Space group determination in EXPO2013

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The symmetry analysis: single crystal data

1) **First step**: identification of the *Laue group* via the recognition of the "symmetry equivalent reflections".

The *Laue group* generally identifies without any doubt the crystal system (pseudo-symmetries, twins etc. are not considered).

b) **Second step**: identification of the *systematically absent reflections*. 
The Laue Group

Symmetry operators: \( C_s \equiv (R_s, T_s), \quad s = 1, \ldots, m \)

Then

\[
    r_{js} = R_s r_j + T_s
\]

are symmetry equivalent positions in direct space.

In the reciprocal space we will observe the symmetry equivalent reflections

\[
    \bar{h}R_s, \quad s = 1, \ldots, m
\]

If the space group is n.c.s., the Friedel opposites should be added: the complete set is

\[
    \bar{h}R_s, \quad s = 1, \ldots, m \quad -\bar{h}R_s, \quad s = 1, \ldots, m
\]
The Laue group $\frac{2}{m} \frac{2}{m} \frac{2}{m}$

\[
R_1 = \begin{bmatrix} 1 & 0 & 0 \\ 0 & 1 & 0 \\ 0 & 0 & 1 \end{bmatrix}, \quad R_2 = \begin{bmatrix} 1 & 0 & 0 \\ 0 & \tilde{1} & 0 \\ 0 & 0 & \tilde{1} \end{bmatrix}, \quad R_3 = \begin{bmatrix} 1 & 0 & 0 \\ 0 & \tilde{1} & 0 \\ 0 & 0 & 1 \end{bmatrix}, \quad R_4 = \begin{bmatrix} 1 & 0 & 0 \\ 0 & 1 & 0 \\ 0 & 0 & \tilde{1} \end{bmatrix}
\]

The equivalent reflections are

$\bar{h}R_1 = (hkl), \quad \bar{h}R_2 = (hkl), \quad \bar{h}R_3 = (hkl), \quad \bar{h}R_4 = (hkl),$ 

$-\bar{h}R_1 = (\bar{h}\bar{k}\bar{l}), \quad -\bar{h}R_2 = (\bar{h}\bar{k}\bar{l}), \quad -\bar{h}R_3 = (\bar{h}\bar{k}\bar{l}), \quad -\bar{h}R_4 = (\bar{h}\bar{k}\bar{l})$
The systematically absent reflections

Since

\[ F_{hR_s} = F_h \exp(-2\pi i h T_s) \]  \hspace{1cm} (1)

we have

\[ |F_{hR_s}| = |F_h|, \quad \varphi_{h,R_s} = \varphi_h - 2\pi i h T_s \]

If \( hR_s = h \) and \( hT_s \neq n \) the relation (1) is violated unless the reflection \( h \) is a systematically absent reflection.

The combination of the information on the Laue Group with that on the systematically absent reflections allows the determination of the Extinction Symbol.
The extinction symbols (ES)

In the first position: cell \textit{Centric type} (e.g., \textit{P - - a}, in orth.)

Then the reflection conditions for each symmetry direction are given.

Symmetry directions not having conditions are represented by a \textbf{dash}.

A symmetry direction with conditions is represented by the symbol of the screw axis or glide plane.

Table of \textbf{ES} are given in \textit{IT per} crystal system. There are 14 \textbf{ES} for monoclinic, 111 for orthorhombic, 31 for tetragonal, 12 for trigonal-hexagonal, 18 for cubic system.
Extinction symbol and compatible space groups

- \( \text{ES} \)

\[
\begin{align*}
\text{P} - - - & \quad \text{P222, Pmmm, Pm2m, P2mm, Pmm2} \\
\text{(in orth.)} & \\
\text{P} - - a & \quad \text{Pm2a, P2_{1}ma, P mma} \\
\text{(in orth.)} & \\
\text{P} - - - & \quad \text{P4, P-4, P4/m, P422, P4mm, P-42m, P-4m2} \\
\text{(in tetrag.)} & \quad \text{P4/mmm} \\
\text{P}6_{1}-- & \quad \text{P6_{1}, P6_{5}} \\
\text{P}6_{1}22 & \quad \text{P6_{1}22, P6_{5}22}
\end{align*}
\]
Space group determination from powder data: systematic overlapping by symmetry

• Orthorombic with
  \( a=10.00 \text{ Å}, \ b=5.77 \text{ Å}, \ c=14.32 \text{ Å} \)

The reflections
  \((h00), (-h,-k,0), (-h,k,0), (h,-k,0)\)
will systematically overlap.

