   - Metric symmetry higher than Laue symmetry
3. Twinning by *reticular merohedry*
   e.g. obverse/reverse twinning in case of a rhombohedral crystal
Reciprocal Space Plot \( l = 1 \)
Reciprocal Space Plot $l = 1$
Reciprocal Space Plot $l = 1$
4. Non-merohedral twins
   Twin operator: arbitrary operator, often rotation of 180°
Reciprocal Space Plot $k = 2$
Reciprocal Space Plot $k = 2$
Reciprocal Space Plot $k = 2$
Four Kinds of Twins (II)

3. Twinning by *reticular merohedry*
   - e.g. obverse/reverse twinning in case of a rhombohedral crystal
   - detection of the lattice centring may be difficult
   - structure solution not as difficult as for merohedral twins.

4. **Non-merohedral twins**
   - Twin operator: arbitrary operator, often rotation of 180°
   - no exact overlap of the reciprocal lattices
   - cell determination problems
   - cell refinement problems
   - some reflections sharp, others split
   - data integration complicated (requires more than one orientation matrix)
   - structure solution not as difficult as for merohedral twins
low value for $<|E^2-1|>$ (expected 0.968 centrosym and 0.736 non-centrosym)

- $R_{int}$ low for the true Laue group and low/medium for the apparent Laue group.

- Todd Yeates Twinning Server:
  
  http://www.doe-mbi.ucla.edu/Services/Twinning

  $J_1 = (1-\alpha)I_1 + \alpha I_2 \quad J_2 = (1-\alpha)I_2 + \alpha I_1$

  $H = (J_1 - J_2)/(J_1 + J_2)$

  non-centrosymmetric structures:

  $\alpha = \frac{1}{2}(1 - 2<|H|>) \quad \text{and} \quad \alpha = \frac{1}{2}[1 - (3<\text{H}^2>)^\frac{1}{2}]$
Tests for Twinning: Perfect Twins

- low value for $\langle |E^2-1| \rangle$ (expected 0.968 centrosym and 0.736 non-centrosym)

- Todd Yeates twinning server: $\langle I^2 \rangle / \langle I \rangle^2$

  (acentric data: 2 for untwinned data, 1.5 for twinned data)


- $L \equiv \frac{I(h_1) - I(h_2)}{I(h_1) + I(h_2)}$

  $h_1$ and $h_2$ proximally located in reciprocal space

  $\langle |L| \rangle \quad \langle L^2 \rangle$

  Acentric, untwinned \hspace{1cm} 1/2 \hspace{1cm} 1/3

  Centric, untwinned \hspace{1cm} 2/\pi \hspace{1cm} 1/2

  Acentric, perfectly twinned \hspace{1cm} 3/8 \hspace{1cm} 1/5

Structure Solution

- Detwinning

\[ J_1 = (1-\alpha) I_1 + \alpha I_2 \quad J_2 = (1-\alpha) I_2 + \alpha I_1 \]

\[ I_1 = \frac{(1-\alpha)J_1 - \alpha J_2}{1-2\alpha} \quad I_2 = \frac{(1-\alpha)J_2 - \alpha J_1}{1-2\alpha} \]

- SHELXD can use the twin law and the fractional contribution

- Molecular Replacement

- MIR

- MAD/ SAD:
Twin Refinement in SHELXL-97

Method of Pratt, Coyle and Ibers:

\[
(F_c^2)^* = \text{osf}^2 \sum_{m=1}^{n} k_m F_{cm}^2
\]

\[
1 = \sum_{m=1}^{n} k_m
\]

\[
k_1 = 1 - \sum_{m=2}^{n} k_m
\]

(n-1) of the fractional contributions can be refined.

TWIN r11 r12 r13 r21 r22 r23 r31 r32 r33 n
BASF k2 k3 ... kn

or

MERG 0
BASF k2 k3 ... kn
HKL F 5

trigonal or hexagonal P with $a = 46.090$, $c = 31.020$ Å.
Space Group Determination

### SPACE GROUP DETERMINATION

Crystal system H and Lattice type P selected

Mean $|E^*E-1| = 0.643$ [expected .968 centrosym and .736 non-centrosym]

Systematic absence exceptions:

<table>
<thead>
<tr>
<th></th>
<th>$6_1/6_5$</th>
<th>$6_2=3_1$</th>
<th>$6_3$</th>
<th>-c-</th>
<th>--c</th>
</tr>
</thead>
<tbody>
<tr>
<td>N</td>
<td>23</td>
<td>20</td>
<td>13</td>
<td>865</td>
<td>499</td>
</tr>
<tr>
<td>$N</td>
<td>&gt;3\sigma$</td>
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<td>1</td>
<td>4</td>
<td>692</td>
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<tr>
<td>$&lt;</td>
<td>l&gt;$</td>
<td>2.3</td>
<td>0.2</td>
<td>3.9</td>
<td>41.5</td>
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<tr>
<td>$&lt;</td>
<td>l</td>
<td>/\sigma&gt;$</td>
<td>2.5</td>
<td>0.9</td>
<td>3.6</td>
</tr>
<tr>
<td>Opt.</td>
<td>Space Group</td>
<td>CSD</td>
<td>R(int)</td>
<td>N(eq)</td>
<td>Syst. Abs.</td>
</tr>
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<td>------</td>
<td>-------------</td>
<td>-----</td>
<td>--------</td>
<td>-------</td>
<td>-----------</td>
</tr>
<tr>
<td>[A]</td>
<td>P3(1)</td>
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<td>0.000</td>
<td>0</td>
<td>0.9 / 2.5</td>
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<tr>
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<td>0.000</td>
<td>0</td>
<td>0.9 / 2.5</td>
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<tr>
<td>[C]</td>
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<td>82</td>
<td>0.195</td>
<td>15855</td>
<td>0.9 / 2.5</td>
</tr>
<tr>
<td>[D]</td>
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<td>82</td>
<td>0.195</td>
<td>15855</td>
<td>0.9 / 2.5</td>
</tr>
<tr>
<td>[E]</td>
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<td>2</td>
<td>0.443</td>
<td>16211</td>
<td>0.9 / 2.5</td>
</tr>
<tr>
<td>[F]</td>
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<td>0.443</td>
<td>16211</td>
<td>0.9 / 2.5</td>
</tr>
<tr>
<td>[G]</td>
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<td>0.442</td>
<td>16285</td>
<td>0.9 / 2.5</td>
</tr>
<tr>
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<td>0.442</td>
<td>16285</td>
<td>0.9 / 2.5</td>
</tr>
<tr>
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<td>P6(1)</td>
<td>62</td>
<td>0.442</td>
<td>16285</td>
<td>3.6 / 14.9</td>
</tr>
<tr>
<td>[J]</td>
<td>P6(5)</td>
<td>62</td>
<td>0.442</td>
<td>16285</td>
<td>3.6 / 14.9</td>
</tr>
<tr>
<td>[K]</td>
<td>P6(2)22</td>
<td>9</td>
<td>0.450</td>
<td>24181</td>
<td>0.9 / 2.5</td>
</tr>
<tr>
<td>[L]</td>
<td>P6(4)22</td>
<td>9</td>
<td>0.450</td>
<td>24181</td>
<td>0.9 / 2.5</td>
</tr>
<tr>
<td>[M]</td>
<td>P6(1)22</td>
<td>20</td>
<td>0.450</td>
<td>24181</td>
<td>3.6 / 14.9</td>
</tr>
<tr>
<td>[N]</td>
<td>P6(5)22</td>
<td>20</td>
<td>0.450</td>
<td>24181</td>
<td>3.6 / 14.9</td>
</tr>
</tbody>
</table>
Resolution: 1.06 Å ➔ Ab initio direct methods

SHELXD

- Ignoring twinning
- detwinned data
- \texttt{TWIN 0 1 0 1 0 0 0 0 -1} and \texttt{BASF 0.2}
6 Mersacidin Molecules
9 MeOH, 58 H₂O

Data 33449
Restraints 10784
Parameters 7438
R1 [I>2σ(I)] 0.1335
wR2 (all data) 0.3338
BASF 0.244


from J. Kärcher, PhD Thesis, 2000
Twinning by Pseudo-Merohedry

Structure of aniline

cell: 21.645 5.833 8.319 90 101.12 90
space group: P2₁/c

R₁ = 0.071 for 1505 F₀ > 4(F₀)
wR₂ = 0.198 for all 1790 data

Analysis of variance for reflections employed in refinement
K = Mean[F₀²] / Mean[Fᶜ²] for group
Fᶜ/Fᶜ(max)  0.000  0.009  0.017  0.026  0.036  0.047
Number in group  197  164  178  188  173  ...
GooF  1.663  1.428  1.579  1.611  1.174  ...
K  6.815  1.807  1.486  1.246  1.096  ...
s.u. (C - C): 0.004 - 0.005
Residual density maximum: 0.26 e/Å³
Opt. A: FOM = 0.040° orthorhombic C  
\[ R(\text{int}) = 0.300 \] [ 5707] 
Cell: 8.319 42.477 5.833 90.00 90.00 90.04  
V: 2061.20 
Matrix: 0.00 0.00 1.00 2.00 0.00 1.00 0.00 1.00 0.00 

