Twinning

Twinning occurs when two or more individuals of the **same phase** exist in the sample. The individuals are related by twin operations.

Set of twinning operators:

$$\left\{\hat{E} \equiv \hat{T}_1, \hat{T}_2, \cdots \hat{T}_n\right\}$$

Their application to the base reciprocal vectors of the first individual:

$$\left\{\mathbf{a}_1^*, \mathbf{a}_2^*, \mathbf{a}_3^*\right\} \xrightarrow{\hat{E}} \left\{\mathbf{a}_1^*, \mathbf{a}_2^*, \mathbf{a}_3^*\right\}$$

$$\left\{\mathbf{a}_1^*, \mathbf{a}_2^*, \mathbf{a}_3^*\right\} \xrightarrow{\hat{T}_2} \left\{\hat{T}_2\mathbf{a}_1^*, \hat{T}_2\mathbf{a}_2^*, \hat{T}_2\mathbf{a}_3^*\right\}$$

....

$$\left\{\mathbf{a}_1^*, \mathbf{a}_2^*, \mathbf{a}_3^*\right\} \xrightarrow{\hat{T}_n} \left\{\hat{T}_n\mathbf{a}_1^*, \hat{T}_n\mathbf{a}_2^*, \hat{T}_n\mathbf{a}_3^*\right\}$$

The operations give rise to *m* independent vectors.

$m=3$	complete overlap
$3 < m \leq 3n$	partial overlap
$m=3n$	no overlap

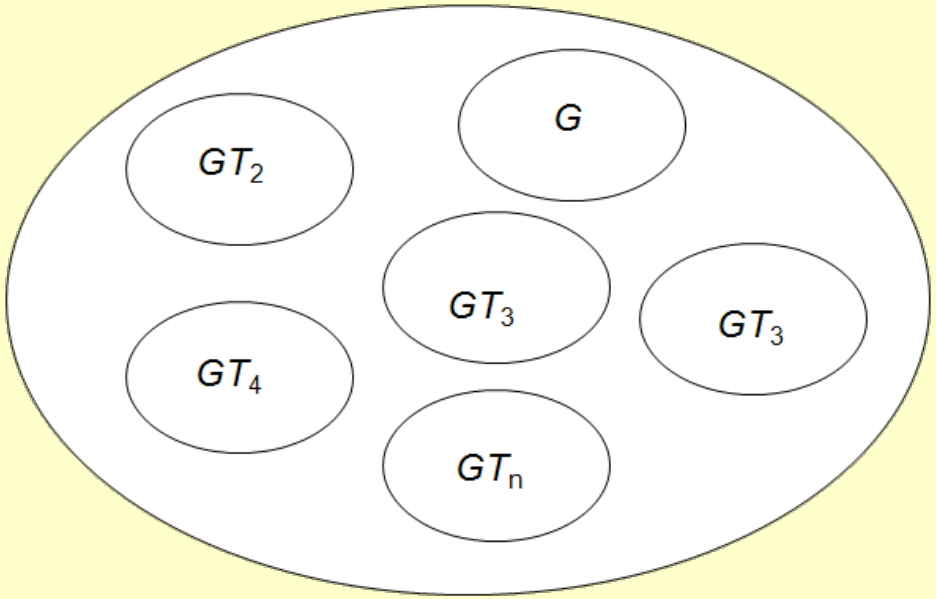
Posible twinning operations can be determined from group → subgroup relations:

G … point group of the structure
H … point group of the crystal lattice

$$G \subset H$$

Twinning operations:

$$\left\{\hat{E} \equiv \hat{T}_1, \hat{T}_2, \cdots \hat{T}_n\right\}$$



With twinning operations we can get indices of a selected reflection in all twin domains

$$\mathbf{h}_i = \mathbf{h} \cdot \mathbf{T}_i$$

Decomposition of H to right classes according to G:
- take element T_1 of H which is not part of G
- create set of elements GT_1 (this is not necessarily subgroup)
- find element T_2 of H which is member neither of G nor of GT_1
- create set of elements GT_2
- Finally H is decomposed to so called right classes. Each element of H belongs to one and only one right class. Each member of the right class can be taken as twinning operation.

Example: *H = 4/mmm, G = mmm*

Order *H* is16, order *G* is 8 $\Rightarrow$ index of the twin is 2 and twin operation can be any from the following symmetry operations of *H*:

$$\begin{bmatrix} 0 & -1 & 0 \\ 1 & 0 & 0 \\ 0 & 0 & 1 \end{bmatrix} \quad \begin{bmatrix} 0 & 1 & 0 \\ -1 & 0 & 0 \\ 0 & 0 & 1 \end{bmatrix} \quad \begin{bmatrix} 0 & 1 & 0 \\ 1 & 0 & 0 \\ 0 & 0 & -1 \end{bmatrix} \quad \begin{bmatrix} 0 & -1 & 0 \\ -1 & 0 & 0 \\ 0 & 0 & -1 \end{bmatrix}$$

$$4^{+}\ 0,0,z \qquad 4^{-}\ 0,0,z \qquad 2\ \ x,x,0 \qquad 2\ \ x,\bar{x},0$$

$$\begin{bmatrix} 0 & 1 & 0 \\ -1 & 0 & 0 \\ 0 & 0 & -1 \end{bmatrix} \quad \begin{bmatrix} 0 & -1 & 0 \\ 1 & 0 & 0 \\ 0 & 0 & -1 \end{bmatrix} \quad \begin{bmatrix} 0 & -1 & 0 \\ -1 & 0 & 0 \\ 0 & 0 & 1 \end{bmatrix} \quad \begin{bmatrix} 0 & 1 & 0 \\ 1 & 0 & 0 \\ 0 & 0 & 1 \end{bmatrix}$$

$$\bar{4}^{+}\ 0,0,z \qquad \bar{4}^{-}\ 0,0,z \qquad m\ \ x,\bar{x},0 \qquad m\ \ x,x,0$$

Diffraction pattern combines **intensities** of twin individuals.

Structure factor of a merohedric twin:

$$\mathcal{F}^2(\mathbf{H}) = v_1 F^2(\mathbf{H}\cdot\mathbf{T}_1) + v_2 F^2(\mathbf{H}\cdot\mathbf{T}_2) + \ldots + v_n F^2(\mathbf{H}\cdot\mathbf{T}_n)$$

where v_i are domain fractions

$\mathbf{T}_i$ are twin operations

The sum of domain fractions $\sum v_i = 1$

Symmetry element of the crystal structure:

$$\mathbf{S}_i \in G \Rightarrow |F(\mathbf{H}\cdot\mathbf{S}_i)| = |F(\mathbf{H})|$$

Transformation of the structure factor of a twin:

$$\mathcal{F}^2(\mathbf{H}\cdot\mathbf{S}_i) = v_1 F^2(\mathbf{H}\cdot\mathbf{S}_i\cdot\mathbf{T}_1) + v_2 F^2(\mathbf{H}\cdot\mathbf{S}_i\cdot\mathbf{T}_2) + \ldots + v_n F^2(\mathbf{H}\cdot\mathbf{S}_i\cdot\mathbf{T}_n)$$

The condition $\mathcal{F}^2(\mathbf{H}\cdot\mathbf{S}_i) = \mathcal{F}^2(\mathbf{H})$ is fullfilled only when

$$\mathbf{H}\cdot\mathbf{S}_i\cdot\mathbf{T}_k = \mathbf{H}\cdot\mathbf{T}_k\cdot\mathbf{S}_j \Rightarrow \mathbf{S}_j = \mathbf{T}_k^{-1}\cdot\mathbf{S}_i\cdot\mathbf{T}_k$$

The conditions

$$\mathbf{H}\cdot\mathbf{S}_i\cdot\mathbf{T}_k = \mathbf{H}\cdot\mathbf{T}_k\cdot\mathbf{S}_j$$

means that the argument of structure factor is always from the same right class of the decomposition. Otherwise the argument would "skip" between domains.

Diffraction symmetry of a twin is given by a set of symmetry operations invariant with respect to the twinning operations. It can be maximally equal to the point group of the lattice, when the domain fractions are equal:

$$v_1 = v_2 = v_3 \ldots = v_n = 1/n$$

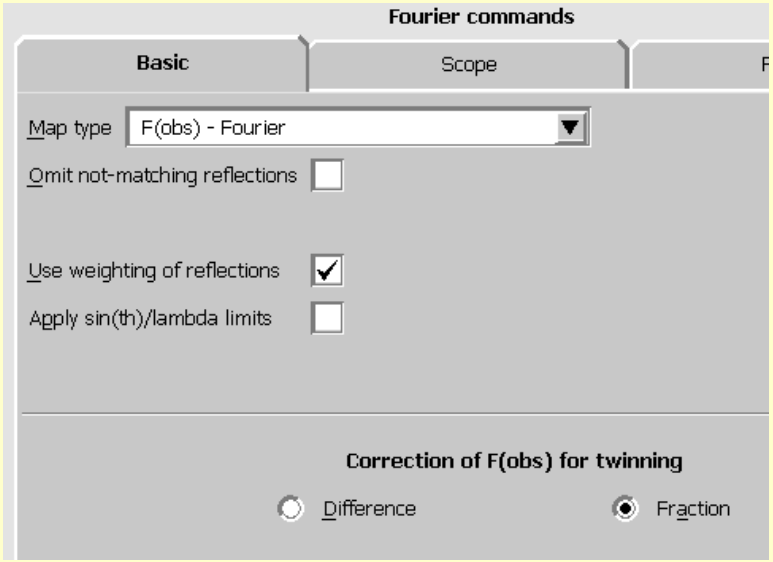
Fourier coefficients for twin

$$F_{obs}(\mathbf{H}) = \mathcal{F}_{obs}(\mathbf{H})\frac{F_{calc}(\mathbf{H})}{\mathcal{F}_{calc}(\mathbf{H})}$$

$$F_{obs}(\mathbf{H}) = \sqrt{\mathcal{F}^2_{obs}(\mathbf{H}) - v_2 F^2_{calc}(\mathbf{H}\cdot\mathbf{T}_2)\ldots - v_n F^2_{calc}(\mathbf{H}\cdot\mathbf{T}_n)}$$

$\mathcal{F}_{obs}(\mathbf{H})$ Structure factors from all domains

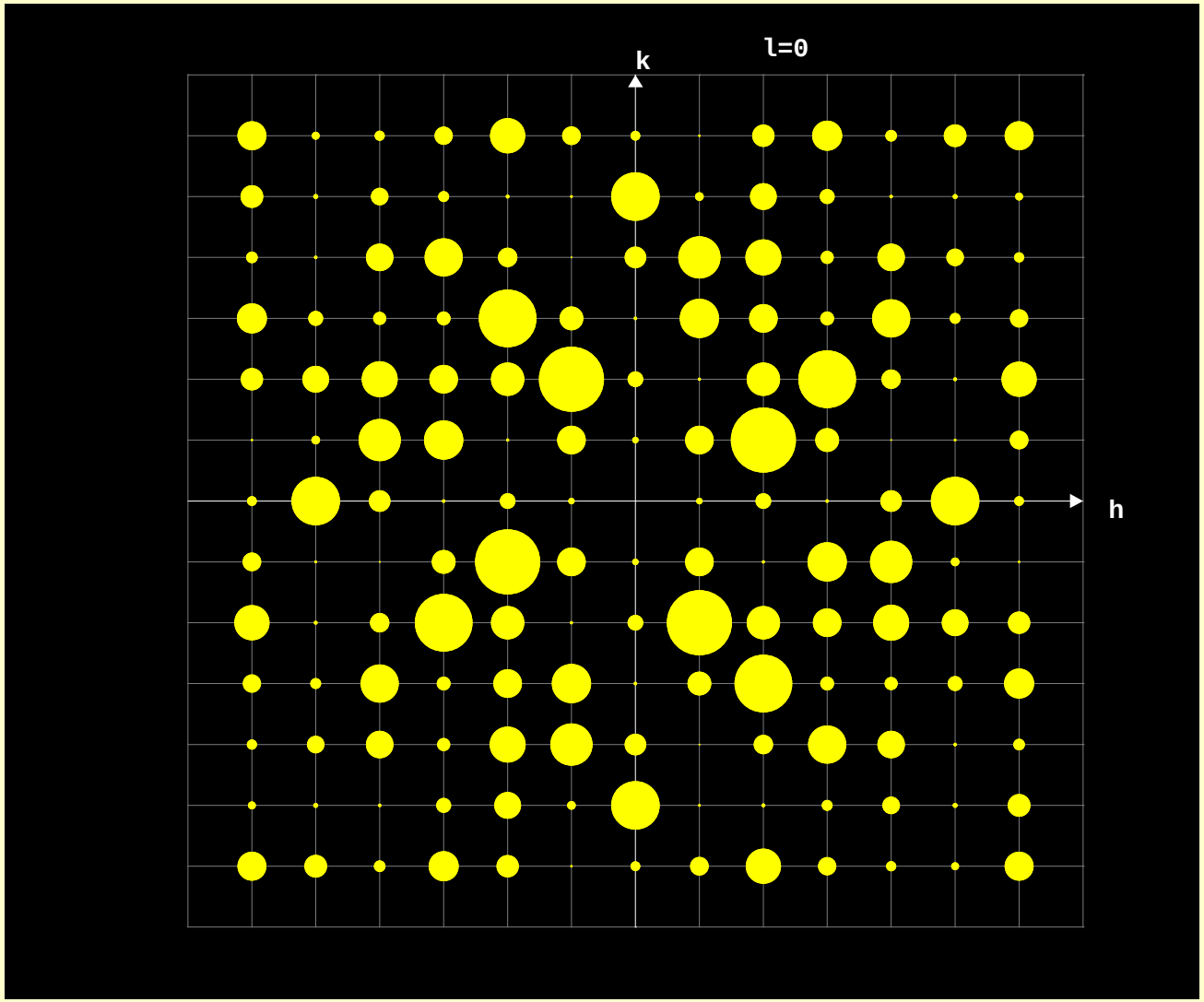
$F_{obs}(\mathbf{H}), F_{calc}(\mathbf{H})$ Structure factors from one domain (needed for calculation of Fourier map)



The precision of “fraction” method grows with completing of the structure model

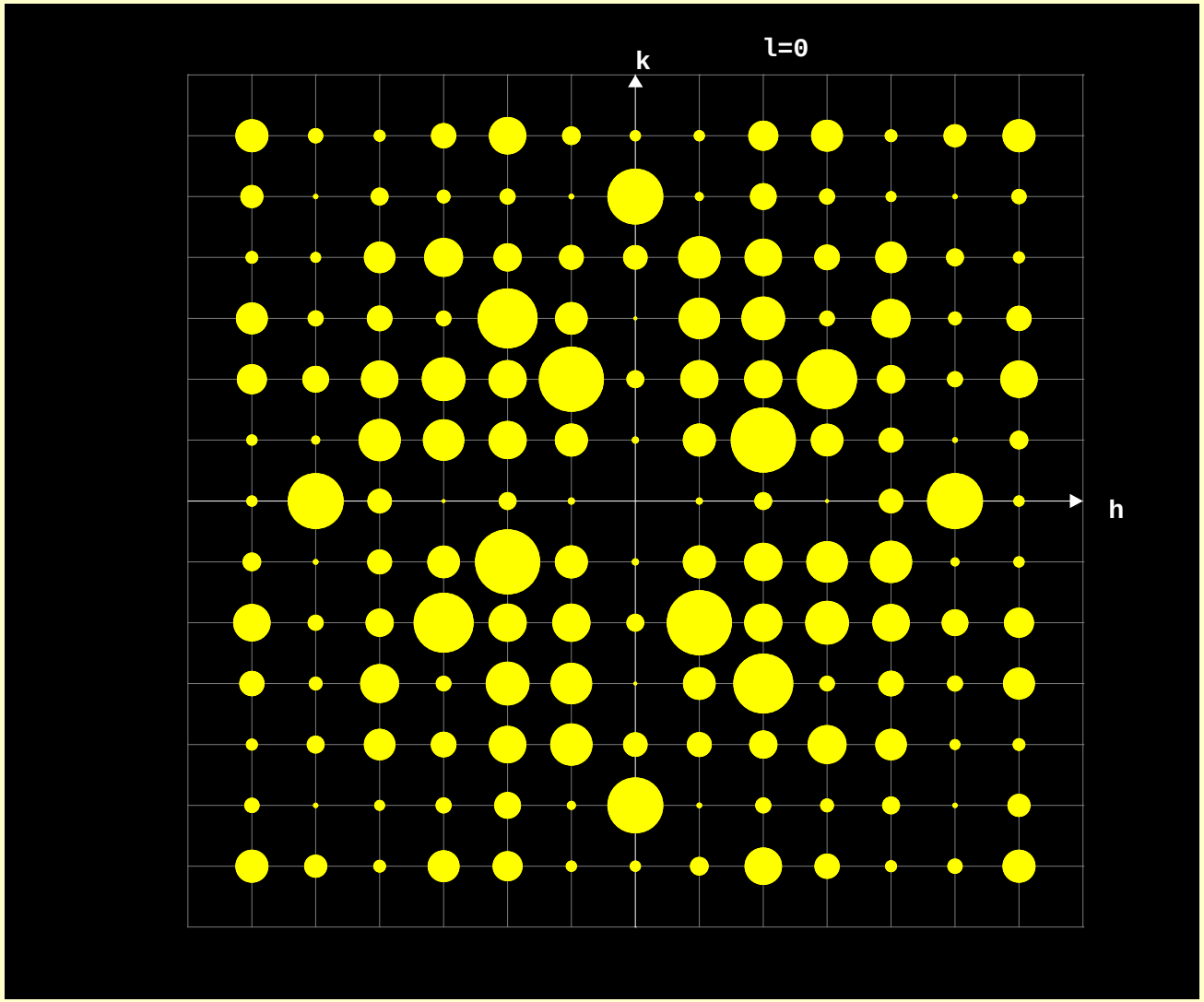
If the Fourier map is not satisfactory switching the method may help

Diffraction symmetry of a merohedric twin: The chance to recognize proper symmetry from the diffraction pattern of merohedric twin depends on the twin domains ratio.