The number of overlapping reflections is given by the multiplicity \( m_h \), which is reflection dependent

• \( m_h = 2 \) for the reflection \((h00)\),
• \( m_h = 4 \) for the reflection \((110)\),
• \( m_h = 8 \) for a generic \((hkl)\) reflection.
The reflection multiplicity $m_h$

- The value of $m_h$ may be calculated by the following algorithm.
- Let 
  
  $C_s = (R_s, T_s), s = 1, \ldots, m$

- be the set of symmetry operators of the space group: the number of distinct vectors

  $h_s = hR_s, \quad s = 1, \ldots, m$

- eventually added to the Friedel opposites

  $-h_s = -hR_s$

- if the space group is non-centric, corresponds to the multiplicity $m_h$ of the reflection.
• Non-symmetry-equivalent reflections can occasionally overlap because of special metric relationships. E.g., in the orthorhombic cell defined by

- \( a = 10.00 \ \text{Å}, b = 5.77 \ \text{Å}, c = 14.32 \ \text{Å} \)
- \( b \approx a / \sqrt{3} \)
- (200) and (110) overlap, as well as (400) and (220), etc.
- Of course, according to the \( c \) values, reflections of (hkl) type can occasionally overlap with (hk0)
- reflections.
Occasional Overlapping

The occasional overlapping of two reflections may be total or partial according to the misfit of the corresponding $2\theta$ angles.
Systematic overlapping of reflections which are not symmetry equivalents

- In high symmetry crystal systems (i.e., in trigonal, tetragonal, hexagonal and cubic systems), where the lattice symmetry may be higher than the Laue symmetry, **systematic overlapping** may occur between reflections which are not symmetry equivalent.

- Ex.
  - Laue group $\overline{3}$, lattice symmetry $\overline{3}m$
  - Laue group $4/m$, lattice symmetry $4/mmm$
Systematic overlapping

For space groups of the Laue class 4/m the symmetry equivalent reflections are

\[(hk\bar{l}), (\bar{h}k\bar{l}), (\bar{k}h\bar{l}), (k\bar{h}l), (\bar{h}k\bar{l}), (hk\bar{l}), (kh\bar{l}), (\bar{k}h\bar{l})\]

Owing to the lattice symmetry [i.e., 4/mmm], the following reflections systematically overlap:

\[(hk\bar{l}), (\bar{h}k\bar{l}), (\bar{k}h\bar{l}), (k\bar{h}l), (\bar{h}k\bar{l}), (hk\bar{l}), (kh\bar{l}), (\bar{k}h\bar{l}), (hk\bar{l}), (\bar{h}k\bar{l}), (hk\bar{l}), (kh\bar{l}), (\bar{k}h\bar{l}), (h\bar{k}l), (\bar{k}h\bar{l}), (k\bar{h}l), (\bar{k}h\bar{l})\]

The first eight are symmetry equivalent to \((hk\bar{l})\) and the second eight to \((kh\bar{l})\).

\(I_{hk\bar{l}}\) and \(I_{k\bar{h}l}\) are uncorrelated. Then the measured overall intensity (summing the contributions of the 16 reflections) cannot be reliably partitioned into \(I_{hk\bar{l}}\) and \(I_{k\bar{h}l}\).
Systematic overlapping

- For space groups belonging to the Laue class $m\overline{3}$ the 24 equivalent reflections are $(hkl), (\overline{h}k\overline{l}), (\overline{h}k\overline{l})$, $(lhk), (l\overline{h}k), (l\overline{h}k)$, $(k\overline{h}), (k\overline{l}h), (k\overline{l}h), (k\overline{l}h)$
  
  + Friedel opposites (1)

Owing to the lattice symmetry (i.e., $m\overline{3}m$) there are 48 reflections which have the same value of $d_{hkl}$: besides the reflections (1), also the following ones overlap:

$(k\overline{l}h), (k\overline{l}h), (k\overline{l}h), (k\overline{l}h), (l\overline{k}h), (l\overline{k}h), (l\overline{k}h), (h\overline{l}k), (h\overline{l}k), (h\overline{l}k), (h\overline{l}k)$

  + Friedel opposites (2)

- $I_{hkl}$ and $I_{khl}$ are uncorrelated. Then the measured overall intensity (summing the contributions of the 48 reflections) cannot be reliably partitioned into $I_{hkl}$ and $I_{khl}$. 
DETERMINATION OF THE SPACE GROUP

- Experimental powder diffraction diagrams are decomposed via LeBail algorithm into single diffraction intensities in the space group having the largest Laue symmetry and no extinction conditions.