Opt. B: FOM = 0.000° monoclinic P  
\[ R(\text{int}) = 0.110 \] [ 4798] 
Cell: 8.319 5.833 21.639 90.00 101.04 90.00  
V: 1030.60 
Matrix: 0.00 0.00 1.00 0.00 1.00 0.00 -1.00 0.00 -1.00 

Mean |E*E-1| = 0.922  
[expected .968 centrosym and .736 non-centrosym]
ROTAX - Output

180.0 degree rotation about 1. 0. 0. reciprocal lattice direction:
[  1.000   0.000   1.004]
[  0.000  -1.000   0.000]
[  0.000   0.000  -1.000]
Figure of merit = 0.34 **********

180.0 degree rotation about 0. 1. 0. direct lattice direction:
[ -1.000   0.000   0.000]
[  0.000   1.000   0.000]
[  0.000   0.000  -1.000]
Figure of merit = 0.00 **********

180.0 degree rotation about 0. 0. 1. direct lattice direction:
[ -1.000   0.000  -1.004]
[  0.000  -1.000   0.000]
[  0.000   0.000   1.000]
Figure of merit = 0.34 **********
Comparison of the results:

<table>
<thead>
<tr>
<th></th>
<th>without TWIN</th>
<th>with TWIN</th>
</tr>
</thead>
<tbody>
<tr>
<td>R1 (F &gt; 4σ(F))</td>
<td>0.071</td>
<td>0.047</td>
</tr>
<tr>
<td>wR2 (all data)</td>
<td>0.198</td>
<td>0.123</td>
</tr>
<tr>
<td>K2</td>
<td>-</td>
<td>0.0734(1)</td>
</tr>
<tr>
<td>Res. electron density</td>
<td>0.26</td>
<td>0.20</td>
</tr>
<tr>
<td>s.u.(C - C)</td>
<td>0.004 - 0.005</td>
<td>0.003</td>
</tr>
<tr>
<td>K (weakest reflections)</td>
<td>6.815</td>
<td>0.956</td>
</tr>
</tbody>
</table>
Twinning by Reticular Merohedry

Structure of K[Au(CN)₂]
cell: 7.240 7.240 26.445 90 90 120, space group R 3̅

A. Rosenzweig, D. T. Cromer,
\[ \text{K[Au(CN)\textsubscript{2}]} \]

\[ R_1 = 0.074 \text{ for } 640 \text{ } F_\text{o} > 4\sigma(F_\text{o}), \quad wR_2 = 0.170 \text{ for all 648 data} \]

\[ R_1 = 0.027 \text{ for } 640 \text{ } F_\text{o} > 4\sigma(F_\text{o}), \quad wR_2 = 0.076 \text{ for all 648 data} \]

Residual density: 1.18/-1.48 e/\text{A}^3
### Warning Signs

#### Systematic Absences Violations:

<table>
<thead>
<tr>
<th>h</th>
<th>k</th>
<th>l</th>
<th>( F_o^2 )</th>
<th>( F_c^2 )</th>
<th>( \Delta(F^2)/\sigma )</th>
<th>( F_c/F_{c_{\text{max}}} )</th>
<th>Res. (Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>3</td>
<td>0</td>
<td>1907.04</td>
<td>407.73</td>
<td>11.79</td>
<td>0.026</td>
<td>2.09</td>
</tr>
<tr>
<td>-1</td>
<td>2</td>
<td>6</td>
<td>7075.12</td>
<td>11145.69</td>
<td>6.78</td>
<td>0.137</td>
<td>2.80</td>
</tr>
<tr>
<td>1</td>
<td>1</td>
<td>6</td>
<td>1275.08</td>
<td>818.27</td>
<td>3.69</td>
<td>0.037</td>
<td>2.80</td>
</tr>
<tr>
<td>-1</td>
<td>2</td>
<td>0</td>
<td>27026.22</td>
<td>32870.20</td>
<td>3.53</td>
<td>0.235</td>
<td>3.60</td>
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<tr>
<td>-1</td>
<td>2</td>
<td>3</td>
<td>47884.52</td>
<td>56252.36</td>
<td>2.98</td>
<td>0.307</td>
<td>3.35</td>
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<tr>
<td>-1</td>
<td>2</td>
<td>12</td>
<td>7698.09</td>
<td>9417.93</td>
<td>2.98</td>
<td>0.126</td>
<td>1.88</td>
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<tr>
<td>-5</td>
<td>4</td>
<td>6</td>
<td>642.68</td>
<td>966.24</td>
<td>2.77</td>
<td>0.040</td>
<td>1.31</td>
</tr>
</tbody>
</table>