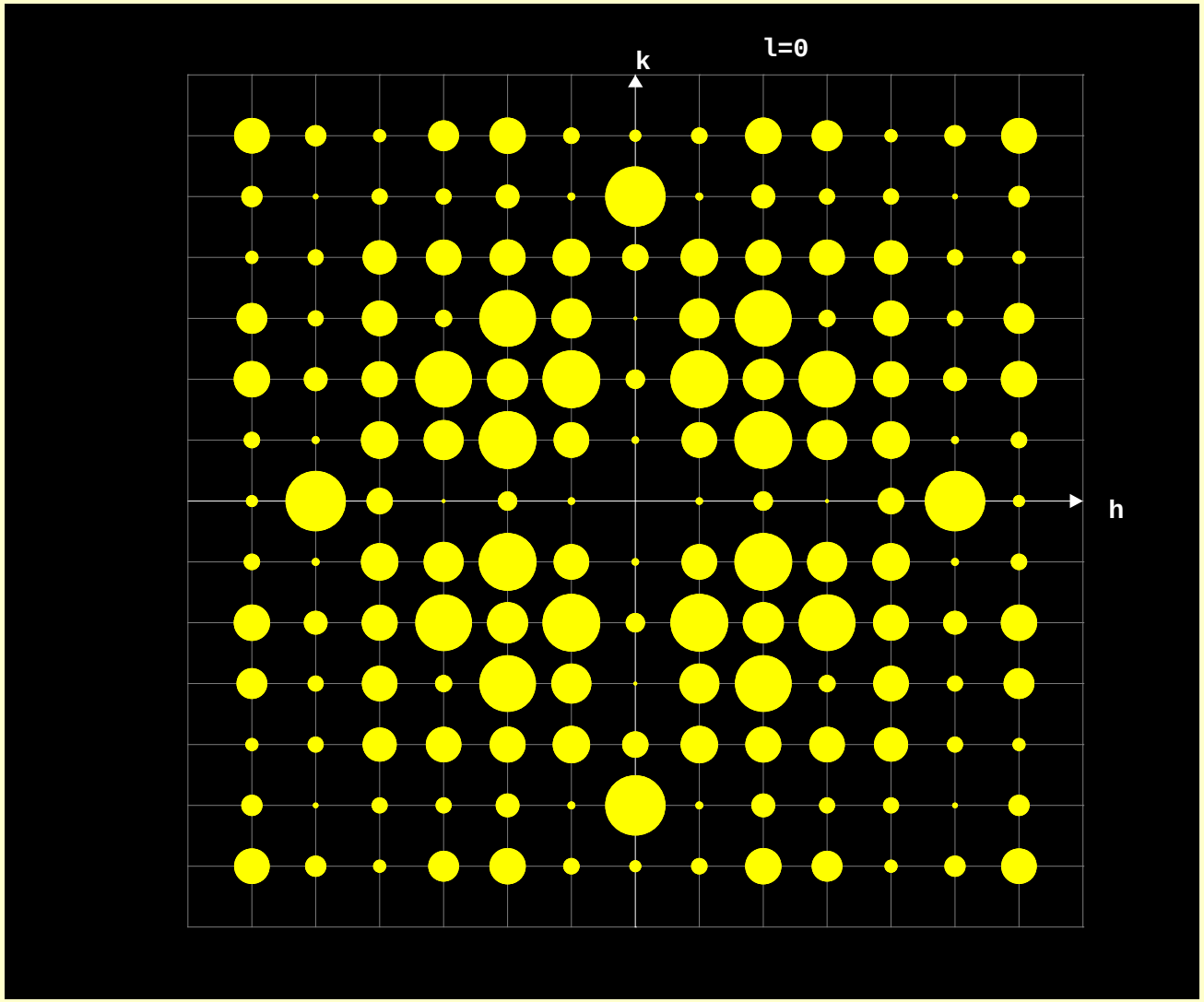
Lattice:
4/mmm
Structure:
4/m
Twin operation:
none

For non-equal domains ratio the proper symmetry is still evident.



Lattice:
4/mmm
Structure:
4/m
Twin operation:
m_x
2nd domain fraction:
25%

For equal domains ratio false higher symmetry is detected.

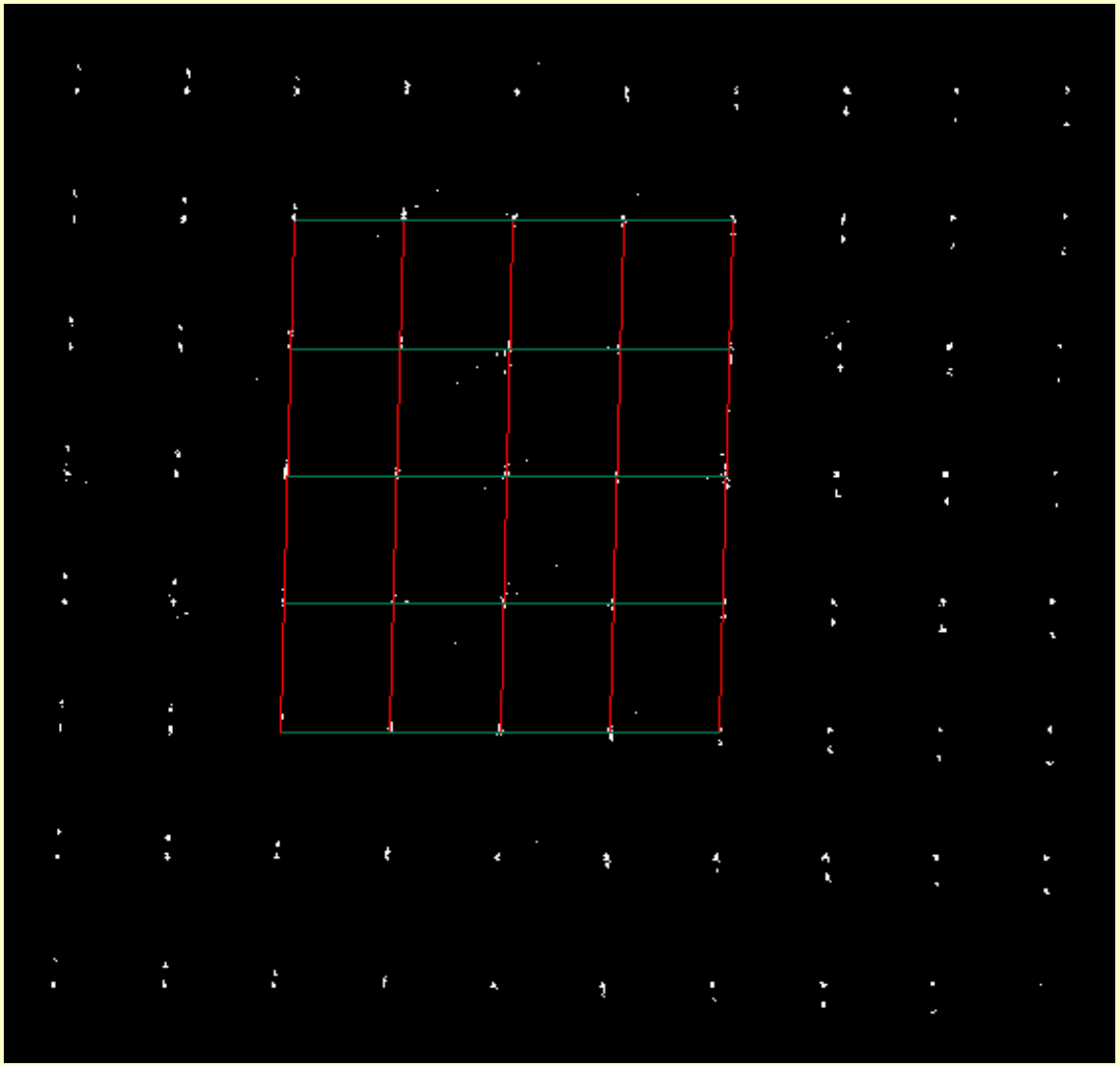


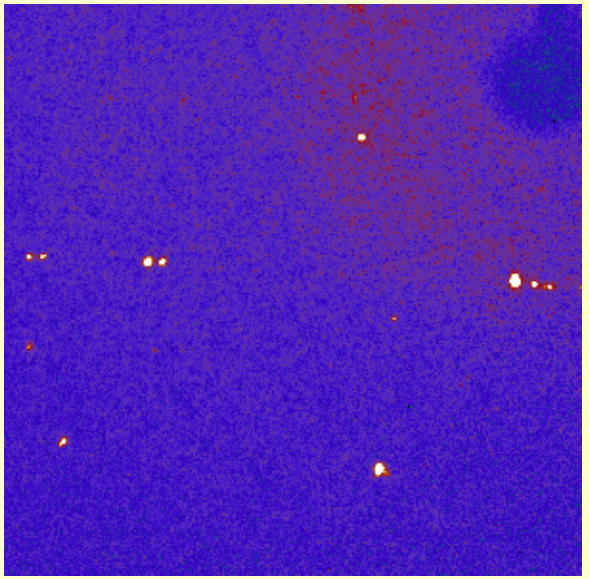
Lattice:
4/mmm
Structure:
4/m
Twin operation:
m_x
2nd domain fraction:
50%

Twins with partial overlaps (m > 3)

$Y(HP_2O_7)(H_2O)_3$

Cell parameters: 6.422 6.890 9.817 81.65 80.27 88.35; symmetry: P-1





Twinning matrix:

-1.0000 0.0517 0.0000
0.0000 1.0000 0.0000
0.0000 0.4161 -1.0000

(**h**' = **T***h, h is a column)

Rot= 180º around **b**

$\mathbf{T}$ applies to indices as a column, therefore $\mathbf{T}$ inverted transposed will apply to reciprocal base vectors as a column. For two fold twin $\mathbf{T}^{-1} = \mathbf{T}$.

$$\begin{pmatrix} -1 & 0 & 0 \\ 0.0517 & 1 & 0.4161 \\ 0 & 0 & -1 \end{pmatrix} \begin{pmatrix} \mathbf{a}^* \\ \mathbf{b}^* \\ \mathbf{c}^* \end{pmatrix} = \begin{pmatrix} -\mathbf{a}^* \\ 0.0517\mathbf{a}^* + \mathbf{b}^* + 0.4161\mathbf{c}^* \\ -\mathbf{c}^* \end{pmatrix}$$

The twinning operation yields one new reciprocal vector:

$$\mathbf{q} = \left(0.0517\mathbf{a}^* + \mathbf{b}^* + 0.4161\mathbf{c}^*\right)$$

We can index the complete diffraction pattern using four indices h,k,l,m. The first twin individual will have indices h,k,l,0; the second twin individual will have indices –h,m,-l,0. The relationship between the individuals can be expressed by 4x4 matrix:

$$\mathbf{W} = \begin{pmatrix} -1 & 0 & 0 & 0 \\ 0 & 0 & 0 & 1 \\ 0 & 0 & -1 & 0 \\ 0 & 1 & 0 & 0 \end{pmatrix}, \qquad \mathbf{H}^1 = \mathbf{H}^2\mathbf{W}$$

Sometimes the splitting is tiny.
In this case it would be hidden for normal CCD measurement.

$NiCd(CN)_4O_3$
Cell parameters: 8.522 16.012 7.651 90 90.32 90
Symmetry: C2/c(α0γ)0s
q vector (0.4346, 0, 0.13)

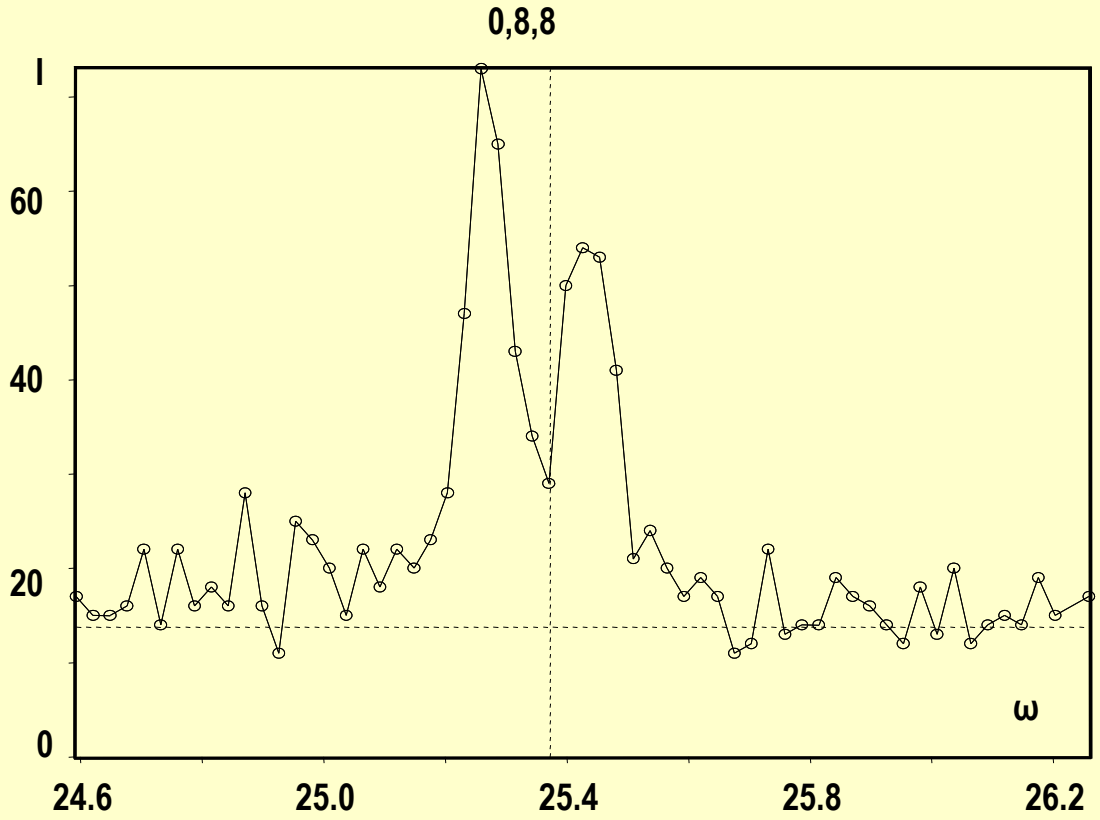
Twinning matrix:

$$\begin{pmatrix} -1 & 0 & 0 \\ 0 & -1 & 0 \\ -0.0124 & 0 & 1 \end{pmatrix}$$

Transformed indices:

$$(-h-0.0124l, \quad -k, \quad l)$$

Almost complete overlap of main reflections, clear separation of satellites.

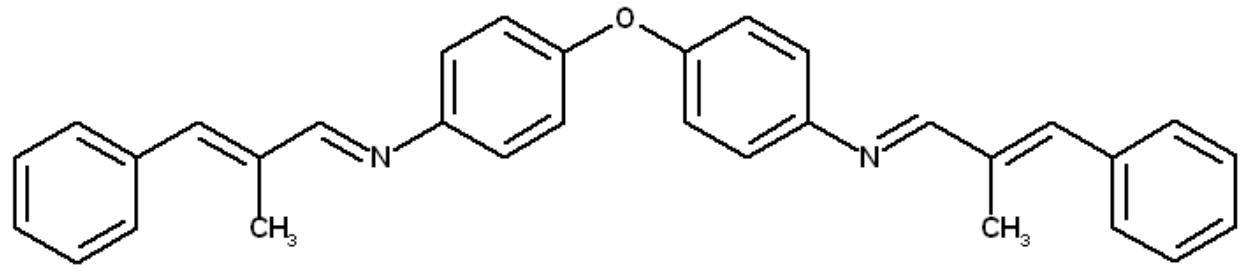


Example 3: AD3

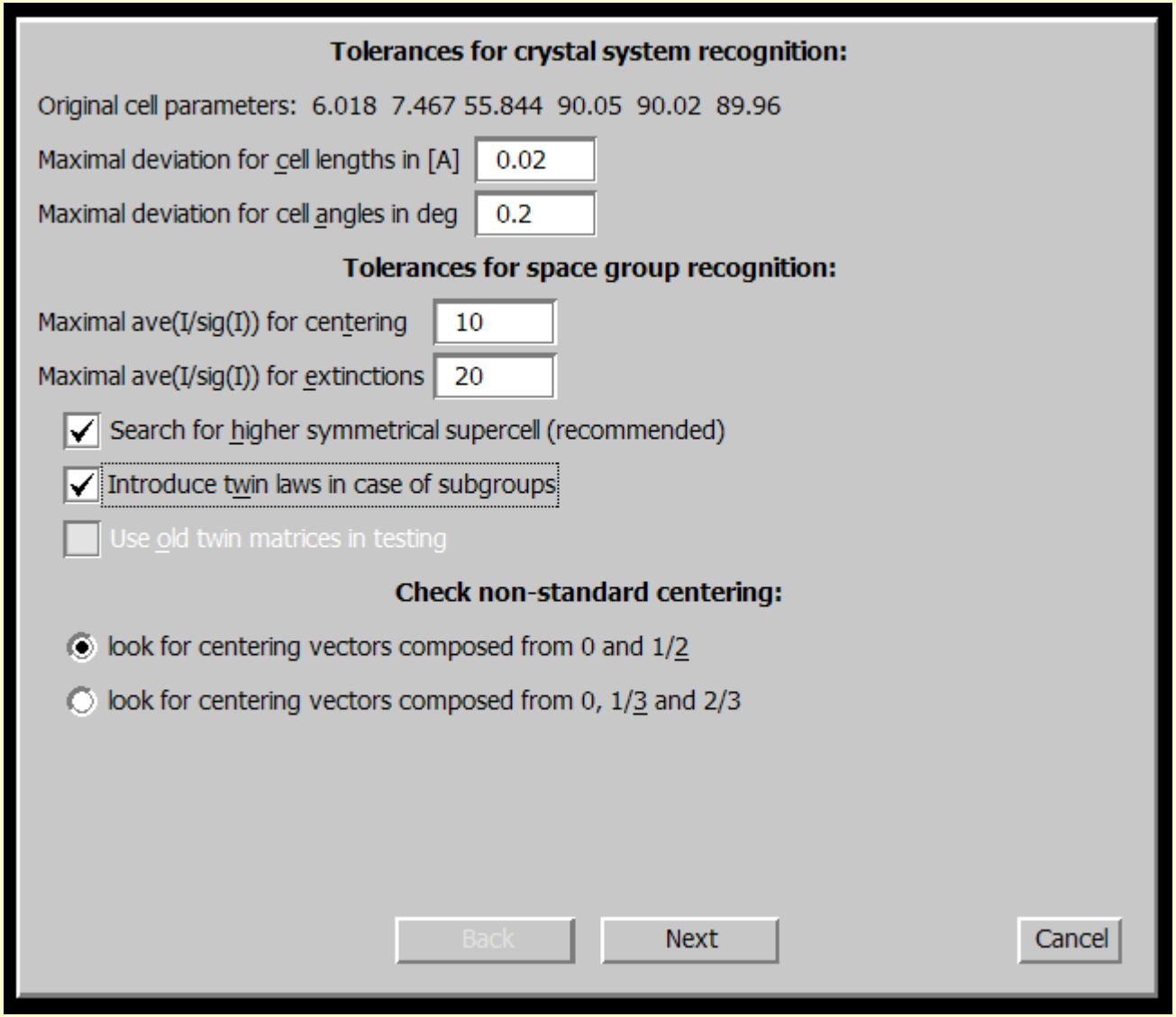
Simple structure with pseudo-merohedric twinning. Finding twinning matrix from group-subgroup transformation. Creating publication CIF.

