User guide

ANALYSIS OF CRYSTALS TWINNED BY NON-MEROHEDRY

Using the programs APEX2, Cell Now, Twinabs, and Shelxl

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TWINNING

Twinning of crystals poses a substantial complication in single crystal X-ray structure determination. Twins are regular aggregates consisting of individual crystallites of the same species joined together in some definite mutual orientation. There are four common kinds of twinning: by merohedry (this includes racemic twinning), by pseudo-merohedry, by reticular merohedry, and non-merohedral twins. Crystals twinned by merohedry and pseudo-merohedry are characterized by completely superimposed lattices, i.e. every reflection of one twin moiety overlaps with another reflection of another twin moiety. For non-merohedral twins this is not the case. While a large fraction of reflections are overlapped, at least some reflections are not or only partially overlapped. For structures with complete overlap of reflections, data collection can be performed as with normal untwinned samples. Here, twinning has to be taken into consideration only at the structure solution and refinement stage (by identification and use of an appropriate twin law). For non-merohedral twins, however, more than one unit cell and orientation matrix has to be identified and intensities integrated for all of the lattices. This type of twinning thus has to be identified during the data collection process and requires a different data collection and processing strategy.

This user guide is intended towards collection and analysis of data from non-merohedrally twinned crystals using the Bruker AXS' Apex2 software suite, including Cell Now and Twinabs, and for solving and refining of such structures using the Shelxtl suite of programs.

Unit cell Determination

Twinned crystals can sometimes be identified optically under a microscope when selecting crystals for diffraction, especially when using a polarizing microscope. If this is the case, it is best to avoid twinned crystals and select an untwinned specimen, or to try to cut an untwinned fragment from a larger twinned sample. Often however twinning is not obvious by optical inspection of the crystals, or crystals are composed of too many twin domains and no sufficiently large piece can be isolated. If no untwinned sample can be found or cut from a larger crystal, data will have to be collected from a twinned sample.

The initial steps for data collection are the same as for an untwinned sample (for the use of the basic functions of the Apex2 software, please refer to the Bruker Apex2 manual, or to one of the many manuals available online, e.g. http://web.ysu.edu/gen/stem/Instrumentation_Manuals_p132.html):

- Collect data for <Unit Cell Determination> as usual.
- Try to <Index> the sample to obtain a unit cell.

Failure to obtain a correct (or any!) unit cell can have many causes. One of the reasons could be the presence of twinning by non-merohedry.

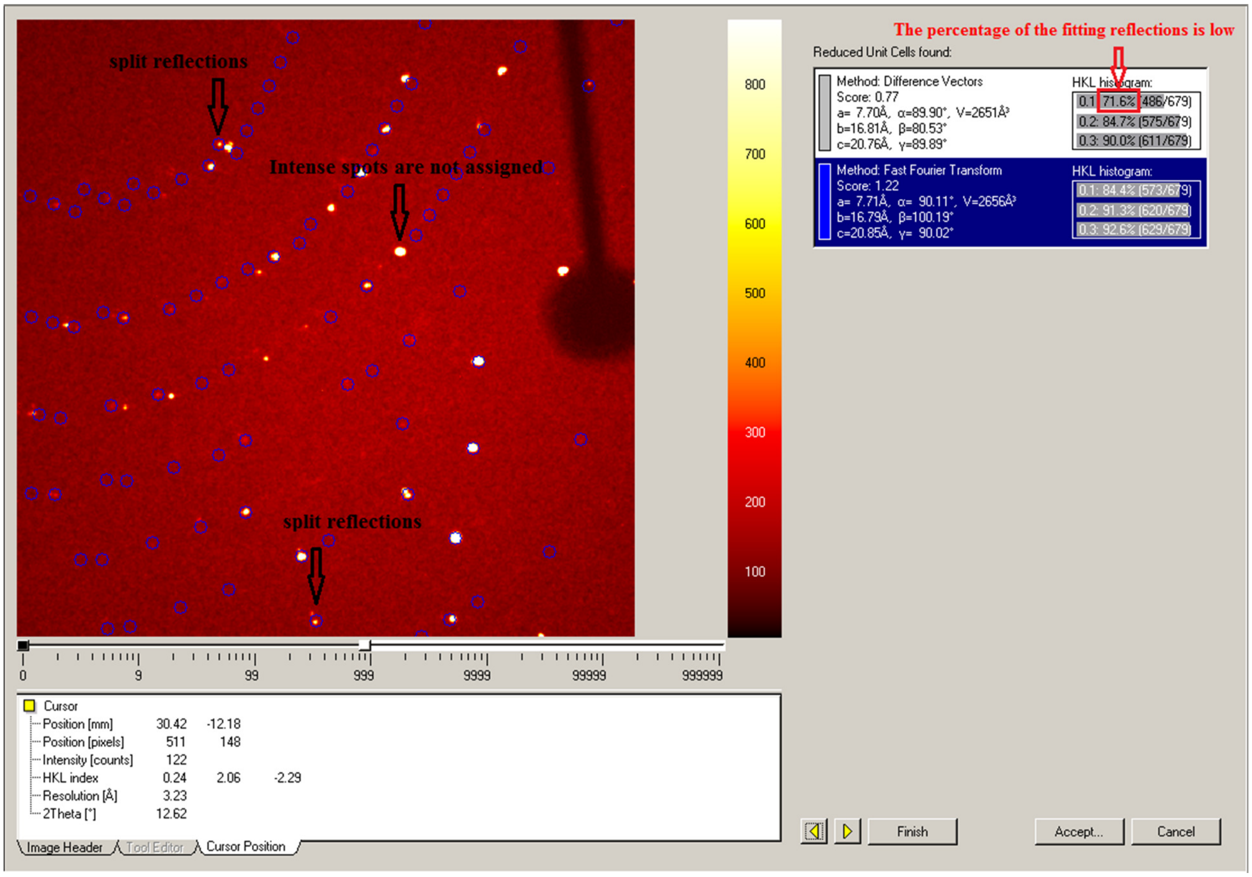
Non-merohedral twinning is not always immediately evident at the unit cell determination steps. Often problems arise only at a later stage: structure solution might fail; structure quality is lower than expected from the experimental R values; large residuals are present; thermal parameters are ill-defined; apparent disorder that cannot be refined well; and other complications. While neither of these complications has to be associated by twinning, it is usually worth checking for presence of twinning by both merohedry (use programs such as Platon or Rotax), or by non-merohedry.

If non-merohedral twinning is present, it is best to recognize it at the unit cell determination stage. Warning signs for non-merohedral twinning at this stage are:

- The unit cell determination fails.
- The unit cell found is unusually large, but many predicted spot positions show no intensity.
- The unit cell size is normal, but many intense spots are not assigned to the predicted cell.
- Some reflections are unusually close or appear “split”.
- In the hkl histogram the percentage of the fitting reflections is low (under 90% for a well diffracting crystal with no other obvious problems).

Not all warning signs have to be present at the same time. Without close inspection of the diffraction data twinning by non-merohedry can often be overlooked at this stage if the minor twin moiety(ies) are substantially less prevalent than the major one.

Figure 1 shows a screenshot of a sample with easily recognizable warning signs for twinning by non-merohedry.



Reduced Unit Cells found:

Method: Difference Vectors
Score: 0.77
a= 7.70Å, α=89.90°, V=2651Å³
b=16.81Å, β=80.53°
c=20.76Å, γ=89.89°

HKL histogram:
0.1 71.6% (486/679)
0.2: 84.7% (575/679)
0.3: 90.0% (611/679)

Method: Fast Fourier Transform
Score: 1.22
a= 7.71Å, α= 90.11°, V=2656Å³
b=16.79Å, β=100.19°
c=20.85Å, γ= 90.02°

HKL histogram:
0.1: 84.4% (573/679)
0.2: 91.3% (620/679)
0.3: 92.6% (629/679)

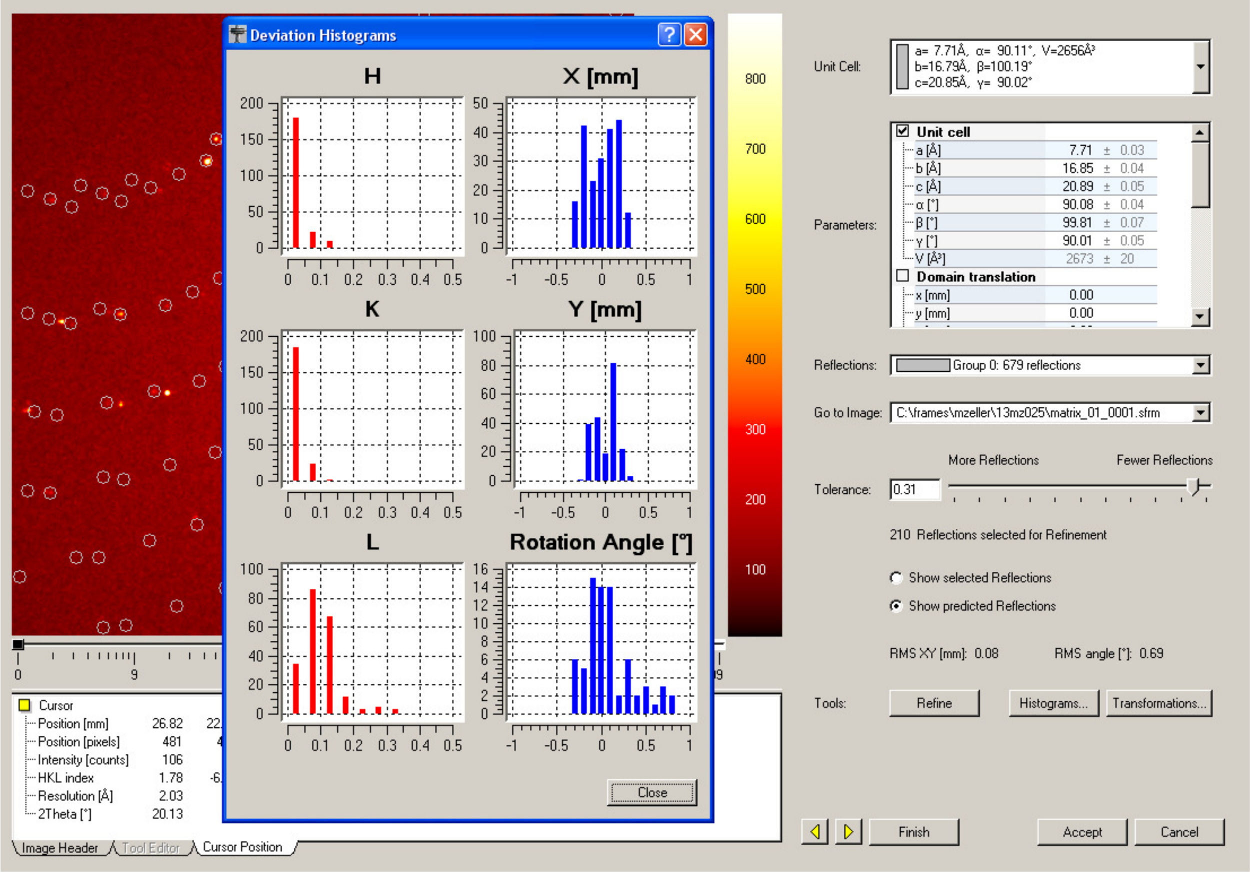
Cursor

Position [mm]	30.42	-12.18	
Position [pixels]	511	148	
Intensity [counts]	122		
HKL index	0.24	2.06	-2.29
Resolution [Å]	3.23		
2Theta [°]	12.62		

Figure 1. Unit cell indexing result window with easily recognizable warning signs for non-merohedral twinning.

If any warning signs are present (or if you have any other reason to suspect twinning, such as failure to obtain a good refinement at a later stage), proceed as follows:

- Choose the best unit cell(s) from the options displayed and then click <Accept>.
- The refinement window will appear.
- Click <Refine> to optimize the unit cell parameters.
- To further check for the possible presence of twinning you can use the <Histograms> tool.



Unit Cell: a= 7.71Å, α= 90.11°, V=2656Å³
b=16.79Å, β=100.19°
c=20.85Å, γ= 90.02°

Parameters:

☑ **Unit cell**		
a [Å]	7.71	± 0.03
b [Å]	16.85	± 0.04
c [Å]	20.89	± 0.05
α [°]	90.08	± 0.04
β [°]	99.81	± 0.07
γ [°]	90.01	± 0.05
V [Å³]	2673	± 20
☐ **Domain translation**		
x [mm]	0.00	
y [mm]	0.00	

Reflections: Group 0: 679 reflections

Go to Image: C:\frames\mzeller\13mz025\matrix_01_0001.sfrm

Tolerance: 0.31

210 Reflections selected for Refinement

○ Show selected Reflections

● Show predicted Reflections

RMS XY [mm]: 0.08 RMS angle [°]: 0.69

☐ Cursor	
Position [mm]	26.82
Position [pixels]	481
Intensity [counts]	106
HKL index	1.78
Resolution [Å]	2.03
2Theta [°]	20.13

Figure. 2. The refinement window with <Histograms> tool

The Histograms show deviations of spots from the expected h k l values for the found cell. Large numbers of reflections that deviate substantially (e.g. above for the <l> Miller index) indicate presence of a second crystal domain (i.e. twinning).

- If there is strong indication for twinning the program Cell Now should be used to establish the correct unit cells, their orientation matrices and the twin laws/transformation matrices.
- The number of available reflections obtained through a normal unit cell determination in Apex2 is usually too low to reliably determine the unit cells for twinned structures ⇒ more data have to be collected first.

Set up a hemisphere or a sphere measurement. When enough data are collected (at least 200 frames for compounds with normal sized unit cells), harvest spots from the newly collected frames:

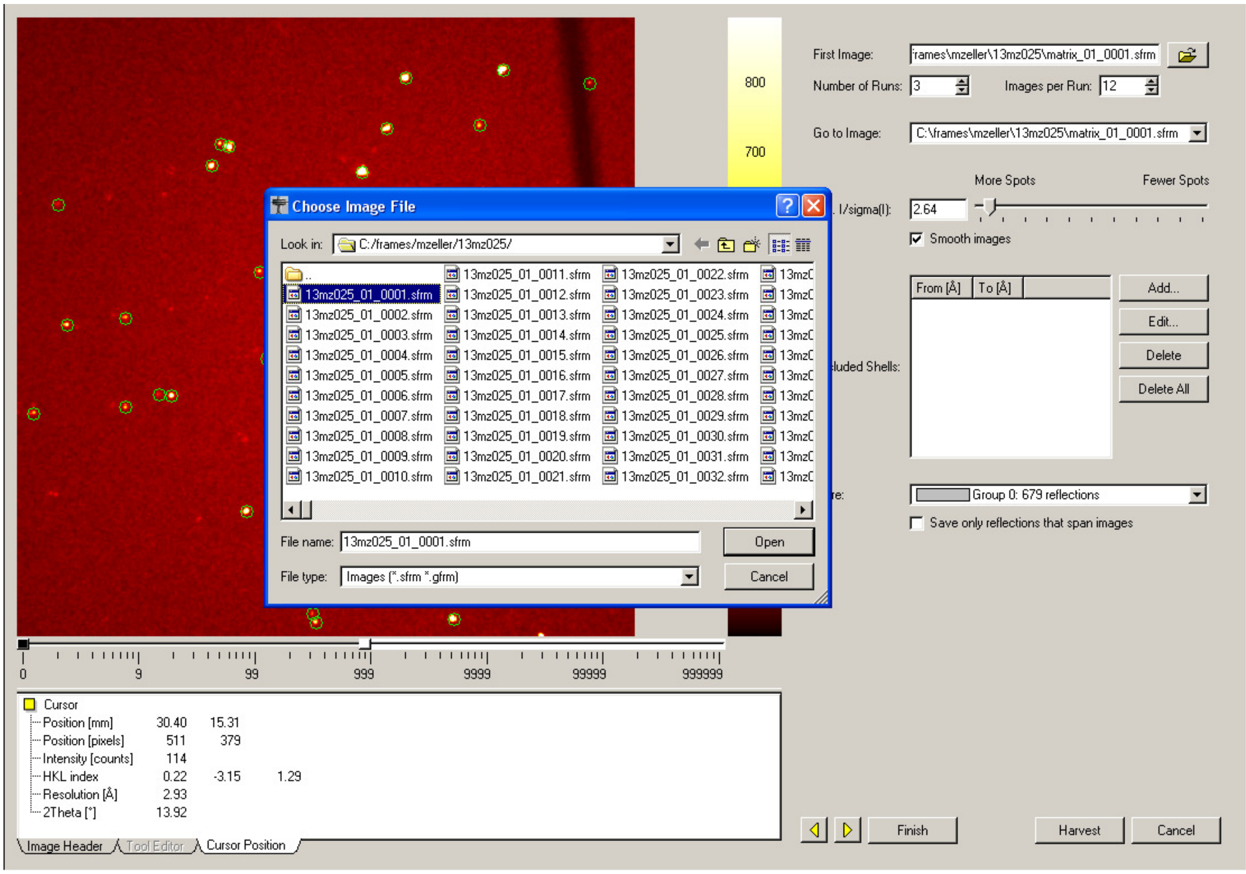
- Return to <Determine Unit Cell>, <Delete> the old unit cell and reflections.
- Click the <Harvest> button and change the image file.

⇒ In the <First Image> line click the browser symbol and choose the first frame from the newly collected data (choose e.g.13mz025_01_0001.sfrm in the current example)

⇒ Change <Number of Runs> to 1 and the <Images per Run> to 200 or more.

⇒ Move the <Min. I/sigma(I)> sliding bar to the left so that more and weaker spots from minor twin moieties are read in. A value of 5 is usually sufficient (this also helps to account for supercell reflections).

⇒ Then click <Harvest>.



Choose Image File

Look in: C:/frames/mzeller/13mz025/

..
13mz025_01_0001.sfrm
13mz025_01_0002.sfrm
13mz025_01_0003.sfrm
13mz025_01_0004.sfrm
13mz025_01_0005.sfrm
13mz025_01_0006.sfrm
13mz025_01_0007.sfrm
13mz025_01_0008.sfrm
13mz025_01_0009.sfrm
13mz025_01_0010.sfrm
13mz025_01_0011.sfrm
13mz025_01_0012.sfrm
13mz025_01_0013.sfrm
13mz025_01_0014.sfrm
13mz025_01_0015.sfrm
13mz025_01_0016.sfrm
13mz025_01_0017.sfrm
13mz025_01_0018.sfrm
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13mz025_01_0025.sfrm
13mz025_01_0026.sfrm
13mz025_01_0027.sfrm
13mz025_01_0028.sfrm
13mz025_01_0029.sfrm
13mz025_01_0030.sfrm
13mz025_01_0031.sfrm
13mz025_01_0032.sfrm

File name: 13mz025_01_0001.sfrm

File type: Images (*.sfrm *.gfrm)

First Image: rames\mzeller\13mz025\matrix_01_0001.sfrm

Number of Runs: 3

Images per Run: 12

Go to Image: C:\frames\mzeller\13mz025\matrix_01_0001.sfrm

. I/sigma(I): 2.64

☑ Smooth images

Group 0: 679 reflections

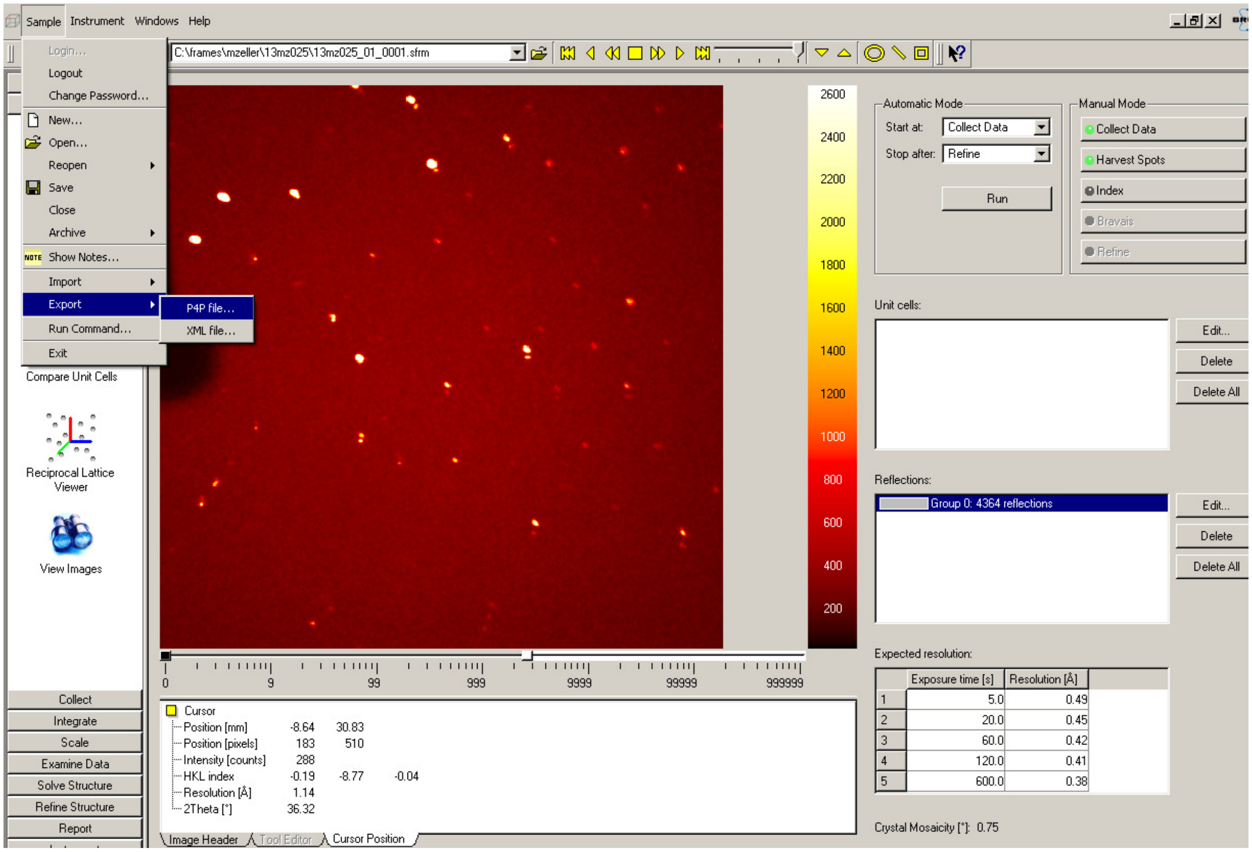
☐ Save only reflections that span images

Cursor
Position [mm] 30.40 15.31
Position [pixels] 511 379
Intensity [counts] 114
HKL index 0.22 -3.15 1.29
Resolution [Å] 2.93
2Theta [°] 13.92

Figure. 3. Unit cell window.

Create a P4P file

- Go to the <Sample> menu on top of the Apex2 window, choose <Export>, click on <P4P file>, this will open the <Export P4P file> window.



C:\frames\mzeller\13mz025\13mz025_01_0001.sfrm

Start at: Collect Data

Stop after: Refine

Group 0: 4364 reflections

Cursor			
Position [mm]	-8.64	30.83	
Position [pixels]	183	510	
Intensity [counts]	288		
HKL index	-0.19	-8.77	-0.04
Resolution [Å]	1.14		
2Theta [°]	36.32		

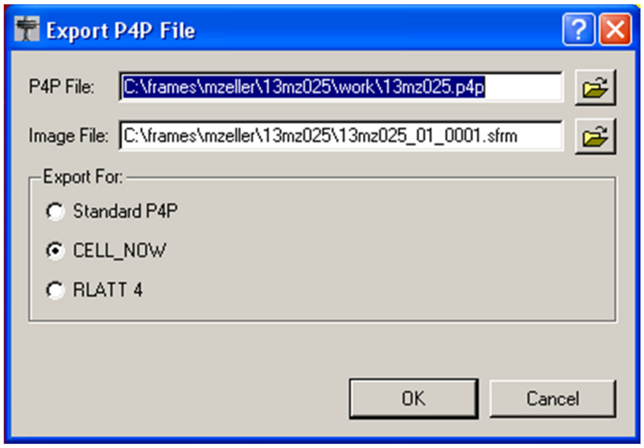
Expected resolution:

	Exposure time [s]	Resolution [Å]
1	5.0	0.49
2	20.0	0.45
3	60.0	0.42
4	120.0	0.41
5	600.0	0.38

Crystal Mosaicity [°]: 0.75

Figure. 4. Unit cell window.

- Choose <CELL_NOW> in the export options, then click OK.



P4P File: C:\frames\mzeller\13mz025\work\13mz025.p4p

Image File: C:\frames\mzeller\13mz025\13mz025_01_0001.sfrm

Export For:

○ Standard P4P

● CELL_NOW

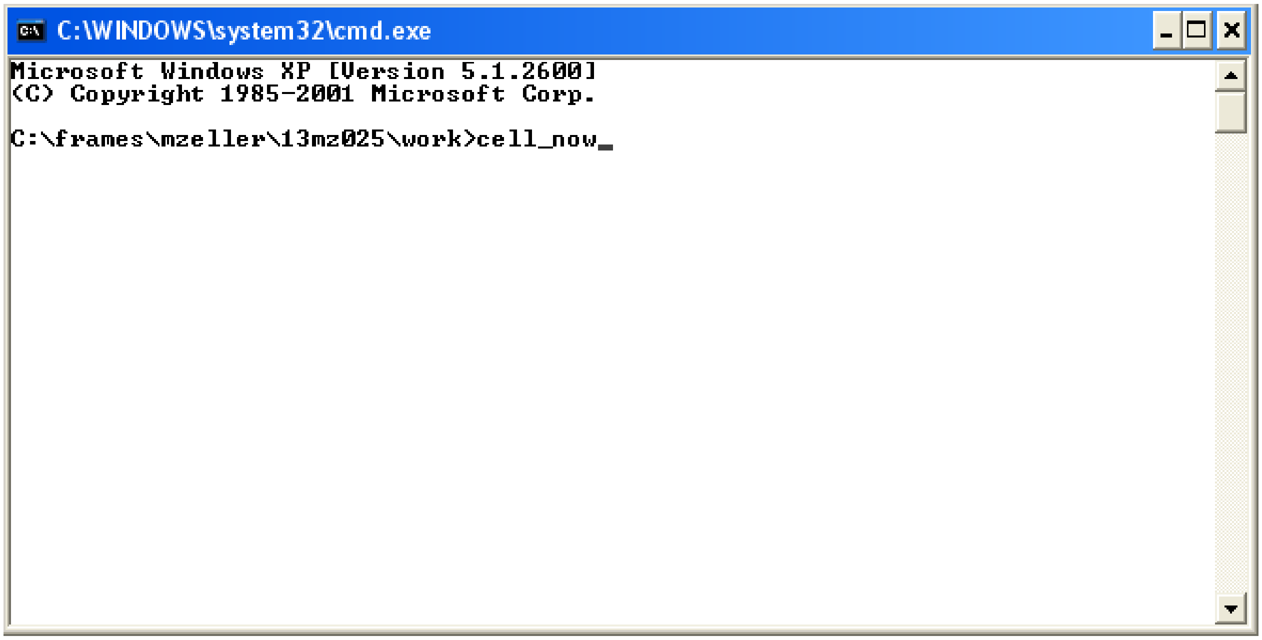
○ RLATT 4

Figure. 5. The p4p file window.

CELL_NOW

Cell Now is used to establish the unit cells for twinned or split crystals, their orientation matrices and the twin laws/transformation matrices.