#### Most Disagreeable Reflections

<table>
<thead>
<tr>
<th>h</th>
<th>k</th>
<th>l</th>
<th>( F_o^2 )</th>
<th>( F_c^2 )</th>
<th>( \Delta(F^2)/\sigma )</th>
<th>( F_c/F_{c_{\text{max}}} )</th>
<th>Res. (Å)</th>
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<tr>
<td>2</td>
<td>0</td>
<td>0</td>
<td>8.08</td>
<td>2.00</td>
<td>observed but should be systematically absent</td>
<td></td>
<td></td>
</tr>
<tr>
<td>-1</td>
<td>0</td>
<td>1</td>
<td>507.42</td>
<td>32.65</td>
<td>observed but should be systematically absent</td>
<td></td>
<td></td>
</tr>
<tr>
<td>-1</td>
<td>0</td>
<td>1</td>
<td>610.89</td>
<td>37.97</td>
<td>observed but should be systematically absent</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>0</td>
<td>-1</td>
<td>517.12</td>
<td>34.48</td>
<td>observed but should be systematically absent</td>
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<td></td>
</tr>
<tr>
<td>0</td>
<td>-1</td>
<td>-1</td>
<td>540.26</td>
<td>33.43</td>
<td>observed but should be systematically absent</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>-1</td>
<td>1</td>
<td>512.14</td>
<td>35.24</td>
<td>observed but should be systematically absent</td>
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<td></td>
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<tr>
<td>10</td>
<td>-1</td>
<td>-1</td>
<td>557.75</td>
<td>34.37</td>
<td>observed but should be systematically absent</td>
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<td></td>
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</tbody>
</table>

---

Reciprocal Space Plot $l = 0$
Reciprocal Space Plot $l = 2$
**Refinement as Twin (I)**

**Merging Results**

<table>
<thead>
<tr>
<th>MERG</th>
<th>BASF</th>
<th>HKLF</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0.2</td>
<td>5</td>
</tr>
</tbody>
</table>

**Comparison of Original and New hkl-Files**

**Original hkl-file**

<table>
<thead>
<tr>
<th>h</th>
<th>k</th>
<th>l</th>
<th>F²</th>
<th>(\sigma(F²))</th>
</tr>
</thead>
<tbody>
<tr>
<td>-1</td>
<td>1</td>
<td>1</td>
<td>1413.81</td>
<td>33.69</td>
</tr>
<tr>
<td>0</td>
<td>1</td>
<td>1</td>
<td>542.01</td>
<td>12.15</td>
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<td>1.06</td>
<td>0.84</td>
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<td>25.72</td>
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<td>1.60</td>
<td>1.62</td>
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<tr>
<td>-5</td>
<td>4</td>
<td>3</td>
<td>1287.24</td>
<td>31.66</td>
</tr>
<tr>
<td>-2</td>
<td>4</td>
<td>3</td>
<td>3234.82</td>
<td>71.41</td>
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</table>

**New hkl-file**

<table>
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<tr>
<th>h</th>
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<th>l</th>
<th>F²</th>
<th>(\sigma(F²))</th>
<th>N</th>
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<tr>
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<td>1</td>
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<tr>
<td>-4</td>
<td>5</td>
<td>-3</td>
<td>1287.24</td>
<td>31.66</td>
<td>-2</td>
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<tr>
<td>-5</td>
<td>4</td>
<td>3</td>
<td>1287.24</td>
<td>31.66</td>
<td>1</td>
</tr>
<tr>
<td>-4</td>
<td>2</td>
<td>-3</td>
<td>3234.82</td>
<td>71.41</td>
<td>-2</td>
</tr>
<tr>
<td>-2</td>
<td>4</td>
<td>3</td>
<td>3234.82</td>
<td>71.41</td>
<td>1</td>
</tr>
</tbody>
</table>
R1 = 0.0178 for 640 $F_o > 4 \sigma(F_o)$
wR2 = 0.0430 for 648 data
K2 = 0.290(4)