Bis[N-(2-benzylidenepropylidene)phenyl]ether

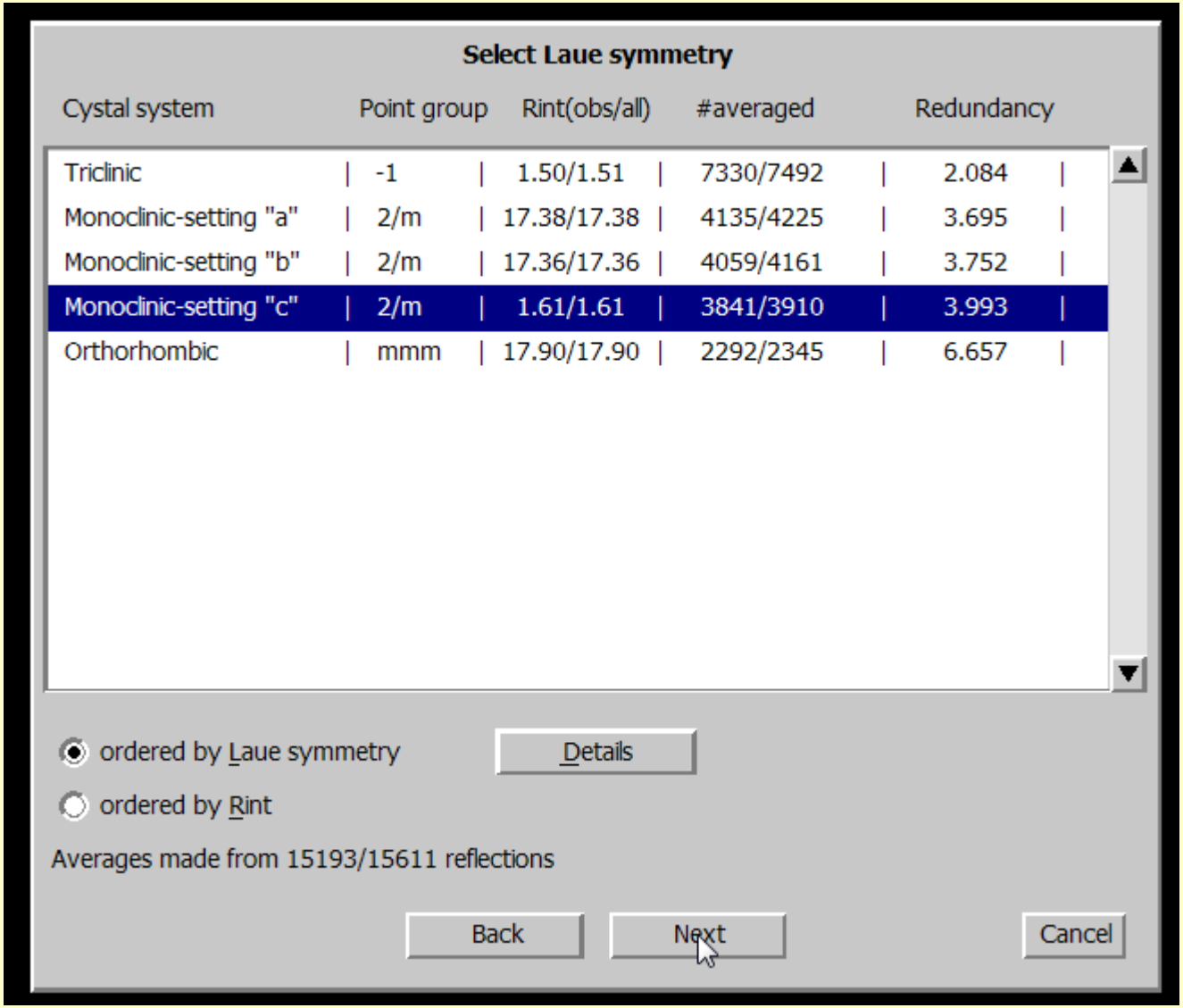
Single crystal data measured with Oxford Diffraction four-circle diffractometer
Input files: AD3.hkl, AD3.sum
Frame scaling, absorption correction: done with software of diffractometer



Test of space group: the option “Introduce twin law” is selected based on the “suspicious” unit cell parameters



Meaning of “Introduce twin law” : if the selected Laue symmetry is lower than the highest possible for the given unit cell, the program will introduce twinning by the lost symmetry elements

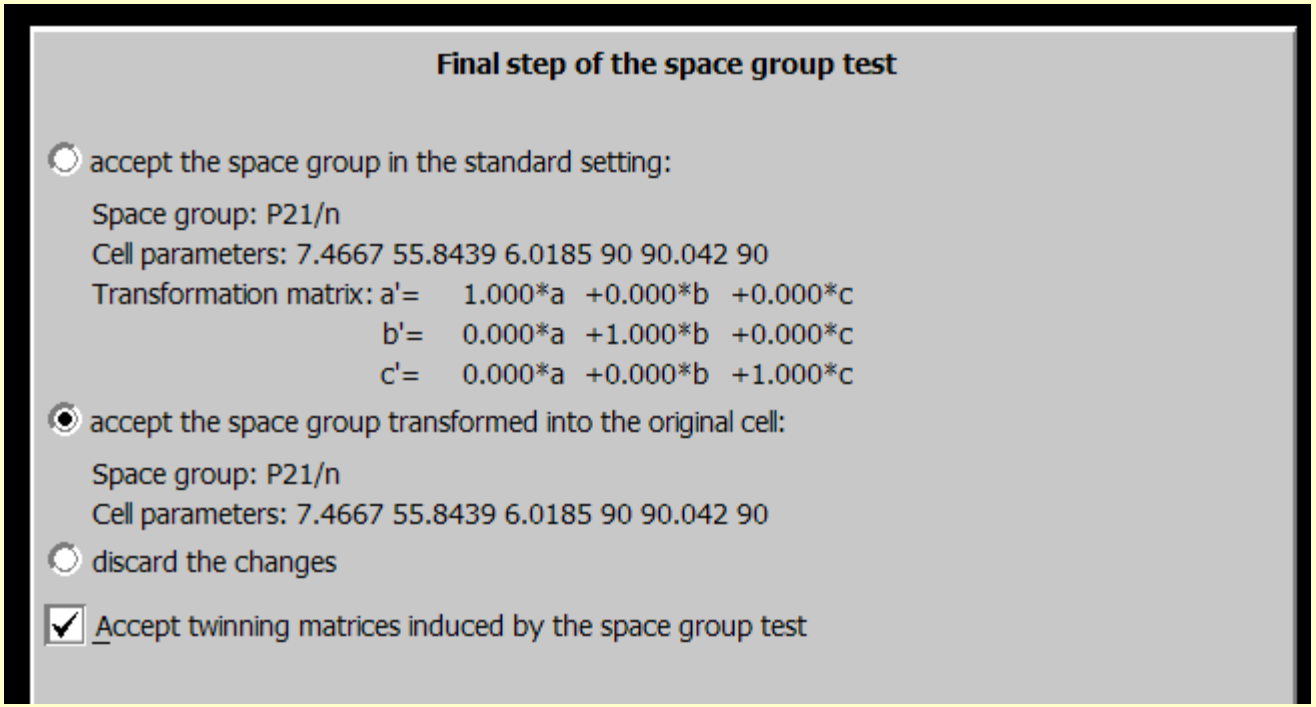


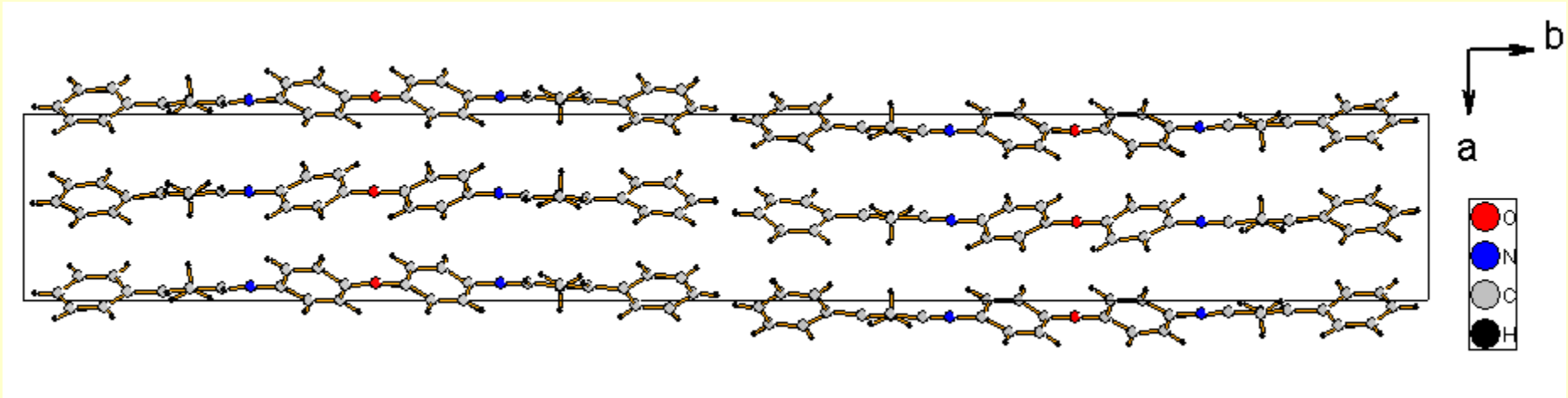
Cell parameters:
7.4728 55.89 6.0235
90 90.0418 90

R_{int} for mmm: 17.9%
R_{int} for 2/m: 1.6%
(setting b)

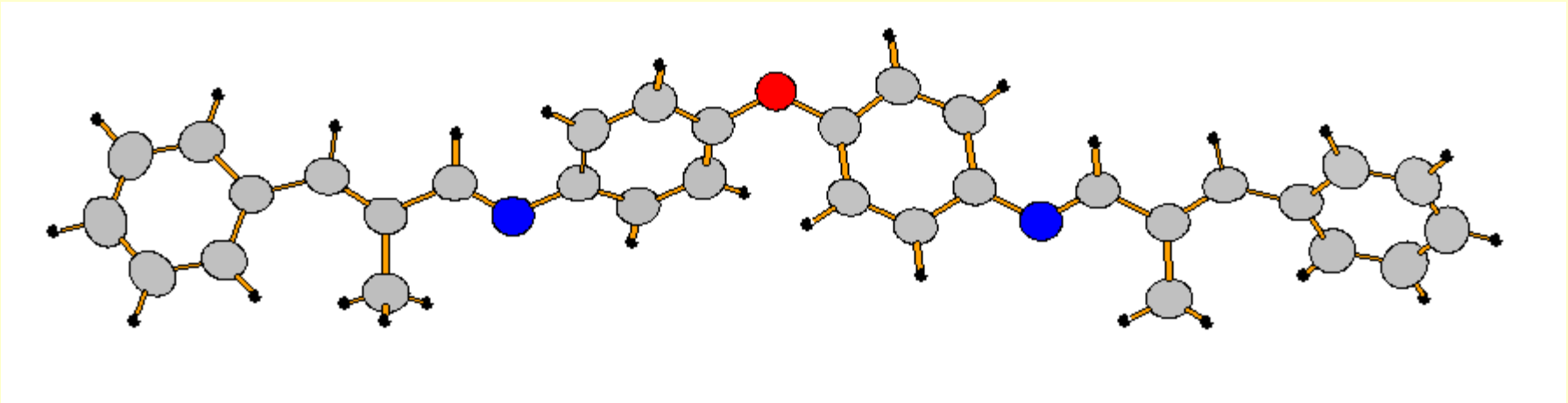
Lost symmetry mmm -> 2/m: x -y -z can be used like twinning operation

Here the twinning is finally accepted





Charge flipping
Correcting chemical types
Anisotropic ADP
Hydrogens

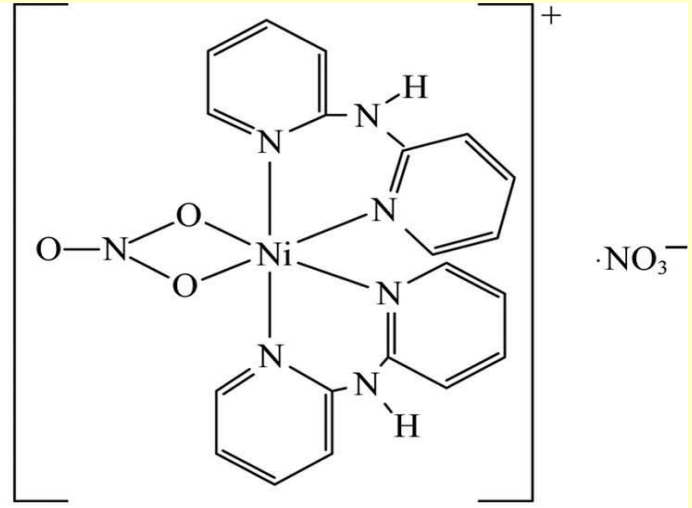


R=20% without twinning, 3.44% with twinning - Refined twin fractions: 0.72, 0.28

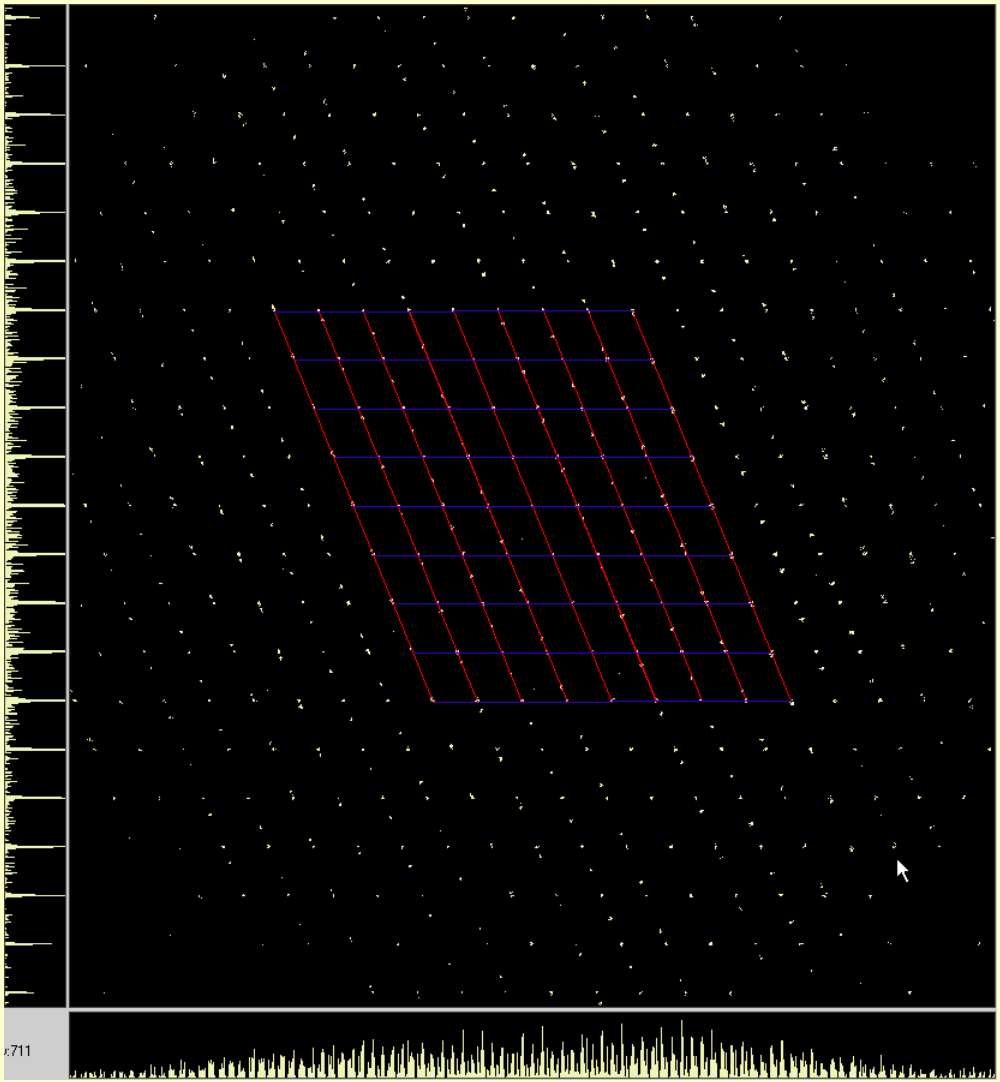
Example 3.2: PyNinit

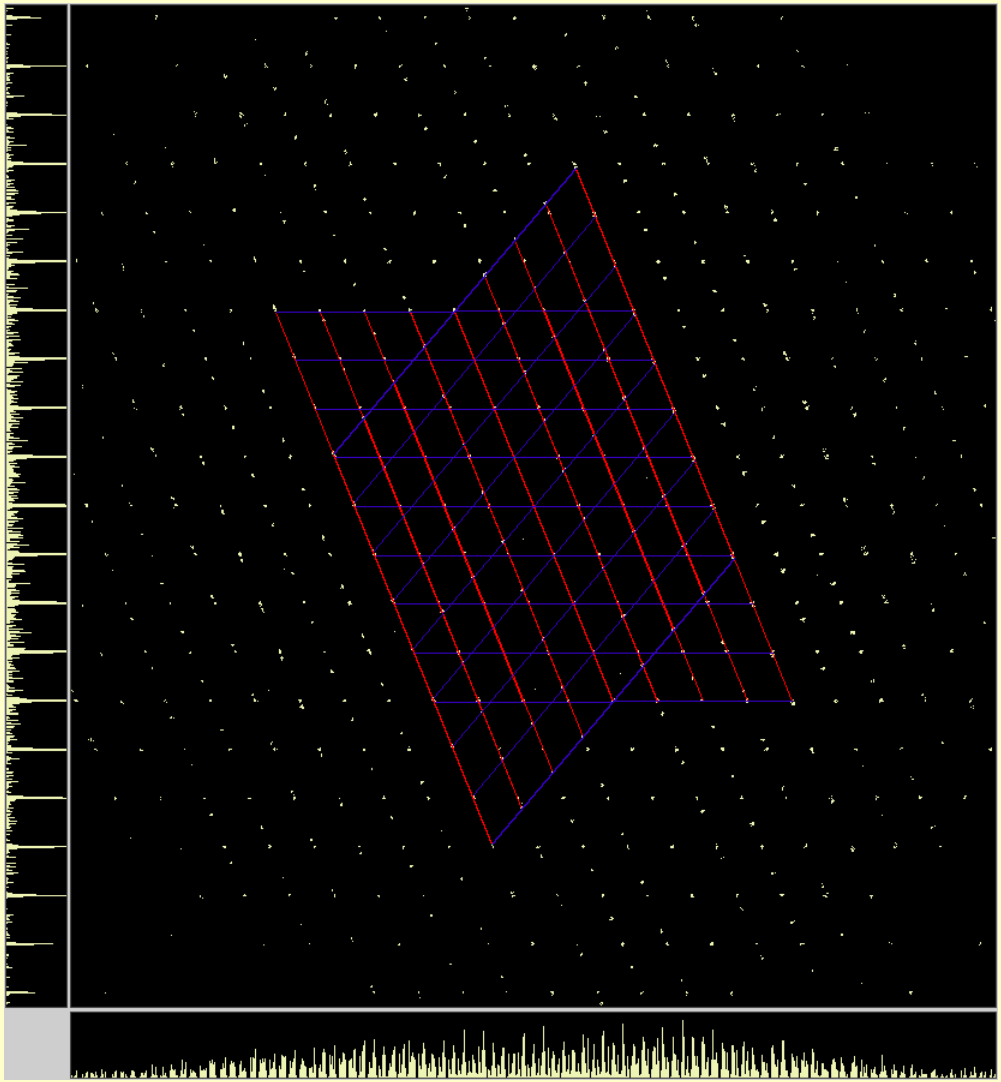
Simple structure with non-merohedric twinning. Handling twin overlaps in Jana2006