- (e.g., $P12/m1$ in monoclinic, $P2/m2/m2/m$ for orthorhombic, $P4/mmm$ for tetragonal, $P6/mmm$ for trigonal-hexagonal systems, and $Pm3m$ for the cubic system.)
DETERMINATION OF THE SPACE GROUP

• The intensities are first normalized
• and then
• submitted to statistical analysis for the determination of the space group symmetry.
• The algorithm provides a probability value for each extinction symbol compatible with the previously established lattice symmetry.
A REPRESENTATIVE EXAMPLE

• In the orthorhombic system any space group may be represented by the string

\[ M \ r_1 / S_1 \ r_2 / S_2 \ r_3 / S_3 \]

• M denotes the unit cell
• \( r_j, \ j = 1, \ldots, 3 \) are the symmetry elements along the three axes
• \( S_j, \ j = 1, \ldots, 3 \) are the symmetry elements perpendicular to the axes
The occurrence probabilities for the axes are

\[ p(2_{1[100]}) = 1 - \langle |E_{h00}|^2 \rangle_{h=2n+1}; \quad p(2_{1[010]}) = 1 - \langle |E_{0k0}|^2 \rangle_{k=2n+1}; \]
\[ p(2_{1[001]}) = 1 - \langle |E_{00l}|^2 \rangle_{l=2n+1} \]

The probabilities are equal to unity if the \( |E|^2 \) averages are equal to zero, and vanish if the averages are equal or larger than unity.

Conversely, for the twofold axes

\[ p(2_{[100]}) = 1 - p(2_{1[100]}); \quad p(2_{[010]}) = 1 - p(2_{1[010]}); \]
\[ p(2_{[001]}) = 1 - p(2_{1[001]}) \]
Occurrence probabilities

- For the planes $S$ normal to $[100]$:

$$p(b) = 1 - \langle |E_{0kl}|^2 \rangle_{k=2n+1}; \quad p(c) = 1 - \langle |E_{0kl}|^2 \rangle_{l=2n+1};$$

$$p(n) = 1 - \langle |E_{0kl}|^2 \rangle_{k+l=2n+1}; \quad p(d) = 1 - \langle |E_{0kl}|^2 \rangle_{k+l\neq4n}$$

$$p(m) = 1 - \max[p(b), p(c), p(n), p(d)]$$
• The **probabilities** for the different types of unit cell are the following:

\[
p(A) = 1 - \langle |E_{hkl}|^2 \rangle_{k+l=2n+1}; \quad p(B) = 1 - \langle |E_{hkl}|^2 \rangle_{h+l=2n+1}; \quad p(C) = 1 - \langle |E_{hkl}|^2 \rangle_{h+k=2n+1}; \quad p(I) = 1 - \langle |E_{hkl}|^2 \rangle_{h+k+l=2n+1}; \quad p(F) = 1 - \langle |E_{hkl}|^2 \rangle_{[g]}, \]

\[
p(P) = 1 - \max[p(A), p(B), p(C), p(I), p(F)]
\]

• \([g]\) means not all the indices odd or even.