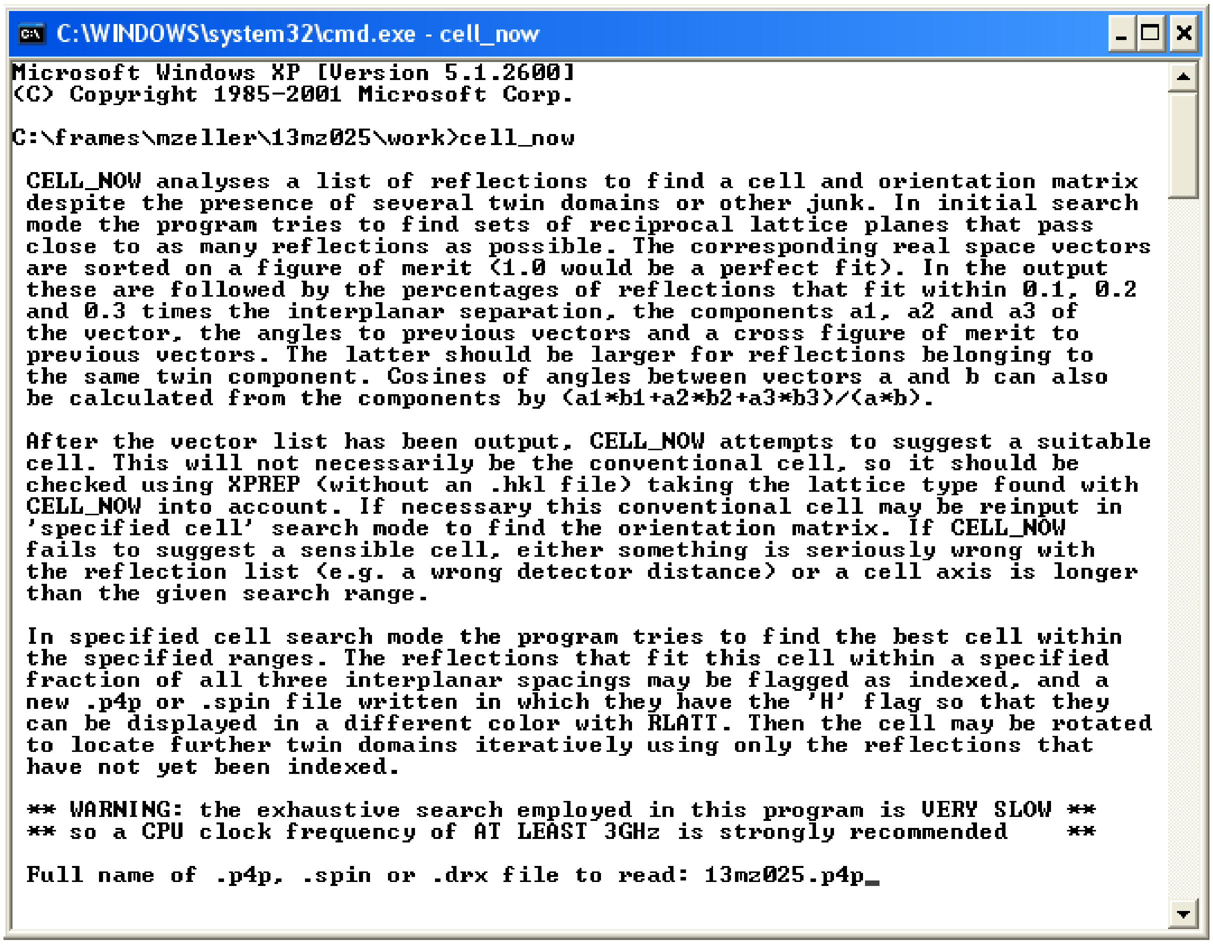
- Go again to <Sample> on top of the Apex2 window ⇒ choose <Run Command>. A command prompt window will appear.
- Type <cell_now>, ⇒ click Enter.



```
Microsoft Windows XP [Version 5.1.2600]
(C) Copyright 1985-2001 Microsoft Corp.

C:\frames\mzeller\13mz025\work>cell_now_
```

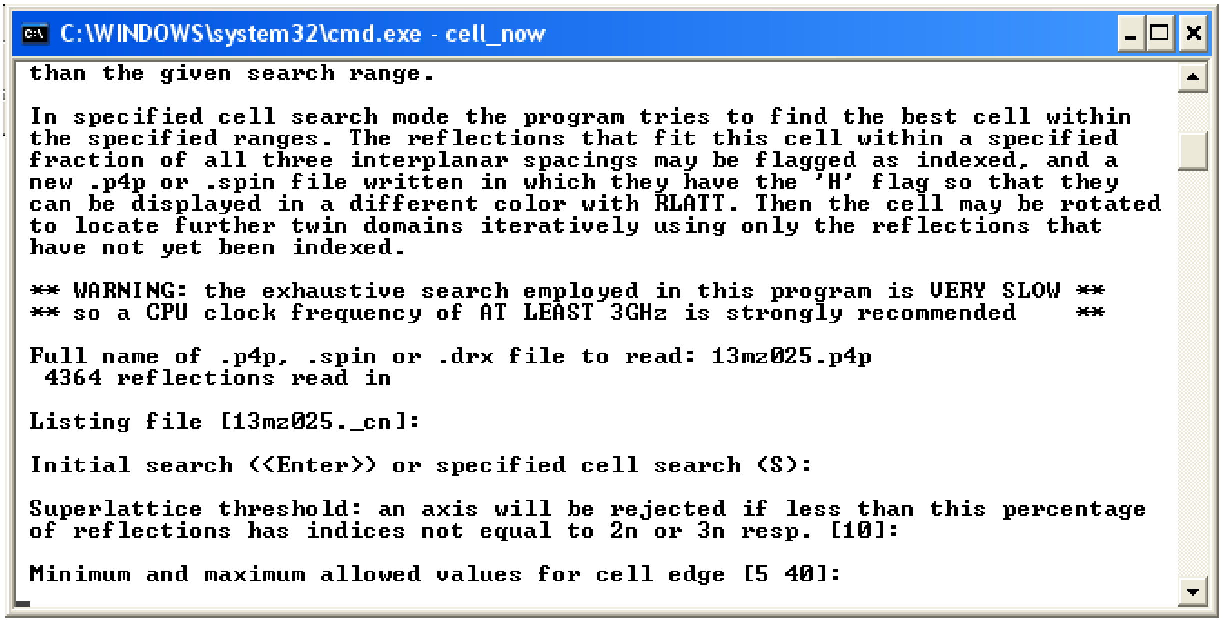
Figure. 6. The Run command window.

- Type the name of the exported p4p file (e.g. 13mz025.p4p) ⇒ click Enter.



```
Microsoft Windows XP [Version 5.1.2600]
(C) Copyright 1985-2001 Microsoft Corp.

C:\frames\mzeller\13mz025\work>cell_now

 CELL_NOW analyses a list of reflections to find a cell and orientation matrix
 despite the presence of several twin domains or other junk. In initial search
 mode the program tries to find sets of reciprocal lattice planes that pass
 close to as many reflections as possible. The corresponding real space vectors
 are sorted on a figure of merit (1.0 would be a perfect fit). In the output
 these are followed by the percentages of reflections that fit within 0.1, 0.2
 and 0.3 times the interplanar separation, the components a1, a2 and a3 of
 the vector, the angles to previous vectors and a cross figure of merit to
 previous vectors. The latter should be larger for reflections belonging to
 the same twin component. Cosines of angles between vectors a and b can also
 be calculated from the components by (a1*b1+a2*b2+a3*b3)/(a*b).

 After the vector list has been output, CELL_NOW attempts to suggest a suitable
 cell. This will not necessarily be the conventional cell, so it should be
 checked using XPREP (without an .hkl file) taking the lattice type found with
 CELL_NOW into account. If necessary this conventional cell may be reinput in
 'specified cell' search mode to find the orientation matrix. If CELL_NOW
 fails to suggest a sensible cell, either something is seriously wrong with
 the reflection list (e.g. a wrong detector distance) or a cell axis is longer
 than the given search range.

 In specified cell search mode the program tries to find the best cell within
 the specified ranges. The reflections that fit this cell within a specified
 fraction of all three interplanar spacings may be flagged as indexed, and a
 new .p4p or .spin file written in which they have the 'H' flag so that they
 can be displayed in a different color with RLATT. Then the cell may be rotated
 to locate further twin domains iteratively using only the reflections that
 have not yet been indexed.

 ** WARNING: the exhaustive search employed in this program is VERY SLOW **
 ** so a CPU clock frequency of AT LEAST 3GHz is strongly recommended    **

 Full name of .p4p, .spin or .drx file to read: 13mz025.p4p_
```

Figure. 7. The Run command window.



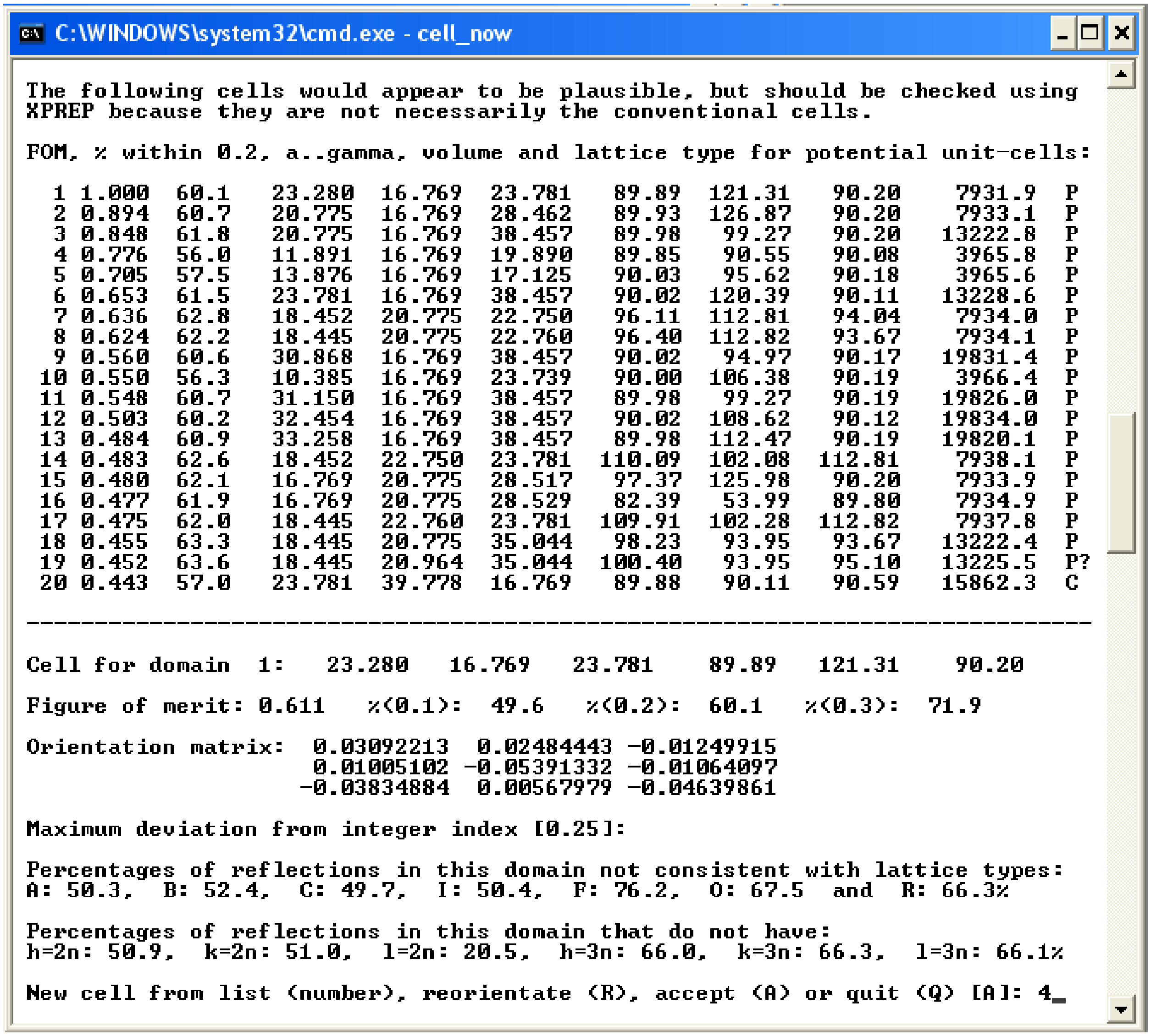
```
 than the given search range.

 In specified cell search mode the program tries to find the best cell within
 the specified ranges. The reflections that fit this cell within a specified
 fraction of all three interplanar spacings may be flagged as indexed, and a
 new .p4p or .spin file written in which they have the 'H' flag so that they
 can be displayed in a different color with RLATT. Then the cell may be rotated
 to locate further twin domains iteratively using only the reflections that
 have not yet been indexed.

 ** WARNING: the exhaustive search employed in this program is VERY SLOW **
 ** so a CPU clock frequency of AT LEAST 3GHz is strongly recommended    **

 Full name of .p4p, .spin or .drx file to read: 13mz025.p4p
  4364 reflections read in

 Listing file [13mz025._cn]:

 Initial search (<Enter>) or specified cell search (S):

 Superlattice threshold: an axis will be rejected if less than this percentage
 of reflections has indices not equal to 2n or 3n resp. [10]:

 Minimum and maximum allowed values for cell edge [5 40]:
_
```

Figure. 8. The Run command window.

Initially, use the default setting of Cell Now for cell search, superlattice threshold and cell edge limits (change the latter if very small or large cell edges are expected). Cell Now will calculate possible unit cells. With larger reflection arrays this might take several minutes, depending on the processor speed of your computer.

Cell Now usually provides more than one solution. Often several of the solutions displayed are equivalent (e.g. when they have the same volume). Of equivalent solutions usually the first is the one in the crystallographic standard setting (but not always). If Cell Now detects crystallographic symmetry (e.g. two 90 degree angles), it displays the cells in the setting of the expected symmetry (e.g. monoclinic).

In simple cases, often the first solution displayed (that with the highest figure of merit, FOM) is the “correct” solution, but this needs to be verified.



```
The following cells would appear to be plausible, but should be checked using
XPREP because they are not necessarily the conventional cells.

FOM, % within 0.2, a..gamma, volume and lattice type for potential unit-cells:

  1 1.000  60.1   23.280  16.769  23.781   89.89  121.31   90.20    7931.9  P
  2 0.894  60.7   20.775  16.769  28.462   89.93  126.87   90.20    7933.1  P
  3 0.848  61.8   20.775  16.769  38.457   89.98   99.27   90.20   13222.8  P
  4 0.776  56.0   11.891  16.769  19.890   89.85   90.55   90.08    3965.8  P
  5 0.705  57.5   13.876  16.769  17.125   90.03   95.62   90.18    3965.6  P
  6 0.653  61.5   23.781  16.769  38.457   90.02  120.39   90.11   13228.6  P
  7 0.636  62.8   18.452  20.775  22.750   96.11  112.81   94.04    7934.0  P
  8 0.624  62.2   18.445  20.775  22.760   96.40  112.82   93.67    7934.1  P
  9 0.560  60.6   30.868  16.769  38.457   90.02   94.97   90.17   19831.4  P
 10 0.550  56.3   10.385  16.769  23.739   90.00  106.38   90.19    3966.4  P
 11 0.548  60.7   31.150  16.769  38.457   89.98   99.27   90.19   19826.0  P
 12 0.503  60.2   32.454  16.769  38.457   90.02  108.62   90.12   19834.0  P
 13 0.484  60.9   33.258  16.769  38.457   89.98  112.47   90.19   19820.1  P
 14 0.483  62.6   18.452  22.750  23.781  110.09  102.08  112.81    7938.1  P
 15 0.480  62.1   16.769  20.775  28.517   97.37  125.98   90.20    7933.9  P
 16 0.477  61.9   16.769  20.775  28.529   82.39   53.99   89.80    7934.9  P
 17 0.475  62.0   18.445  22.760  23.781  109.91  102.28  112.82    7937.8  P
 18 0.455  63.3   18.445  20.775  35.044   98.23   93.95   93.67   13222.4  P
 19 0.452  63.6   18.445  20.964  35.044  100.40   93.95   95.10   13225.5  P?
 20 0.443  57.0   23.781  39.778  16.769   89.88   90.11   90.59   15862.3  C

------------------------------------------------------------------------------

Cell for domain  1:   23.280   16.769   23.781    89.89   121.31    90.20

Figure of merit: 0.611   %(0.1):  49.6   %(0.2):  60.1   %(0.3):  71.9

Orientation matrix:   0.03092213  0.02484443 -0.01249915
                      0.01005102 -0.05391332 -0.01064097
                     -0.03834884  0.00567979 -0.04639861

Maximum deviation from integer index [0.25]:

Percentages of reflections in this domain not consistent with lattice types:
A: 50.3,  B: 52.4,  C: 49.7,  I: 50.4,  F: 76.2,  O: 67.5  and  R: 66.3%

Percentages of reflections in this domain that do not have:
h=2n: 50.9,  k=2n: 51.0,  l=2n: 20.5,  h=3n: 66.0,  k=3n: 66.3,  l=3n: 66.1%

New cell from list (number), reorientate (R), accept (A) or quit (Q) [A]: 4_
```

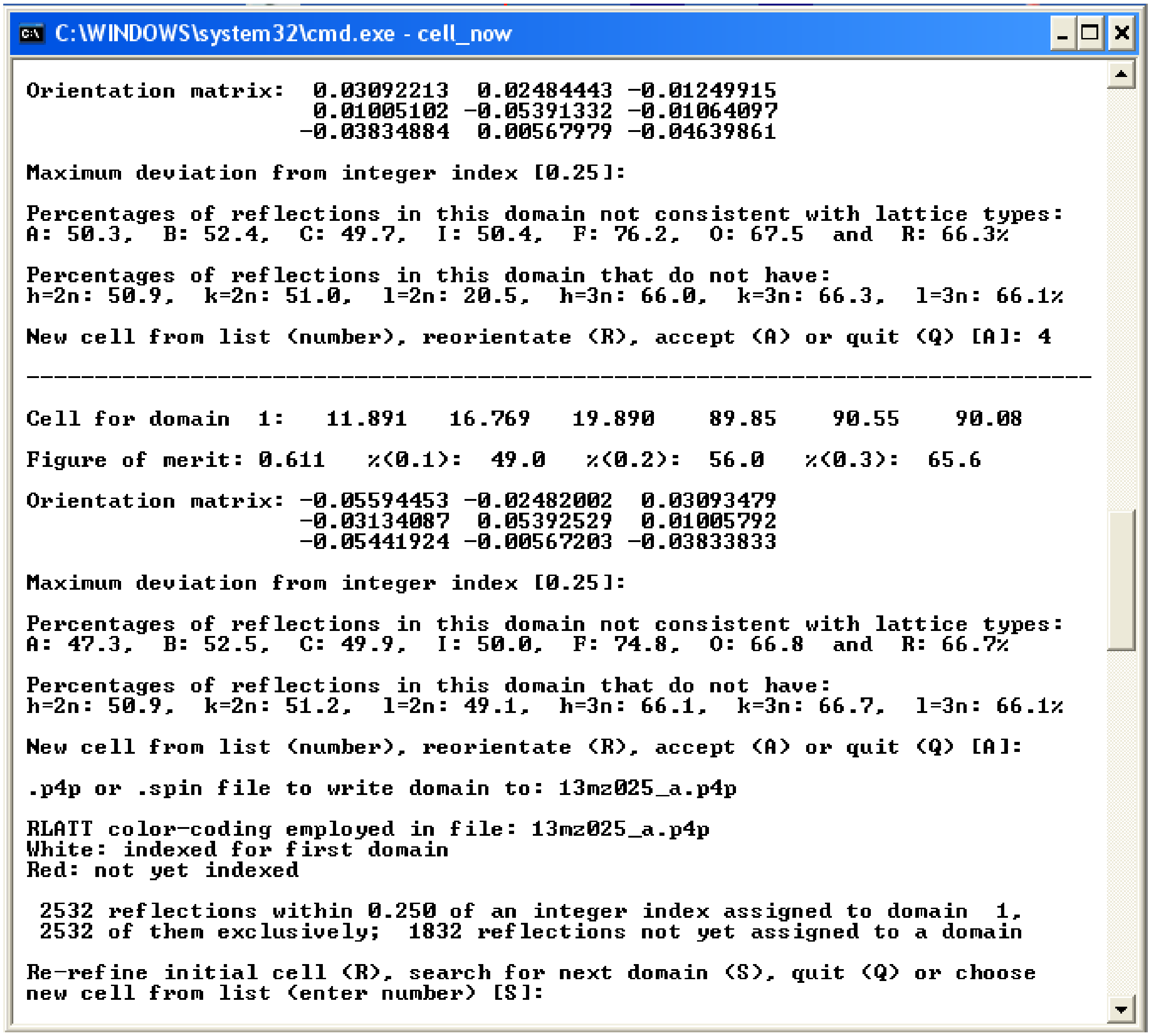
Figure. 9. The Run command window.

Indicators for which solution is correct (if any) are:

- The percentage of fitting reflections (column 3). Solutions with high values are more likely to be correct.
- The symmetry of the unit cell (columns 4-9). Higher symmetry solutions are ranked higher by cell now.
- The volume of the unit cell (second last column). For cells with similar percentages of fitting reflections the smaller unit cell with the same lattice centering should is more likely to be correct.

After inspection of the possible solutions:

- Choose the most likely unit cell (number 4, in the above example: It is the highest ranked solution of the smallest unit cells while having a similar percentage of fitting reflections as the larger volume unit cells).
- To select solution number one, enter a value for <maximum deviation from integer index [0.25]> (0.25 is usually suitable, at least initially), then click <Enter> to accept solution 1. To select any other solution, do the same, but then enter the number of the chosen solution (<new cell from list>).
- <Accept> or change the value for <maximum deviation from integer index [0.25]> (0.25 is usually suitable, at least initially).
- Enter the chosen filename followed by .p4p to indicate the file format (e.g. 13mz025_a.p4p) ⇒ Enter.



```
Orientation matrix:  0.03092213  0.02484443 -0.01249915
                     0.01005102 -0.05391332 -0.01064097
                    -0.03834884  0.00567979 -0.04639861

Maximum deviation from integer index [0.25]:

Percentages of reflections in this domain not consistent with lattice types:
A: 50.3,  B: 52.4,  C: 49.7,  I: 50.4,  F: 76.2,  O: 67.5  and  R: 66.3%

Percentages of reflections in this domain that do not have:
h=2n: 50.9,  k=2n: 51.0,  l=2n: 20.5,  h=3n: 66.0,  k=3n: 66.3,  l=3n: 66.1%

New cell from list (number), reorientate (R), accept (A) or quit (Q) [A]: 4

------------------------------------------------------------------------------

Cell for domain  1:   11.891   16.769   19.890    89.85    90.55    90.08

Figure of merit: 0.611   %(0.1):  49.0   %(0.2):  56.0   %(0.3):  65.6

Orientation matrix: -0.05594453 -0.02482002  0.03093479
                    -0.03134087  0.05392529  0.01005792
                    -0.05441924 -0.00567203 -0.03833833

Maximum deviation from integer index [0.25]:

Percentages of reflections in this domain not consistent with lattice types:
A: 47.3,  B: 52.5,  C: 49.9,  I: 50.0,  F: 74.8,  O: 66.8  and  R: 66.7%

Percentages of reflections in this domain that do not have:
h=2n: 50.9,  k=2n: 51.2,  l=2n: 49.1,  h=3n: 66.1,  k=3n: 66.7,  l=3n: 66.1%

New cell from list (number), reorientate (R), accept (A) or quit (Q) [A]:

.p4p or .spin file to write domain to: 13mz025_a.p4p

RLATT color-coding employed in file: 13mz025_a.p4p
White: indexed for first domain
Red: not yet indexed

 2532 reflections within 0.250 of an integer index assigned to domain  1,
 2532 of them exclusively;  1832 reflections not yet assigned to a domain

Re-refine initial cell (R), search for next domain (S), quit (Q) or choose
new cell from list (enter number) [S]:
```

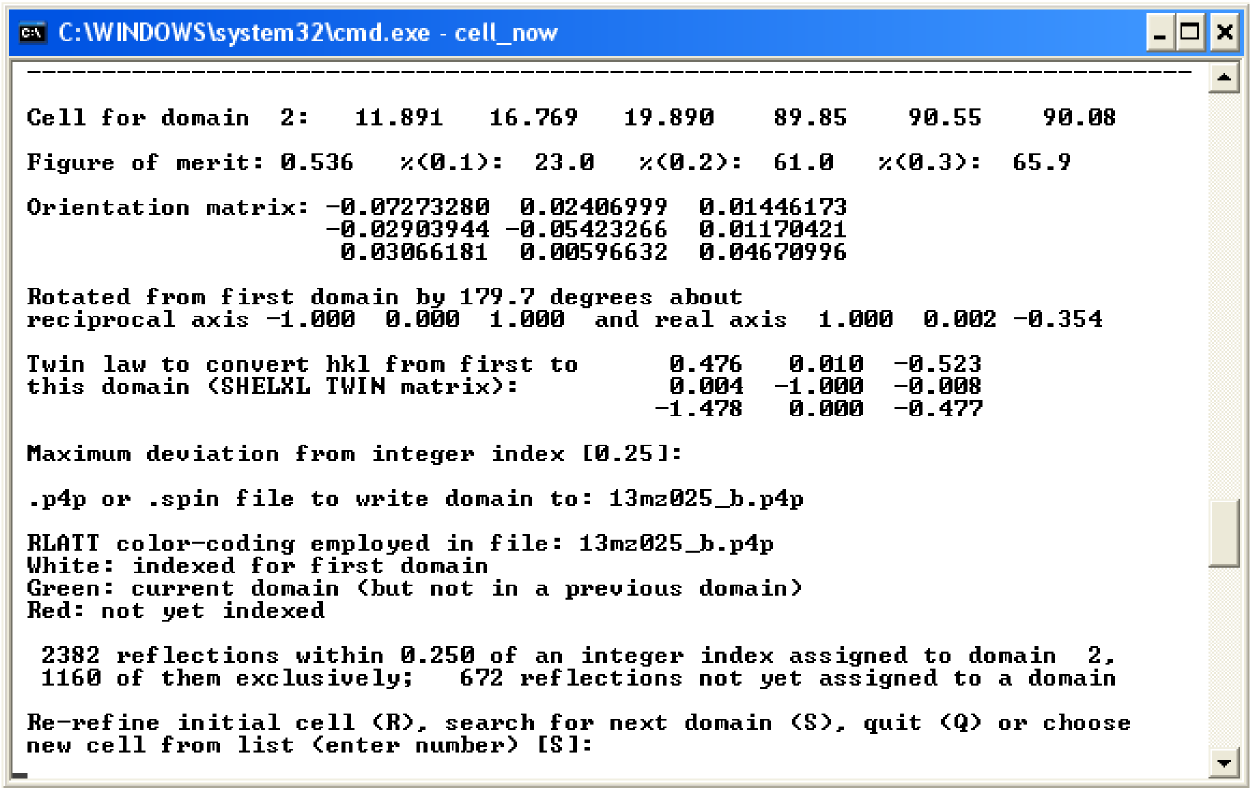
Figure. 10. The Run command window.

Cell Now writes the file and will display the number of reflections that fit and don’t fit the chosen cell (within the selected maximum deviation). In the written p4p file, reflections are coded so that reflections belonging to one domain are displayed in the same colour if analyzed in RLATT or the Lattice Viewer of Apex2.

- If a substantial number of reflections is not yet indexed, type <S> to search for another domain ⇒ Enter

Cell now will attempt to find another domain with the same cell parameters, but with a different orientation matrix. If the second domain is created indeed by a twin operation, the domains are usually related by a simple mathematical operation, e.g. a 180 degree rotation around a low index axis (e.g. one of the real space or reciprocal axis, or simple combinations of them such as a face or space diagonal). Also possible are 90 or 120 degree rotations. Solutions rotated by different values, or around non-integer fractions of an axis are usually not created by twinning, but the crystal might be split or cracked (small angle rotations around an odd axis), or simply more than one crystal is present (random rotation around an odd axis).

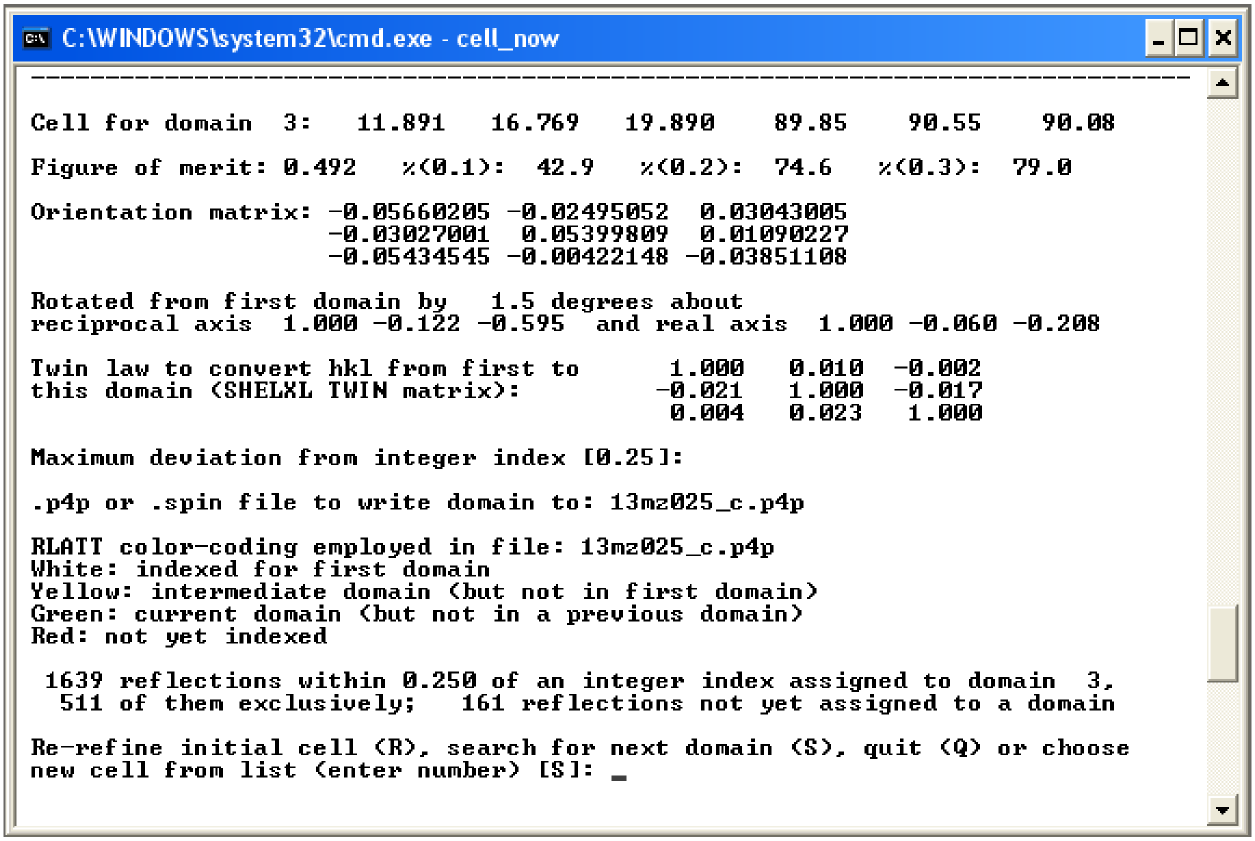
After Cell Now found a second domain, it will display the rotation angle and axis for both real and reciprocal space. Closely inspect the values. If the result makes sense, proceed as for the first domain and name the new file (e.g. 13mz025_b.p4p).



```
-----------------------------------------------------------------------------

Cell for domain  2:   11.891   16.769   19.890    89.85    90.55    90.08

Figure of merit: 0.536   %(0.1):  23.0   %(0.2):  61.0   %(0.3):  65.9

Orientation matrix: -0.07273280  0.02406999  0.01446173
                    -0.02903944 -0.05423266  0.01170421
                     0.03066181  0.00596632  0.04670996

Rotated from first domain by 179.7 degrees about
reciprocal axis -1.000  0.000  1.000  and real axis  1.000  0.002 -0.354

Twin law to convert hkl from first to       0.476   0.010  -0.523
this domain (SHELXL TWIN matrix):           0.004  -1.000  -0.008
                                           -1.478   0.000  -0.477

Maximum deviation from integer index [0.25]:

.p4p or .spin file to write domain to: 13mz025_b.p4p

RLATT color-coding employed in file: 13mz025_b.p4p
White: indexed for first domain
Green: current domain (but not in a previous domain)
Red: not yet indexed

 2382 reflections within 0.250 of an integer index assigned to domain  2,
 1160 of them exclusively;   672 reflections not yet assigned to a domain

Re-refine initial cell (R), search for next domain (S), quit (Q) or choose
new cell from list (enter number) [S]:
_
```

Figure. 11. The Run command window.