Single crystal data measured with Oxford Diffraction four-circle diffractometer
Input files:
1st twin domain: pyNinit_twin1.hkl, pyNinit_twin1.sum
2nd twin domain pyNinit_twin2.hkl, pyNinit_twin2.sum
hklf5: pyNinit_twin1_hklf5.hkl
Twinning matrix: (-1 0 -0.733| 0 -1 0| 0 0 1)
Frame scaling, absorption correction: done with software of diffractometer



J. Černák, M. Dušek, K. Fejfarová, Acta Cryst. (2009). C65, m260-m262.





LATTICE

Current cell

14.5940(15) 9.8526(5) 16.0519(11) 90.005(5) 113.739(8) 90.016(6) 2112.8(3)

Constrained current cell

14.6005(13) 9.8578(7) 16.0454(17) 90.0 113.814(12) 90.0 2112.8(3)

Lattice reduction

selected cell

14.5993 9.8527 16.0514 90.0061 113.7692 90.0239 mP 35

reduced cell

9.8527 14.5993 16.0514 113.7692 90.0061 90.0239 2113.0

Twin information

1: 14.5934 9.8530 16.0502 90.007 113.720 90.008 2112.9

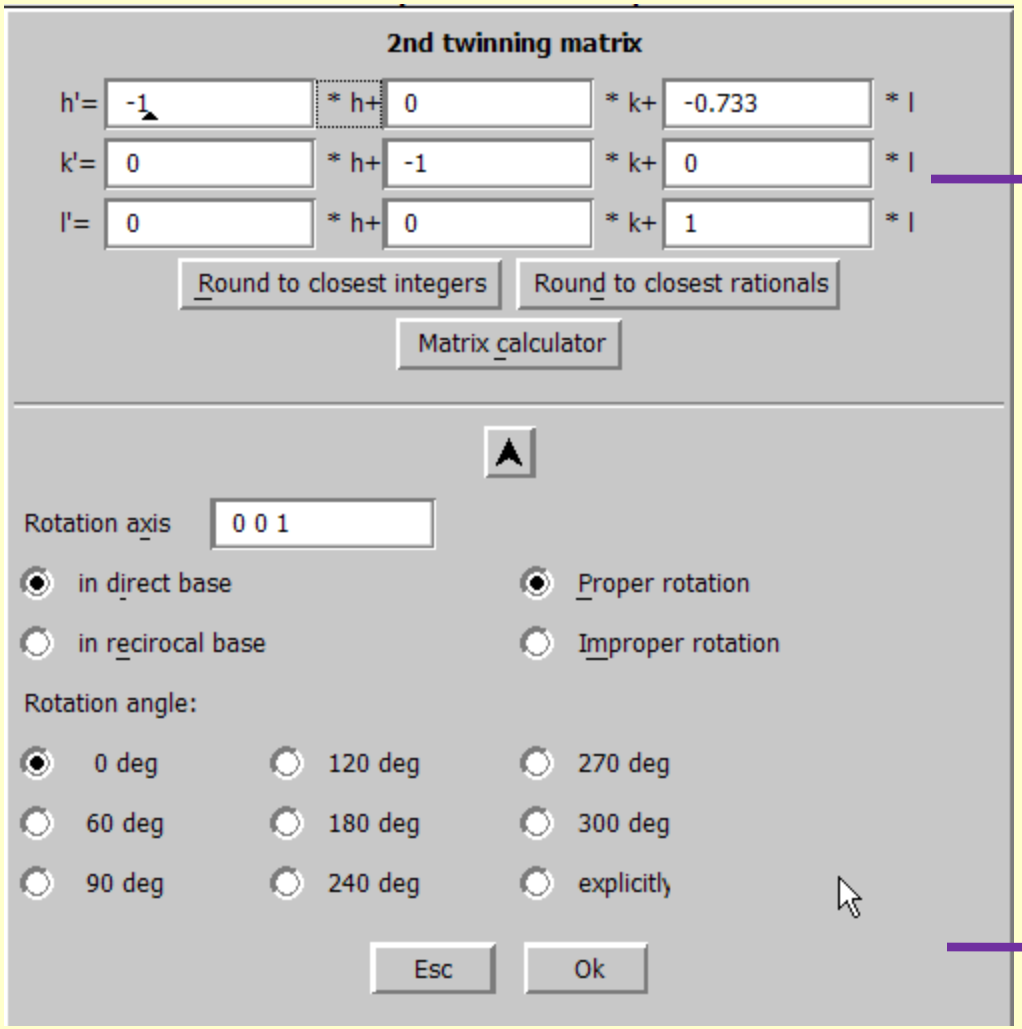
2: 14.6015 9.8542 16.0544 90.002 113.764 90.030 2114.1

1: Total: 4970(61.1%) Separate: 3201(39.3%) Overlapped: 1769(21.7%)

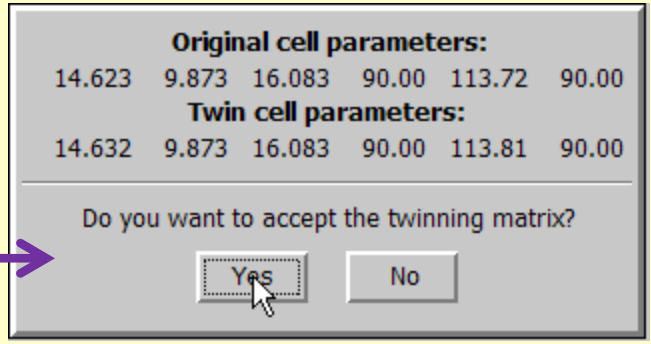
2: Total: 4930(60.6%) Separate: 3161(38.8%) Overlapped: 1769(21.7%)

Unindexed: 9 (0.1%)

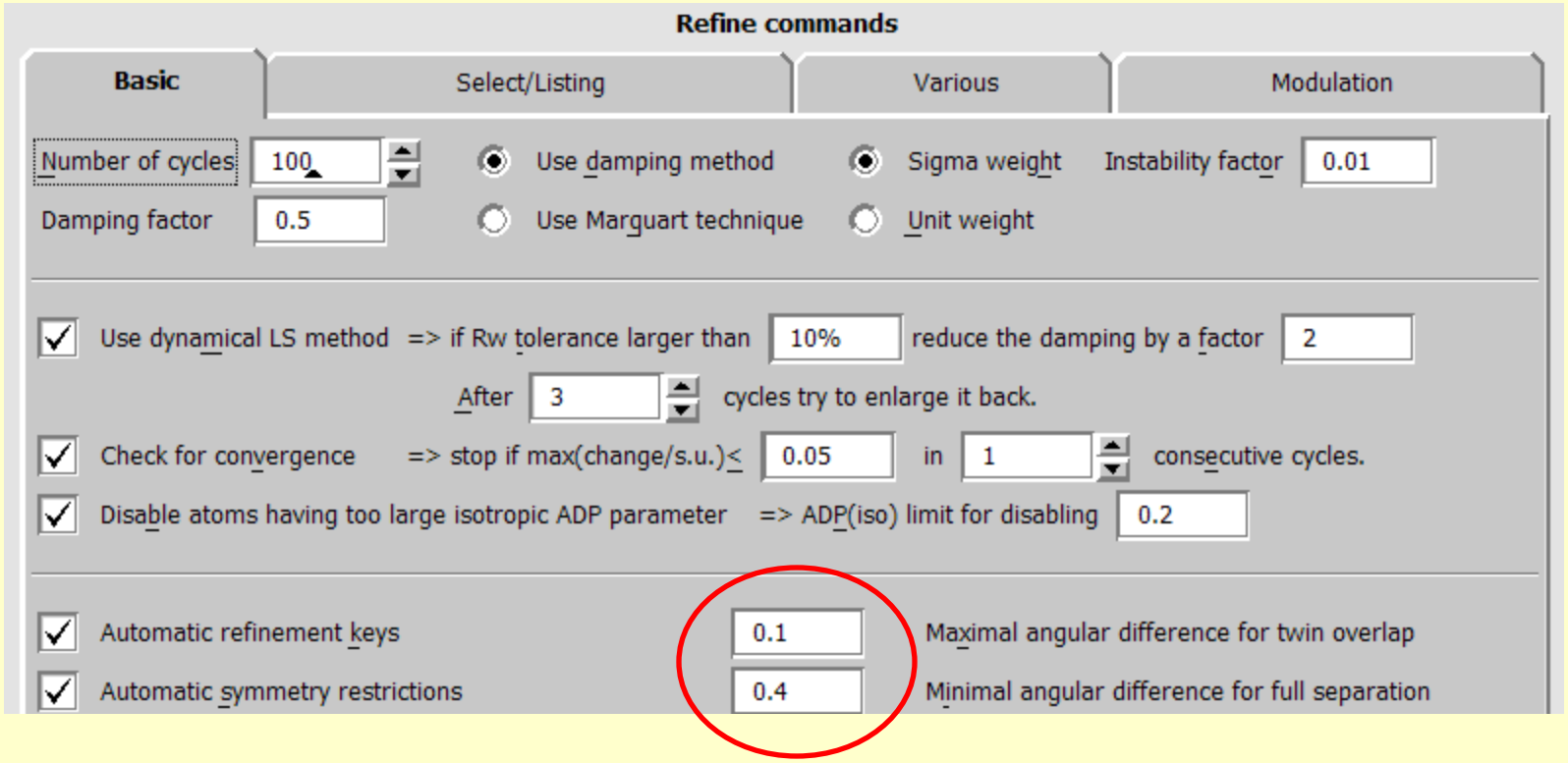
1. Structure solution using 1st twin domain only: R ~ 17%
2. Introduction of twinning: R ~ 13%



$$\begin{pmatrix} h' \\ k' \\ l' \end{pmatrix} = \begin{pmatrix} -1 & 0 & -0.733 \\ 0 & -1 & 0 \\ 0 & 0 & 1 \end{pmatrix} \begin{pmatrix} h \\ k \\ l \end{pmatrix}$$



3. Discarding partially overlapped reflections: R ~ 4%



R factors : [2993=1921+1072/317], Damping factor: 0.5000
GOF(obs)= 1.36 GOF(all)= 1.30
Number of reflections excluded due to refinement options: 0+1898
R(obs)= 3.83 Rw(obs)= 3.11 R(all)= 8.63 Rw(all)= 3.84
Last Rw(all): 3.84
Maximum change/s.u. : -0.0190 for U23[N7]

Where to stop?
We want to discard as little reflections as possible

L1	L2	R_{obs}	GOF	discarded
0	0	15.2	8.2	0
0.1	0	12.7	4.7	0
0.2	0	7.4	3.6	0
0.3	0	7.1	3.5	0
0.4	0	9.8	5.5	0
0.35	0	9.6	5.4	0
0.31	0	9.3	5.3	0
0.3	0.4	6.9	3.6	778
0.3	0.5	7.4	3.8	1264
0.3	0.6	7.9	4.1	1916
0.2	0.3	6.2	3.0	415
0.2	0.4	5.7	3.0	1193
0.15	0.3	5.3	2.4	709
0.1	0.3	4.8	1.9	1120
0.1	0.4	3.9	1.4	1848

4. Using data of the second domain
5. Testing scale of domains

6. Using HKLF5 file: R ~ 5.5% without discarding reflections

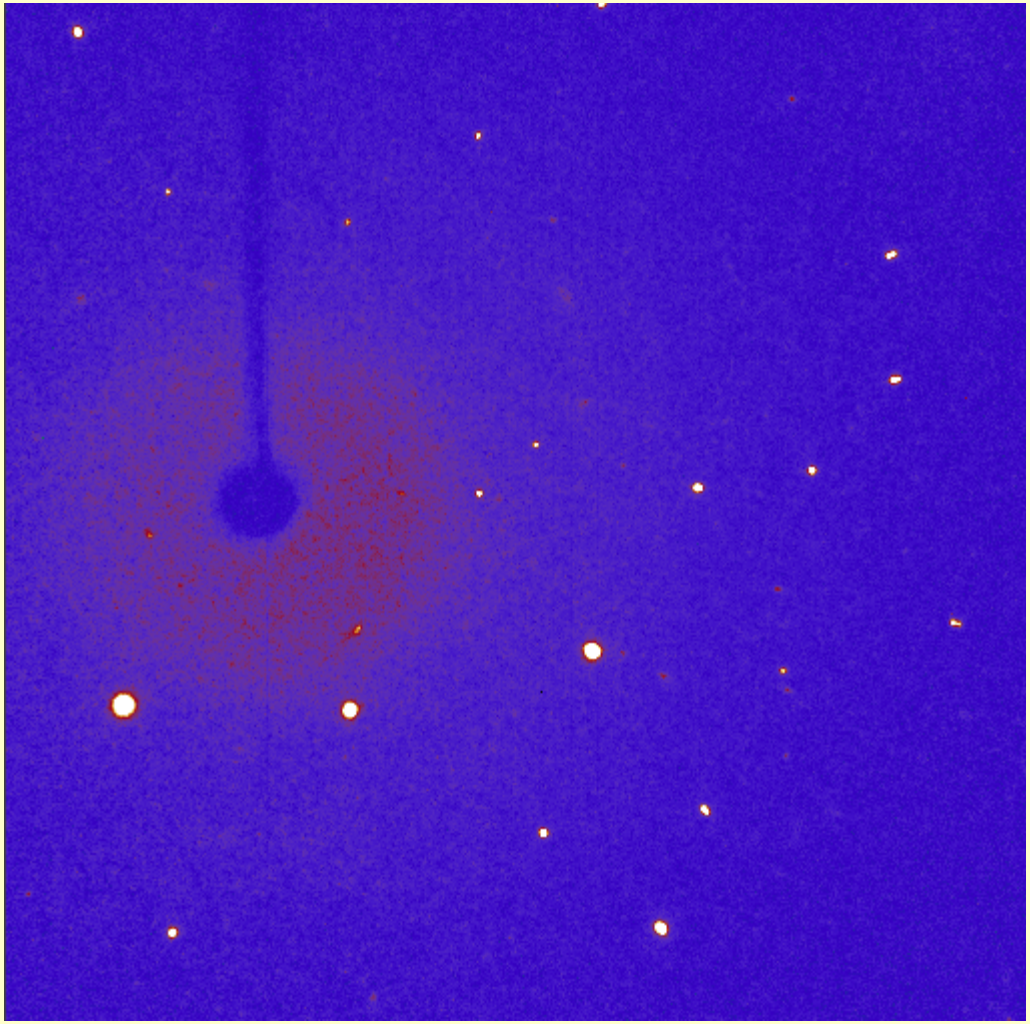
The diffractometer software should know more about overlaps than Jana2006

```
  -4  -7 -12 132.825 55.5172  -2
  13   7 -12 132.825 55.5172   1
  -5  -7 -12 19.9661 56.6859  -2
  14   7 -12 19.9661 56.6859   1
  -6  -7 -12 24.2907 52.1818  -2
  15   7 -12 24.2907 52.1818   1
   0  -4 -11 421.765 31.9614  -2
   8   4 -11 421.765 31.9614   1
  -1  -4 -11 42.9233 24.2092  -2
   9   4 -11 42.9233 24.2092   1
  -2  -4 -11 322.224 34.3002  -2
  10   4 -11 322.224 34.3002   1
  -3  -4 -11 48.0177 31.7279  -2
  11   4 -11 48.0177 31.7279   1
  -4  -4 -11 471.881 52.0069  -2
  12   4 -11 471.881 52.0069   1
  -5  -4 -11 383.447 52.9024  -2
  13   4 -11 383.447 52.9024   1
  -6  -4 -11 348.428 62.3163  -2
  14   4 -11 348.428 62.3163   1
  -7  -4 -11 162.334 70.3329  -2
  15   4 -11 162.334 70.3329   1
  -8  -4 -11 289.451 54.9913  -2
  16   4 -11 289.451 54.9913   1
  -9  -4 -11 81.6937 39.9522  -2
  17   4 -11 81.6937 39.9522   1
```

Example 3.3: $CsLiSO_4$

Simple structure with pseudo-merohedric 3-fold twinning. Finding twinning matrix from group->subgroup transformation. Transformation to four times larger reciprocal cell.