• In the practice it is important to calculate

\[
<Z_w> = (\sum w_j Z_j) / \sum w_j
\]

• where \(w\) takes the overlapping into account.
The probability of an extinction symbol

- In the orthorhombic system

\[ p(P - - -) = p(P)p(2_{[100]})p(m \perp a)p(2_{[010]})p(m \perp b)p(2_{[001]})p(m \perp c) \]

- Calculate now \( p(Bb-b) \). The string \( b-b \) implies the following symmetry elements:

\[ b \perp a, \ b \perp c, \ 2_{1[010]} \]

- The presence of \( B \) additionally implies

\[ c \perp a, \ n \perp b, \ a \perp c, \ 2_{1[100]}, \ 2_{1[001]} \]

- Then

\[ p(Bb-b) = p(B)p(2_{1[100]})p(b, c \perp a)p(2_{1[010]})p(n \perp b)p(2_{1[001]})p(a, b \perp c) \]
For each ES the list of compatible space groups is made available. Once the space group is accepted, a new pattern decomposition is made.
The limit of the $|E|^2$-statistics

- A limit of the $z$-statistics is that the reliability of an $ES$ is calculated via the analysis of the average $|E|^2$-values corresponding to specific subsets of reflections.
- The method is unable to distinguish between the case in which all the peak intensities of a given subset are quite small and the case in which very few of them are medium or large. This may cause substantial errors in the identification of the correct $ES$. 
The control peak list

• Reflections non-overlapping or in weak overlapping with others are selected. Their number is expected to be sufficiently high to include a sufficiently large number of reflections, belonging to the various subsets of reflections involved in the extinction rules.

• Such a number may vary from few unities for quite small structures to one-two hundreds for larger structures.
The use of the control peak list

• Suppose that $p(ES)$ has been already estimated.

• Then each reflection belonging to the control peak list is checked: if its indices contradict an extinction rule of the $ES$ then $p(ES)$ is multiplied by 0.5. $p(ES)$ is multiplied by $0.5^n$ if $n$ reflections contradict the extinction rules of $ES$.

• If the probability of a symmetry element is reduced, the same is made for the derived symmetry elements.
An algorithm has been introduced to automatically exclude from the calculations $2\theta$ intervals for which there is no evidence of an appreciable diffraction signal. In general they lie at high $2\theta$ values, where small intensities can be transformed in large normalized intensities.
Experimental applications

We applied *EXPO2008* and *EXPO2004* to 75 test structures.

In 49/75 cases *orn=1* for both the programs.

In the other cases the main problems are:

- *Pseudosymmetry* (often pseudotranslational symmetry)
- *Impurity peaks*
<table>
<thead>
<tr>
<th>Codename</th>
<th>$cES$</th>
<th>EXPO2004</th>
<th>EXPO2008</th>
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<td>AGPZ</td>
<td>P b c a</td>
<td>25</td>
<td>7</td>
</tr>
<tr>
<td>BAMO</td>
<td>P 1 2 1 1</td>
<td>3</td>
<td>2</td>
</tr>
<tr>
<td>CARBA</td>
<td>C m c a</td>
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<td>1</td>
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<tr>
<td>CeO$_2$</td>
<td>F _ _ _</td>
<td>3</td>
<td>1</td>
</tr>
<tr>
<td>CFCL</td>
<td>F d d _</td>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>CUPZ</td>
<td>P b c a</td>
<td>16</td>
<td>1</td>
</tr>
<tr>
<td>DADA</td>
<td>P 2$_1$ 2$_1$ 2$_1$</td>
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<td>1</td>
</tr>
<tr>
<td>F1A</td>
<td>R _ _</td>
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<td>7</td>
</tr>
<tr>
<td>LaB$_6$</td>
<td>P _ _ _</td>
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<td>1</td>
</tr>
<tr>
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<td>P 1 2 1 1</td>
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<td>1</td>
</tr>
<tr>
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<td>P 1 2$_1$/a 1</td>
<td>2</td>
<td>1</td>
</tr>
<tr>
<td>MCM</td>
<td>P _ _ _</td>
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<td>1</td>
</tr>
<tr>
<td>NICKEL</td>
<td>P 1 2/c 1</td>
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<td>2</td>
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<td>EXPO2004</td>
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<td>--------------</td>
<td>----------</td>
<td>----------</td>
</tr>
<tr>
<td>NIZR</td>
<td>P 1 21/n 1</td>
<td>4</td>
<td>1</td>
</tr>
<tr>
<td>NORB2</td>
<td>P 1 21/c 1</td>
<td>4</td>
<td>1</td>
</tr>
<tr>
<td>OTHYM</td>
<td>P 1 21/n 1</td>
<td>2</td>
<td>1</td>
</tr>
<tr>
<td>RUCO</td>
<td>I b a _</td>
<td>2</td>
<td>2</td>
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<tr>
<td>SALBU</td>
<td>P b c a</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>SBPO</td>
<td>P 1 21/n 1</td>
<td>5</td>
<td>1</td>
</tr>
<tr>
<td>SCAMPHOR1</td>
<td>P 21 21 21</td>
<td>5</td>
<td>6</td>
</tr>
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<td>2</td>
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<td>P 41 21 _</td>
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<td>12</td>
</tr>
<tr>
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<td>3</td>
</tr>
<tr>
<td>YONO</td>
<td>P 1 21 1</td>
<td>2</td>
<td>2</td>
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</table>
### RUNT - Ten extinction symbols in order of $p(ES)$ as calculated by:

<table>
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<tr>
<th></th>
<th>$P(ES)$</th>
<th></th>
<th>$P(ES)$</th>
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<tbody>
<tr>
<td>I b c a</td>
<td>0.559</td>
<td>P 2_1 2_1 2_1</td>
<td>0.127</td>
</tr>
<tr>
<td>I c_ _ a</td>
<td>0.546</td>
<td>P n _ a</td>
<td>0.123</td>
</tr>
<tr>
<td>I_ _ c b</td>
<td>0.530</td>
<td>P n n _</td>
<td>0.041</td>
</tr>
<tr>
<td>I b a _</td>
<td>0.526</td>
<td>P n n a</td>
<td>0.038</td>
</tr>
<tr>
<td>I_ _ (ab)</td>
<td>0.517</td>
<td>P n _ n</td>
<td>0.025</td>
</tr>
<tr>
<td>I(bc) _ _</td>
<td>0.513</td>
<td>P b n _</td>
<td>0.018</td>
</tr>
<tr>
<td>I_ _ (ac)_</td>
<td>0.498</td>
<td>P n a _</td>
<td>0.017</td>
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<tr>
<td>I_ _ _</td>
<td>0.486</td>
<td>P b n a</td>
<td>0.017</td>
</tr>
<tr>
<td>B b (ac) b</td>
<td>0.475</td>
<td>P n a a</td>
<td>0.016</td>
</tr>
<tr>
<td>B b _ b</td>
<td>0.463</td>
<td>P n c a</td>
<td>0.016</td>
</tr>
</tbody>
</table>
**ZOPI**: a small interval around $2\theta = 10^\circ$.

The reflection positions are represented by red vertical bars; The reflection $(1\ 0\ -2)$ [violating the extinction rules of the true symbol ($P\ 21/c$)] completely overlaps with an impurity peak.
Look at the pattern

MCM-22, space group P6/mmm, ES P---. The space groups in order of orn are shown in the figure. The first peak excludes ES up to orn=5
Correct s.g. I222, orn=2; orn=1 for I – (ac) –

$N_s a r$ (number of syst. abs ref) for $I,a,c$ operators (blue and red colour for single and for overlapping syst. abs. refl.);

Interval ($9^\circ$ - $14.5^\circ$).

- Vertical bars at the bottom show reflection positions;
- upper black bars show single extinct reflections for the $I – (ac) –$
**APOFERRITIN**

*Spacegroup* $F432 - ES = F\_\_\_\_ - a = 184.305\ \AA$

*N-TREOR*: $a = 183.116\ \AA$, $N_{PEAK} = 40$, $M_{20} = 13$, $NIX = 2$

RANK = 1

*Sp. Group Determ.*: $RES = 6.02\ \AA$, $N_{REFL} = 2911$

$ES_1 = F\_\_\_\_, \ P(ES_1) = 0.537$

$ES_2 = F4_1\_\_, \ P(ES_2) = 0.208$
**HEWL**

**Spacegroup** $P4_32_12$ - ES = $P4_32_12$

$a = 78.844$ Å, $c = 38.188$ Å

**N-TREOR:** $a = 78.782$ Å, $c = 38.140$ Å,
$N_{PEAK} = 81$, $M_{20} = 29$, $NIX = 0$  RANK = 1

**Sp. Group Determ.:** RES = 6.02 Å, $N_{REFL} = 382$

$ES_1 = P4_32_12$, $P(ES_1) = 0.383$

$ES_2 = P4_22_12$, $P(ES_2) = 0.338$
INSULIN CUBIC

Spacegroup $I2_13 - ES = I\_\_\_\_ - a = 79.00 \text{ Å}$

$N$-TREOR: $a = 78.972 \text{ Å}, N_{\text{PEAK}} = 80, M_{20} = 76, N_{\text{IX}} = 0$ \hspace{1cm} RANK = 2