- If a substantial number of reflections is not yet indexed, type <S> to search for another domain ⇒ Enter.

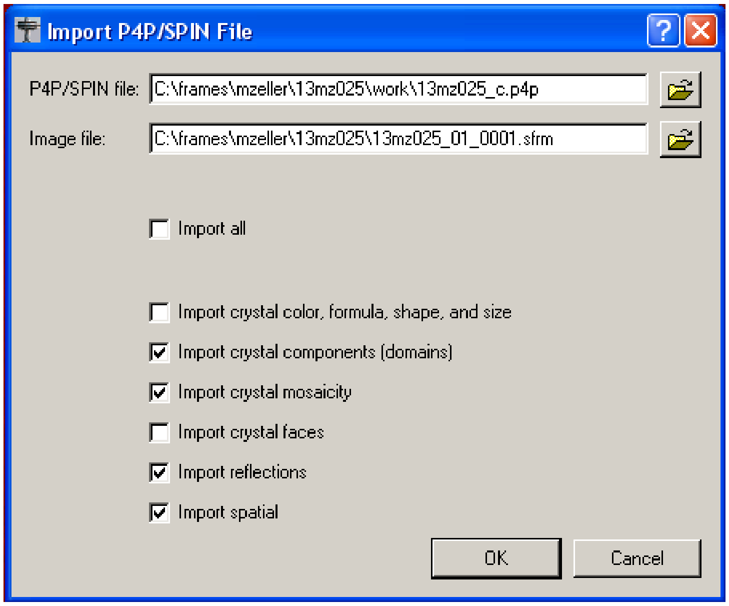


```
 ------------------------------------------------------------------------------

 Cell for domain  3:   11.891   16.769   19.890    89.85    90.55    90.08

 Figure of merit: 0.492   %(0.1):  42.9   %(0.2):  74.6   %(0.3):  79.0

 Orientation matrix: -0.05660205 -0.02495052  0.03043005
                     -0.03027001  0.05399809  0.01090227
                     -0.05434545 -0.00422148 -0.03851108

 Rotated from first domain by   1.5 degrees about
 reciprocal axis  1.000 -0.122 -0.595  and real axis  1.000 -0.060 -0.208

 Twin law to convert hkl from first to      1.000   0.010  -0.002
 this domain (SHELXL TWIN matrix):         -0.021   1.000  -0.017
                                            0.004   0.023   1.000

 Maximum deviation from integer index [0.25]:

 .p4p or .spin file to write domain to: 13mz025_c.p4p

 RLATT color-coding employed in file: 13mz025_c.p4p
 White: indexed for first domain
 Yellow: intermediate domain (but not in first domain)
 Green: current domain (but not in a previous domain)
 Red: not yet indexed

  1639 reflections within 0.250 of an integer index assigned to domain  3,
   511 of them exclusively;   161 reflections not yet assigned to a domain

 Re-refine initial cell (R), search for next domain (S), quit (Q) or choose
 new cell from list (enter number) [S]: _
```

Figure. 12. The Run command window.

Cell Now can colour code up to three domains for inspection in RLATT or the Apex2 reciprocal lattice viewer. After writing three domains to file (or when no new or valid domains are found any more), import the written p4p file of your choice back into Apex2:

- In Apex2, go to <Sample> on top of the window ⇒ <Import> ⇒ <Load File> (13mz025_b.p4p in the current example).
- Check the <Import Reflections> check box.



Import P4P/SPIN File

P4P/SPIN file: C:\frames\mzeller\13mz025\work\13mz025_c.p4p

Image file: C:\frames\mzeller\13mz025\13mz025_01_0001.sfrm

☐ Import all

☐ Import crystal color, formula, shape, and size
☑ Import crystal components (domains)
☑ Import crystal mosaicity
☐ Import crystal faces
☑ Import reflections
☑ Import spatial

Figure. 13. The ImportP4P/SPIN File window.

- In Apex2, open the <Reciprocal Lattice Viewer> in the <Evaluate> menue.
- In the <RLATT> drop down menue, choose <Visualization>.

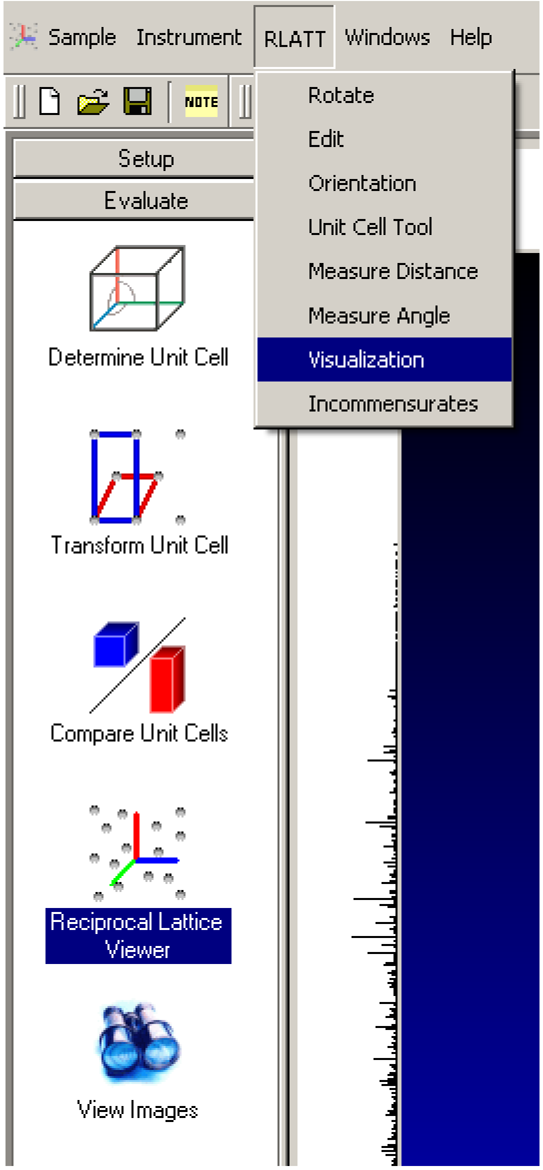
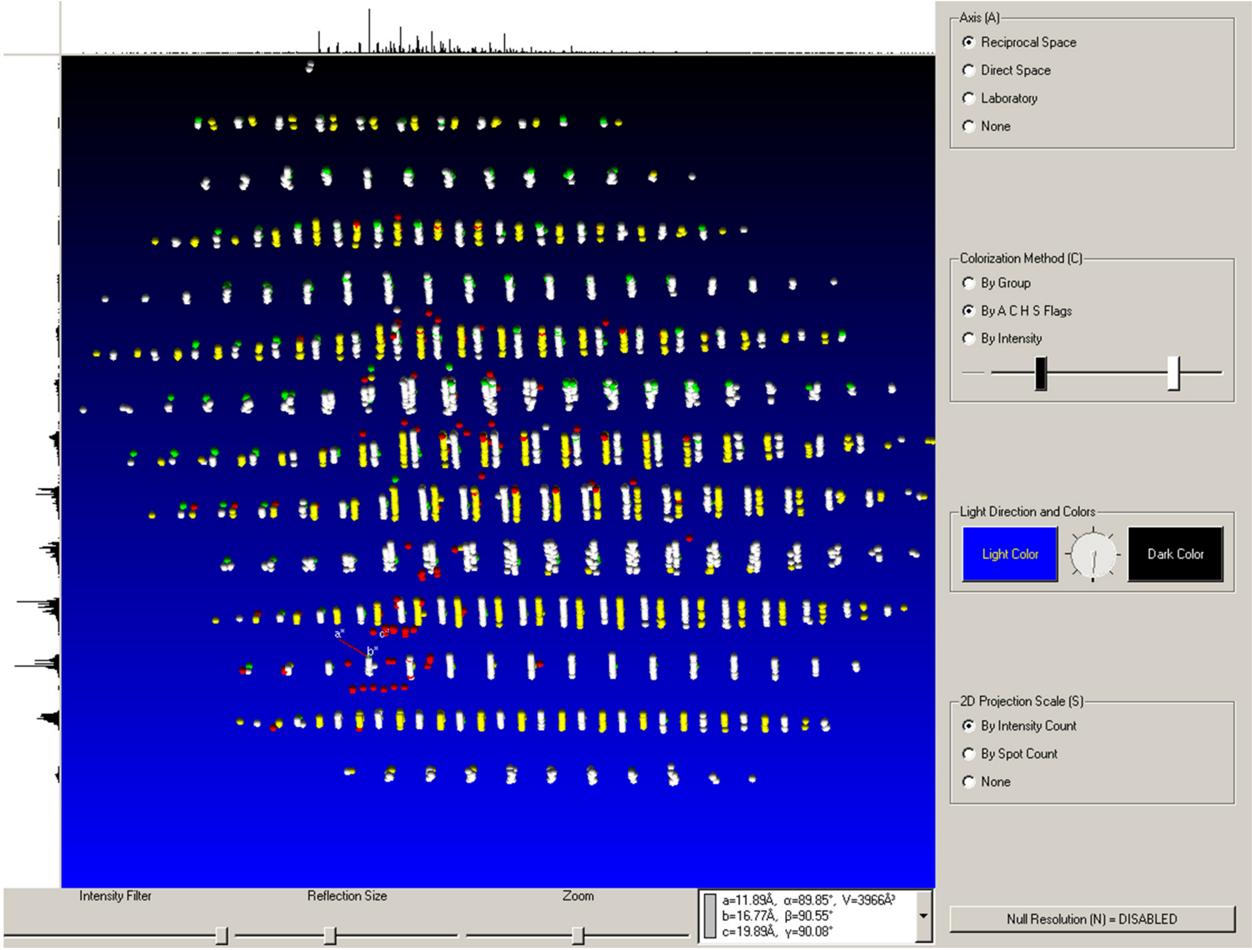


Figure. 14. Open <RLATT> tools.

- In the interface that opens, check <By A C H S Flags> in the <Colorization Method> menue to display the Cell Now solutions in different colours (white for the first domain, green for the last assigned domain, yellow for all in between domains).

  Adjust <Intensity Filter>, <Reflection Size> and <Zoom> using the slide rules at the bottom. Switch between <Rotate>, <Select> and <Measure> via a right mouse click.



Axis (A)
● Reciprocal Space
○ Direct Space
○ Laboratory
○ None

Colorization Method (C)
○ By Group
● By A C H S Flags
○ By Intensity

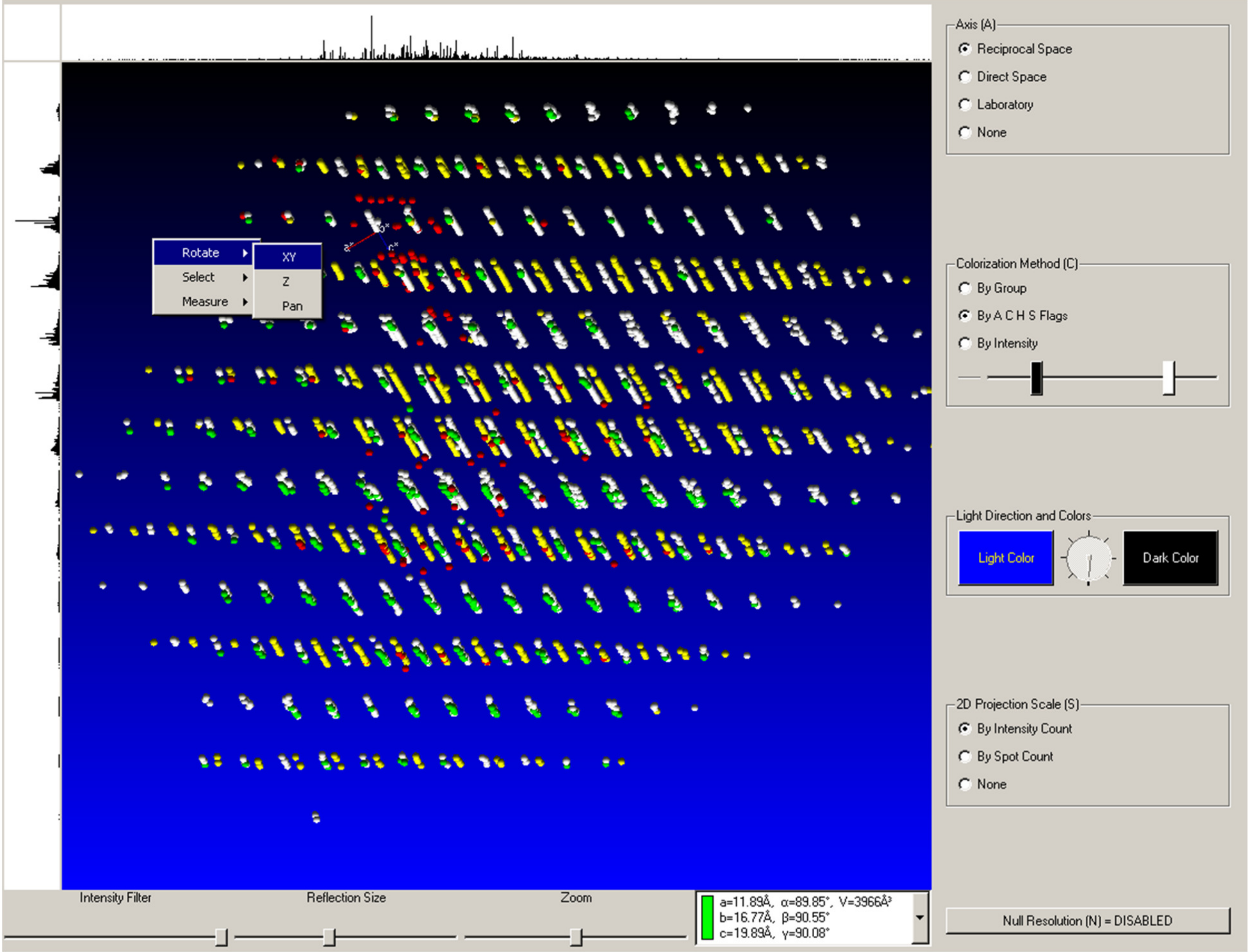
Light Direction and Colors

2D Projection Scale (S)
● By Intensity Count
○ By Spot Count
○ None

a=11.89Å, α=89.85°, V=3966Å³
b=16.77Å, β=90.55°
c=19.89Å, γ=90.08°

Figure. 15. A typical reciprocal lattice view of a non-merohedrally twinned crystal. Two lattices are clearly visible (white and yellow), with reflections overlapping in some rows, and displaying separate moieties in other rows.

To test if the unit cells obtained from Cell Now are correct the unit cell axis lengths should be measured. Orient the view so as to look down one of the unit cell axes. Then switch to <Measure> and <Distance> (right mouse click).



Axis (A)
● Reciprocal Space
○ Direct Space
○ Laboratory
○ None

Colorization Method (C)
○ By Group
● By A C H S Flags
○ By Intensity

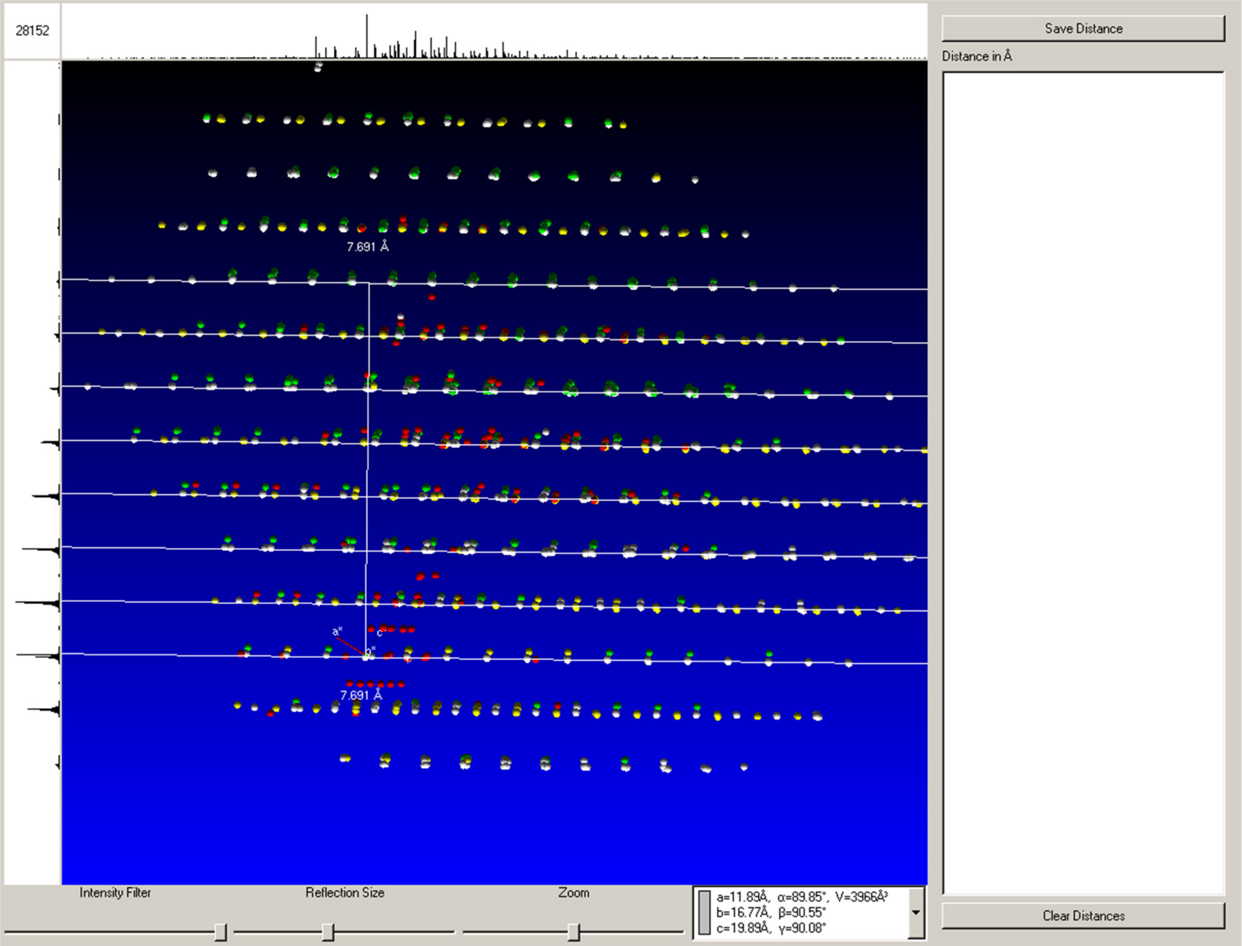
Light Direction and Colors

2D Projection Scale (S)
● By Intensity Count
○ By Spot Count
○ None

🟩 a=11.89Å, α=89.85°, V=3966Å³
b=16.77Å, β=90.55°
c=19.89Å, γ=90.08°

Figure. 16. Above shows a typical reciprocal lattice view of a non-merohedrally twinned crystal.

Place the cursor at one of the reflections of the main moiety (white lattice) and drag the mouse along one of the axis directions to line up with a parallel line of reflections. Press the plus button of the keyboard as often as needed to line up rows of reflections as good as possible with the displayed lines as shown in Fig. 17 below. Read the displayed value (in Å) and compare it with the axis values obtained by Cell Now. If all three values agree, the unit cell from Cell Now is a valid solution. If not (as in the current example, below), the unit cell has to be corrected, e.g. by running Cell Now again with the new information taken into account.



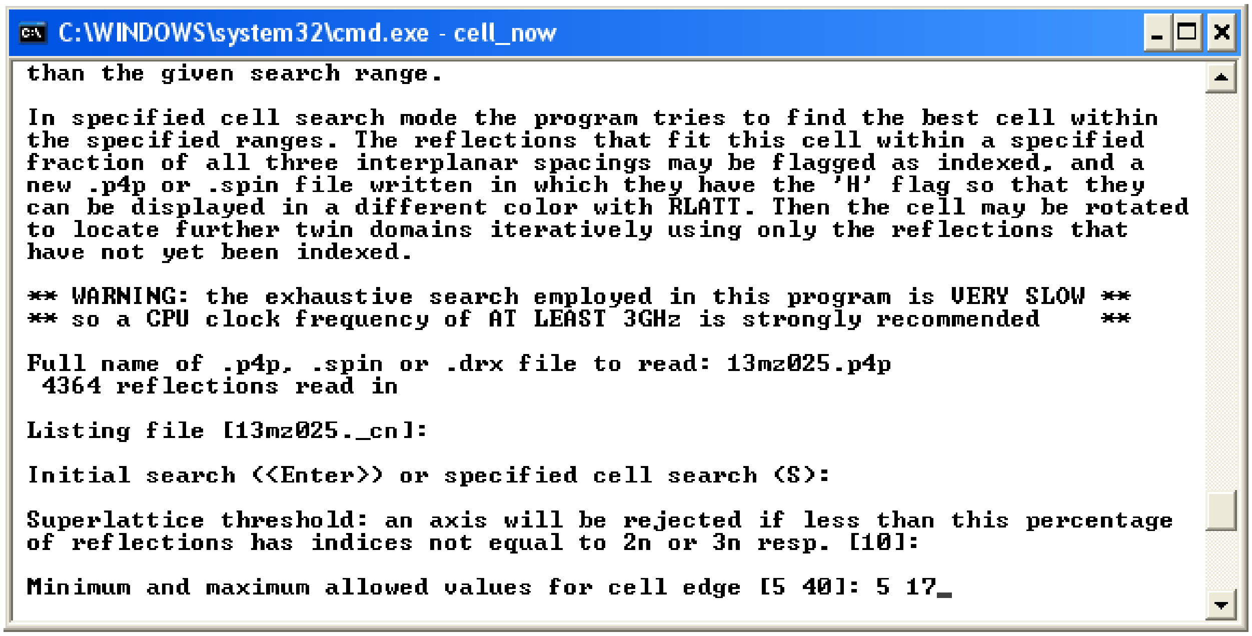
```
a=11.89Å,  α=89.85°,  V=3966Å³
b=16.77Å,  β=90.55°
c=19.89Å,  γ=90.08°
```

Figure. 17. The Reciprocal Lattice window with lattice parameter measurement, view down the b-axis from Cell Now. Note the disagreement of the directions of the a- and b-axes with the observed patterns, and the measured lattice constant value of 7.691 Å which is not found in the unit cell from Cell Now (displayed at the bottom right).

In the current case, close inspection shows that the unit cell axes measured in the reciprocal lattice viewer are all smaller than 17 Å, and the beta angle of 90 degree from Cell Now appears to be incorrect. To obtain a corrected unit cell:

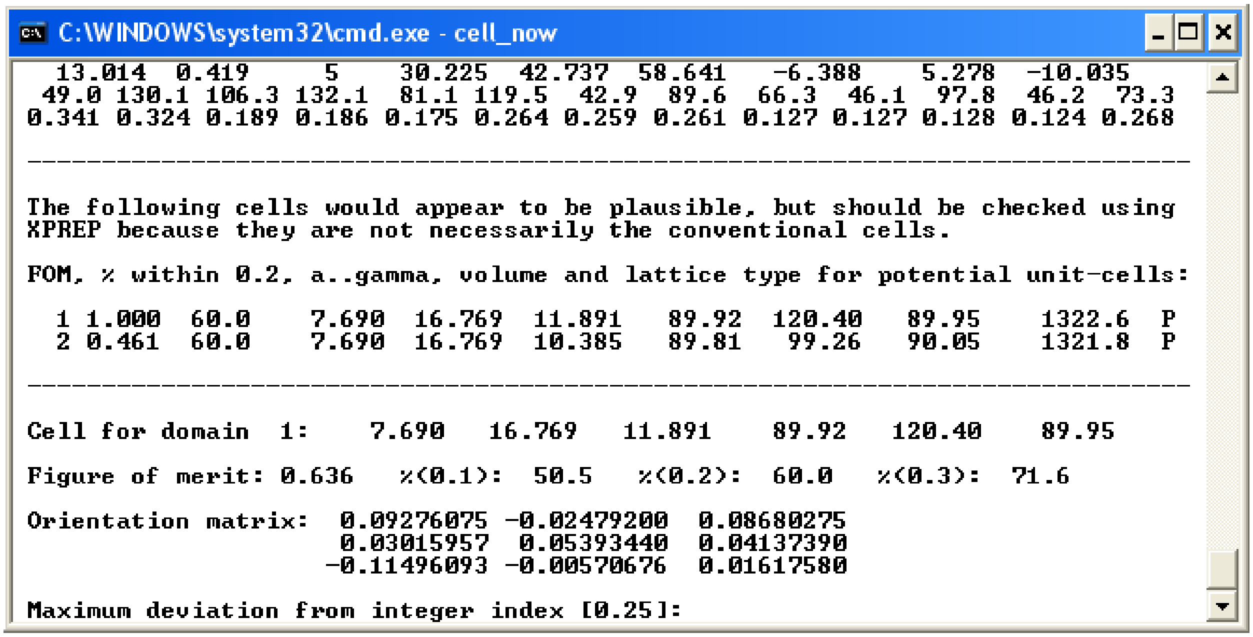
- Go back to the Command prompt ⇒ Quit the previous run of Cell Now (Type <q>), start a new run of Cell Now.
- Start a new run of Cell Now. Proceed as before. Adjust values such as indicated by the reciprocal lattice analysis (e.g. change the superlattice threshold if a supercell had been found, change the <minimum and maximum allowed value for cell edge> if the cell found previously has been too large or too small, or use the <specified cell search> if you know the approximate unit cell parameters (in the current case the value for <minimum and

maximum allowed value for cell edge> was changed from <4 40> to <5 17> to force Cell Now to consider smaller unit cells).



```
than the given search range.

In specified cell search mode the program tries to find the best cell within
the specified ranges. The reflections that fit this cell within a specified
fraction of all three interplanar spacings may be flagged as indexed, and a
new .p4p or .spin file written in which they have the 'H' flag so that they
can be displayed in a different color with RLATT. Then the cell may be rotated
to locate further twin domains iteratively using only the reflections that
have not yet been indexed.

** WARNING: the exhaustive search employed in this program is VERY SLOW **
** so a CPU clock frequency of AT LEAST 3GHz is strongly recommended    **

Full name of .p4p, .spin or .drx file to read: 13mz025.p4p
 4364 reflections read in

Listing file [13mz025._cn]:

Initial search (<Enter>) or specified cell search (S):

Superlattice threshold: an axis will be rejected if less than this percentage
of reflections has indices not equal to 2n or 3n resp. [10]:

Minimum and maximum allowed values for cell edge [5 40]: 5 17_
```

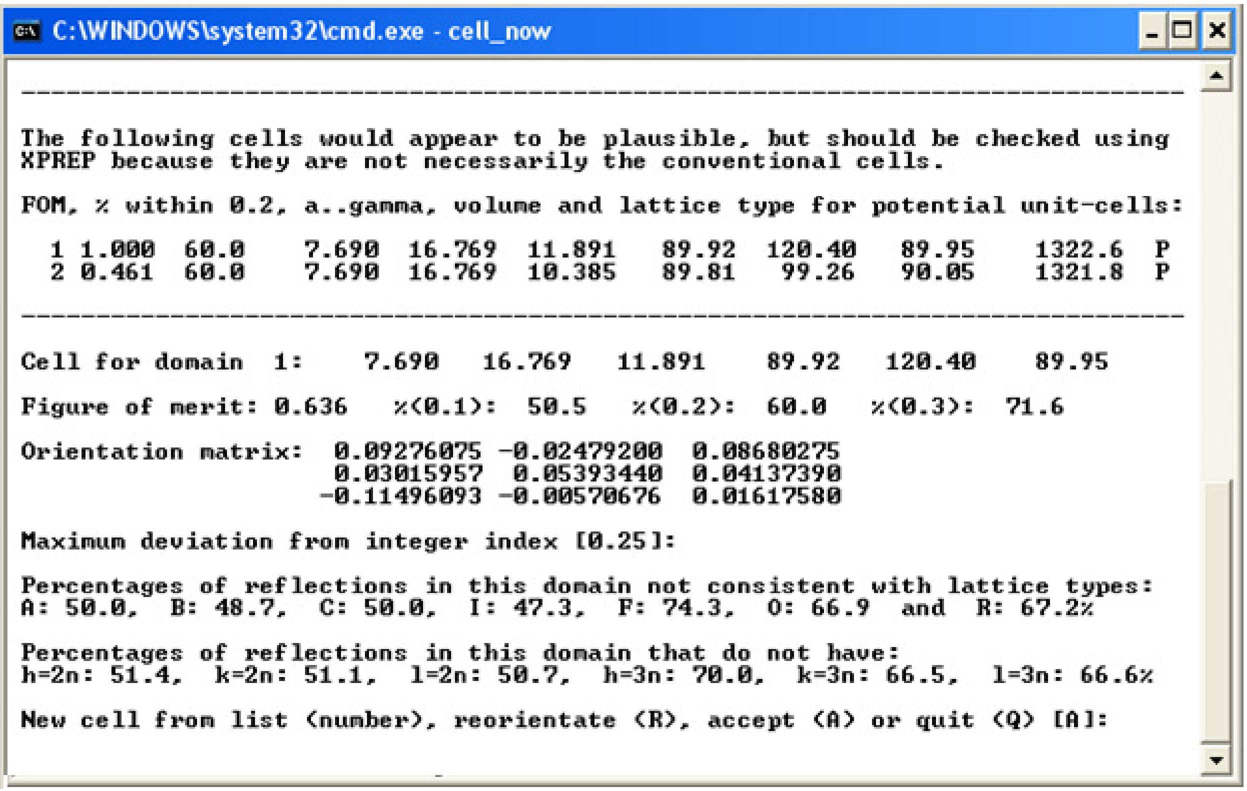
Figure. 18. The Run command window.



```
   13.014  0.419      5     30.225  42.737  58.641   -6.388    5.278  -10.035
  49.0 130.1 106.3 132.1  81.1 119.5  42.9  89.6  66.3  46.1  97.8  46.2  73.3
 0.341 0.324 0.189 0.186 0.175 0.264 0.259 0.261 0.127 0.127 0.128 0.124 0.268

 ------------------------------------------------------------------------------

 The following cells would appear to be plausible, but should be checked using
 XPREP because they are not necessarily the conventional cells.

 FOM, % within 0.2, a..gamma, volume and lattice type for potential unit-cells:

   1 1.000  60.0    7.690  16.769  11.891   89.92  120.40   89.95    1322.6  P
   2 0.461  60.0    7.690  16.769  10.385   89.81   99.26   90.05    1321.8  P

 ------------------------------------------------------------------------------

 Cell for domain  1:    7.690   16.769   11.891    89.92   120.40    89.95

 Figure of merit: 0.636   %(0.1):  50.5   %(0.2):  60.0   %(0.3):  71.6

 Orientation matrix:  0.09276075 -0.02479200  0.08680275
                      0.03015957  0.05393440  0.04137390
                     -0.11496093 -0.00570676  0.01617580

 Maximum deviation from integer index [0.25]:
```

Figure. 19. The Run command window.