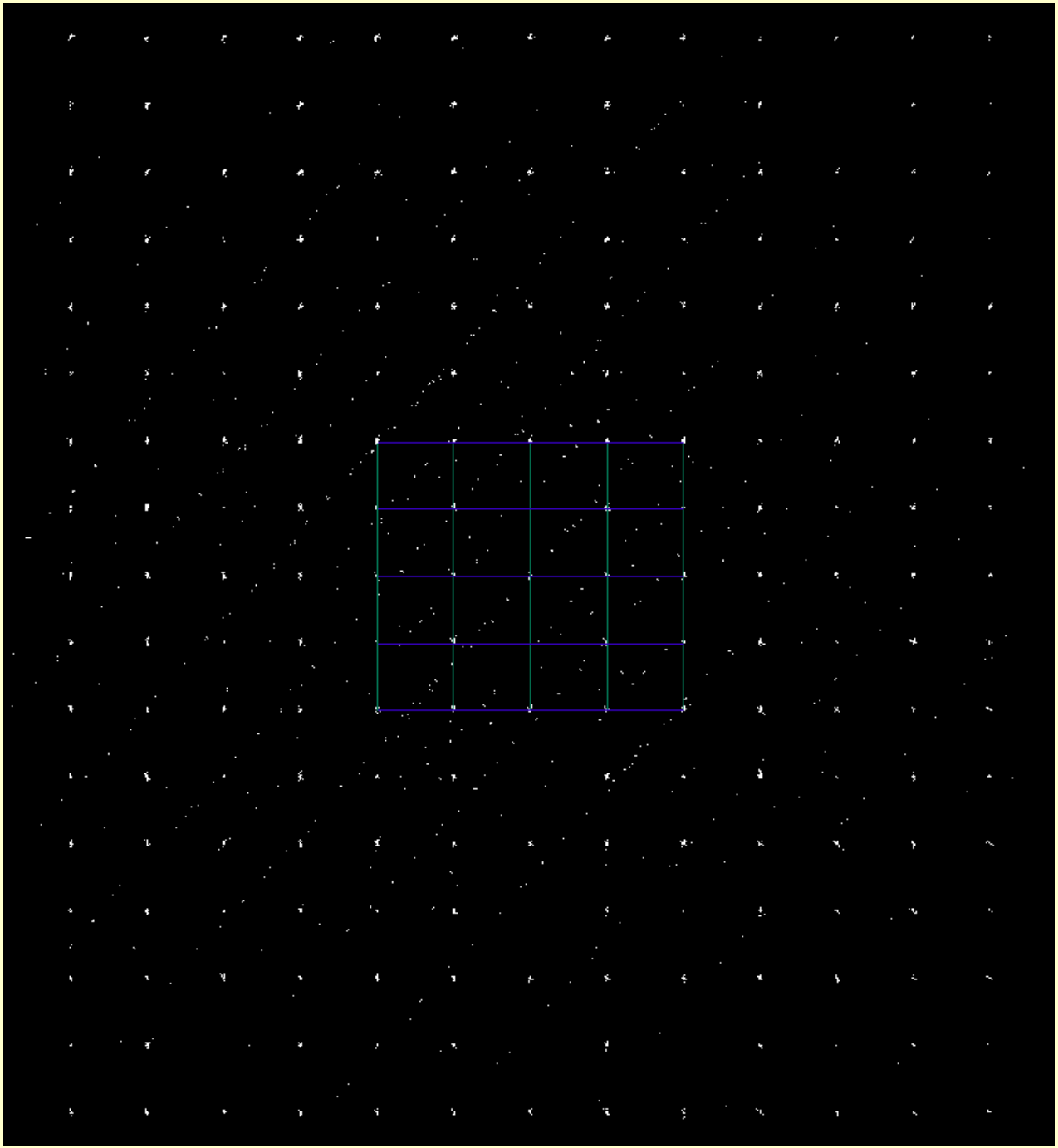
Single crystal data measured with Oxford Diffraction four-circle diffractometer
Input files: CsLiSO4.hkl, CsLiSO4.sum
Frame scaling, absorption correction: done with software of diffractometer



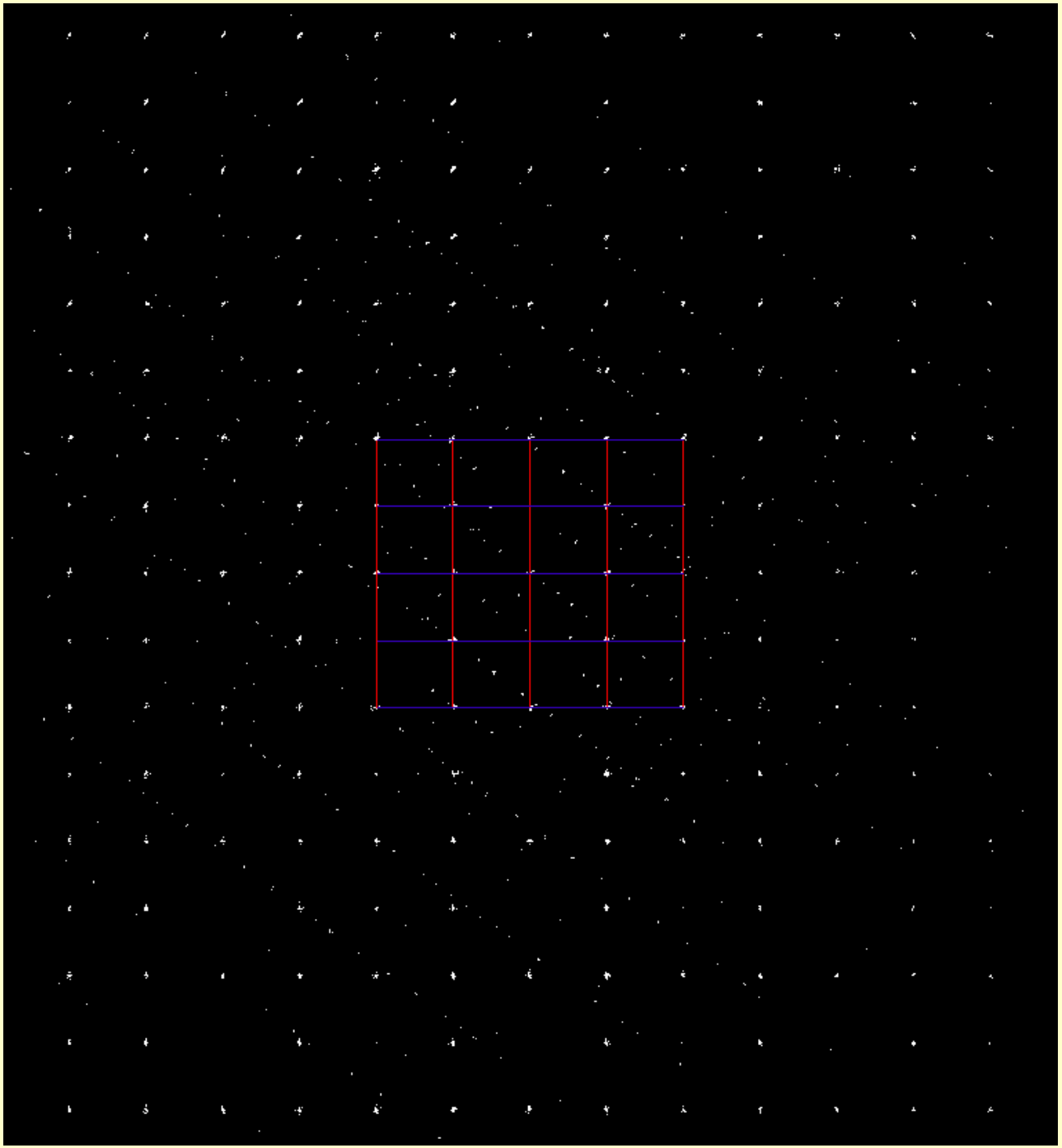
Good data

No split reflections

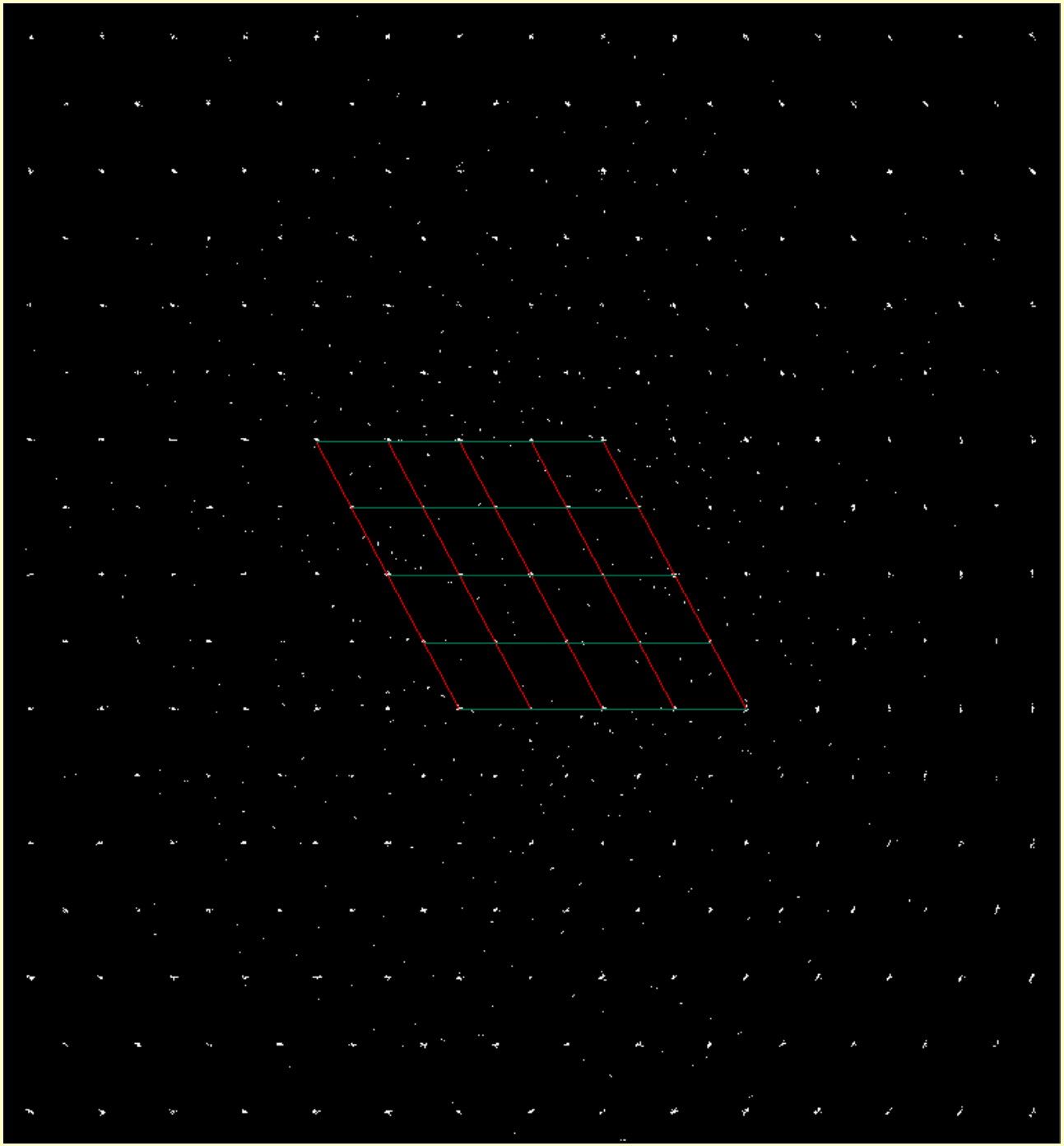
No overlaps



Peaks projected along a*



Peaks projected along b*

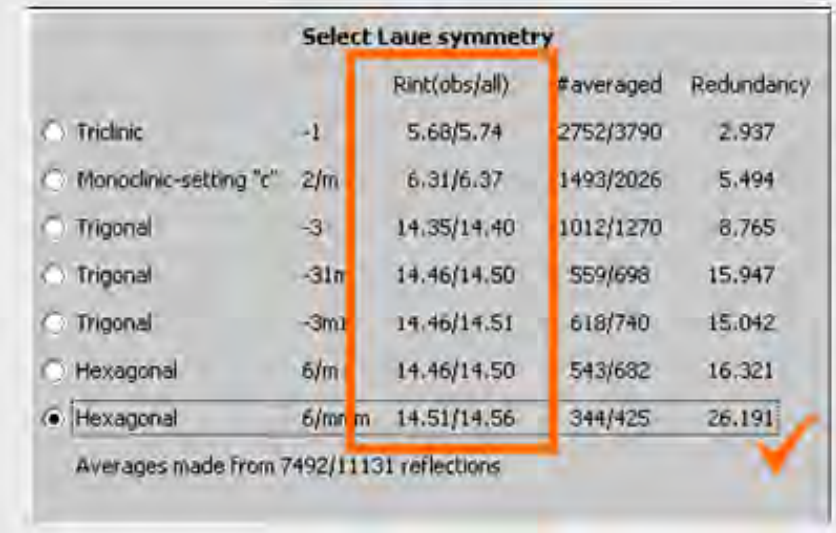


Peaks projected along c*

hexagonal unit cell

a = 10.89451(0.00058)
b = 10.88937(0.00058)
c = 8.80485(0.00041)
α = 90.00104(0.00407)
β = 90.02358(0.00406)
γ = 119.94547(0.00557)
V = 905.11

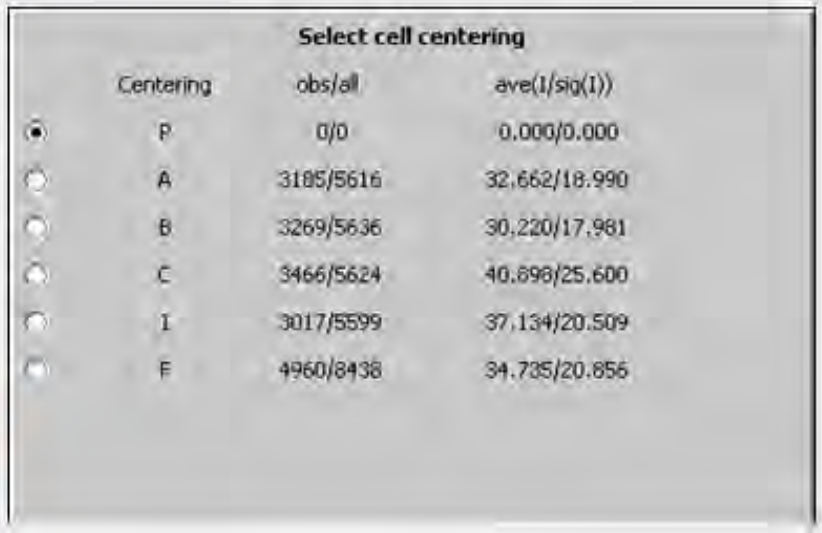
Symmetry wizard



Select Laue symmetry

		Rint(obs/all)	#averaged	Redundancy
Triclinic	-1	5.68/5.74	2752/3790	2.937
Monoclinic-setting "c"	2/m	6.31/6.37	1493/2026	5.494
Trigonal	-3	14.35/14.40	1012/1270	8.765
Trigonal	-31m	14.46/14.50	559/698	15.947
Trigonal	-3m1	14.46/14.51	618/740	15.042
Hexagonal	6/m	14.46/14.50	543/682	16.321
Hexagonal	6/mmm	14.51/14.56	344/425	26.191