Residual density maximum = 1.03 $e/\AA^3$

Bovine Insulin

51 amino acids in the asymmetric unit
<table>
<thead>
<tr>
<th>Cell for domain 1:</th>
<th>78.040</th>
<th>77.986</th>
<th>78.024</th>
<th>89.99</th>
<th>89.94</th>
<th>90.01</th>
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</thead>
<tbody>
<tr>
<td>Figure of merit:</td>
<td>0.560</td>
<td>0.516</td>
<td>0.552</td>
<td>0.626</td>
<td></td>
<td></td>
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<tr>
<td>Orientation matrix:</td>
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<td>0.00347148</td>
<td>0.01005931</td>
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<td>-0.00035888</td>
<td>0.00749939</td>
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<tr>
<td></td>
<td>0.00230569</td>
<td>-0.01233875</td>
<td>0.00261444</td>
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<tr>
<td>4072 reflections within 0.250 of an integer index assigned to domain 1,</td>
<td></td>
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<table>
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<th>Cell for domain 2:</th>
<th>78.040</th>
<th>77.986</th>
<th>78.024</th>
<th>89.99</th>
<th>89.94</th>
<th>90.01</th>
</tr>
</thead>
<tbody>
<tr>
<td>Figure of merit:</td>
<td>0.910</td>
<td>0.914</td>
<td>0.936</td>
<td>0.945</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Orientation matrix:</td>
<td>0.00745656</td>
<td>0.000032791</td>
<td>0.01040996</td>
<td>0.00017398</td>
<td>-0.01281862</td>
<td>0.00027851</td>
</tr>
<tr>
<td></td>
<td>0.01041953</td>
<td>-0.00001700</td>
<td>-0.00747135</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Rotated from first domain by 89.2 degrees about reciprocal axis 0.928 0.207 1.000 and real axis 0.927 0.208 1.000</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Twin law to convert hkl from first to this domain (SHELXL TWIN matrix):</td>
<td>0.459</td>
<td>-0.625</td>
<td>0.631</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>0.824</td>
<td>0.036</td>
<td>-0.565</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>0.330</td>
<td>0.780</td>
<td>0.532</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3564 reflections within 0.250 of an integer index assigned to domain 2, 2751 of them exclusively; 184 reflections not yet assigned to a domain</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<td></td>
</tr>
</tbody>
</table>
Data Processing and Solution

• integrated using both orientation matrices

• each component scaled separately using non-overlapped reflections

• Detwinned HKLF4 format file for structure solution and refinement

• HKLF5 for final refinement

  Resolution to 1.60 Å

  Cubic symmetry (space group I2\textsubscript{1}3): high redundancy

  6 sulfur atoms easily found by S-SAD, excellent experimental map
Refinement Statistics

R1 (Fo > 4\sigma (Fo)) \hspace{1cm} 0.1050
wR2 (all data) \hspace{1cm} 0.2856
R1 (after merging for Fourier) \hspace{1cm} 0.1175
BASF (Twinning Fraction) \hspace{1cm} 0.427
Solvent Content (%) \hspace{1cm} 58
Mean B value (Å²) Main Chain Atoms \hspace{1cm} 18.64
Mean B value (Å²) Side Chain Atoms and Solvent \hspace{1cm} 29.94
Number of Protein Atoms \hspace{1cm} 389
Number of Solvent Atoms \hspace{1cm} 51
Warning Signs for Merohedral Twinning

- Metric symmetry higher than Laue symmetry
- $R_{int}$ for the higher symmetry Laue group only slightly higher than for the lower symmetry one
- Different $R_{int}$ values for the higher symmetry Laue group for different crystals of the same compound
- Mean value for $|E^2 - 1| \ll 0.736$
- Apparent trigonal or hexagonal space group
- Systematic absences not consistent with any known space group
- No structure solution
- Patterson function physically impossible (for heavy atom structures)
- High R-Values
Warning Signs for Non-merohedral Twinning

- An unusually long axis
- Problems with cell refinement
- Some reflections sharp, others split
- $K = \text{mean}(F_o^2)/\text{mean}(F_c^2)$ is systematically high for reflections with low intensity
- For all of the most disagreeable reflections $F_o >> F_c$.
- Strange residual density, which could not be resolved as solvent or disorder.


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