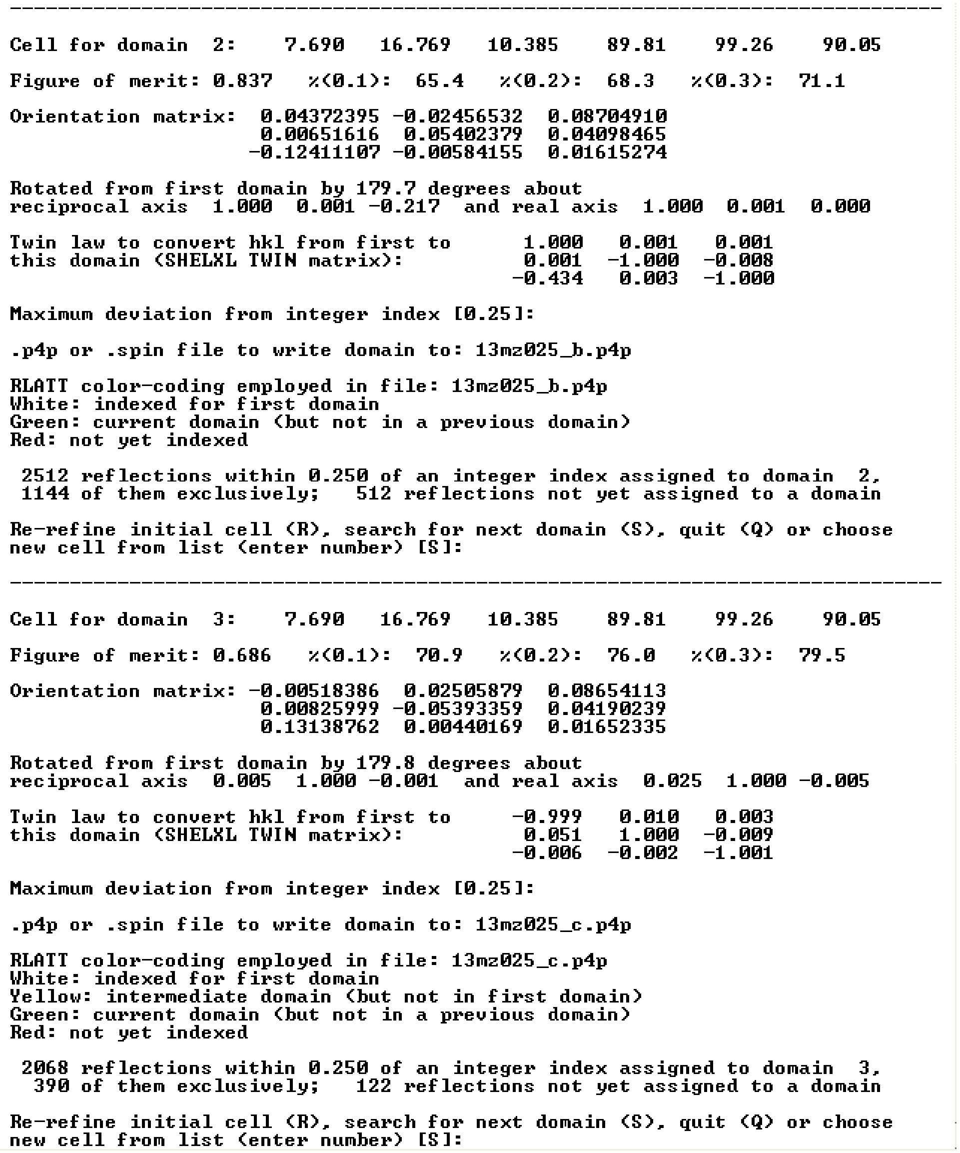
A new set of unit cells is displayed. Proceed as described before and select the most suitable unit cell (in this case, two equivalent monoclinic solutions were found. The solution with the smaller beta angle was chosen following IUCR recommendations).



```
---------------------------------------------------------------------------

The following cells would appear to be plausible, but should be checked using
XPREP because they are not necessarily the conventional cells.

FOM, % within 0.2, a..gamma, volume and lattice type for potential unit-cells:

  1 1.000  60.0    7.690  16.769  11.891   89.92  120.40   89.95    1322.6  P
  2 0.461  60.0    7.690  16.769  10.385   89.81   99.26   90.05    1321.8  P

---------------------------------------------------------------------------

Cell for domain  1:    7.690   16.769   11.891    89.92   120.40    89.95

Figure of merit: 0.636   %(0.1):  50.5   %(0.2):  60.0   %(0.3):  71.6

Orientation matrix:  0.09276075 -0.02479200  0.08680275
                     0.03015957  0.05393440  0.04137390
                    -0.11496093 -0.00570676  0.01617580

Maximum deviation from integer index [0.25]:

Percentages of reflections in this domain not consistent with lattice types:
A: 50.0,  B: 48.7,  C: 50.0,  I: 47.3,  F: 74.3,  O: 66.9  and  R: 67.2%

Percentages of reflections in this domain that do not have:
h=2n: 51.4,  k=2n: 51.1,  l=2n: 50.7,  h=3n: 70.0,  k=3n: 66.5,  l=3n: 66.6%

New cell from list (number), reorientate (R), accept (A) or quit (Q) [A]:
```

Figure. 20. The Run command window.

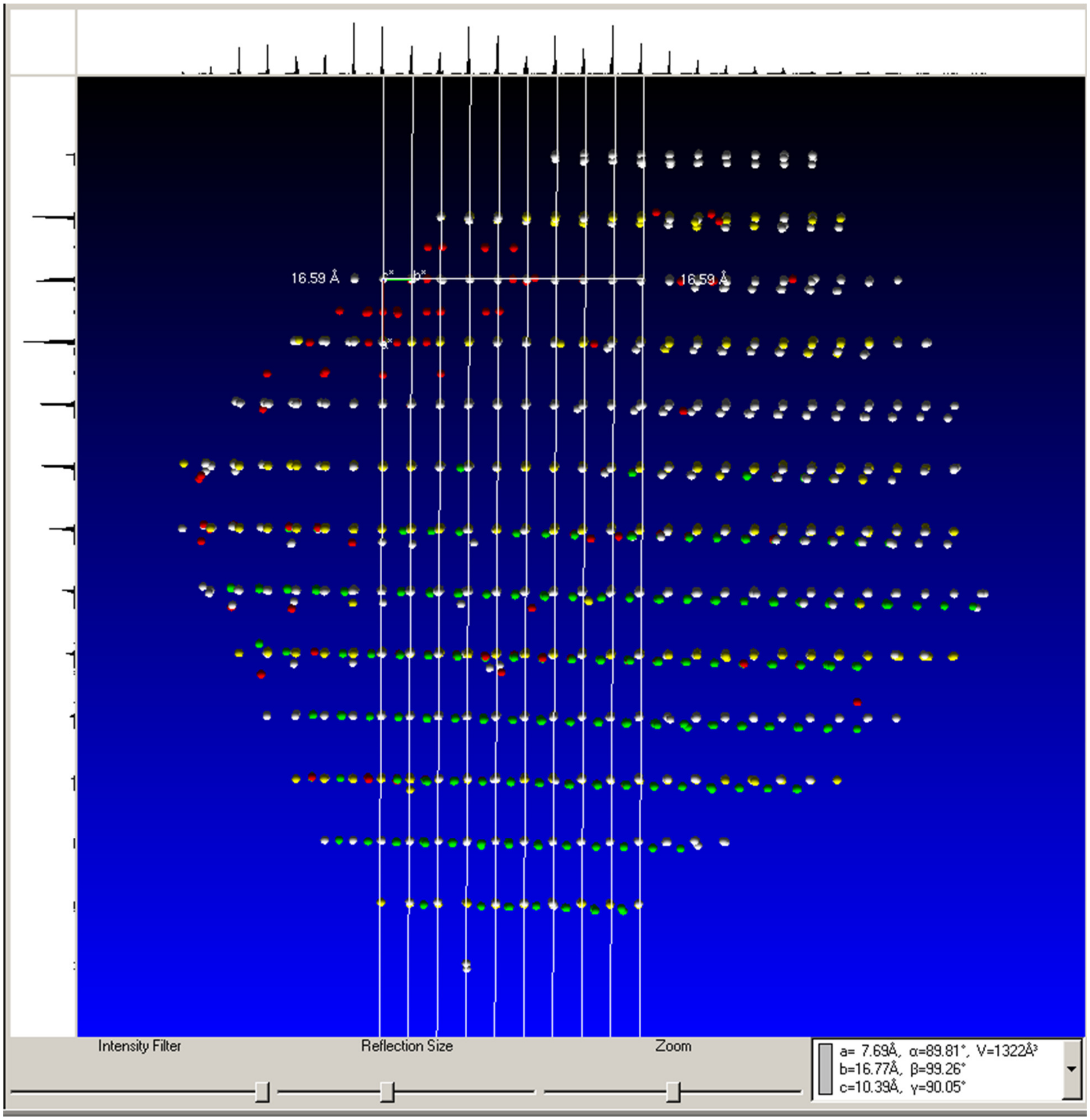
Repeat the steps described earlier to obtain a p4p file with two (or more) twin moieties. Here two additional twin moieties were found. The second is related to the first by a 180 degree rotation around the real a-axis. Note the transformation matrix having one off diagonal element different from zero (this or similar patterns are typical for most twins). The third moiety is related to the first by a close to 180 degree rotation around the b-axis. With a monoclinic crystal this cannot be a twin operation (a two fold axis around b is a symmetry element already present in the monoclinic system). This indicates that the crystal might be split in two nearly aligned parts rather than being twinned a second time. Cell Now and Apex2 can be used to handle both twins as well as split crystals.



```
------------------------------------------------------------------------------

Cell for domain  2:    7.690   16.769   10.385    89.81    99.26    90.05

Figure of merit: 0.837   %(0.1):  65.4   %(0.2):  68.3   %(0.3):  71.1

Orientation matrix:  0.04372395 -0.02456532  0.08704910
                     0.00651616  0.05402379  0.04098465
                    -0.12411107 -0.00584155  0.01615274

Rotated from first domain by 179.7 degrees about
reciprocal axis  1.000  0.001 -0.217  and real axis  1.000  0.001  0.000

Twin law to convert hkl from first to      1.000   0.001   0.001
this domain (SHELXL TWIN matrix):          0.001  -1.000  -0.008
                                          -0.434   0.003  -1.000

Maximum deviation from integer index [0.25]:

.p4p or .spin file to write domain to: 13mz025_b.p4p

RLATT color-coding employed in file: 13mz025_b.p4p
White: indexed for first domain
Green: current domain (but not in a previous domain)
Red: not yet indexed

 2512 reflections within 0.250 of an integer index assigned to domain  2,
 1144 of them exclusively;   512 reflections not yet assigned to a domain

Re-refine initial cell (R), search for next domain (S), quit (Q) or choose
new cell from list (enter number) [S]:

------------------------------------------------------------------------------

Cell for domain  3:    7.690   16.769   10.385    89.81    99.26    90.05

Figure of merit: 0.686   %(0.1):  70.9   %(0.2):  76.0   %(0.3):  79.5

Orientation matrix: -0.00518386  0.02505879  0.08654113
                     0.00825999 -0.05393359  0.04190239
                     0.13138762  0.00440169  0.01652335

Rotated from first domain by 179.8 degrees about
reciprocal axis  0.005  1.000 -0.001  and real axis  0.025  1.000 -0.005

Twin law to convert hkl from first to     -0.999   0.010   0.003
this domain (SHELXL TWIN matrix):          0.051   1.000  -0.009
                                          -0.006  -0.002  -1.001

Maximum deviation from integer index [0.25]:

.p4p or .spin file to write domain to: 13mz025_c.p4p

RLATT color-coding employed in file: 13mz025_c.p4p
White: indexed for first domain
Yellow: intermediate domain (but not in first domain)
Green: current domain (but not in a previous domain)
Red: not yet indexed

 2068 reflections within 0.250 of an integer index assigned to domain  3,
  390 of them exclusively;   122 reflections not yet assigned to a domain

Re-refine initial cell (R), search for next domain (S), quit (Q) or choose
new cell from list (enter number) [S]:
```

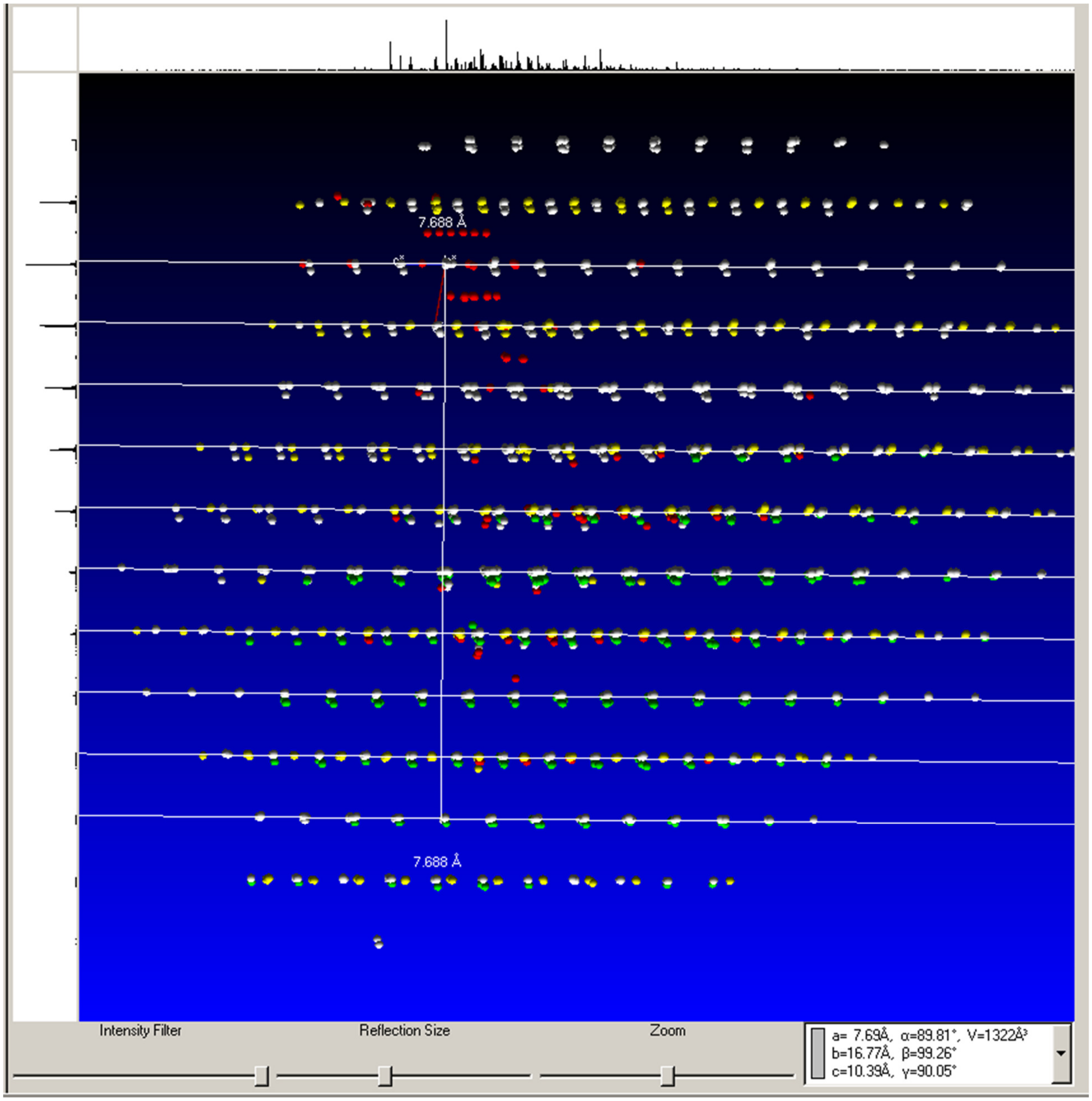
Figure. 21. The Run command window.

- To once again check the results, the data are re-imported into Apex2 as described above. For the example used here, re-inspection via the reciprocal lattice viewer now shows agreement of axis orientations and unit cell dimensions (Figures 18 to 20).



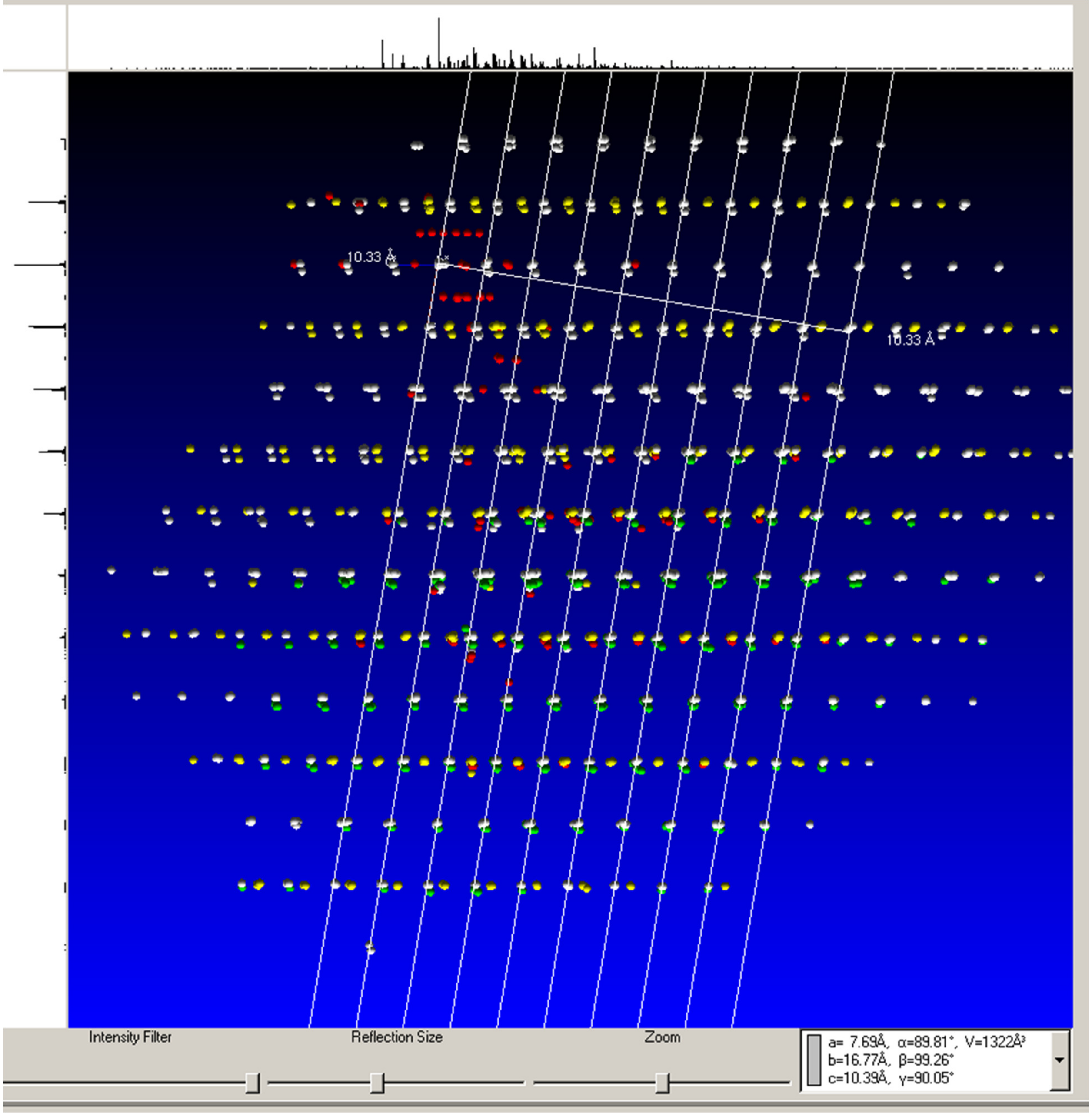
```
a= 7.69Å, α=89.81°, V=1322Å³
b=16.77Å, β=99.26°
c=10.39Å, γ=90.05°
```

Figure. 22. The Reciprocal Lattice window with a non-merohedrally twinned crystal.



a= 7.69Å, α=89.81°, V=1322Å³
b=16.77Å, β=99.26°
c=10.39Å, γ=90.05°

Figure. 23. The Reciprocal Lattice window with a non-merohedrally twinned crystal.

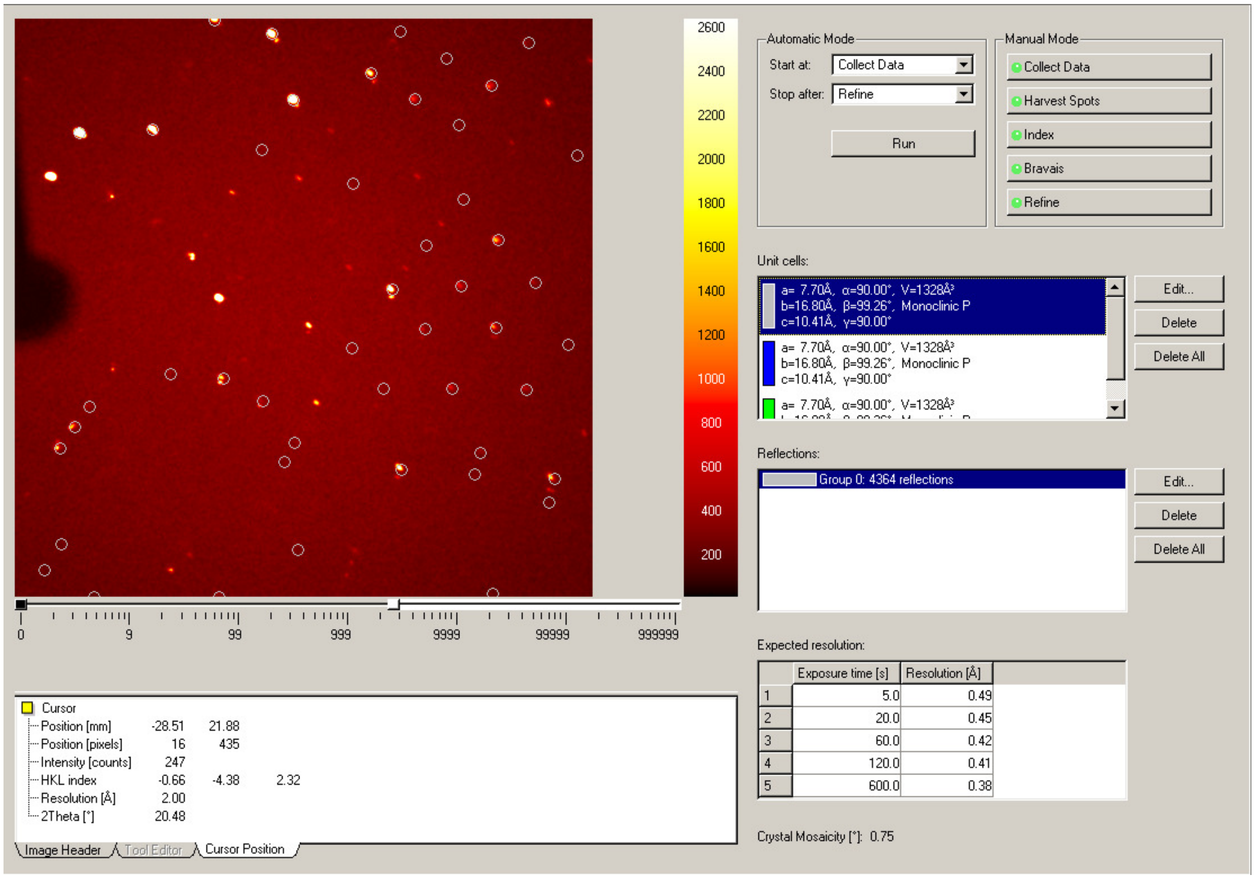


a= 7.69Å, α=89.81°, V=1322Å³
b=16.77Å, β=99.26°
c=10.39Å, γ=90.05°

Figure. 24. The Reciprocal Lattice window with a non-merohedrally twinned crystal.

- To further check the accuracy of the unit cells obtained, go back <Determine Unit Cell>.
- Right click on the diffraction image, select <show overlay> to display predicted spot positions for each moiety

The interface will display the unit cells obtained from Cell Now. Different colours are used by Apex2 for different moieties. For correct solutions, circles should closely match the spots. In the example, every spot corresponds to the position of a reflection of at least one of the three twin moieties.