Averages made from 7492/11131 reflections

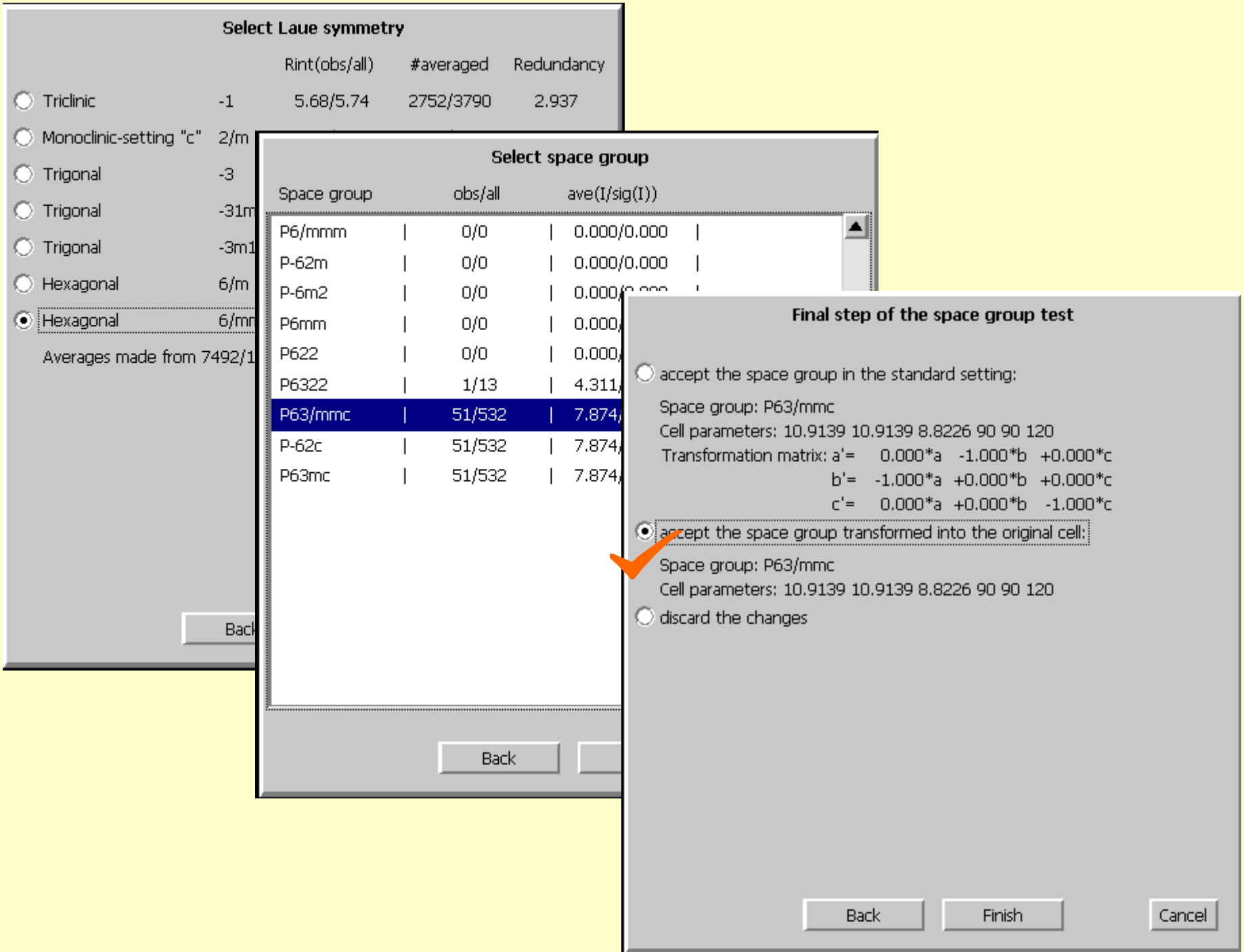


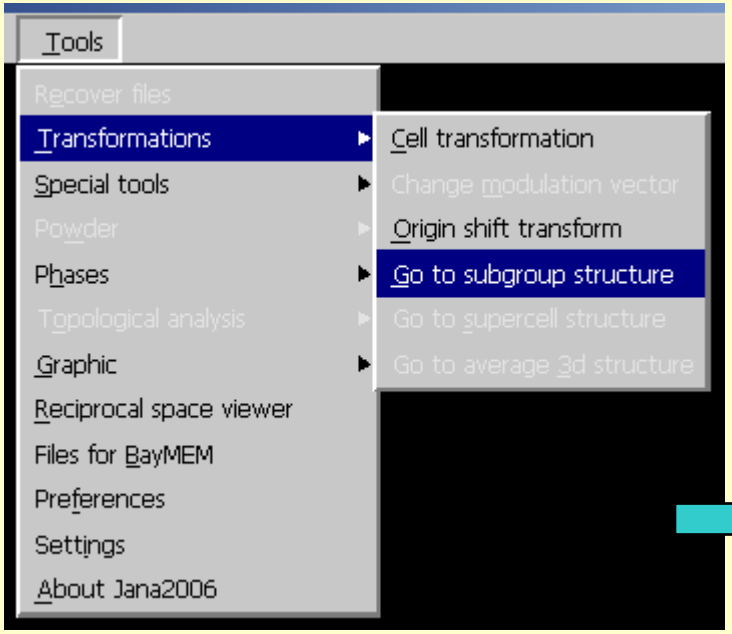
Select cell centering

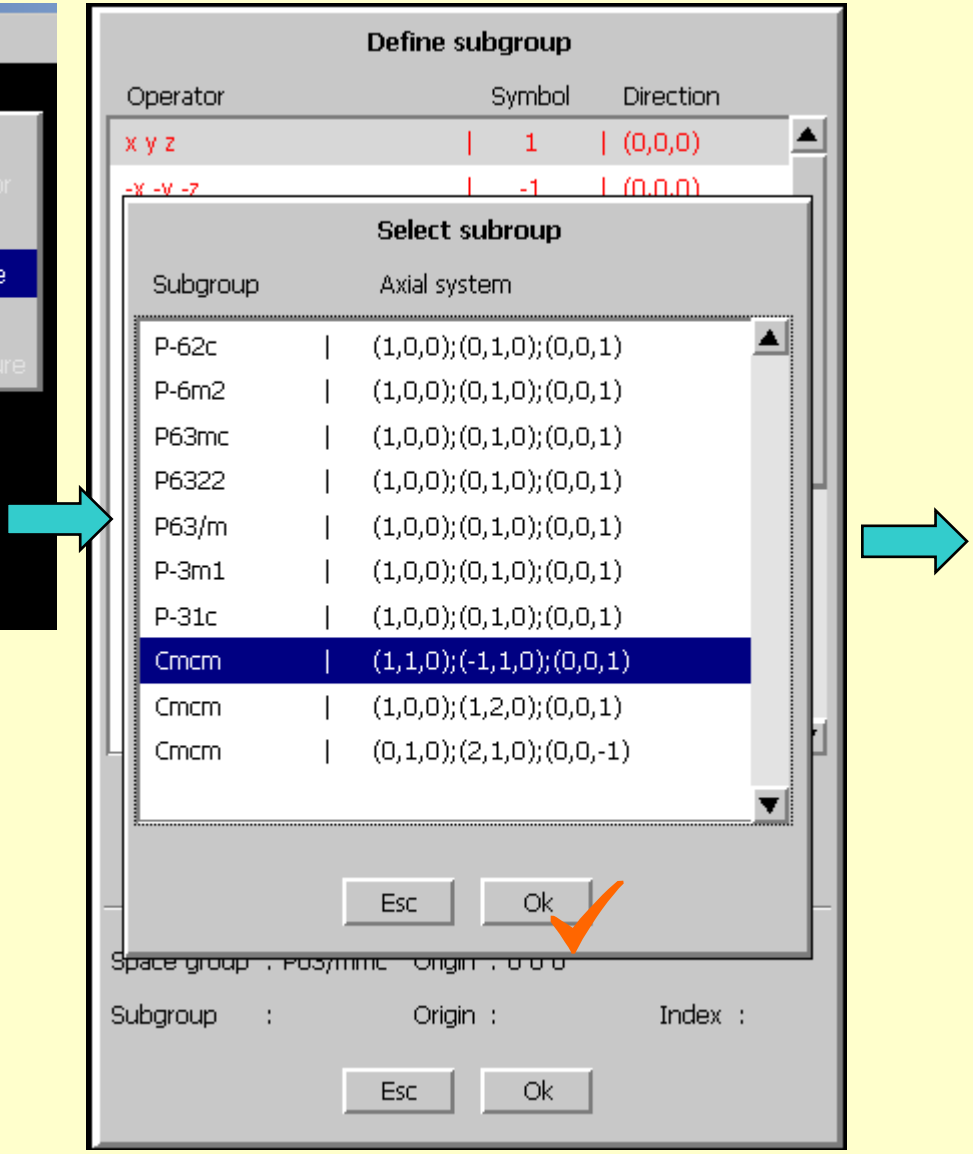
Centering	obs/all	ave(I/sig(I))
P	0/0	0.000/0.000
A	3185/5616	32.662/18.990
B	3269/5636	30.220/17.981
C	3466/5624	40.898/25.600
I	3017/5599	37.134/20.509
F	4960/8438	34.735/20.856

From symmetry wizard this is evident that hexagonal symmetry is violated. Because cell parameters are exactly hexagonal merohedric twinning is highly probable. In order to get twinning matrices easily we shall use the highest hexagonal symmetry and use group-subgroup transformation tool.

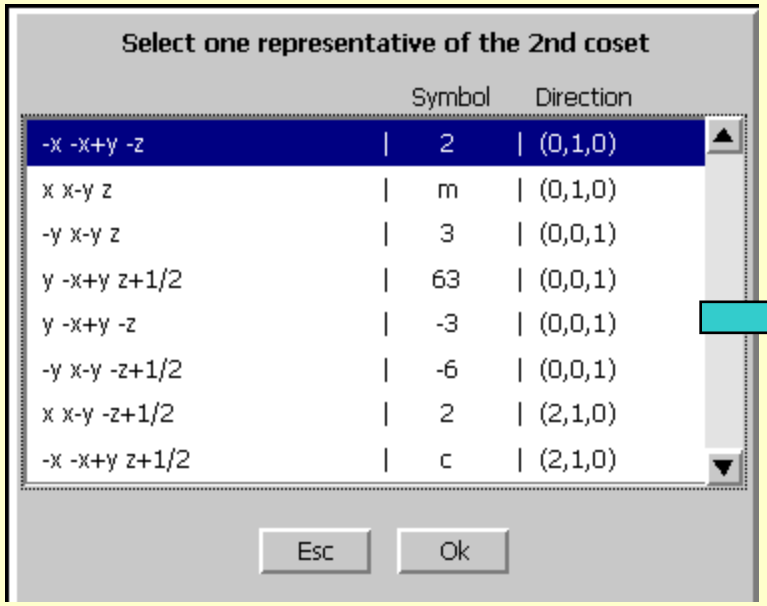
The symmetry wizard does not list orthorhombic possibilities because they are in contradiction with cell parameters. Orthorhombic cells will be available in group-subgroup transformation.

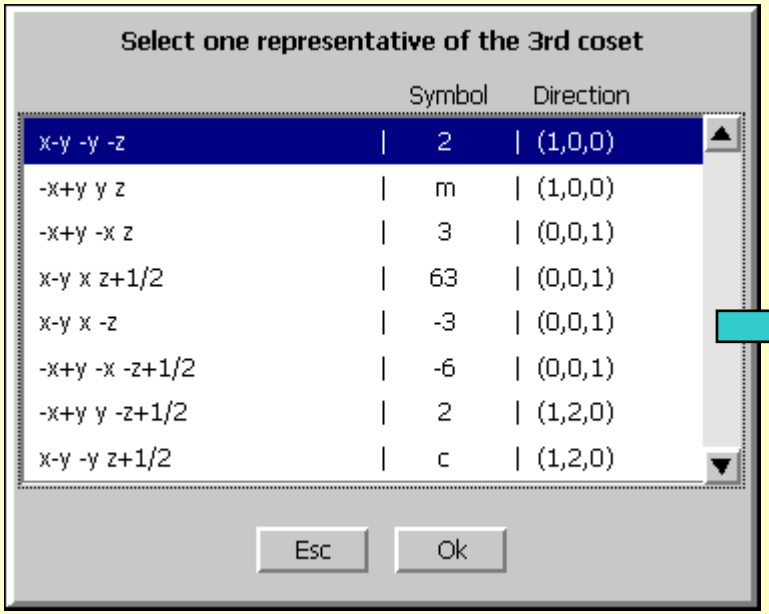

Select Laue symmetry
Rint(obs/all)
#averaged
Redundancy
Triclinic -1 5.68/5.74 2752/3790 2.937
Monoclinic-setting "c" 2/m
Trigonal -3
Trigonal -31m
Trigonal -3m1
Hexagonal 6/m
Hexagonal 6/mm
Averages made from 7492/1
Back
Select space group
Space group
obs/all
ave(I/sig(I))
P6/mmm | 0/0 | 0.000/0.000
P-62m | 0/0 | 0.000/0.000
P-6m2 | 0/0 | 0.000/
P6mm | 0/0 | 0.000/
P622 | 0/0 | 0.000/
P6322 | 1/13 | 4.311/
P63/mmc | 51/532 | 7.874/
P-62c | 51/532 | 7.874/
P63mc | 51/532 | 7.874/
Back
Final step of the space group test
accept the space group in the standard setting:
Space group: P63/mmc
Cell parameters: 10.9139 10.9139 8.8226 90 90 120
Transformation matrix: a'= 0.000*a -1.000*b +0.000*c
b'= -1.000*a +0.000*b +0.000*c
c'= 0.000*a +0.000*b -1.000*c
accept the space group transformed into the original cell:
Space group: P63/mmc
Cell parameters: 10.9139 10.9139 8.8226 90 90 120
discard the changes
Back
Finish
Cancel

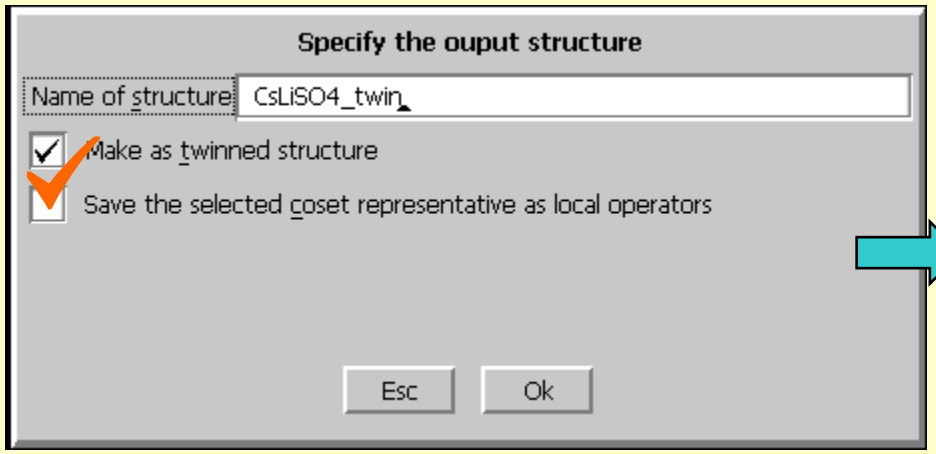


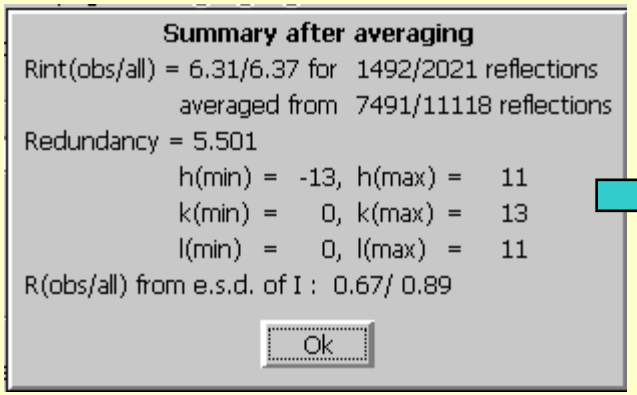


Transformation to orthorhombic cell

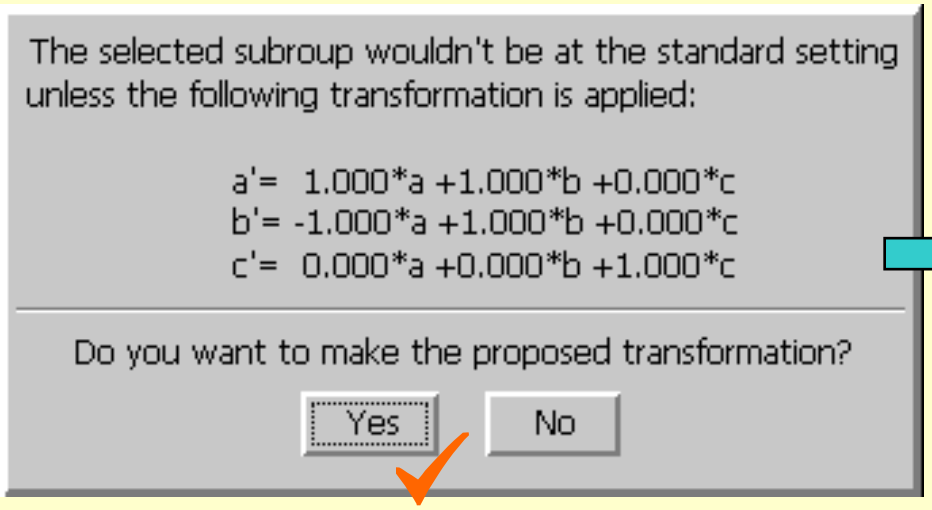


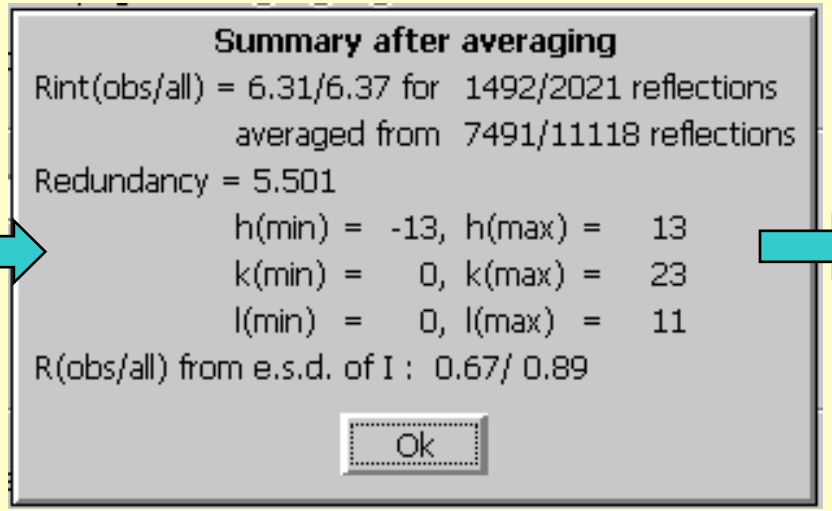


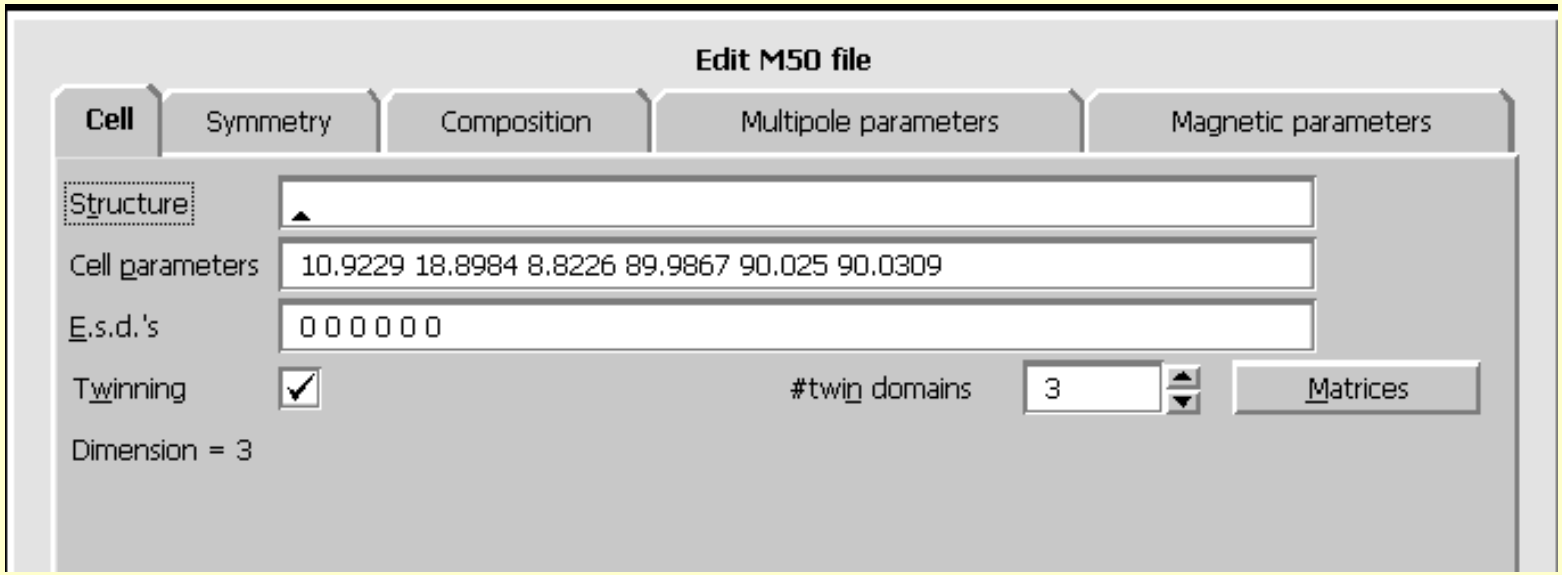




The discarded symmetry operations will be used as twinning operations. They are two -> we have created three-fold twin.