Sp. Group Determ.: RES = 6.02 Å, $N_{\text{REFL}} = 289$

$ES_1 = I\_\_\_, P(ES_1) = 0.767$

$ES_2 = I4_1\_\_\_ P(ES_2) = 0.355$
INSULIN HEXAGONAL

Spacegroup $R3 - ES = R\_\_\_\_$

$a = 82.495 \, \text{Å}, \quad c = 33.650 \, \text{Å}$

$N\text{-TREOR: } a = 82.486 \, \text{Å}, \quad c = 33.636 \, \text{Å}, \quad N_{\text{PEAK}} = 98, \quad M_{20} = 37, \quad N_{\text{IX}} = 1 \quad \text{RANK} = 1$

$Sp. \text{ Group Determ. : } RES = 6.02 \, \text{Å}, \quad N_{\text{REFL}} = 228$

$ES_1 = P6_2 \_ \_ \_ , \quad P(ES_1) = 0.770$
$ES_2 = P3_1 \_ \_ \_ , \quad P(ES_2) = 0.770$
$ES_3 = P6_1 \_ \_ \_ , \quad P(ES_3) = 0.703$
$ES_4 = P6_3 \_ \_ \_ , \quad P(ES_4) = 0.549$
$ES_5 = P\_ \_ \_ \_ , \quad P(ES_5) = 0.086$
$ES_6 = R(\text{obv}) \_ \_ , \quad P(ES_6) = 0.042$
$ES_7 = R(\text{rev}) \_ \_ , \quad P(ES_7) = 0.042$
$ES_8 = P\_ \_ \_ c , \quad P(ES_8) = 0.014$
Insulin hexagonal
Resolution = 6.02 Å
Insulin hexagonal, Resolution = 6.02 Å
**TEWL**

**Spacegroup** \( P6_122 \) - \( ES = P6_1 \_\_\_\_\_\_ \)
\[ a = 71.198 \, \text{Å}, \quad c = 85.205 \, \text{Å} \]

**N-TREOR:** \( a = 71.093 \, \text{Å}, \quad c = 85.023 \, \text{Å}, \quad N_{\text{PEAK}} = 95, \quad M_{20} = 55, \quad N_{\text{IX}} = 0. \quad \text{RANK} = 1 \)

**Sp. Group Determ.:** \( RES = 6.02 \, \text{Å}, \quad N_{\text{REFL}} = 412 \)

\[
\begin{align*}
ES_1 & = P6_2 \_\_\_, \quad P(ES_1) = 0.884 \\
ES_2 & = P3_1 \_\_\_, \quad P(ES_2) = 0.884 \\
ES_3 & = P6_3 \_\_\_, \quad P(ES_3) = 0.884 \\
ES_4 & = P6_1 \_\_\_, \quad P(ES_4) = 0.884 \\
ES_5 & = P\_\_\_\_\_, \quad P(ES_5) = 0.098
\end{align*}
\]
THAUMATIN

Spacegroup $P4_122$ - $ES = P4_1__$

$a = 58.731$ Å, $c = 151.614$ Å

N-TREOR: $a = 58.553$ Å, $c = 151.320$ Å, $N_{\text{PEAK}} = 70$, $M_{20} = 31$, $N_{\text{IX}} = 0$  
RANK = 1

Sp. Group Determ.: $RES = 6.02$ Å, $N_{\text{REFL}} = 412$

$ES_1 = P4_2\ 2_1\ _,\ \ P(ES_1) = 0.515$

$ES_2 = P4_1\ 2_1\ _,\ \ P(ES_2) = 0.515$

$ES_3 = P_\ \ 2_1\ _,\ \ P(ES_3) = 0.057$

$ES_4 = P4_1\ _\ _\ _\ ,\ \ P(ES_4) = 0.057$

$ES_5 = P4_2\ _\ _\ _\ ,\ \ P(ES_5) = 0.057$

$ES_6 = P4_2/n\ _\ _\ _\ ,\ \ P(ES_6) = 0.027$
Thaumatin
Resolution = 6.02 Å

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