Start at: Collect Data

Stop after: Refine

Unit cells:

a= 7.70Å, α=90.00°, V=1328Å³
b=16.80Å, β=99.26°, Monoclinic P
c=10.41Å, γ=90.00°

a= 7.70Å, α=90.00°, V=1328Å³
b=16.80Å, β=99.26°, Monoclinic P
c=10.41Å, γ=90.00°

a= 7.70Å, α=90.00°, V=1328Å³

Reflections:

Group 0: 4364 reflections

Expected resolution:

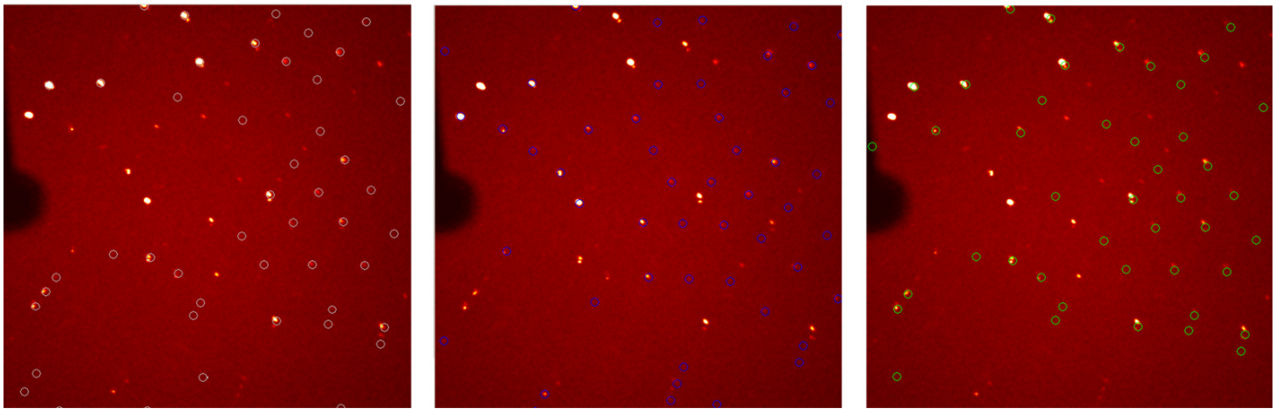
	Exposure time [s]	Resolution [Å]
1	5.0	0.49
2	20.0	0.45
3	60.0	0.42
4	120.0	0.41
5	600.0	0.38

Crystal Mosaicity [°]: 0.75

Cursor			
Position [mm]	-28.51	21.88	
Position [pixels]	16	435	
Intensity [counts]	247		
HKL index	-0.66	-4.38	2.32
Resolution [Å]	2.00		
2Theta [°]	20.48		

Figure. 25. Unit cell window. Predicted spot positions for moiety 1 shown.

The diffraction data acquired from a twinned crystal, diffraction spots from different domains are observed simultaneously.



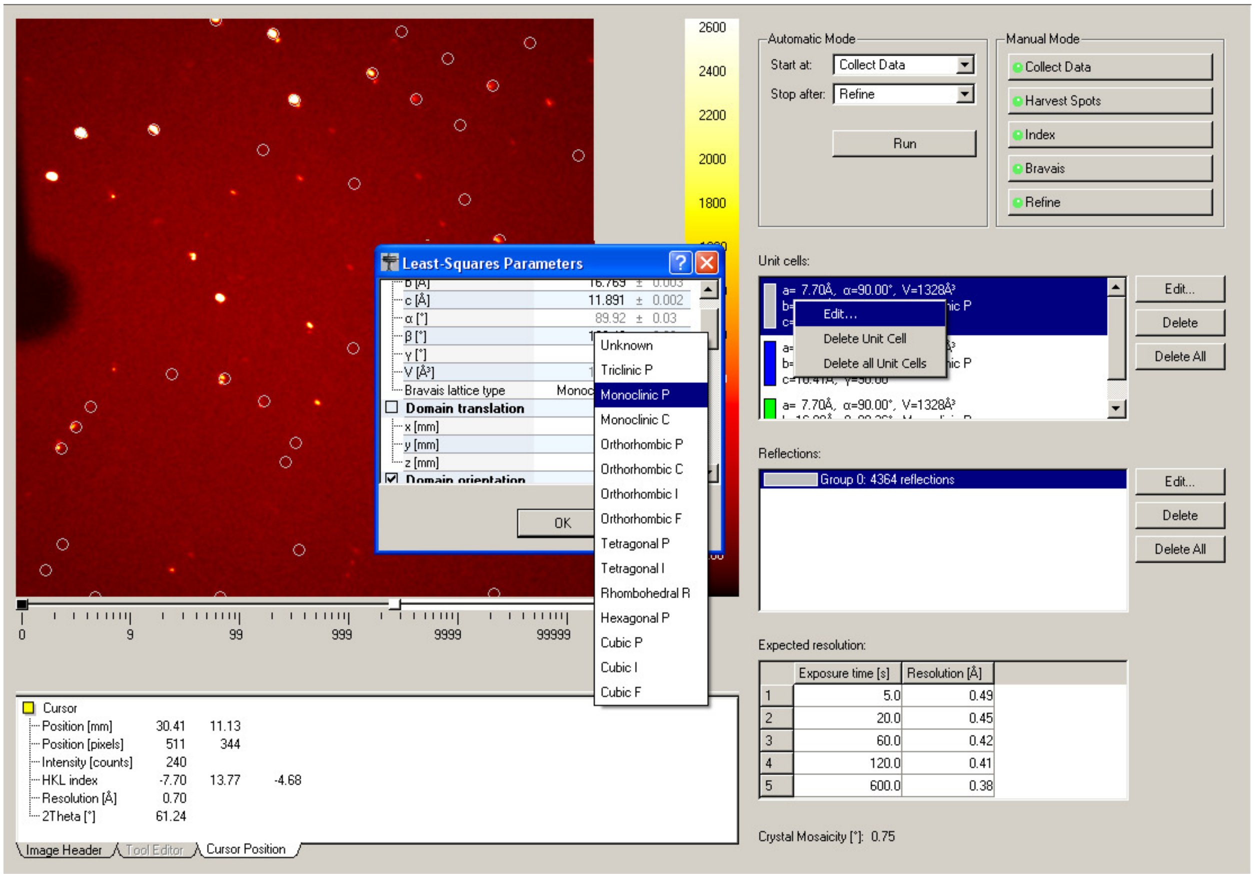
Component 1 Component 2 Component 3

Figure. 26. Comparison of the three components.

If predicted and observed the spot positions do not match closely perform a least square refinement for each of the unit cells.

The crystal symmetry such as Bravais setting and lattice centering are not imported from Cell Now into Apex2 and have to be set by hand.

- Right click on component a ⇒ click <Edit>, then choose the expected crystal symmetry from <Bravais lattice type> (here: monoclinic primitive as two of the angles are almost 90 degrees).
- Repeat for the other components.
  (Crystal symmetry can be also set using the <Bravais> option in the main Unit cell determination window; the lattice centering has to be set manually in all cases).



Least-Squares Parameters:

c [Å]	11.891 ± 0.002
α [°]	89.92 ± 0.03
Bravais lattice type	Monoc
☐ Domain translation	
x [mm]	
y [mm]	
z [mm]	
☑ Domain orientation	

Unit cells:

a= 7.70Å, α=90.00°, V=1328Å³

c=10.41Å, γ=90.00°

a= 7.70Å, α=90.00°, V=1328Å³

Reflections:

Group 0: 4364 reflections

Expected resolution:

	Exposure time [s]	Resolution [Å]
1	5.0	0.49
2	20.0	0.45
3	60.0	0.42
4	120.0	0.41
5	600.0	0.38

Crystal Mosaicity [°]: 0.75

Cursor			
Position [mm]	30.41	11.13	
Position [pixels]	511	344	
Intensity [counts]	240		
HKL index	-7.70	13.77	-4.68
Resolution [Å]	0.70		
2Theta [°]	61.24		

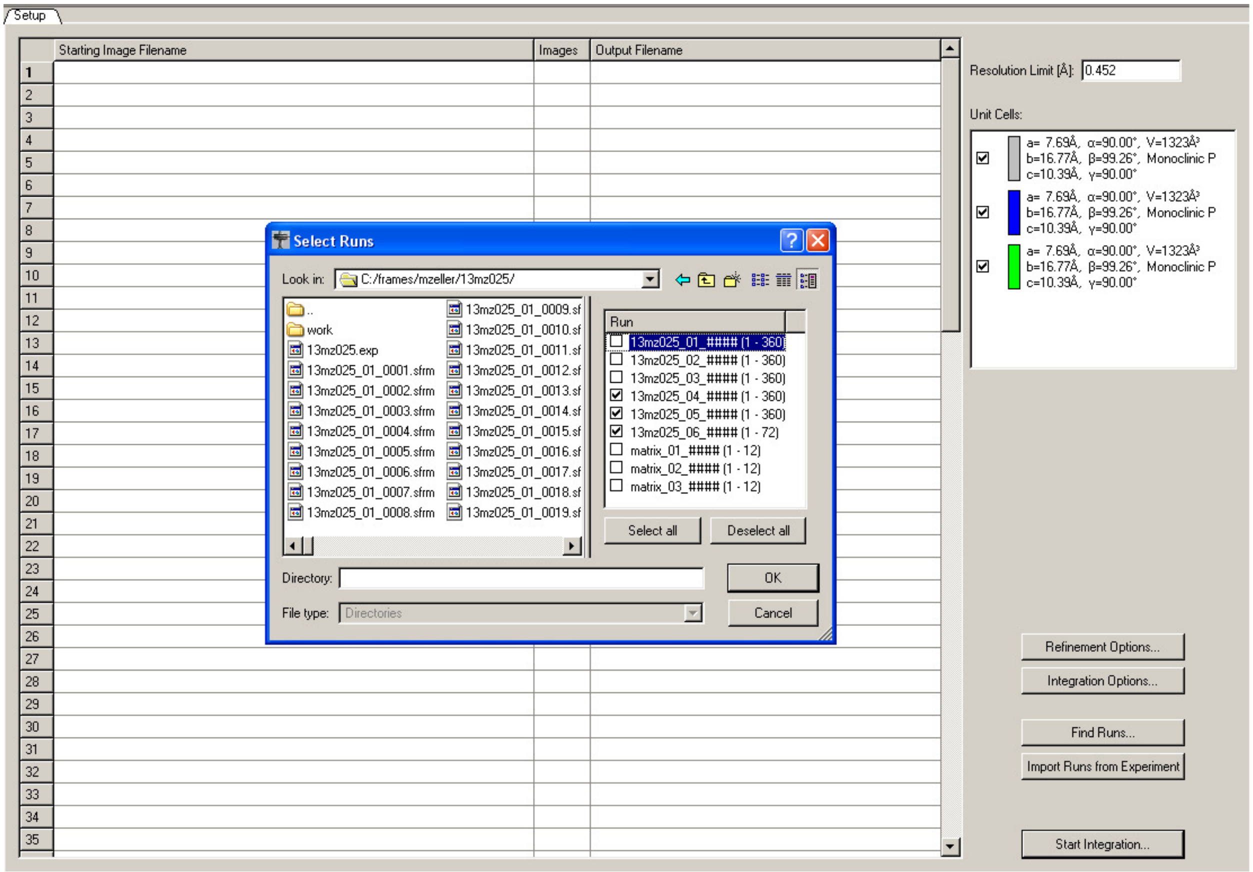
Figure. 27. Unit cell window.

If no data collection has been started yet, proceed to <Collect> and set up a sufficiently large data collection. For twinned structures, a higher redundancy (multiplicity of observation, MoO) is usually required for meaningful multi-scan absorption correction than for untwinned similar systems. Set up a hemisphere or sphere data collection for any but the highest symmetry systems.

It is advisable to check completeness of the dataset via a trial solution and refinement before terminating a data collection or dismounting the crystal. Additional data collection runs might be required due to rejected data[1] or need for better absorption correction!

When enough data for a trial solution and integration are collected, proceed to <Integrate> (do not yet stop the data collection!).

- Click on <Find Runs>.
- If the beam stop has to be mapped for all or some of the collected data, integrate these first. Check these data runs, then click <OK>.



Look in: C:/frames/mzeller/13mz025/

Run
☐ 13mz025_01_#### (1 - 360)
☐ 13mz025_02_#### (1 - 360)
☐ 13mz025_03_#### (1 - 360)
☑ 13mz025_04_#### (1 - 360)
☑ 13mz025_05_#### (1 - 360)
☑ 13mz025_06_#### (1 - 72)
☐ matrix_01_#### (1 - 12)
☐ matrix_02_#### (1 - 12)
☐ matrix_03_#### (1 - 12)

Directory:
File type: Directories

Resolution Limit [Å]: 0.452

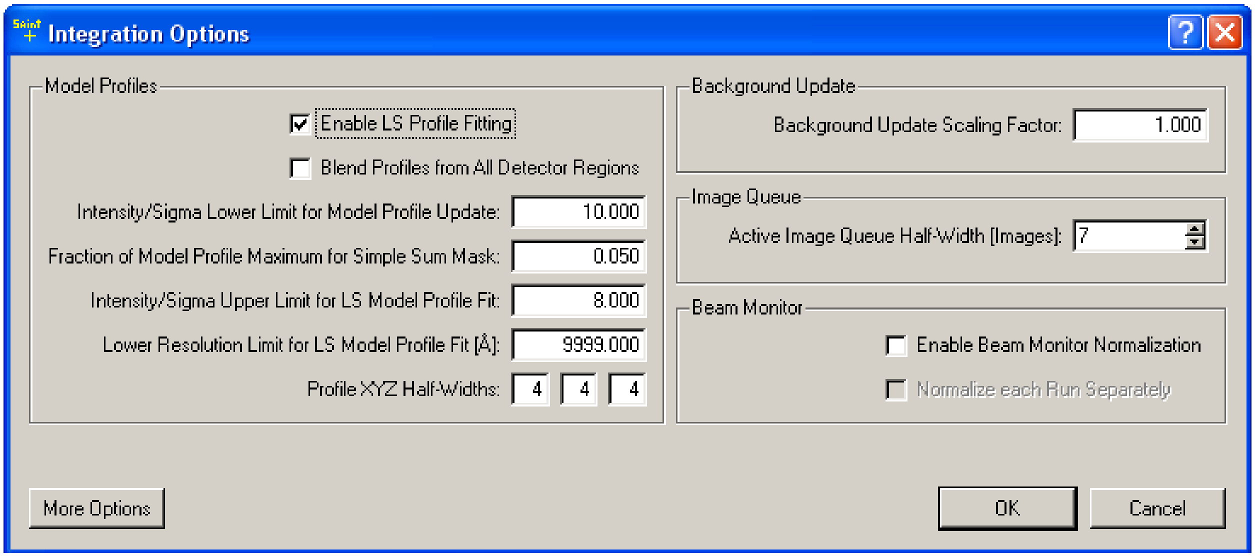
Unit Cells:
☑ a= 7.69Å, α=90.00°, V=1323Å³ b=16.77Å, β=99.26°, Monoclinic P c=10.39Å, γ=90.00°
☑ a= 7.69Å, α=90.00°, V=1323Å³ b=16.77Å, β=99.26°, Monoclinic P c=10.39Å, γ=90.00°
☑ a= 7.69Å, α=90.00°, V=1323Å³ b=16.77Å, β=99.26°, Monoclinic P c=10.39Å, γ=90.00°

---

[1] A common problem with rejected data in Apex2 involves excessive overlap of reflections in a “chain of pearls” fashion. If reflections from different twin moieties that successively overlap exceed the “maximum queue size” set in SAINT (the integration program used in Apex2), the reflections are rejected by SAINT as the intensities cannot be accurately measured any more. The settings for “maximum queue size” and other parameters can be edited in SAINT so that fewer reflections are rejected, but usually at the expense of data quality. In such cases, the best approach usually involves to collect more data, e.g. a full sphere of data for even a high symmetry crystal system. For low symmetry cases sometimes no complete data set can be obtained.

Figure. 28. Integration Start Window with Fine Runs window open.

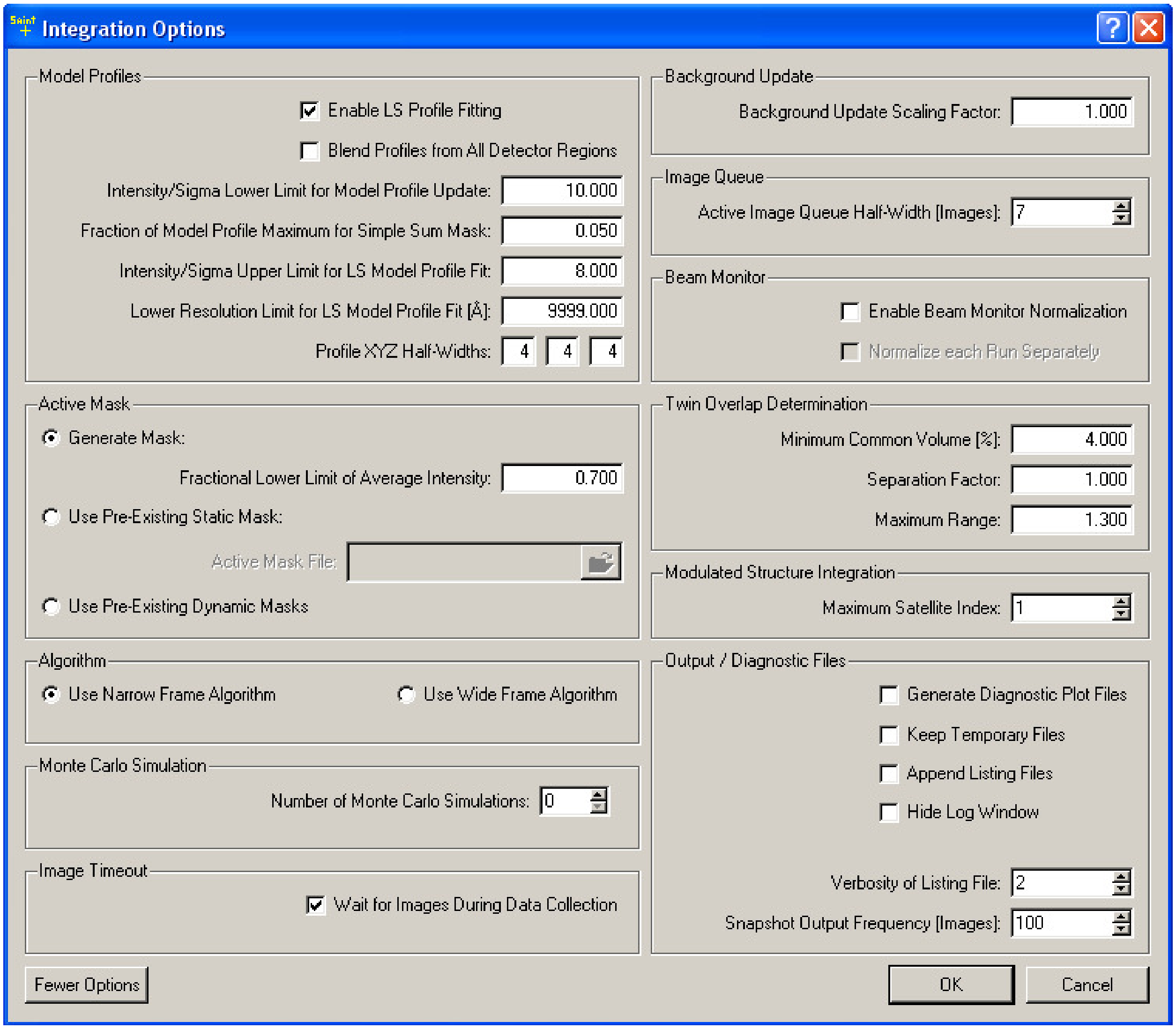
- Click <Integrate Options> ⇒ click <More Options>.



☑ Enable LS Profile Fitting
☐ Blend Profiles from All Detector Regions
Intensity/Sigma Lower Limit for Model Profile Update: 10.000
Fraction of Model Profile Maximum for Simple Sum Mask: 0.050
Intensity/Sigma Upper Limit for LS Model Profile Fit: 8.000
Lower Resolution Limit for LS Model Profile Fit [Å]: 9999.000
Profile XYZ Half-Widths: 4 4 4
Background Update Scaling Factor: 1.000
Active Image Queue Half-Width [Images]: 7
☐ Enable Beam Monitor Normalization
☐ Normalize each Run Separately

Figure. 29. Integration Option window.

- Change Fractional Lower Limit of Average Intensity of Generate Mask to e.g. 0.700.



Integration Options

Model Profiles

☑ Enable LS Profile Fitting

☐ Blend Profiles from All Detector Regions

Intensity/Sigma Lower Limit for Model Profile Update: 10.000

Fraction of Model Profile Maximum for Simple Sum Mask: 0.050

Intensity/Sigma Upper Limit for LS Model Profile Fit: 8.000

Lower Resolution Limit for LS Model Profile Fit [Å]: 9999.000

Profile XYZ Half-Widths: 4 4 4

Active Mask

● Generate Mask:

Fractional Lower Limit of Average Intensity: 0.700

○ Use Pre-Existing Static Mask:

Active Mask File:

○ Use Pre-Existing Dynamic Masks

Algorithm

● Use Narrow Frame Algorithm ○ Use Wide Frame Algorithm

Monte Carlo Simulation

Number of Monte Carlo Simulations: 0

Image Timeout

☑ Wait for Images During Data Collection

Background Update

Background Update Scaling Factor: 1.000

Image Queue

Active Image Queue Half-Width [Images]: 7

Beam Monitor

☐ Enable Beam Monitor Normalization

☐ Normalize each Run Separately

Twin Overlap Determination

Minimum Common Volume [%]: 4.000

Separation Factor: 1.000

Maximum Range: 1.300

Modulated Structure Integration

Maximum Satellite Index: 1

Output / Diagnostic Files

☐ Generate Diagnostic Plot Files

☐ Keep Temporary Files

☐ Append Listing Files

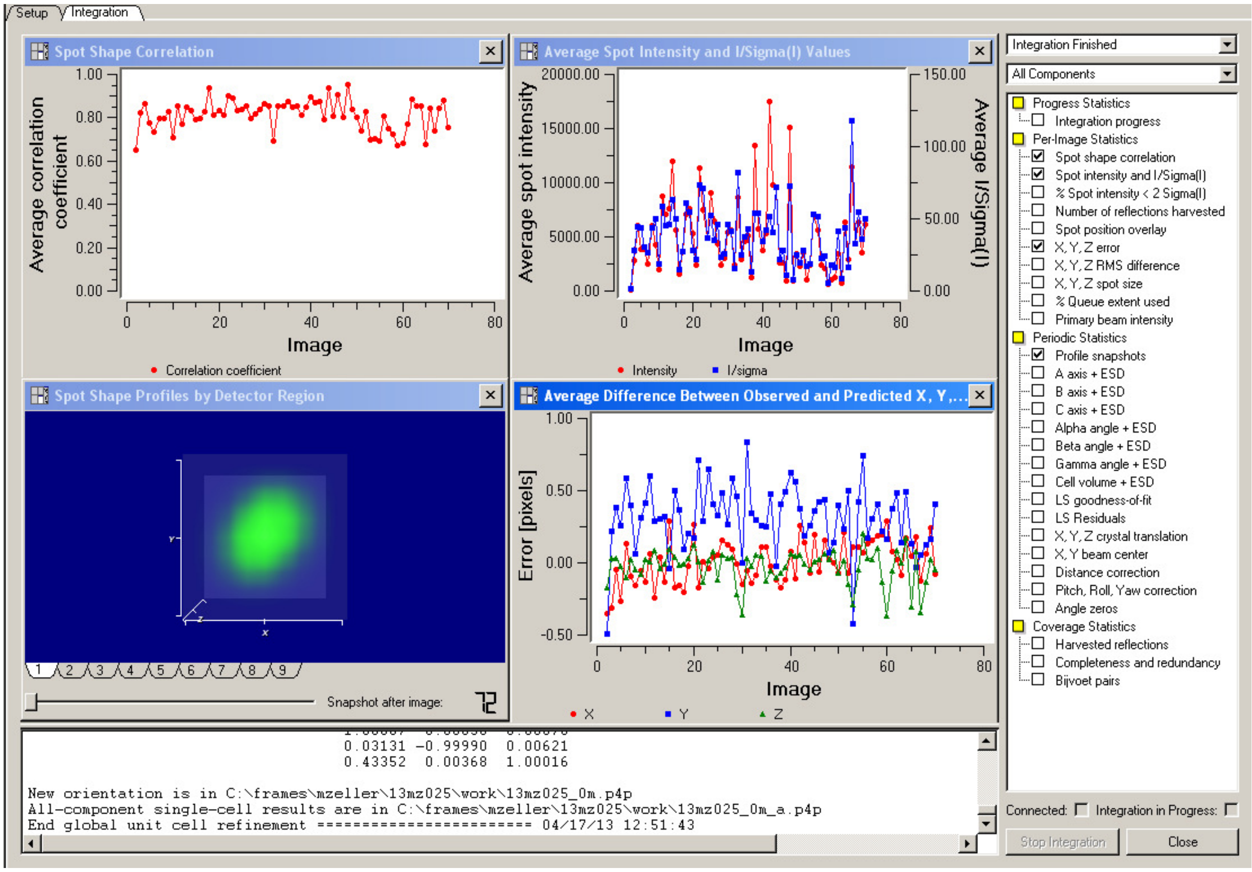
☐ Hide Log Window

Verbosity of Listing File: 2

Snapshot Output Frequency [Images]: 100

Figure. 30. Integration Option window.

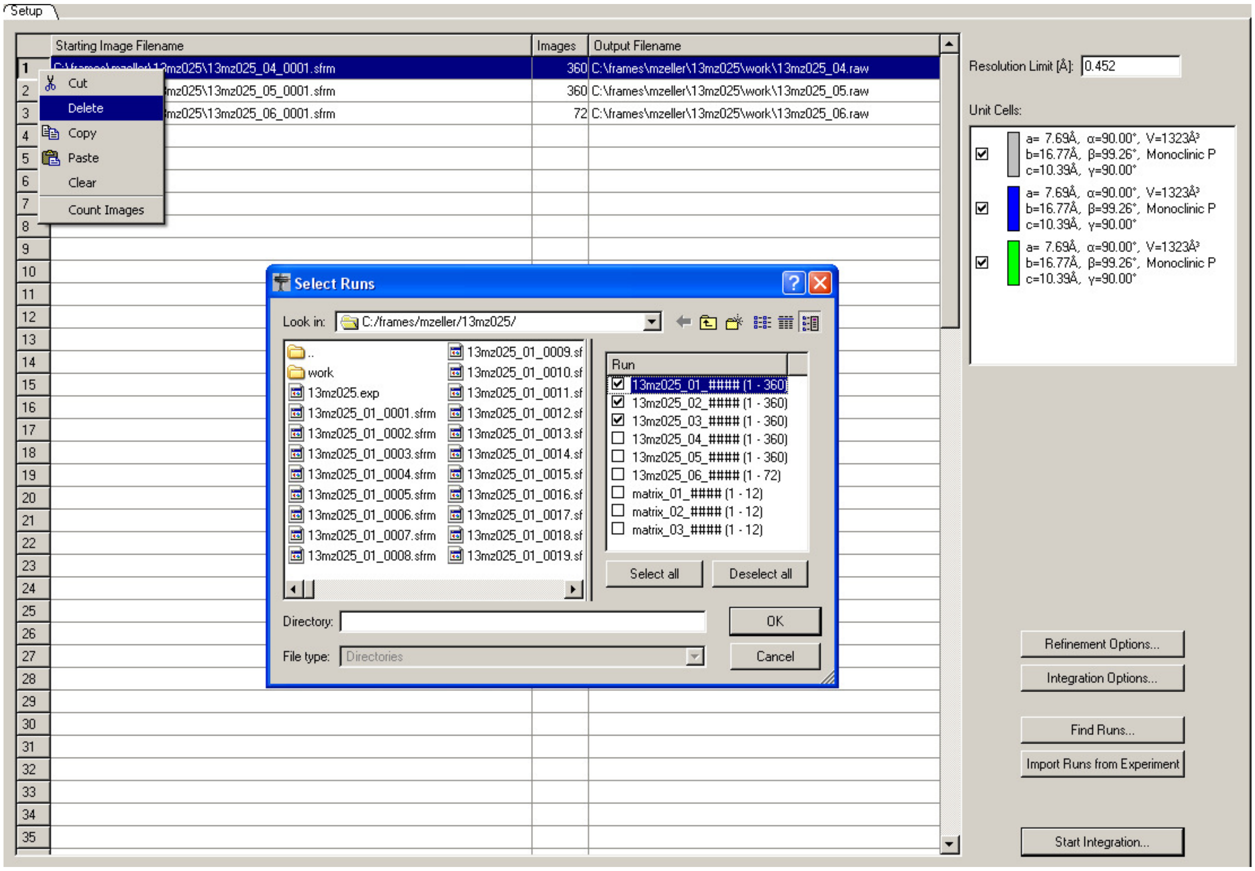
- Click on <Start integration> button.



```
                                                 0.03131 -0.99990  0.00621
                                                 0.43352  0.00368  1.00016

New orientation is in C:\frames\mzeller\13mz025\work\13mz025_0m.p4p
All-component single-cell results are in C:\frames\mzeller\13mz025\work\13mz025_0m_a.p4p
End global unit cell refinement ======================== 04/17/13 12:51:43
```

Figure. 31. Integration Option window with active Integration running.

- Click on <Close>.
- Delete the old three file run, Click on <Find Run>, then choose the first three row, then click on <OK>.