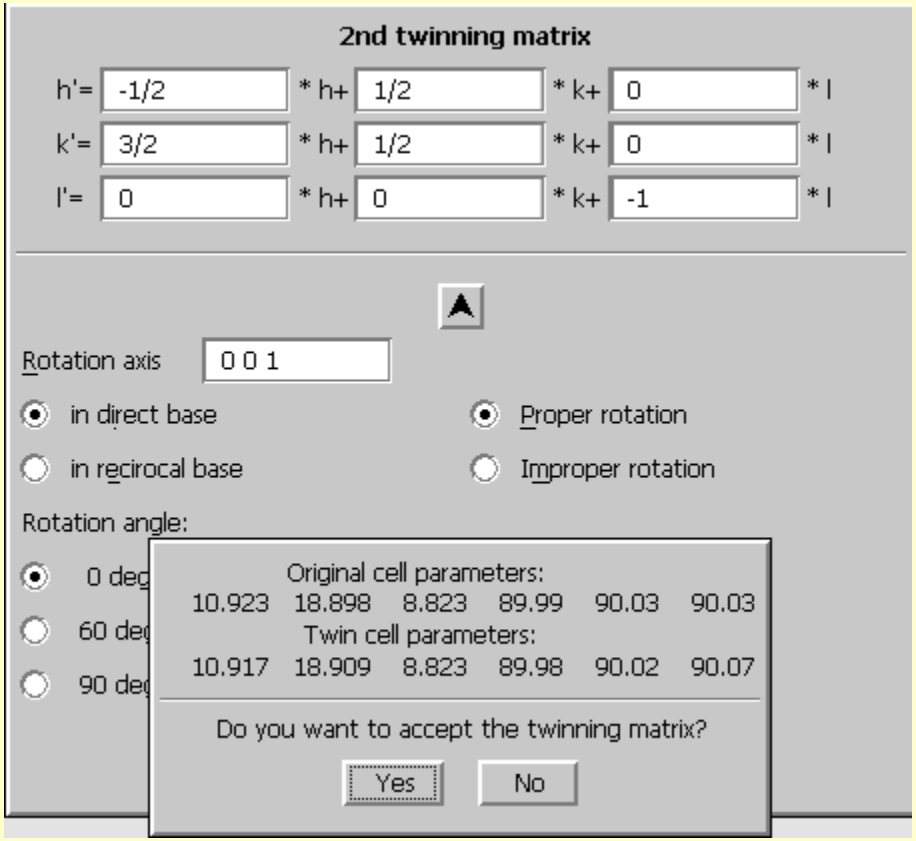
Finally a transformation is offered to standard symmetry Cmcm. We can check new cell parameters in EditM50.

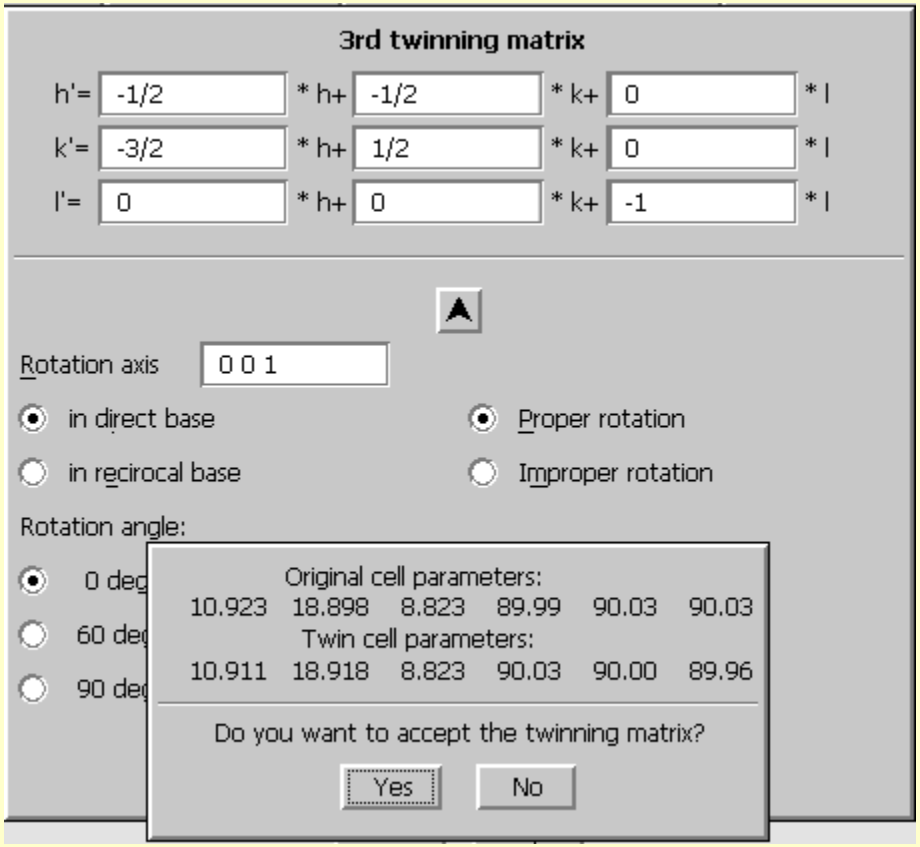
```
commcell 10.9165 10.9114 8.8226 90.001 90.024 119.946
trcell
     1.0000    -1.0000     0.0000
     1.0000     1.0000     0.0000
     0.0000     0.0000     1.0000
refblock Block1
sourcefile CsLiSO4.hkl
filedate 23/01/2008 filetime 10:46:40
difcode 5 correspond 1 corrlp -1 corrabs 0
lambda 0.71073 radtype 1 polarization 1 datcolltemp 293
monangle 6.0821 perfmono 0.5
nref 11131 ndim95 3 nlines 22262 twread 1 scmax 1 scale 1
cell 10.9165 10.9114 8.8226 90.001 90.024 119.946
ormat
    0.071904   -0.022060    0.042037
   -0.045981   -0.092164   -0.055632
    0.062385    0.046966   -0.089360
end
Data Block1
     1 -13   3   0   0.00   0.00  26.30  26.30   0.111946E+03   0.838333E+03     1.000   1   1
   0.100000E+01   0.100000E+01  0.0000  0.5782  0.7640  0.6042 -0.2860  0.5019  0.6038  0.0000
```

Header of M95 now contains transformation from hexagonal to new orthorhombic elementary cell. It is applied when making M90 before merging of reflections.

```
datblock Block1
nref 2021 obslim 3 norefitems 1 diffscales 1 dataave 1 scalelim 10
indslowest 3 indfastest 1 addcentrsymm 0 sigimethod 1 multave 1
flimprint 5 flimcull -1
lambda 0.71073 radtype 1 dattype 1 lpfactor 1 monangle 6.082 perfmono 0.5
datcolltemp 293
end
Data Block1
   2    0    0    166.8      3.4    1    0    1  0.0000
   4    0    0 115151.2    357.3    1    0    1  0.0000
   6    0    0     13.8      9.2    1    0    1  0.0000
   8    0    0  56835.4    139.6    1    0    1  0.0000
  10    0    0     -8.5     16.5    1    0    1  0.0000
  12    0    0  10219.7     72.2    1    0    1  0.0000
 -13    1    0    -20.7     30.6    1    0    1  0.0000
 -11    1    0      9.2     17.3    1    0    1  0.0000
  -9    1    0     -3.9     34.2    1    0    1  0.0000
  -7    1    0      9.1     10.9    1    0    1  0.0000
  -5    1    0      8.5      7.2    1    0    1  0.0000
```

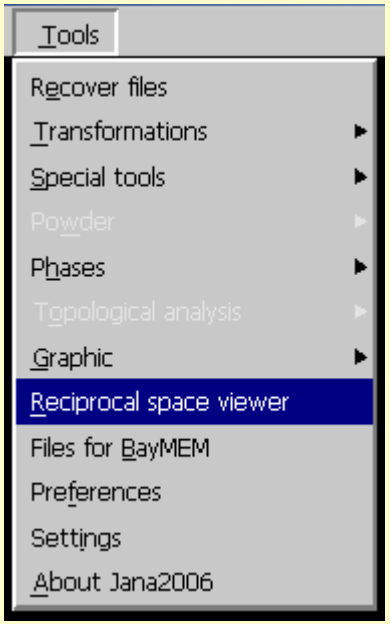
Flags in M90 show that all reflections are indexed in the first domain. This is correct because the twin is merohedric.

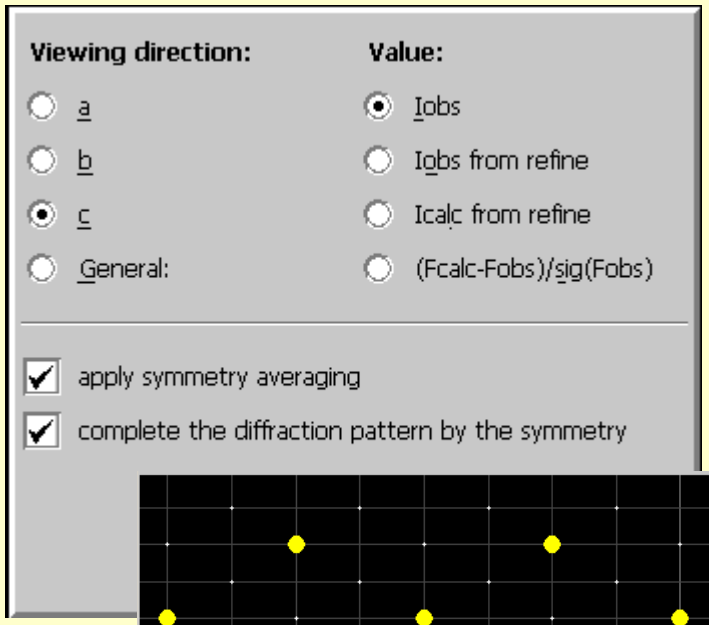


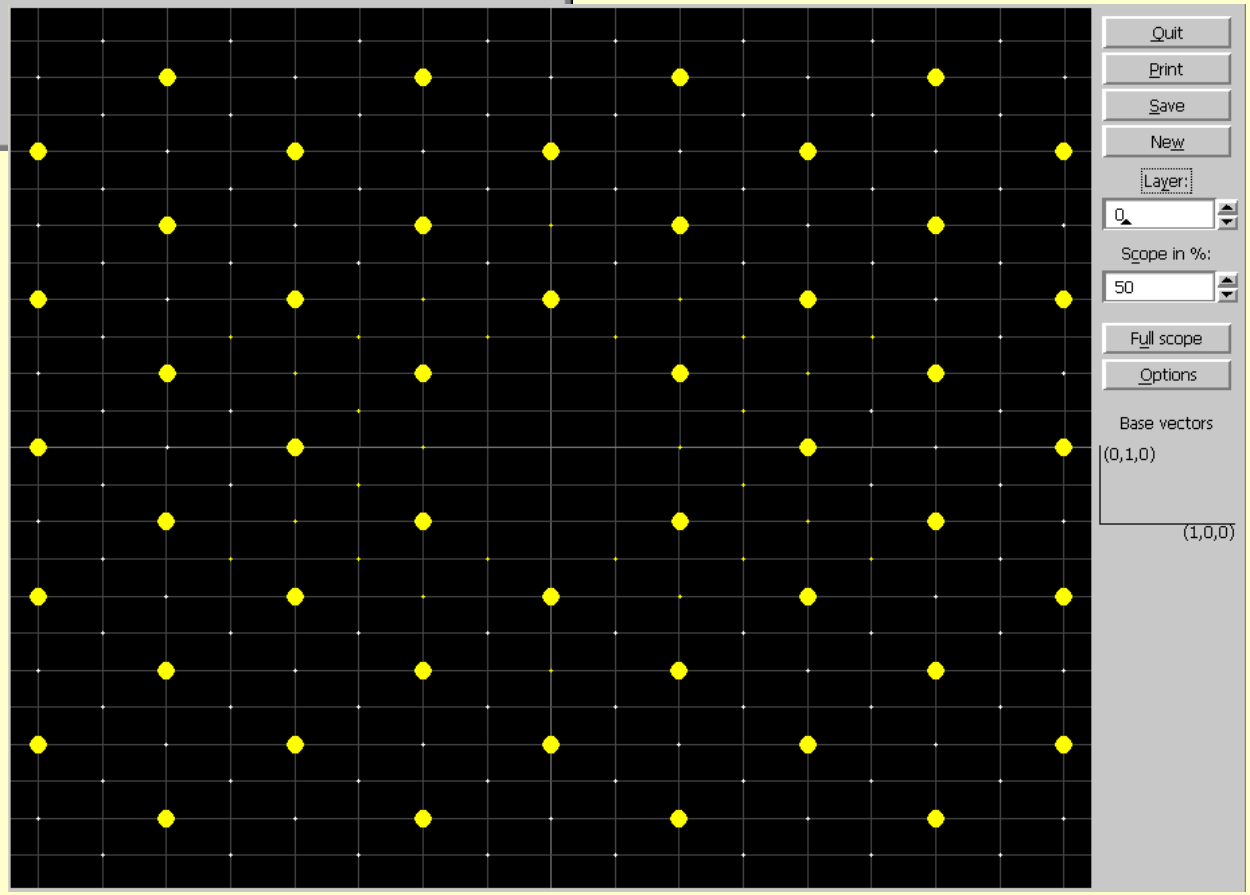


Twinning matrices

Attempts to solve structure fail!!

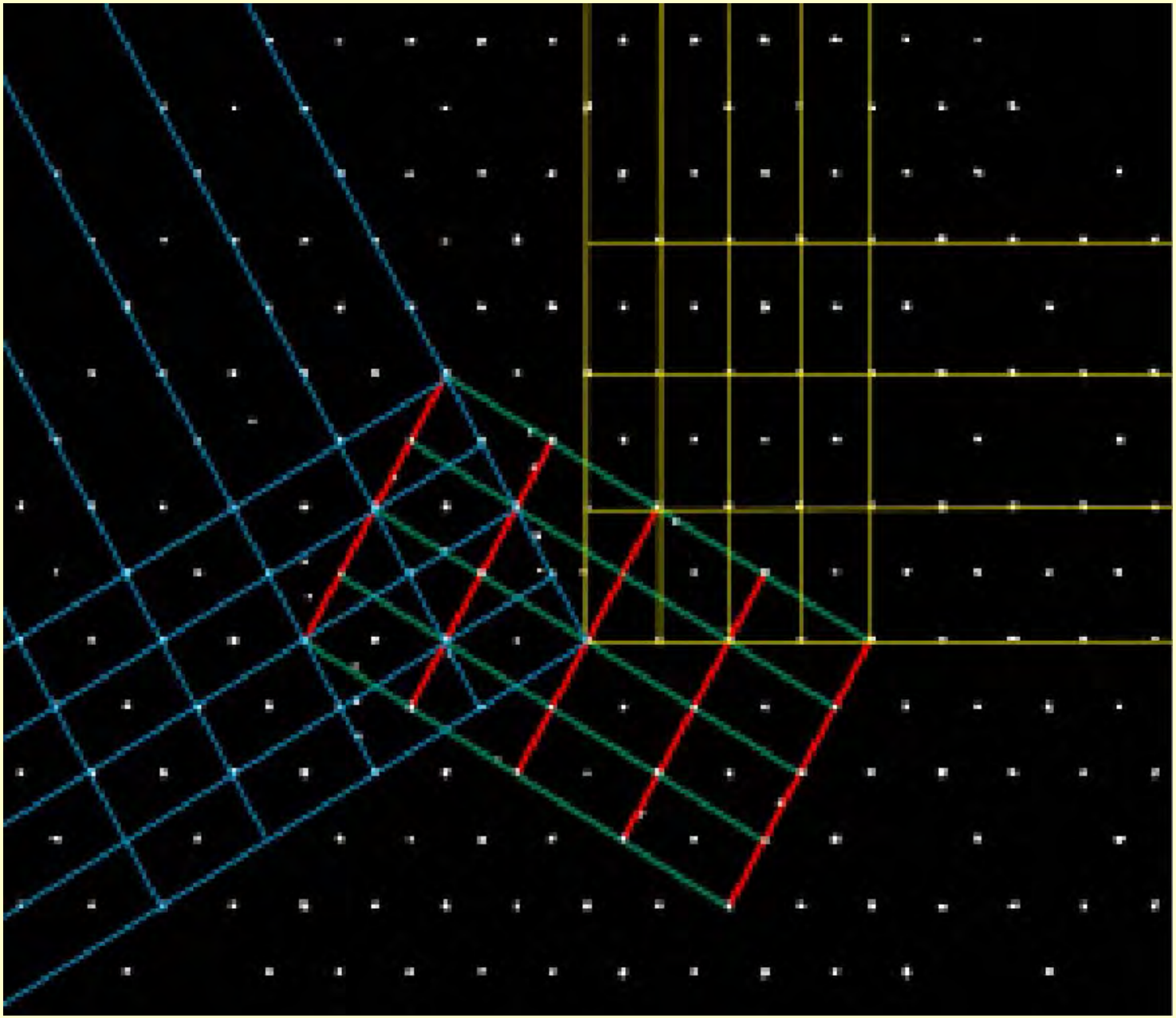




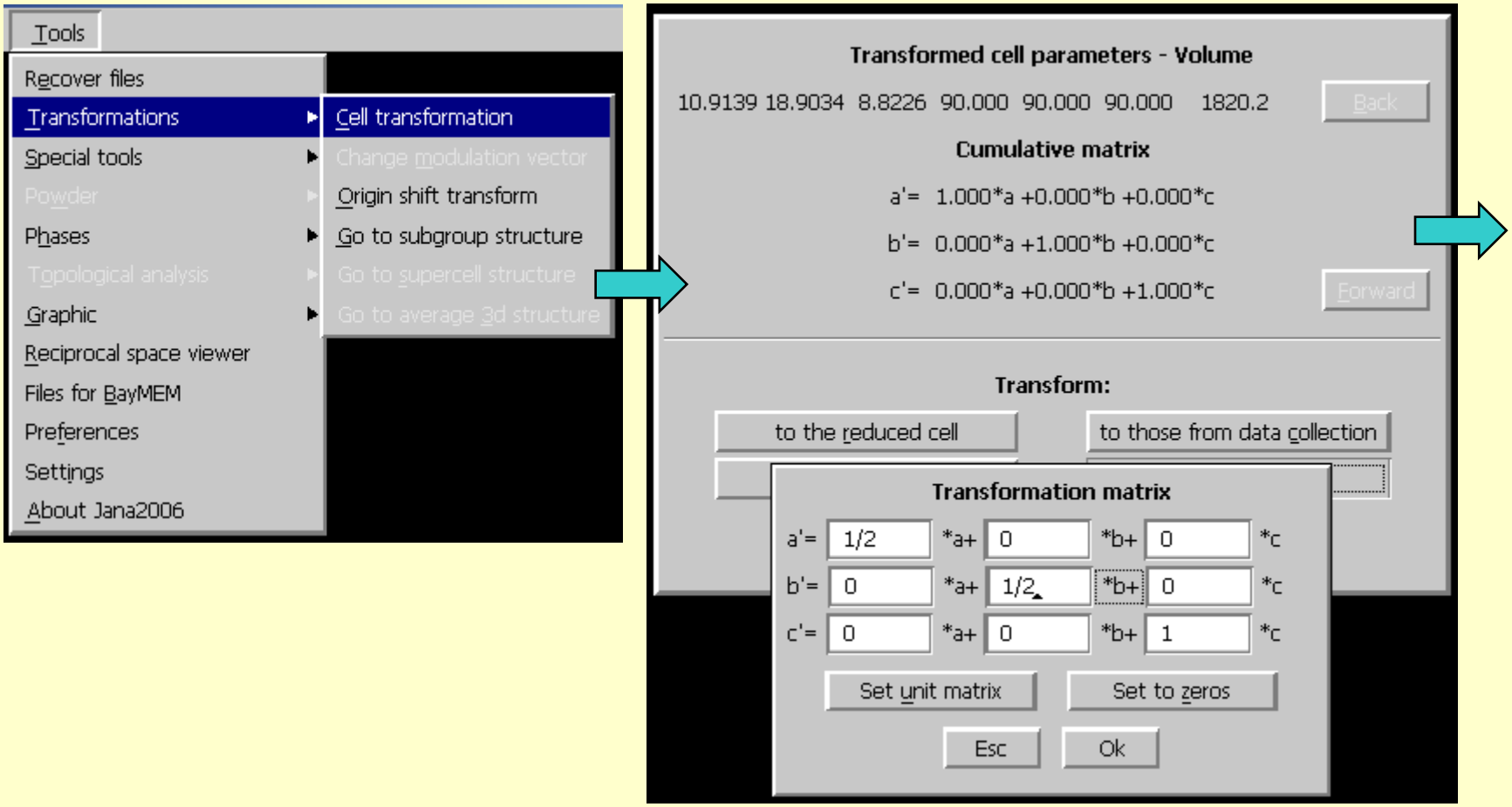


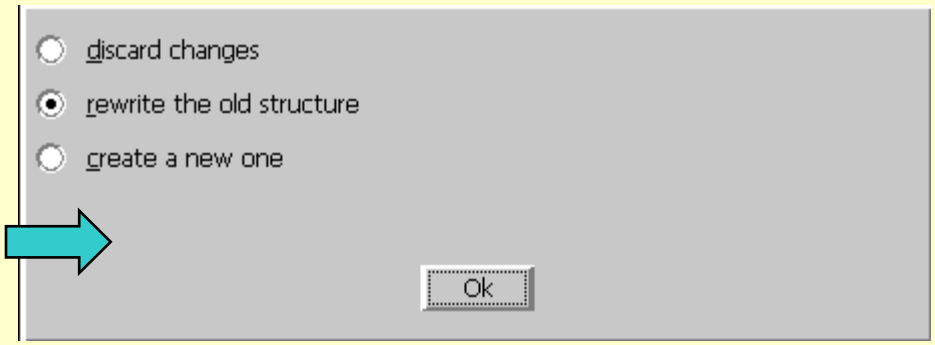
This will start Jana2006 to show Reciprocal space viewer.