	Starting Image Filename	Images	Output Filename
1	C:\frames\mzeller\13mz025\13mz025_04_0001.sfrm	360	C:\frames\mzeller\13mz025\work\13mz025_04.raw
2	mz025\13mz025_05_0001.sfrm	360	C:\frames\mzeller\13mz025\work\13mz025_05.raw
3	mz025\13mz025_06_0001.sfrm	72	C:\frames\mzeller\13mz025\work\13mz025_06.raw

Resolution Limit [Å]: 0.452

Unit Cells:

☑ a= 7.69Å, α=90.00°, V=1323Å³ b=16.77Å, β=99.26°, Monoclinic P c=10.39Å, γ=90.00°

☑ a= 7.69Å, α=90.00°, V=1323Å³ b=16.77Å, β=99.26°, Monoclinic P c=10.39Å, γ=90.00°

☑ a= 7.69Å, α=90.00°, V=1323Å³ b=16.77Å, β=99.26°, Monoclinic P c=10.39Å, γ=90.00°

Look in: C:/frames/mzeller/13mz025/

..
work
13mz025.exp
13mz025_01_0001.sfrm
13mz025_01_0002.sfrm
13mz025_01_0003.sfrm
13mz025_01_0004.sfrm
13mz025_01_0005.sfrm
13mz025_01_0006.sfrm
13mz025_01_0007.sfrm
13mz025_01_0008.sfrm
13mz025_01_0009.sf
13mz025_01_0010.sf
13mz025_01_0011.sf
13mz025_01_0012.sf
13mz025_01_0013.sf
13mz025_01_0014.sf
13mz025_01_0015.sf
13mz025_01_0016.sf
13mz025_01_0017.sf
13mz025_01_0018.sf
13mz025_01_0019.sf

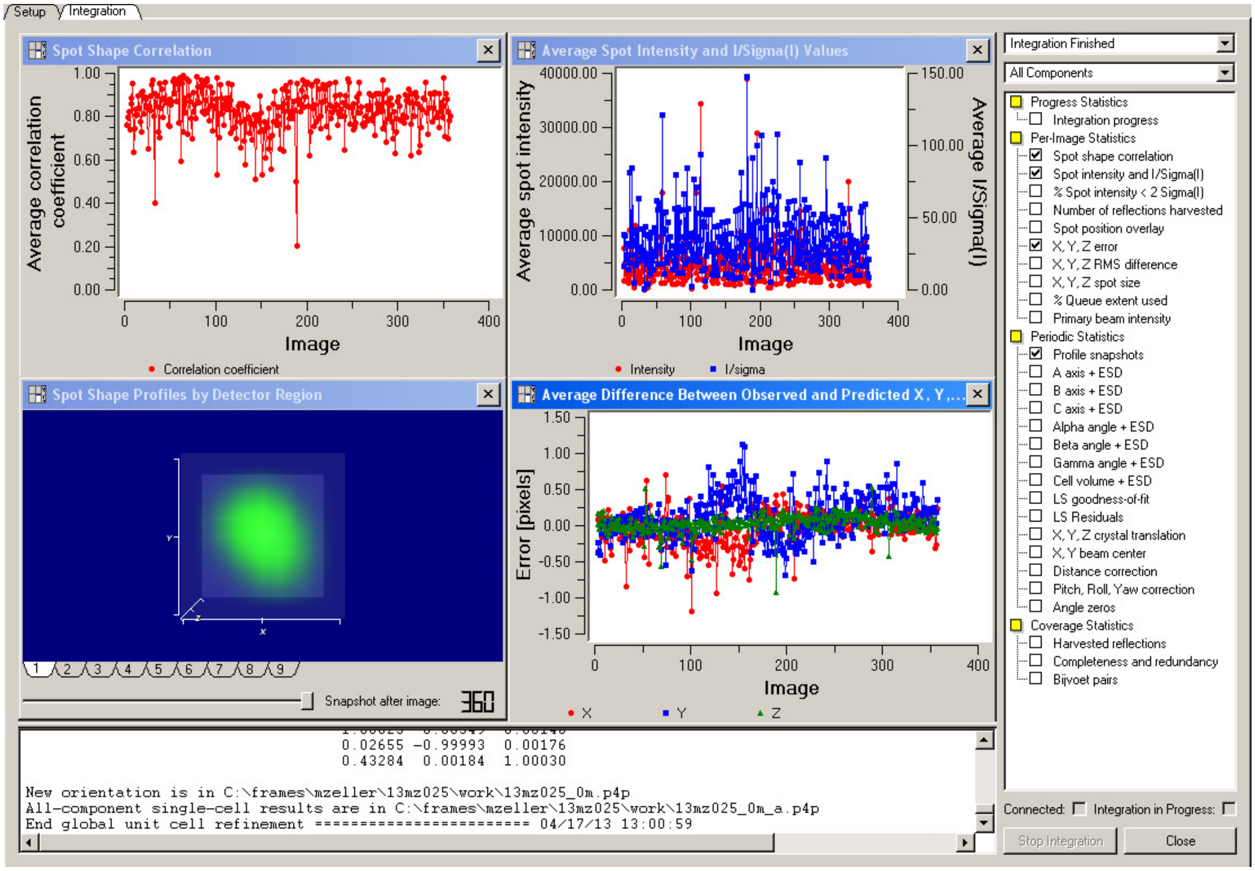
Run
☑ 13mz025_01_#### (1 - 360)
☑ 13mz025_02_#### (1 - 360)
☑ 13mz025_03_#### (1 - 360)
☐ 13mz025_04_#### (1 - 360)
☐ 13mz025_05_#### (1 - 360)
☐ 13mz025_06_#### (1 - 72)
☐ matrix_01_#### (1 - 12)
☐ matrix_02_#### (1 - 12)
☐ matrix_03_#### (1 - 12)

Directory:

File type: Directories

Figure. 32. Integration Start Window with Fine Runs window open.

- Click <Integrate options> ⇒ click on <More Option>.
- Change Fractional Lower Limit of Average Intensity of Generate Mask to 0.000.
- Click on <Start Integration>.



- Integration Finished
- All Components

- ☐ Progress Statistics
  - ☐ Integration progress
- ☐ Per-Image Statistics
  - ☑ Spot shape correlation
  - ☑ Spot intensity and I/Sigma(I)
  - ☐ % Spot intensity < 2 Sigma(I)
  - ☐ Number of reflections harvested
  - ☐ Spot position overlay
  - ☑ X, Y, Z error
  - ☐ X, Y, Z RMS difference
  - ☐ X, Y, Z spot size
  - ☐ % Queue extent used
  - ☐ Primary beam intensity
- ☐ Periodic Statistics
  - ☑ Profile snapshots
  - ☐ A axis + ESD
  - ☐ B axis + ESD
  - ☐ C axis + ESD
  - ☐ Alpha angle + ESD
  - ☐ Beta angle + ESD
  - ☐ Gamma angle + ESD
  - ☐ Cell volume + ESD
  - ☐ LS goodness-of-fit
  - ☐ LS Residuals
  - ☐ X, Y, Z crystal translation
  - ☐ X, Y beam center
  - ☐ Distance correction
  - ☐ Pitch, Roll, Yaw correction
  - ☐ Angle zeros
- ☐ Coverage Statistics
  - ☐ Harvested reflections
  - ☐ Completeness and redundancy
  - ☐ Bijvoet pairs

Connected: ☐ Integration in Progress: ☐

```
                                 0.02655 -0.99993  0.00176
                                 0.43284  0.00184  1.00030

New orientation is in C:\frames\mzeller\13mz025\work\13mz025_0m.p4p
All-component single-cell results are in C:\frames\mzeller\13mz025\work\13mz025_0m_a.p4p
End global unit cell refinement ======================== 04/17/13 13:00:59
```

Figure. 33. Integration Option window with active Integration running.

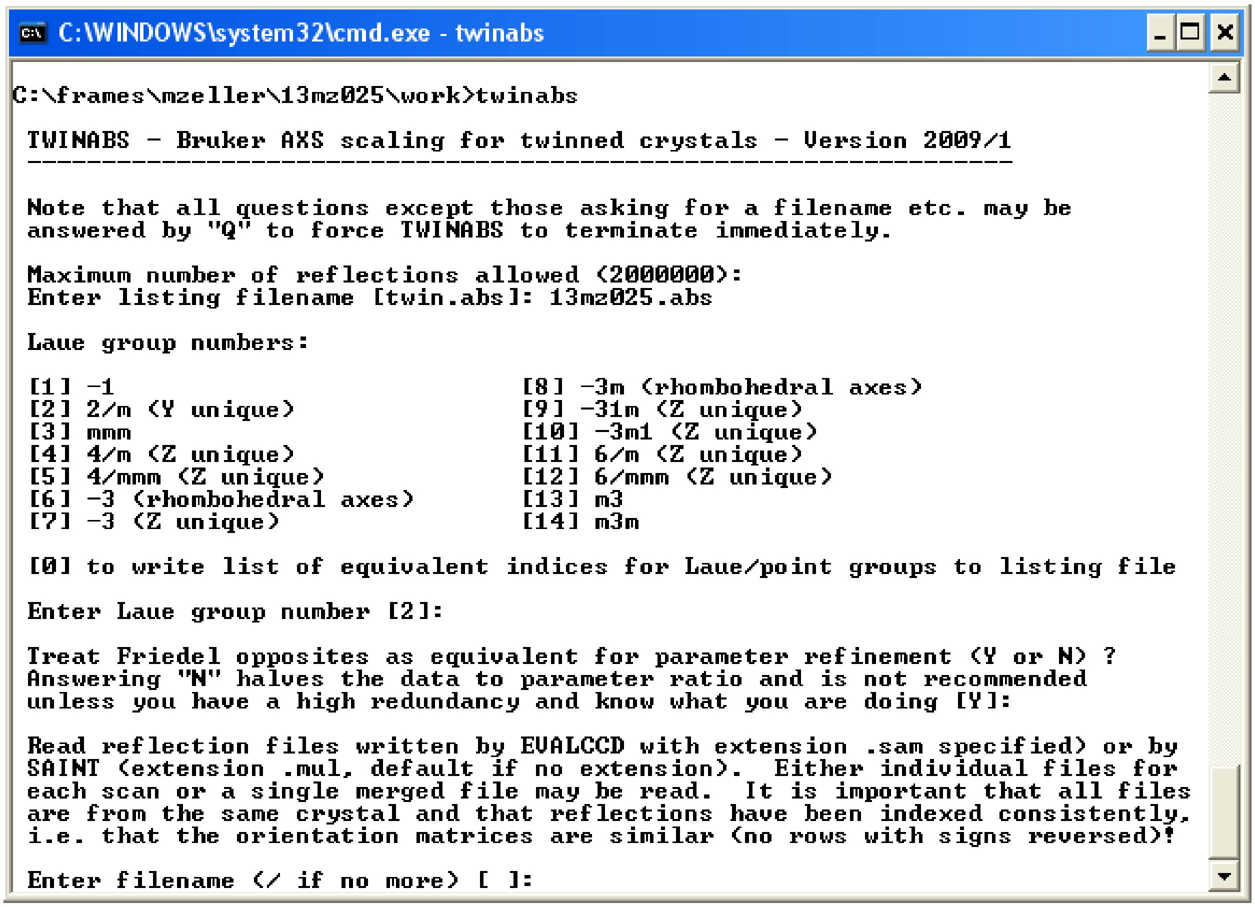
- Click on <Close>.

The results of the integration process:

- <Spots Shape Correlation>
   There are some spots of correlation coefficient display low level that mean the orientation matrix are not described by diffraction pattern.
- <Spot Shape Profiles by Detector Region>
  Split spot shapes display......... indicate to diffraction of twin crystal.
- <Average Difference>
  The errors in X and Y are larger than 1 pixel. The varies during the integration of run that show some of the problem.

TWINABS.

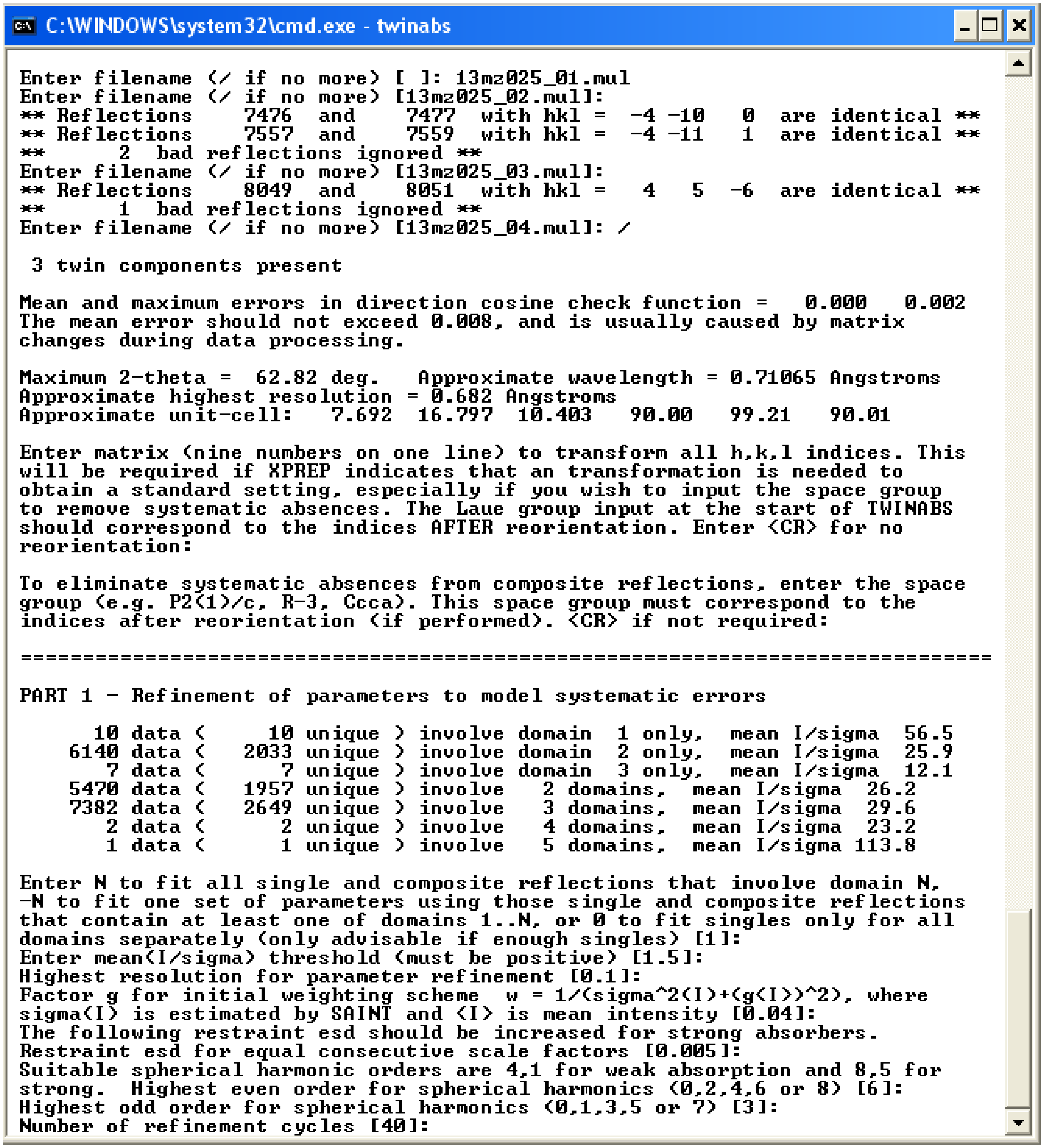
- Open run command ⇒Type <twinabs>, Enter.
- Give name 13mz025.abs, then Enter.
- Choose Laue group numbers:[2], then Enter and answer Y to the next question.



```
C:\frames\mzeller\13mz025\work>twinabs

 TWINABS - Bruker AXS scaling for twinned crystals - Version 2009/1
 ------------------------------------------------------------------

 Note that all questions except those asking for a filename etc. may be
 answered by "Q" to force TWINABS to terminate immediately.

 Maximum number of reflections allowed (2000000):
 Enter listing filename [twin.abs]: 13mz025.abs

 Laue group numbers:

 [1] -1                              [8] -3m (rhombohedral axes)
 [2] 2/m (Y unique)                  [9] -31m (Z unique)
 [3] mmm                             [10] -3m1 (Z unique)
 [4] 4/m (Z unique)                  [11] 6/m (Z unique)
 [5] 4/mmm (Z unique)                [12] 6/mmm (Z unique)
 [6] -3 (rhombohedral axes)          [13] m3
 [7] -3 (Z unique)                   [14] m3m

 [0] to write list of equivalent indices for Laue/point groups to listing file

 Enter Laue group number [2]:

 Treat Friedel opposites as equivalent for parameter refinement (Y or N) ?
 Answering "N" halves the data to parameter ratio and is not recommended
 unless you have a high redundancy and know what you are doing [Y]:

 Read reflection files written by EVALCCD with extension .sam specified) or by
 SAINT (extension .mul, default if no extension).  Either individual files for
 each scan or a single merged file may be read.  It is important that all files
 are from the same crystal and that reflections have been indexed consistently,
 i.e. that the orientation matrices are similar (no rows with signs reversed)!

 Enter filename (/ if no more) [ ]:
```

Figure. 34. The Run command window.

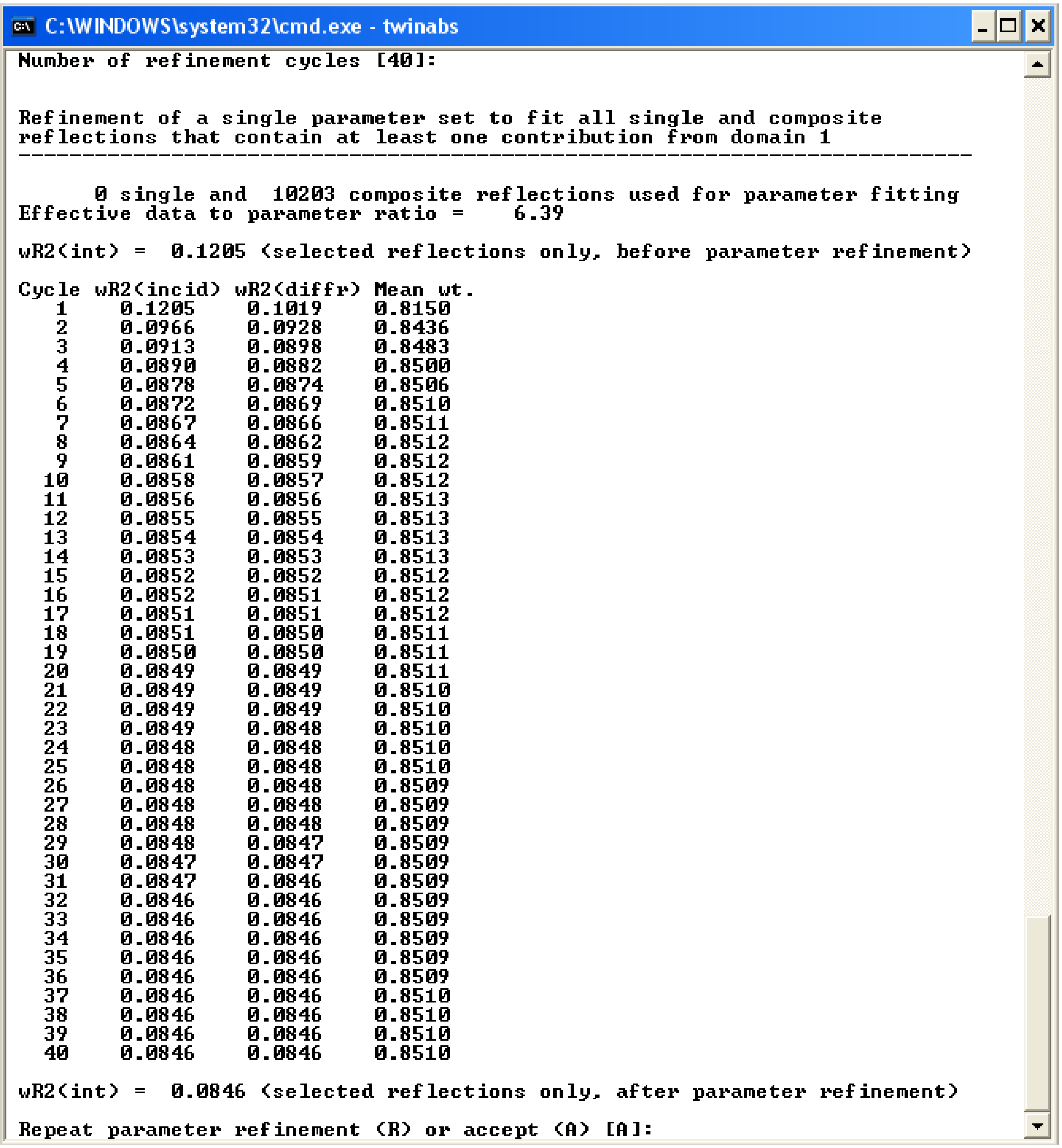
- Enter filename: 13mz023_01.mul for the first data set.
- Enter the rest one by one until you see 1 bad reflections ignored: type / and enter.



```
Enter filename (/ if no more) [ ]: 13mz025_01.mul
Enter filename (/ if no more) [13mz025_02.mul]:
** Reflections    7476  and    7477  with hkl =  -4 -10   0  are identical **
** Reflections    7557  and    7559  with hkl =  -4 -11   1  are identical **
**      2  bad reflections ignored **
Enter filename (/ if no more) [13mz025_03.mul]:
** Reflections    8049  and    8051  with hkl =   4   5  -6  are identical **
**      1  bad reflections ignored **
Enter filename (/ if no more) [13mz025_04.mul]: /

 3 twin components present

Mean and maximum errors in direction cosine check function =   0.000   0.002
The mean error should not exceed 0.008, and is usually caused by matrix
changes during data processing.

Maximum 2-theta =  62.82 deg.    Approximate wavelength = 0.71065 Angstroms
Approximate highest resolution = 0.682 Angstroms
Approximate unit-cell:   7.692  16.797  10.403   90.00   99.21   90.01

Enter matrix (nine numbers on one line) to transform all h,k,l indices. This
will be required if XPREP indicates that an transformation is needed to
obtain a standard setting, especially if you wish to input the space group
to remove systematic absences. The Laue group input at the start of TWINABS
should correspond to the indices AFTER reorientation. Enter <CR> for no
reorientation:

To eliminate systematic absences from composite reflections, enter the space
group (e.g. P2(1)/c, R-3, Ccca). This space group must correspond to the
indices after reorientation (if performed). <CR> if not required:

==============================================================================

PART 1 - Refinement of parameters to model systematic errors

      10 data (     10 unique ) involve domain  1 only,  mean I/sigma  56.5
    6140 data (   2033 unique ) involve domain  2 only,  mean I/sigma  25.9
       7 data (      7 unique ) involve domain  3 only,  mean I/sigma  12.1
    5470 data (   1957 unique ) involve   2 domains,  mean I/sigma  26.2
    7382 data (   2649 unique ) involve   3 domains,  mean I/sigma  29.6
       2 data (      2 unique ) involve   4 domains,  mean I/sigma  23.2
       1 data (      1 unique ) involve   5 domains,  mean I/sigma 113.8

Enter N to fit all single and composite reflections that involve domain N,
-N to fit one set of parameters using those single and composite reflections
that contain at least one of domains 1..N, or 0 to fit singles only for all
domains separately (only advisable if enough singles) [1]:
Enter mean(I/sigma) threshold (must be positive) [1.5]:
Highest resolution for parameter refinement [0.1]:
Factor g for initial weighting scheme  w = 1/(sigma^2(I)+(g<I>)^2), where
sigma(I) is estimated by SAINT and <I> is mean intensity [0.04]:
The following restraint esd should be increased for strong absorbers.
Restraint esd for equal consecutive scale factors [0.005]:
Suitable spherical harmonic orders are 4,1 for weak absorption and 8,5 for
strong.  Highest even order for spherical harmonics (0,2,4,6 or 8) [6]:
Highest odd order for spherical harmonics (0,1,3,5 or 7) [3]:
Number of refinement cycles [40]:
```

Figure. 35. The Run command window.

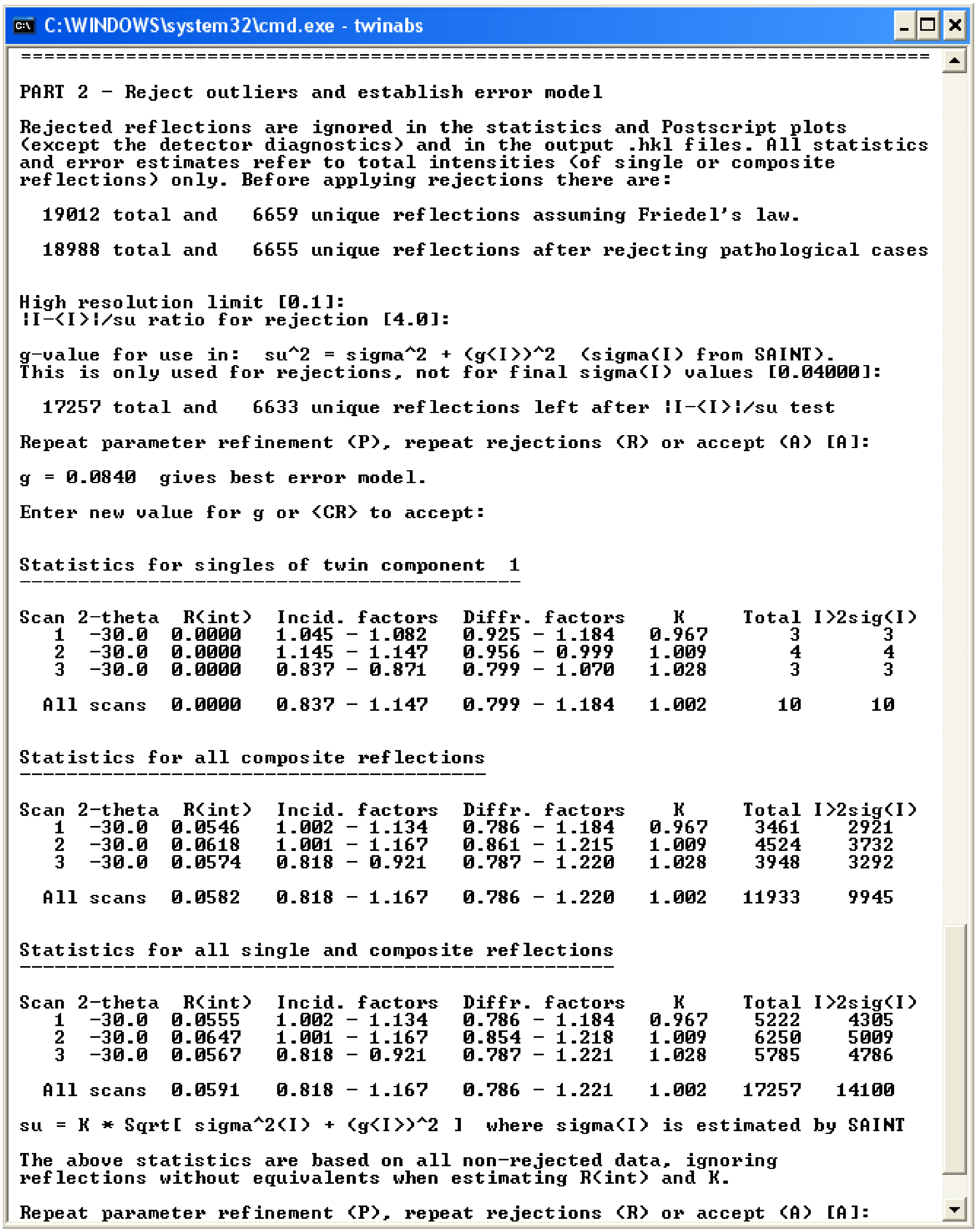
- go to the refinement step, and accept the results of wR2(int) = 0.0846. Do not accept anything over 0.2.



```
Number of refinement cycles [40]:


Refinement of a single parameter set to fit all single and composite
reflections that contain at least one contribution from domain 1
----------------------------------------------------------------------------

      0 single and  10203 composite reflections used for parameter fitting
Effective data to parameter ratio =    6.39

wR2(int) =  0.1205 (selected reflections only, before parameter refinement)

Cycle wR2(incid) wR2(diffr) Mean wt.
   1     0.1205     0.1019     0.8150
   2     0.0966     0.0928     0.8436
   3     0.0913     0.0898     0.8483
   4     0.0890     0.0882     0.8500
   5     0.0878     0.0874     0.8506
   6     0.0872     0.0869     0.8510
   7     0.0867     0.0866     0.8511
   8     0.0864     0.0862     0.8512
   9     0.0861     0.0859     0.8512
  10     0.0858     0.0857     0.8512
  11     0.0856     0.0856     0.8513
  12     0.0855     0.0855     0.8513
  13     0.0854     0.0854     0.8513
  14     0.0853     0.0853     0.8513
  15     0.0852     0.0852     0.8512
  16     0.0852     0.0851     0.8512
  17     0.0851     0.0851     0.8512
  18     0.0851     0.0850     0.8511
  19     0.0850     0.0850     0.8511
  20     0.0849     0.0849     0.8511
  21     0.0849     0.0849     0.8510
  22     0.0849     0.0849     0.8510
  23     0.0849     0.0848     0.8510
  24     0.0848     0.0848     0.8510
  25     0.0848     0.0848     0.8510
  26     0.0848     0.0848     0.8509
  27     0.0848     0.0848     0.8509
  28     0.0848     0.0848     0.8509
  29     0.0848     0.0847     0.8509
  30     0.0847     0.0847     0.8509
  31     0.0847     0.0846     0.8509
  32     0.0846     0.0846     0.8509
  33     0.0846     0.0846     0.8509
  34     0.0846     0.0846     0.8509
  35     0.0846     0.0846     0.8509
  36     0.0846     0.0846     0.8509
  37     0.0846     0.0846     0.8510
  38     0.0846     0.0846     0.8510
  39     0.0846     0.0846     0.8510
  40     0.0846     0.0846     0.8510

wR2(int) =  0.0846 (selected reflections only, after parameter refinement)

Repeat parameter refinement (R) or accept (A) [A]:
```

Figure. 36. The Run command window.

Go to the outlier rejection step.

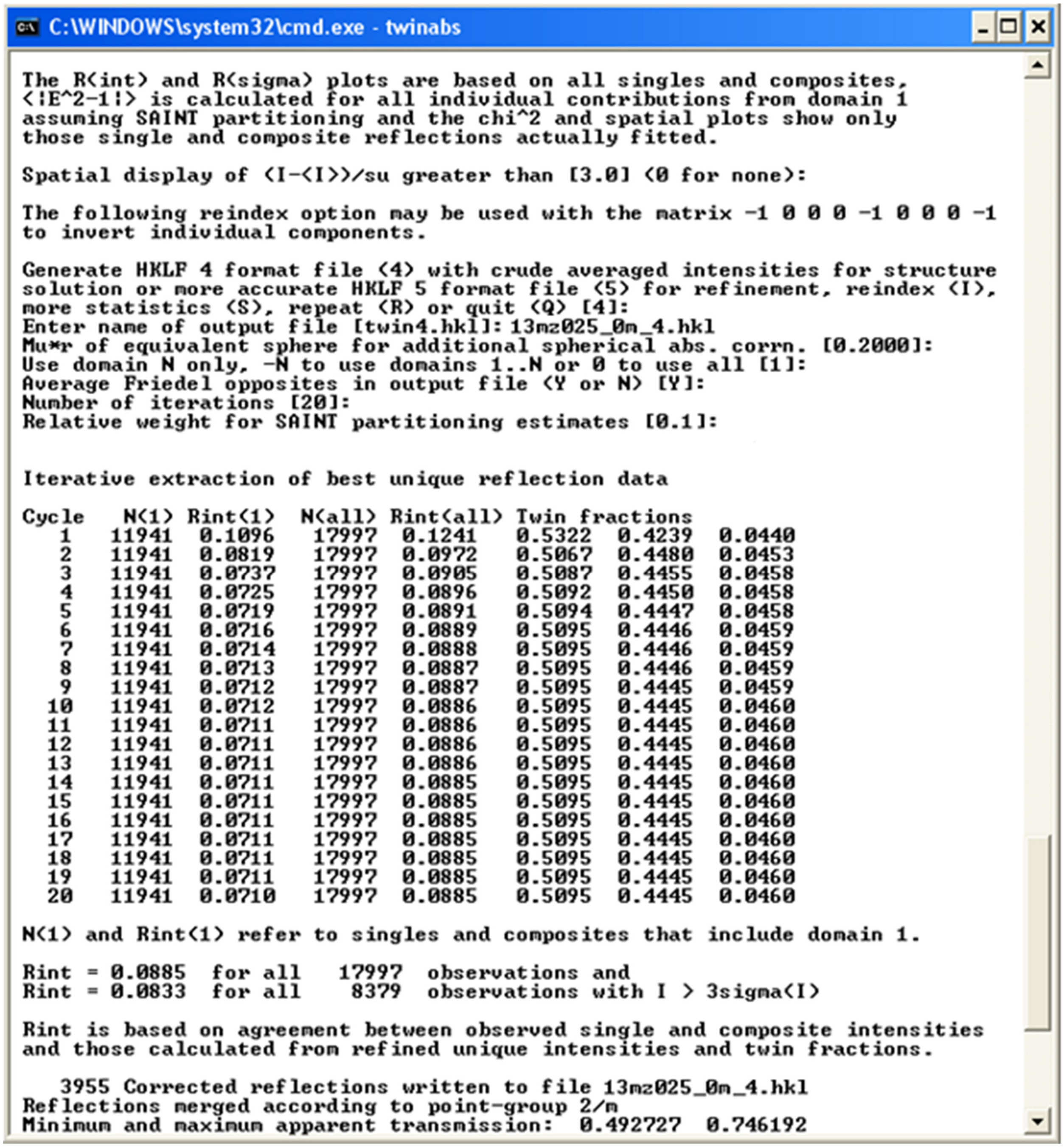


```
==============================================================================

PART 2 - Reject outliers and establish error model

Rejected reflections are ignored in the statistics and Postscript plots
(except the detector diagnostics) and in the output .hkl files. All statistics
and error estimates refer to total intensities (of single or composite
reflections) only. Before applying rejections there are:

  19012 total and   6659 unique reflections assuming Friedel's law.

  18988 total and   6655 unique reflections after rejecting pathological cases


High resolution limit [0.1]:
|I-<I>|/su ratio for rejection [4.0]:

g-value for use in:  su^2 = sigma^2 + (g<I>)^2  (sigma(I) from SAINT).
This is only used for rejections, not for final sigma(I) values [0.04000]:

  17257 total and   6633 unique reflections left after |I-<I>|/su test

Repeat parameter refinement (P), repeat rejections (R) or accept (A) [A]:

g = 0.0840  gives best error model.

Enter new value for g or <CR> to accept:


Statistics for singles of twin component  1
-------------------------------------------

Scan 2-theta  R(int)  Incid. factors  Diffr. factors    K     Total I>2sig(I)
   1  -30.0  0.0000   1.045 - 1.082   0.925 - 1.184   0.967       3       3
   2  -30.0  0.0000   1.145 - 1.147   0.956 - 0.999   1.009       4       4
   3  -30.0  0.0000   0.837 - 0.871   0.799 - 1.070   1.028       3       3

  All scans  0.0000   0.837 - 1.147   0.799 - 1.184   1.002      10      10


Statistics for all composite reflections
----------------------------------------

Scan 2-theta  R(int)  Incid. factors  Diffr. factors    K     Total I>2sig(I)
   1  -30.0  0.0546   1.002 - 1.134   0.786 - 1.184   0.967    3461    2921
   2  -30.0  0.0618   1.001 - 1.167   0.861 - 1.215   1.009    4524    3732
   3  -30.0  0.0574   0.818 - 0.921   0.787 - 1.220   1.028    3948    3292

  All scans  0.0582   0.818 - 1.167   0.786 - 1.220   1.002   11933    9945


Statistics for all single and composite reflections
---------------------------------------------------

Scan 2-theta  R(int)  Incid. factors  Diffr. factors    K     Total I>2sig(I)
   1  -30.0  0.0555   1.002 - 1.134   0.786 - 1.184   0.967    5222    4305
   2  -30.0  0.0647   1.001 - 1.167   0.854 - 1.218   1.009    6250    5009
   3  -30.0  0.0567   0.818 - 0.921   0.787 - 1.221   1.028    5785    4786

  All scans  0.0591   0.818 - 1.167   0.786 - 1.221   1.002   17257   14100

su = K * Sqrt[ sigma^2(I) + (g<I>)^2 ]  where sigma(I) is estimated by SAINT

The above statistics are based on all non-rejected data, ignoring
reflections without equivalents when estimating R(int) and K.

Repeat parameter refinement (P), repeat rejections (R) or accept (A) [A]:
```

Figure. 37. The Run command window.

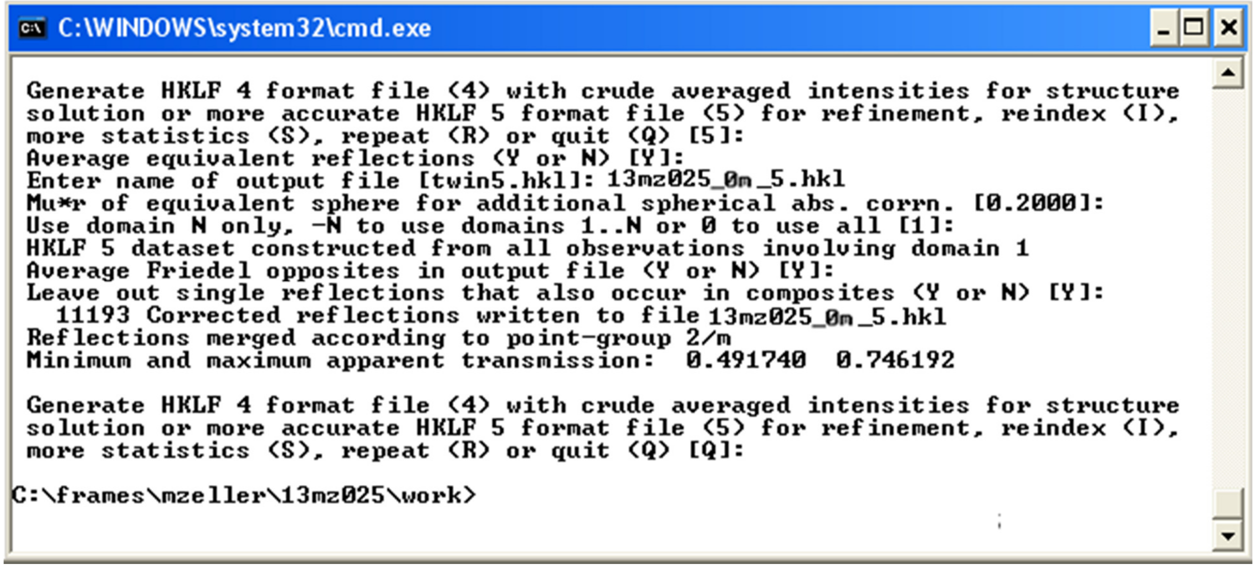
Output reflections

- Choose to output a standard *hkl* file with only one component ⇒ Give name 13mz025_0m_4.hkl.
- and a non-standard *hkl* file with all twin components as 13mz025_0m_5.hkl.



```
The R(int) and R(sigma) plots are based on all singles and composites,
<|E^2-1|> is calculated for all individual contributions from domain 1
assuming SAINT partitioning and the chi^2 and spatial plots show only
those single and composite reflections actually fitted.

Spatial display of (I-<I>)/su greater than [3.0] (0 for none):

The following reindex option may be used with the matrix -1 0 0 0 -1 0 0 0 -1
to invert individual components.

Generate HKLF 4 format file (4) with crude averaged intensities for structure
solution or more accurate HKLF 5 format file (5) for refinement, reindex (I),
more statistics (S), repeat (R) or quit (Q) [4]:
Enter name of output file [twin4.hkl]: 13mz025_0m_4.hkl
Mu*r of equivalent sphere for additional spherical abs. corrn. [0.2000]:
Use domain N only, -N to use domains 1..N or 0 to use all [1]:
Average Friedel opposites in output file (Y or N) [Y]:
Number of iterations [20]:
Relative weight for SAINT partitioning estimates [0.1]:


Iterative extraction of best unique reflection data

Cycle    N(1) Rint(1)   N(all) Rint(all) Twin fractions
   1    11941  0.1096    17997  0.1241    0.5322  0.4239  0.0440
   2    11941  0.0819    17997  0.0972    0.5067  0.4480  0.0453
   3    11941  0.0737    17997  0.0905    0.5087  0.4455  0.0458
   4    11941  0.0725    17997  0.0896    0.5092  0.4450  0.0458
   5    11941  0.0719    17997  0.0891    0.5094  0.4447  0.0458
   6    11941  0.0716    17997  0.0889    0.5095  0.4446  0.0459
   7    11941  0.0714    17997  0.0888    0.5095  0.4446  0.0459
   8    11941  0.0713    17997  0.0887    0.5095  0.4446  0.0459
   9    11941  0.0712    17997  0.0887    0.5095  0.4445  0.0459
  10    11941  0.0712    17997  0.0886    0.5095  0.4445  0.0460
  11    11941  0.0711    17997  0.0886    0.5095  0.4445  0.0460
  12    11941  0.0711    17997  0.0886    0.5095  0.4445  0.0460
  13    11941  0.0711    17997  0.0886    0.5095  0.4445  0.0460
  14    11941  0.0711    17997  0.0885    0.5095  0.4445  0.0460
  15    11941  0.0711    17997  0.0885    0.5095  0.4445  0.0460
  16    11941  0.0711    17997  0.0885    0.5095  0.4445  0.0460
  17    11941  0.0711    17997  0.0885    0.5095  0.4445  0.0460
  18    11941  0.0711    17997  0.0885    0.5095  0.4445  0.0460
  19    11941  0.0711    17997  0.0885    0.5095  0.4445  0.0460
  20    11941  0.0710    17997  0.0885    0.5095  0.4445  0.0460

N(1) and Rint(1) refer to singles and composites that include domain 1.

Rint = 0.0885  for all   17997  observations and
Rint = 0.0833  for all    8379  observations with I > 3sigma(I)

Rint is based on agreement between observed single and composite intensities
and those calculated from refined unique intensities and twin fractions.

   3955 Corrected reflections written to file 13mz025_0m_4.hkl
Reflections merged according to point-group 2/m
Minimum and maximum apparent transmission:  0.492727  0.746192
```

Figure. 38. The Run command window.



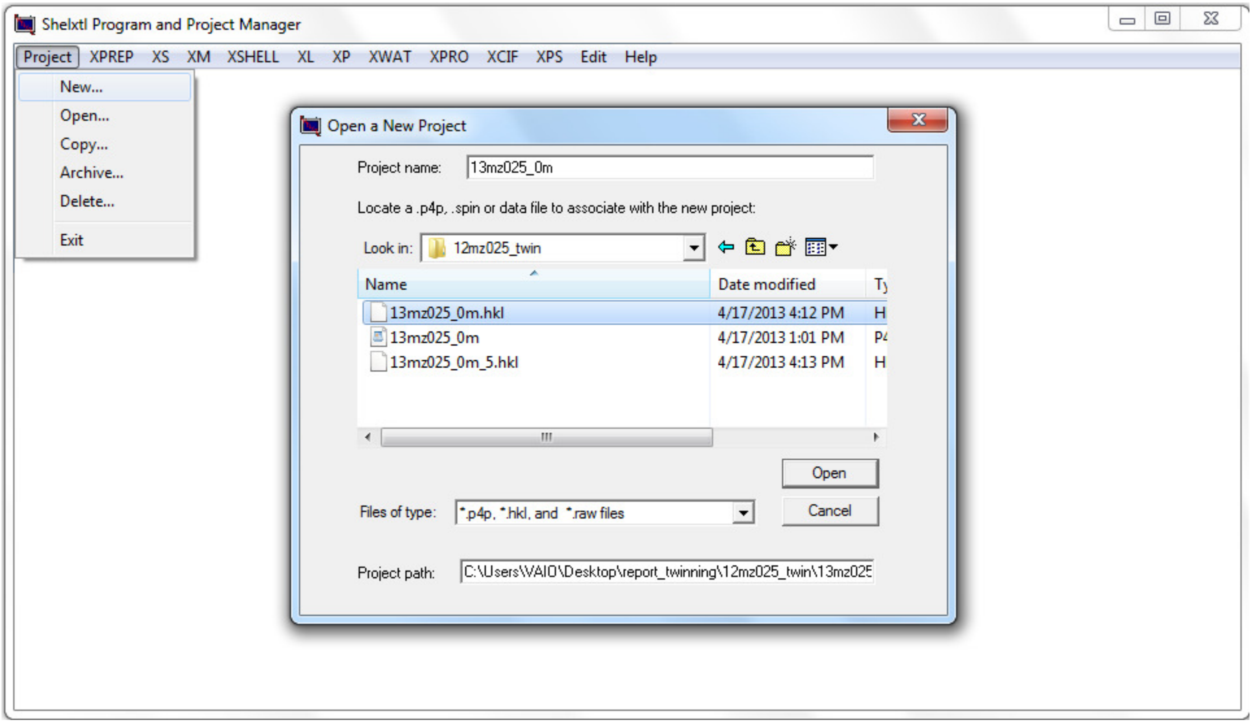
```
 Generate HKLF 4 format file (4) with crude averaged intensities for structure
 solution or more accurate HKLF 5 format file (5) for refinement, reindex (I),
 more statistics (S), repeat (R) or quit (Q) [5]:
 Average equivalent reflections (Y or N) [Y]:
 Enter name of output file [twin5.hkl]: 13mz025_0m_5.hkl
 Mu*r of equivalent sphere for additional spherical abs. corrn. [0.2000]:
 Use domain N only, -N to use domains 1..N or 0 to use all [1]:
 HKLF 5 dataset constructed from all observations involving domain 1
 Average Friedel opposites in output file (Y or N) [Y]:
 Leave out single reflections that also occur in composites (Y or N) [Y]:
   11193 Corrected reflections written to file 13mz025_0m_5.hkl
 Reflections merged according to point-group 2/m
 Minimum and maximum apparent transmission:  0.491740  0.746192

 Generate HKLF 4 format file (4) with crude averaged intensities for structure
 solution or more accurate HKLF 5 format file (5) for refinement, reindex (I),
 more statistics (S), repeat (R) or quit (Q) [Q]:

C:\frames\mzeller\13mz025\work>
```

Figure. 39. The Run command window.

We have two hkl files. We use detwinned HKLF4 format file for structure solution and refiment HKLF5 for final refinement.

Solving structures

- Open the SHELXTL program. Select project, choose new, and then give a name project and open.



Project name: 13mz025_0m

Locate a .p4p, .spin or data file to associate with the new project:

Look in: 12mz025_twin

Name	Date modified	Ty
13mz025_0m.hkl	4/17/2013 4:12 PM	H
13mz025_0m	4/17/2013 1:01 PM	P4
13mz025_0m_5.hkl	4/17/2013 4:13 PM	H

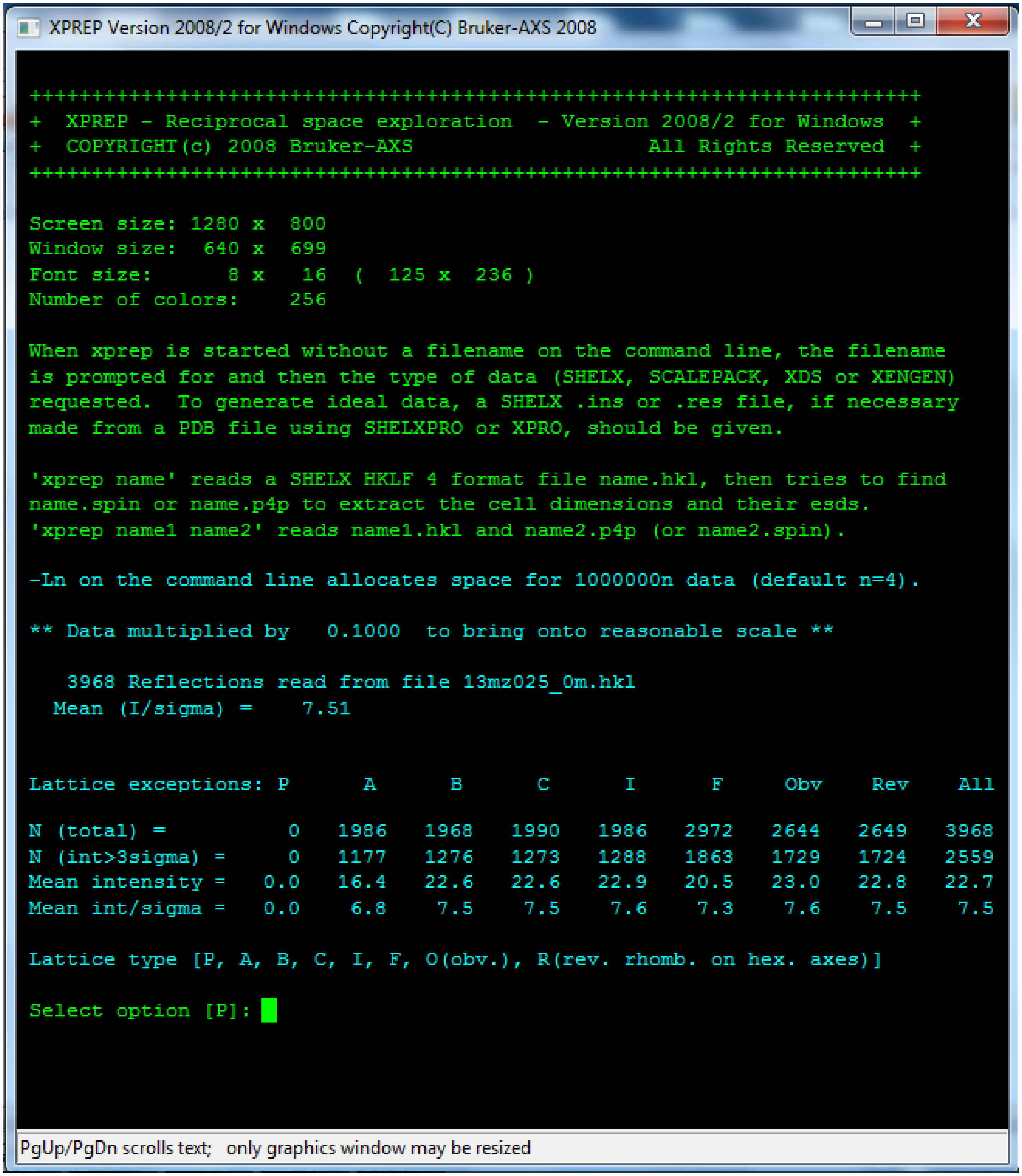
Files of type: *.p4p, *.hkl, and *.raw files

Project path: C:\Users\VAIO\Desktop\report_twinning\12mz025_twin\13mz025

Figure. 40. Shelxtl Program and project manager window.

XPREP

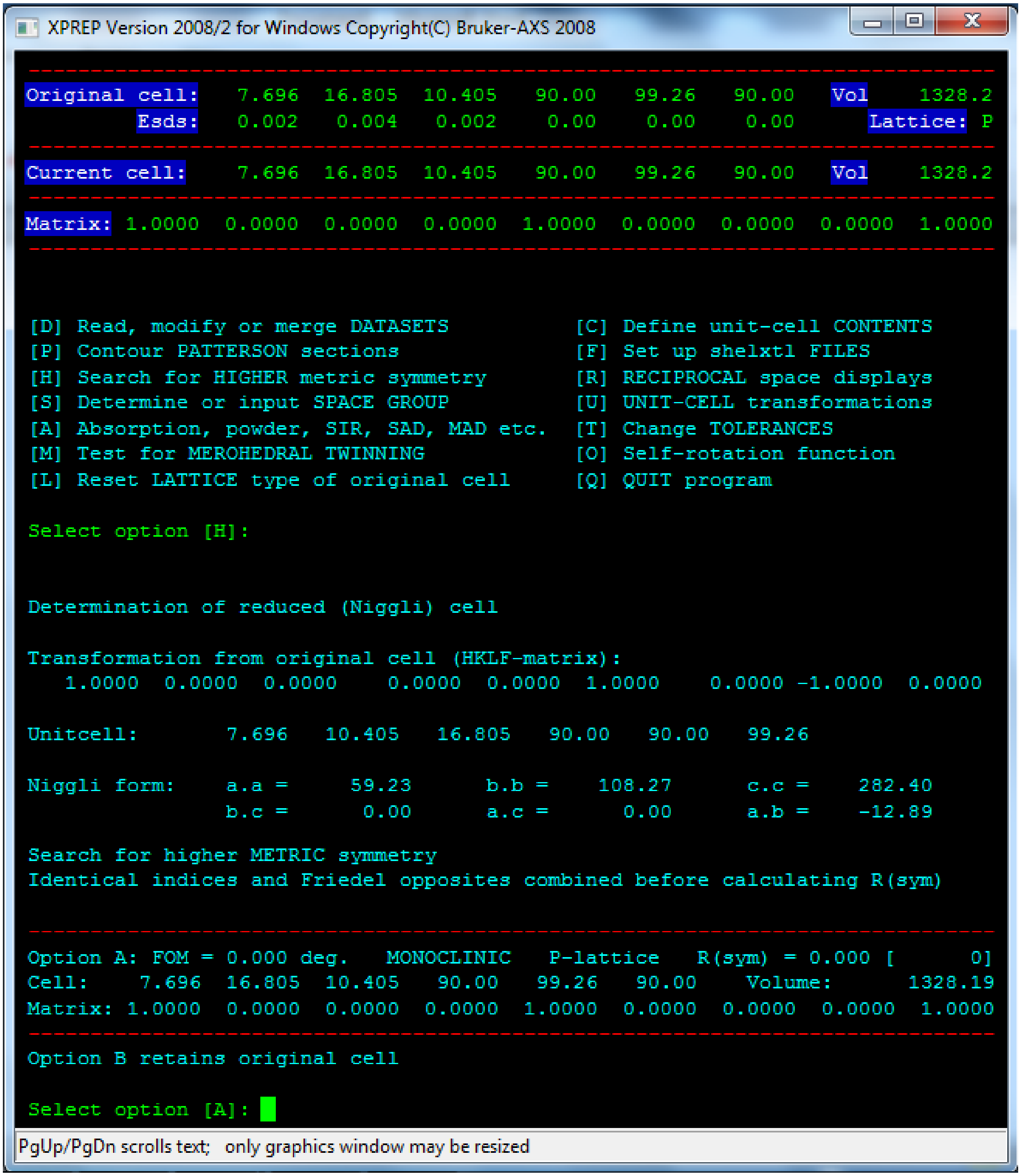
- Select XPREP on the toolbar.
- Select the suggested lattice type, Enter.



```
++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++
+  XPREP - Reciprocal space exploration  - Version 2008/2 for Windows  +
+  COPYRIGHT(c) 2008 Bruker-AXS                   All Rights Reserved  +
++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++

Screen size: 1280 x  800
Window size:  640 x  699
Font size:      8 x   16  (  125 x  236 )
Number of colors:    256

When xprep is started without a filename on the command line, the filename
is prompted for and then the type of data (SHELX, SCALEPACK, XDS or XENGEN)
requested.  To generate ideal data, a SHELX .ins or .res file, if necessary
made from a PDB file using SHELXPRO or XPRO, should be given.

'xprep name' reads a SHELX HKLF 4 format file name.hkl, then tries to find
name.spin or name.p4p to extract the cell dimensions and their esds.
'xprep name1 name2' reads name1.hkl and name2.p4p (or name2.spin).

-Ln on the command line allocates space for 1000000n data (default n=4).

** Data multiplied by   0.1000  to bring onto reasonable scale **

   3968 Reflections read from file 13mz025_0m.hkl
  Mean (I/sigma) =    7.51


Lattice exceptions: P      A      B      C      I      F    Obv    Rev    All

N (total) =          0   1986   1968   1990   1986   2972   2644   2649   3968
N (int>3sigma) =     0   1177   1276   1273   1288   1863   1729   1724   2559
Mean intensity =   0.0   16.4   22.6   22.6   22.9   20.5   23.0   22.8   22.7
Mean int/sigma =   0.0    6.8    7.5    7.5    7.6    7.3    7.6    7.5    7.5

Lattice type [P, A, B, C, I, F, O(obv.), R(rev. rhomb. on hex. axes)]

Select option [P]: █
```

Figure. 41. XPREP window.