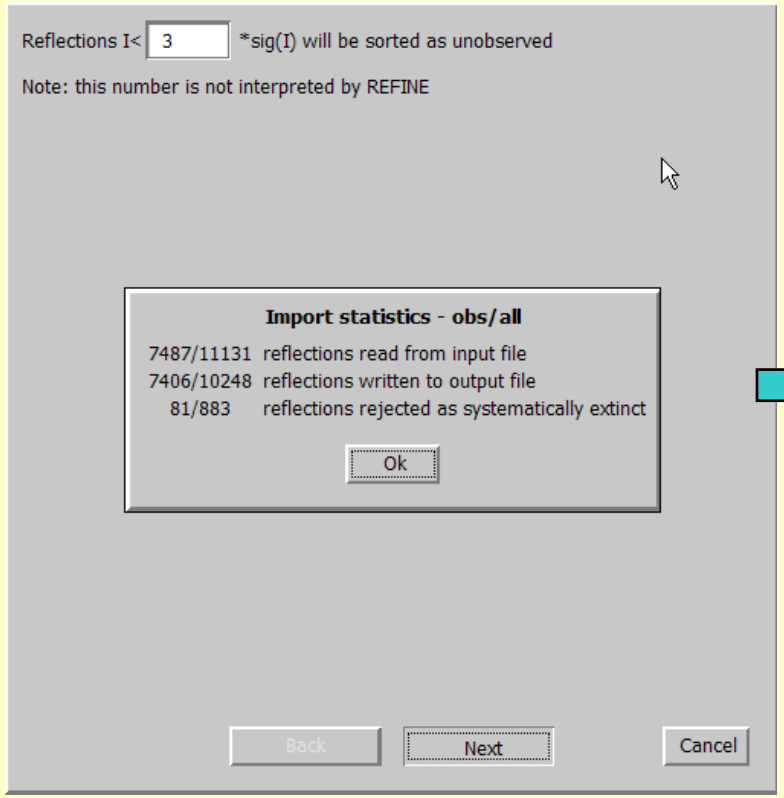
Can we use four times larger reciprocal cell + three-fold twinning?



Transformation







Summary of systematic extinctions

n(all) : 883, n(obs) : 81

Average(I/Sig(I)) : 1.12

List of the strongest absent reflections:

h	k	l	I	sig(I)	I/sig(I)
-1	1	0	20.3	0.7	29.0
0	1	0	17.9	0.8	23.5
0	-1	0	18.9	0.8	22.8
0	1	0	17.2	0.8	21.0
0	-1	0	16.8	0.8	20.8
0	1	0	15.5	0.8	19.9
0	1	0	16.8	0.9	19.3
-1	1	0	9.7	0.5	19.0
0	-1	0	16.7	0.9	18.0
-3	-1	0	14.4	0.8	18.0
0	-1	0	17.1	1.0	17.1
0	-1	0	15.9	0.9	16.8
0	1	0	15.6	0.9	16.8
0	1	0	15.6	0.9	16.4

Ok

Summary after averaging

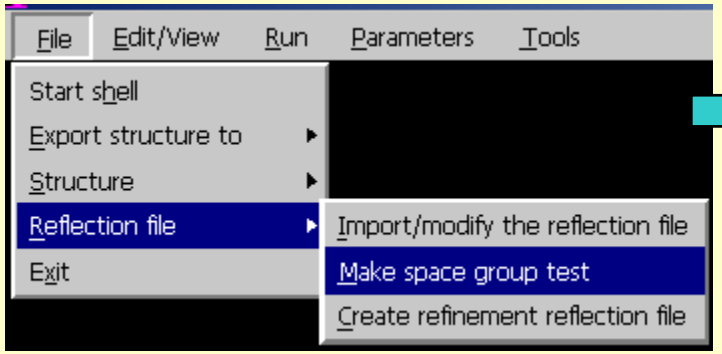
Rint(obs/all) = 6.39/6.43 for 1057/1235 reflections
averaged from 7406/10248 reflections

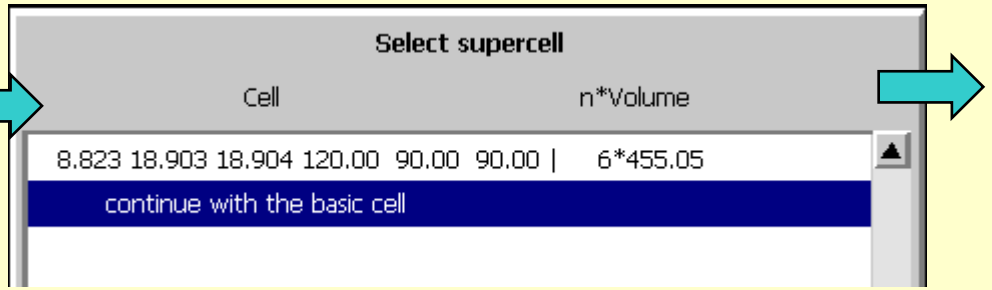
Redundancy = 8.298

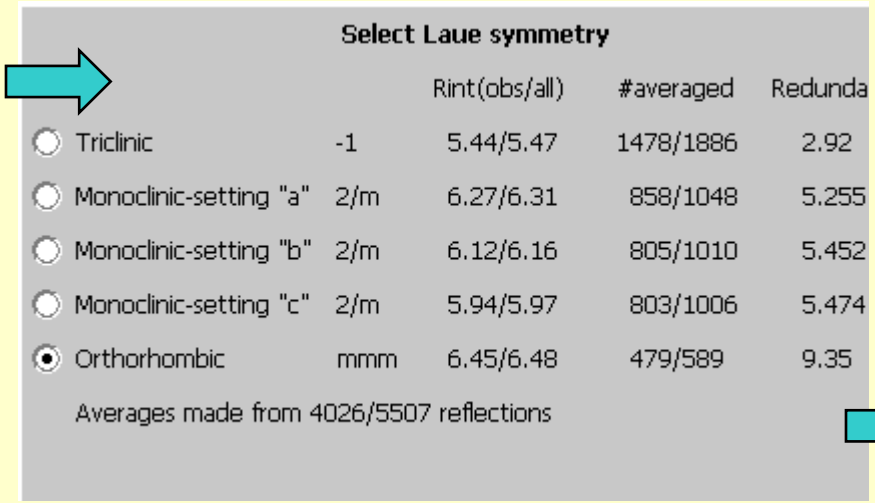
h(min) = 0, h(max) = 11
k(min) = -6, k(max) = 6
l(min) = 0, l(max) = 11

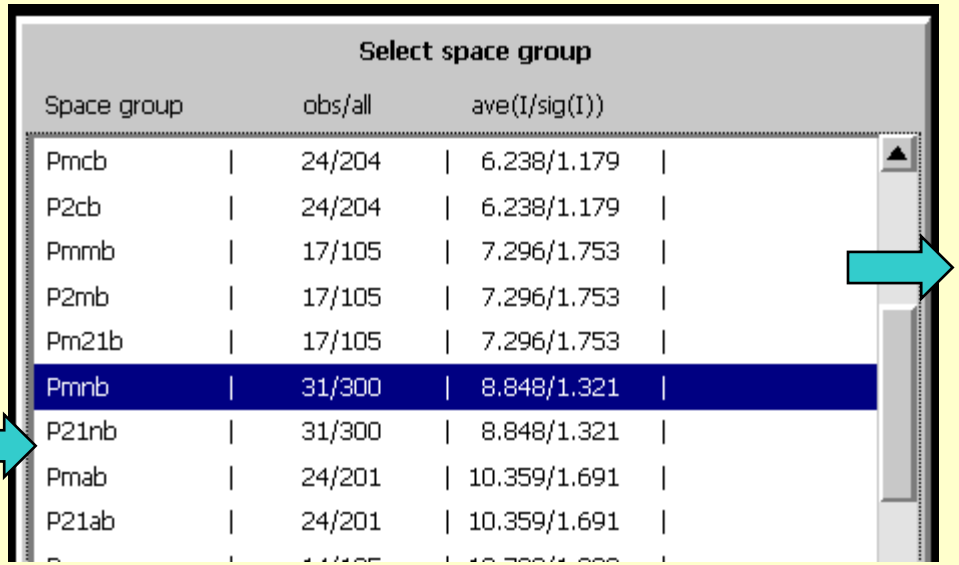
R(obs/all) from e.s.d. of I : 0.55/ 0.64

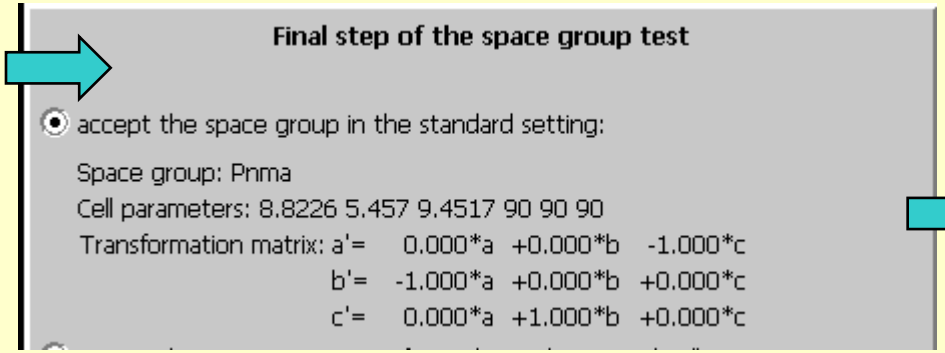
Ok

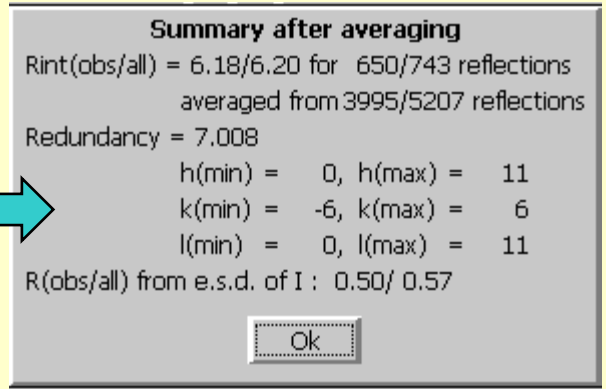

File
Edit/View
Run
Parameters
Tools
Start shell
Export structure to
Structure
Reflection file
Exit
Import/modify the reflection file
Make space group test
Create refinement reflection file

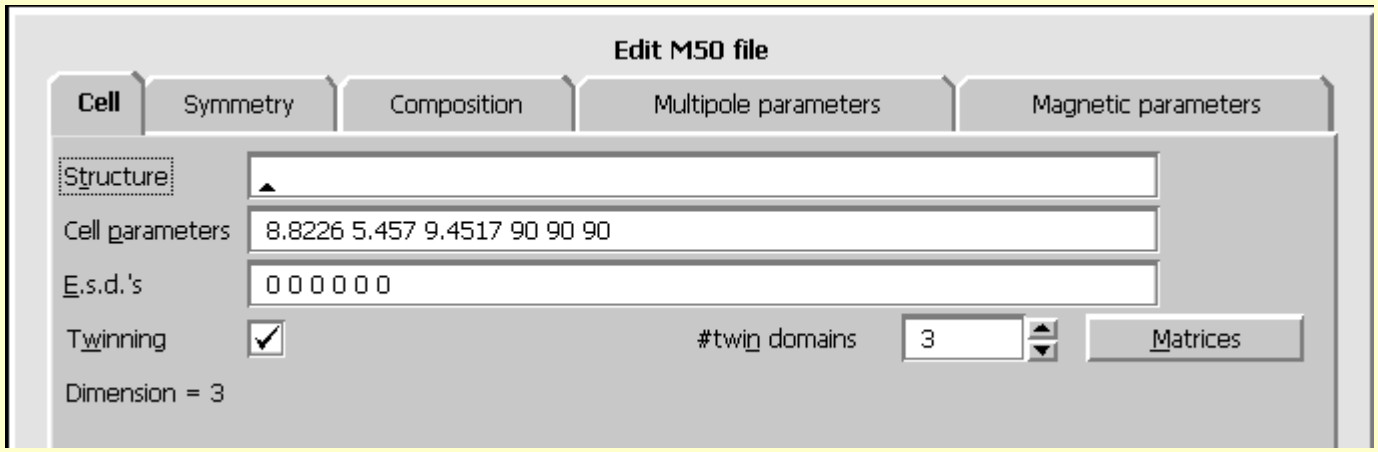

Select supercell
Cell
n*Volume
8.823 18.903 18.904 120.00 90.00 90.00 | 6*455.05
continue with the basic cell


Select Laue symmetry
Rint(obs/all)
#averaged
Redunda
Triclinic -1 5.44/5.47 1478/1886 2.92
Monoclinic-setting "a" 2/m 6.27/6.31 858/1048 5.255
Monoclinic-setting "b" 2/m 6.12/6.16 805/1010 5.452
Monoclinic-setting "c" 2/m 5.94/5.97 803/1006 5.474
Orthorhombic mmm 6.45/6.48 479/589 9.35
Averages made from 4026/5507 reflections


Select space group
Space group | obs/all | ave(I/sig(I))
Pmcb | 24/204 | 6.238/1.179
P2cb | 24/204 | 6.238/1.179
Pmmb | 17/105 | 7.296/1.753
P2mb | 17/105 | 7.296/1.753
Pm21b | 17/105 | 7.296/1.753
Pmnb | 31/300 | 8.848/1.321
P21nb | 31/300 | 8.848/1.321
Pmab | 24/201 | 10.359/1.691
P21ab | 24/201 | 10.359/1.691


Final step of the space group test
accept the space group in the standard setting:
Space group: Pnma
Cell parameters: 8.8226 5.457 9.4517 90 90 90
Transformation matrix: a'= 0.000*a +0.000*b -1.000*c
b'= -1.000*a +0.000*b +0.000*c
c'= 0.000*a +1.000*b +0.000*c


Summary after averaging
Rint(obs/all) = 6.18/6.20 for 650/743 reflections
averaged from 3995/5207 reflections
Redundancy = 7.008
h(min) = 0, h(max) = 11
k(min) = -6, k(max) = 6
l(min) = 0, l(max) = 11
R(obs/all) from e.s.d. of I : 0.50/ 0.57
Ok




```
commcell 10.9165 10.9114 8.8226 90.001 90.024 119.946
trcell
     0.0000     0.5000    -0.5000
     0.0000     0.5000     0.5000
     1.0000     0.0000     0.0000
refblock Block1
sourcefile CsLiSO4.hkl
filedate 23/01/2008 filetime 10:46:40
difcode 5 correspond 1 corrlp -1 corrab
lambda 0.71073 radtype 1 polarization 1
monangle 6.0821 perfmono 0.5
nref 11131 ndim95 3 nlines 22262 twread
cell 10.9165 10.9114 8.8226 90.001 90.0
ormat
    0.071904   -0.022060    0.042037
   -0.045981   -0.092164   -0.055632
    0.062385    0.046966   -0.089360
end
Data Block1
```

```
datblock Block1
nref 743 obslim 3 norefitems 1 diffscales 1 dataave 1 scalelim 10
indslowest 3 indfastest 1 addcentrsymm 0 sigimethod 1 multave 1
flimprint 5 flimcull -1
lambda 0.71073 radtype 1 dattype 1 lpfactor 1 monangle 6.082 perfmono 0.5
datcolltemp 293
end
Data Block1
   2    0    0  21338.2     51.8    1    0    1  0.0000
   4    0    0 120500.5    371.6    1    0    1  0.0000
   6    0    0  29054.0     94.2    1    0    1  0.0000
   8    0    0  19811.9    131.8    1    0    1  0.0000
  10    0    0  14985.7    133.1    1    0    1  0.0000
   2    1    0   1491.4      9.5    1    0    1  0.0000
   4    1    0      8.6      5.6    1    0    1  0.0000
   6    1    0   2380.5     20.9    1    0    1  0.0000
   8    1    0     -1.1     27.3    1    0    1  0.0000
  10    1    0    272.2     29.4    1    0    1  0.0000
   0    2    0 115151.2    357.3    1    0    1  0.0000
```

M95 with all transformations cumulated

M90 should contain three domains: 1,2,3

Solution with Superflip

Correction of false Li

Final refinement: R ~ 2.5%