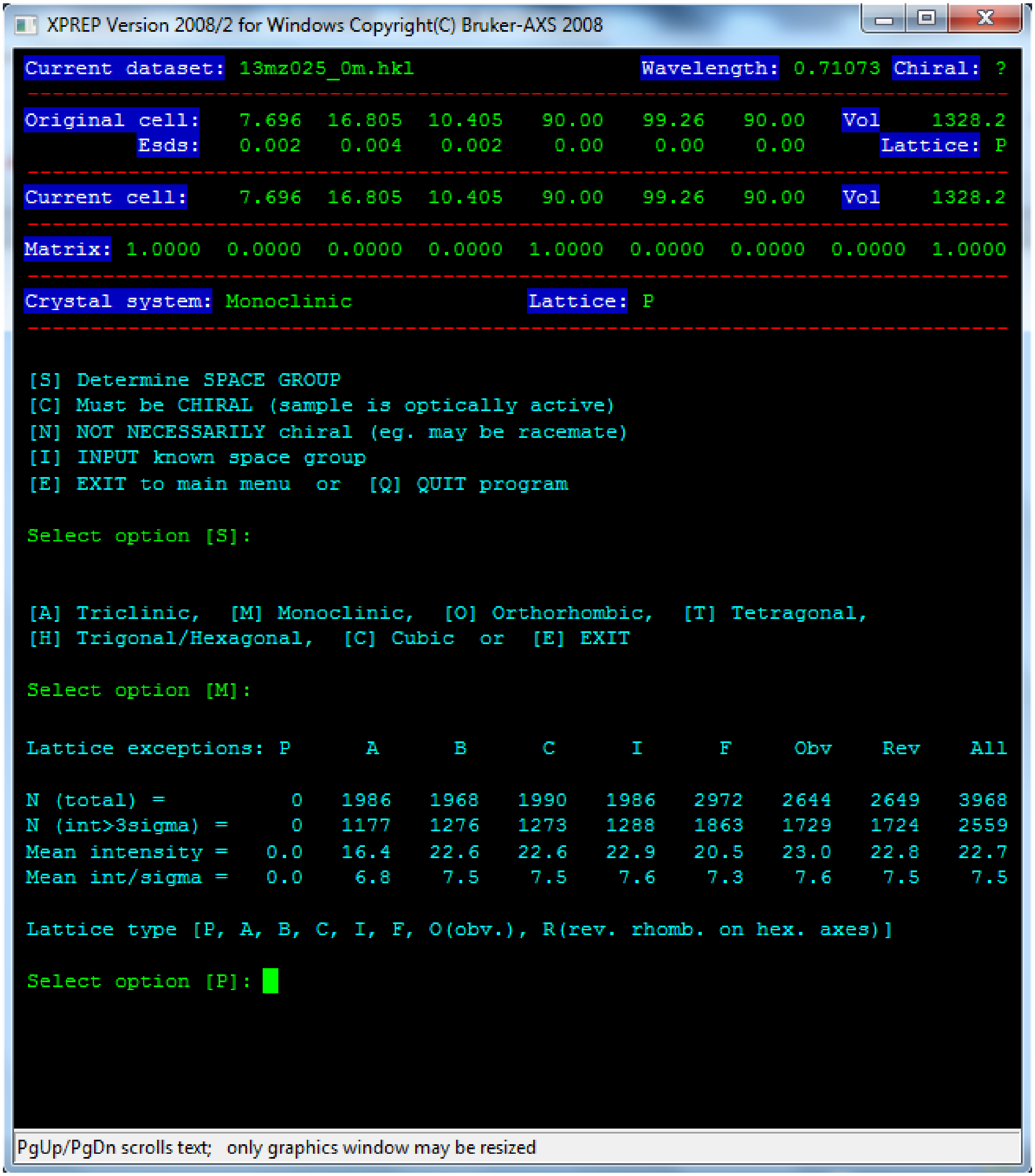
- Choose [H] to search for higher metric symmetry.
- Choose offered choice [A] for Laue group.



```
--------------------------------------------------------------------------------
Original cell:    7.696  16.805  10.405    90.00    99.26    90.00   Vol     1328.2
         Esds:    0.002   0.004   0.002     0.00     0.00     0.00      Lattice: P
--------------------------------------------------------------------------------
Current cell:     7.696  16.805  10.405    90.00    99.26    90.00   Vol     1328.2
--------------------------------------------------------------------------------
Matrix: 1.0000  0.0000  0.0000  0.0000  1.0000  0.0000  0.0000  0.0000  1.0000
--------------------------------------------------------------------------------


[D] Read, modify or merge DATASETS          [C] Define unit-cell CONTENTS
[P] Contour PATTERSON sections              [F] Set up shelxtl FILES
[H] Search for HIGHER metric symmetry       [R] RECIPROCAL space displays
[S] Determine or input SPACE GROUP          [U] UNIT-CELL transformations
[A] Absorption, powder, SIR, SAD, MAD etc.  [T] Change TOLERANCES
[M] Test for MEROHEDRAL TWINNING            [O] Self-rotation function
[L] Reset LATTICE type of original cell     [Q] QUIT program

Select option [H]:


Determination of reduced (Niggli) cell

Transformation from original cell (HKLF-matrix):
   1.0000  0.0000  0.0000    0.0000  0.0000  1.0000    0.0000 -1.0000  0.0000

Unitcell:        7.696   10.405   16.805   90.00   90.00   99.26

Niggli form:     a.a =     59.23      b.b =    108.27      c.c =    282.40
                 b.c =      0.00      a.c =      0.00      a.b =    -12.89

Search for higher METRIC symmetry
Identical indices and Friedel opposites combined before calculating R(sym)

--------------------------------------------------------------------------------
Option A: FOM = 0.000 deg.   MONOCLINIC   P-lattice   R(sym) = 0.000 [      0]
Cell:    7.696  16.805  10.405    90.00    99.26    90.00     Volume:      1328.19
Matrix: 1.0000  0.0000  0.0000  0.0000  1.0000  0.0000  0.0000  0.0000  1.0000
--------------------------------------------------------------------------------
Option B retains original cell

Select option [A]:
```

Figure. 42. XPREP window.

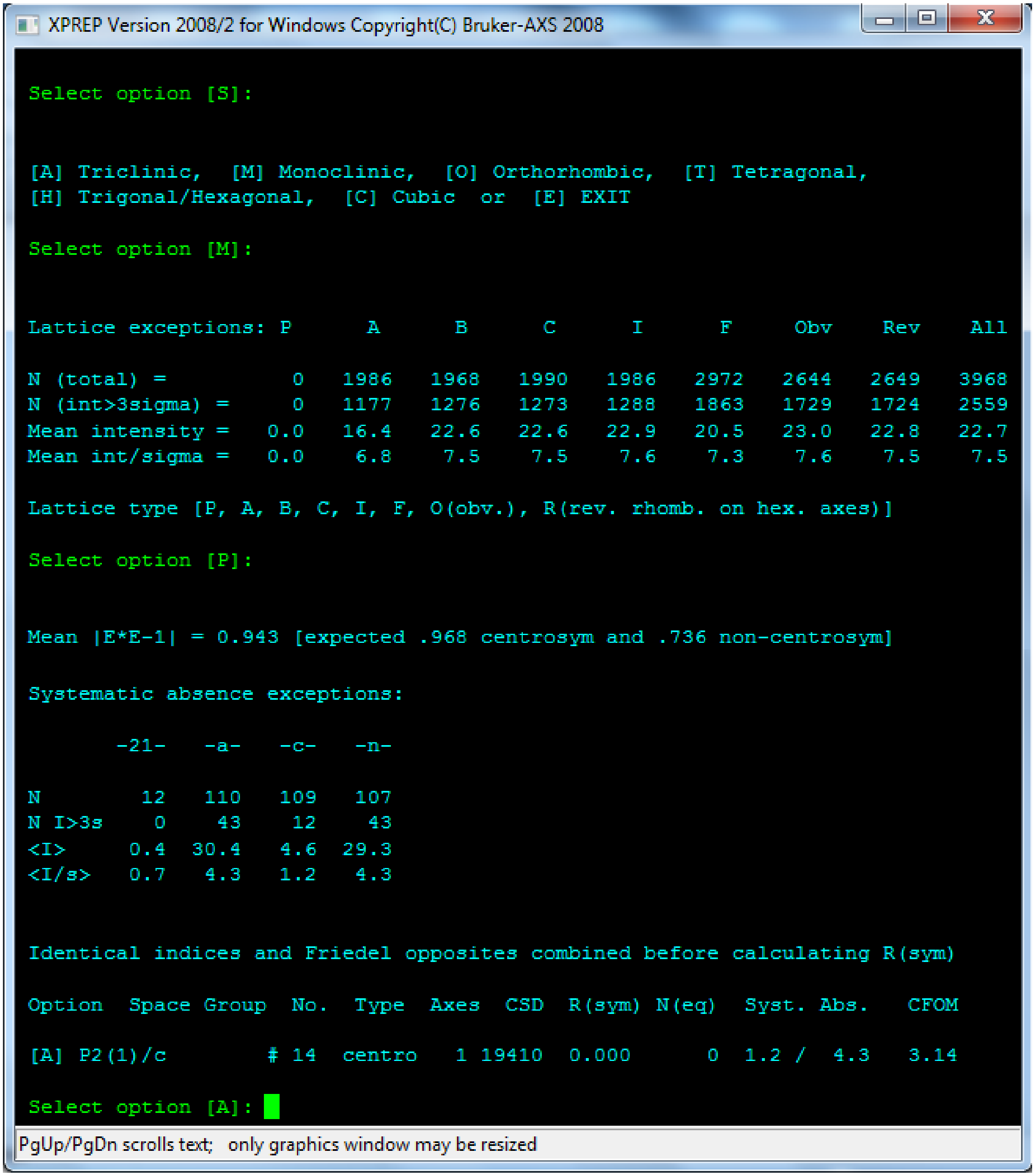
- Select [S] to determine or input space group.
- Select [M] to determine space group monoclinic.



```
Current dataset: 13mz025_0m.hkl                     Wavelength: 0.71073 Chiral: ?
-------------------------------------------------------------------------------
Original cell:    7.696  16.805  10.405   90.00   99.26   90.00   Vol     1328.2
         Esds:    0.002   0.004   0.002    0.00    0.00    0.00      Lattice: P
-------------------------------------------------------------------------------
Current cell:     7.696  16.805  10.405   90.00   99.26   90.00   Vol     1328.2
-------------------------------------------------------------------------------
Matrix: 1.0000  0.0000  0.0000  0.0000  1.0000  0.0000  0.0000  0.0000  1.0000
-------------------------------------------------------------------------------
Crystal system: Monoclinic                  Lattice: P
-------------------------------------------------------------------------------

[S] Determine SPACE GROUP
[C] Must be CHIRAL (sample is optically active)
[N] NOT NECESSARILY chiral (eg. may be racemate)
[I] INPUT known space group
[E] EXIT to main menu  or  [Q] QUIT program

Select option [S]:


[A] Triclinic,  [M] Monoclinic,  [O] Orthorhombic,  [T] Tetragonal,
[H] Trigonal/Hexagonal,  [C] Cubic  or  [E] EXIT

Select option [M]:

Lattice exceptions: P      A      B      C      I      F     Obv    Rev    All

N (total) =         0   1986   1968   1990   1986   2972   2644   2649   3968
N (int>3sigma) =    0   1177   1276   1273   1288   1863   1729   1724   2559
Mean intensity =  0.0   16.4   22.6   22.6   22.9   20.5   23.0   22.8   22.7
Mean int/sigma =  0.0    6.8    7.5    7.5    7.6    7.3    7.6    7.5    7.5

Lattice type [P, A, B, C, I, F, O(obv.), R(rev. rhomb. on hex. axes)]

Select option [P]:
```

Figure. 43. XPREP window.

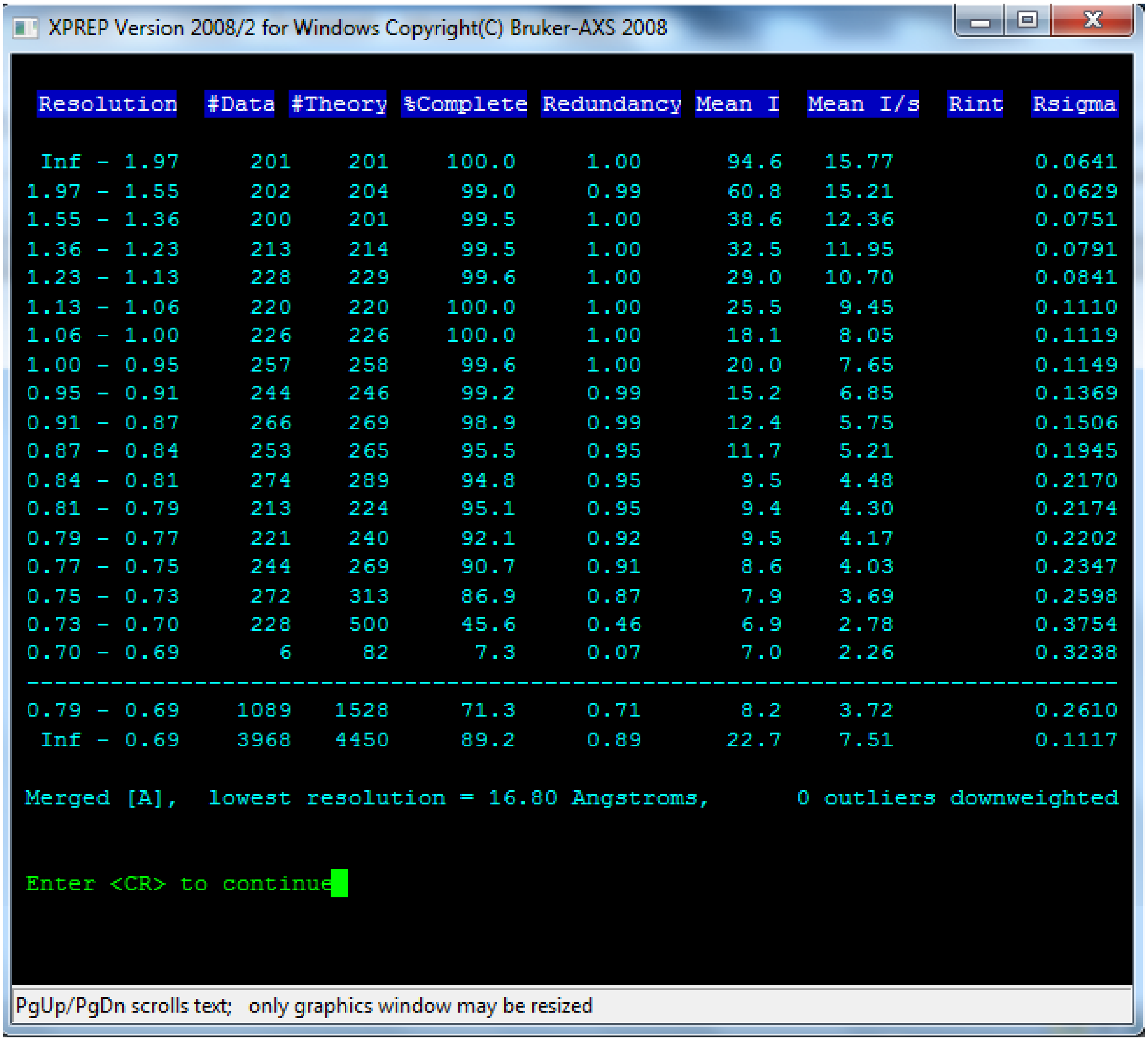
- Select [P]
- Select [A] for determine space group.



```
Select option [S]:


[A] Triclinic,  [M] Monoclinic,  [O] Orthorhombic,  [T] Tetragonal,
[H] Trigonal/Hexagonal,  [C] Cubic  or  [E] EXIT

Select option [M]:


Lattice exceptions: P      A      B      C      I      F     Obv    Rev    All

N (total) =         0   1986   1968   1990   1986   2972   2644   2649   3968
N (int>3sigma) =    0   1177   1276   1273   1288   1863   1729   1724   2559
Mean intensity =  0.0   16.4   22.6   22.6   22.9   20.5   23.0   22.8   22.7
Mean int/sigma =  0.0    6.8    7.5    7.5    7.6    7.3    7.6    7.5    7.5

Lattice type [P, A, B, C, I, F, O(obv.), R(rev. rhomb. on hex. axes)]

Select option [P]:


Mean |E*E-1| = 0.943 [expected .968 centrosym and .736 non-centrosym]

Systematic absence exceptions:

       -21-   -a-    -c-    -n-

N        12   110    109    107
N I>3s    0    43     12     43
<I>     0.4  30.4    4.6   29.3
<I/s>   0.7   4.3    1.2    4.3


Identical indices and Friedel opposites combined before calculating R(sym)

Option  Space Group  No.  Type   Axes  CSD  R(sym) N(eq)  Syst. Abs.   CFOM

[A] P2(1)/c        # 14  centro   1 19410  0.000      0  1.2 /  4.3   3.14

Select option [A]:
```

Figure. 44. XPREP window.

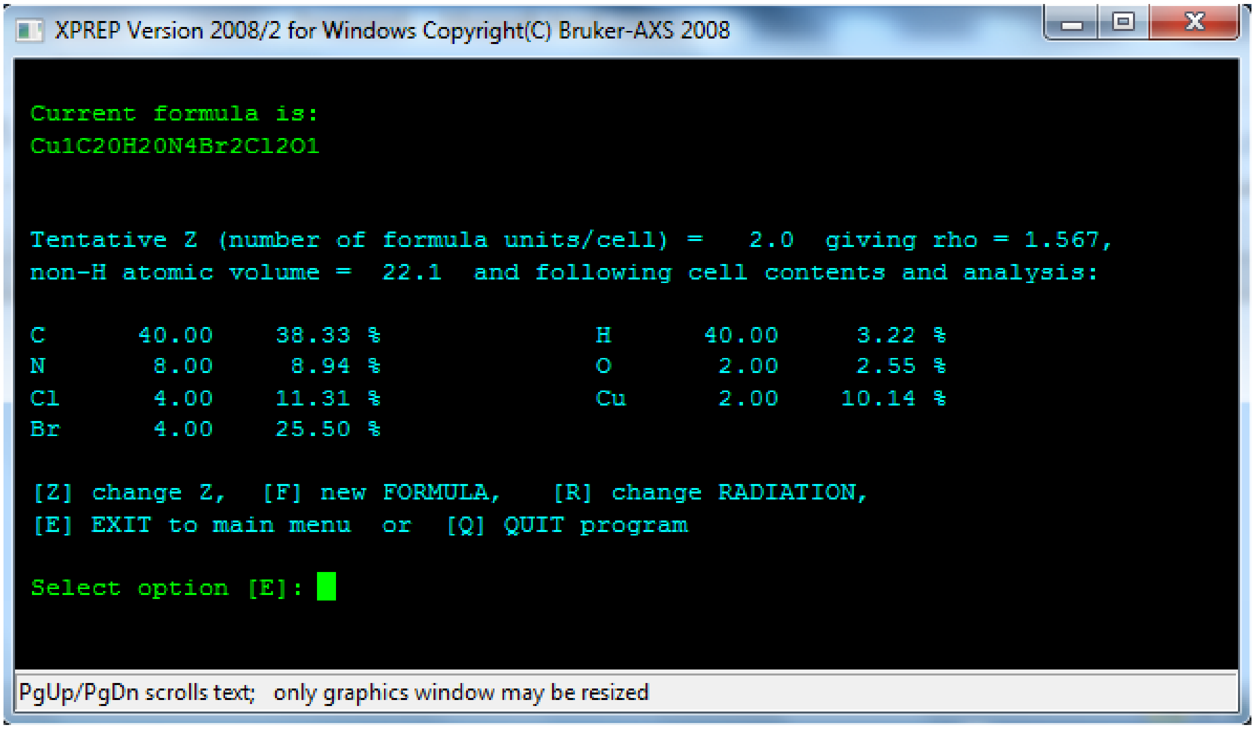
- Select [D] to read, modify or merge datasets.
- Select [S] to display the intensity statistics.



```
 Resolution   #Data #Theory %Complete Redundancy Mean I  Mean I/s  Rint   Rsigma

 Inf - 1.97     201    201    100.0      1.00      94.6   15.77          0.0641
1.97 - 1.55     202    204     99.0      0.99      60.8   15.21          0.0629
1.55 - 1.36     200    201     99.5      1.00      38.6   12.36          0.0751
1.36 - 1.23     213    214     99.5      1.00      32.5   11.95          0.0791
1.23 - 1.13     228    229     99.6      1.00      29.0   10.70          0.0841
1.13 - 1.06     220    220    100.0      1.00      25.5    9.45          0.1110
1.06 - 1.00     226    226    100.0      1.00      18.1    8.05          0.1119
1.00 - 0.95     257    258     99.6      1.00      20.0    7.65          0.1149
0.95 - 0.91     244    246     99.2      0.99      15.2    6.85          0.1369
0.91 - 0.87     266    269     98.9      0.99      12.4    5.75          0.1506
0.87 - 0.84     253    265     95.5      0.95      11.7    5.21          0.1945
0.84 - 0.81     274    289     94.8      0.95       9.5    4.48          0.2170
0.81 - 0.79     213    224     95.1      0.95       9.4    4.30          0.2174
0.79 - 0.77     221    240     92.1      0.92       9.5    4.17          0.2202
0.77 - 0.75     244    269     90.7      0.91       8.6    4.03          0.2347
0.75 - 0.73     272    313     86.9      0.87       7.9    3.69          0.2598
0.73 - 0.70     228    500     45.6      0.46       6.9    2.78          0.3754
0.70 - 0.69       6     82      7.3      0.07       7.0    2.26          0.3238
------------------------------------------------------------------------------
0.79 - 0.69    1089   1528     71.3      0.71       8.2    3.72          0.2610
 Inf - 0.69    3968   4450     89.2      0.89      22.7    7.51          0.1117

Merged [A],  lowest resolution = 16.80 Angstroms,      0 outliers downweighted


Enter <CR> to continue
```

Figure. 45. XPREP window.

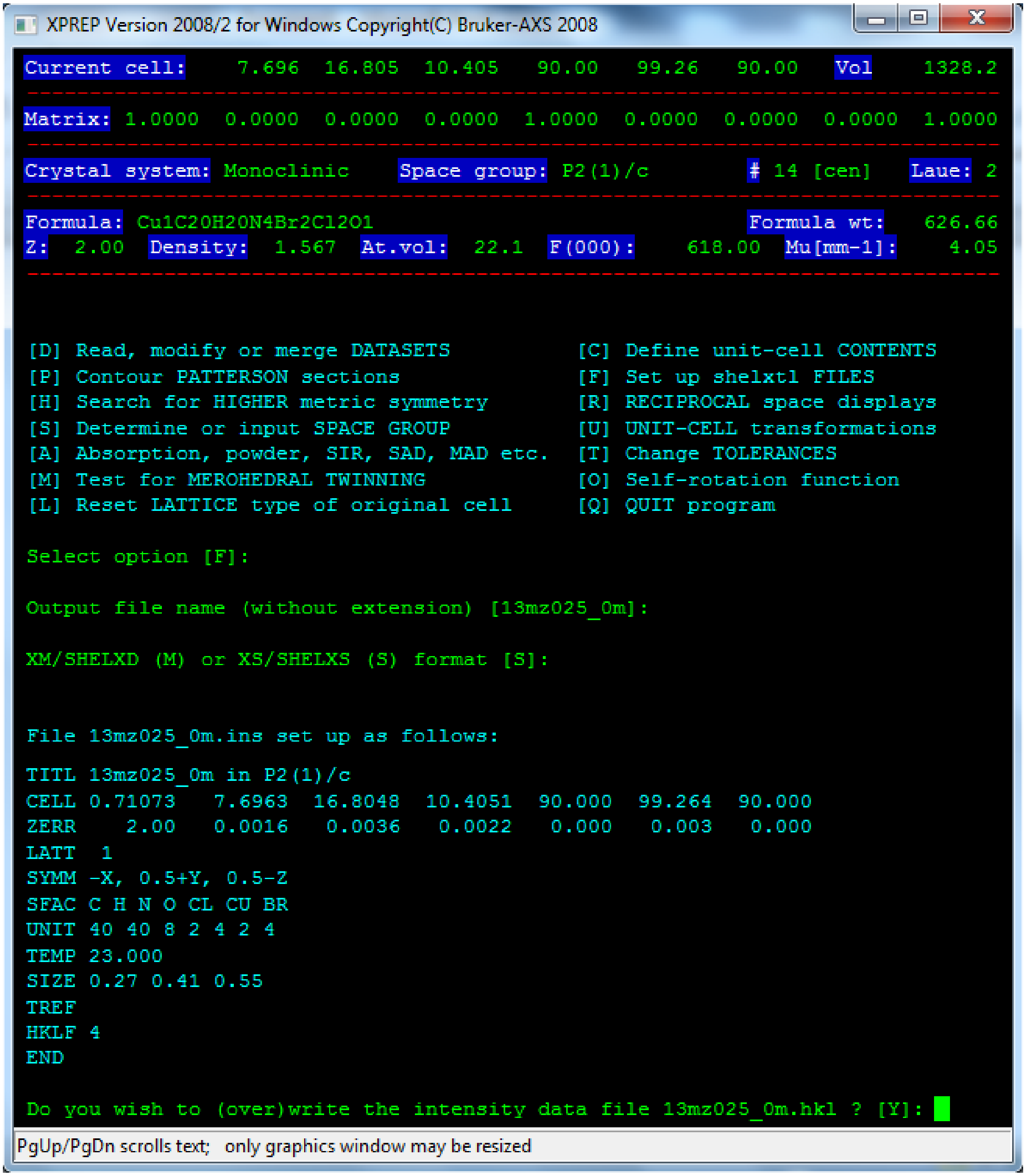
- Select [A] to not merge all equivalent reflections.
- Select [E] to exit to the menu.



```
Current formula is:
Cu1C20H20N4Br2Cl2O1


Tentative Z (number of formula units/cell) =   2.0  giving rho = 1.567,
non-H atomic volume =  22.1  and following cell contents and analysis:

C      40.00    38.33 %              H      40.00     3.22 %
N       8.00     8.94 %              O       2.00     2.55 %
Cl      4.00    11.31 %              Cu      2.00    10.14 %
Br      4.00    25.50 %

[Z] change Z,  [F] new FORMULA,   [R] change RADIATION,
[E] EXIT to main menu  or  [Q] QUIT program

Select option [E]:
```

Figure. 46. XPREP window.

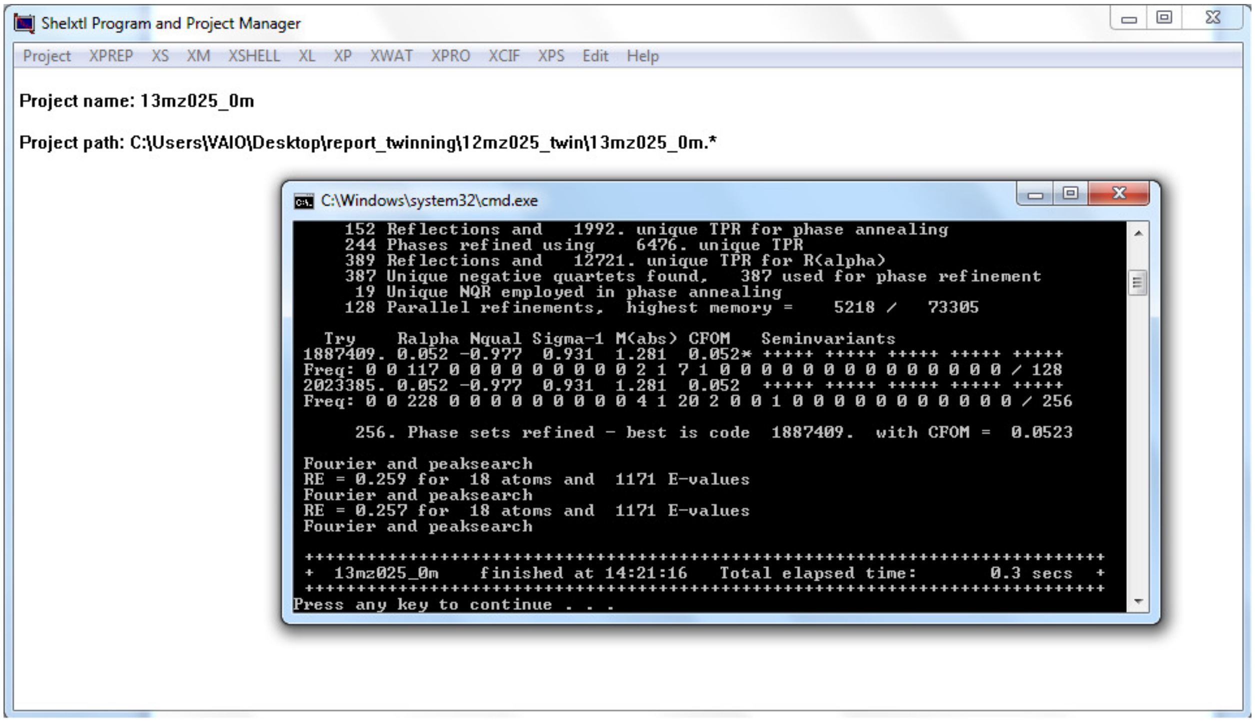
- Select [F] to set up shelxtl file.
- Select [Y] at the prompt to generate an .ins file.



```
Current cell:     7.696  16.805  10.405   90.00   99.26   90.00  Vol     1328.2
------------------------------------------------------------------------------
Matrix: 1.0000  0.0000  0.0000  0.0000  1.0000  0.0000  0.0000  0.0000  1.0000
------------------------------------------------------------------------------
Crystal system: Monoclinic   Space group: P2(1)/c        # 14 [cen]   Laue: 2
------------------------------------------------------------------------------
Formula: Cu1C20H20N4Br2Cl2O1                             Formula wt:   626.66
Z:  2.00  Density:  1.567  At.vol:  22.1  F(000):    618.00  Mu[mm-1]:    4.05
------------------------------------------------------------------------------


[D] Read, modify or merge DATASETS           [C] Define unit-cell CONTENTS
[P] Contour PATTERSON sections               [F] Set up shelxtl FILES
[H] Search for HIGHER metric symmetry        [R] RECIPROCAL space displays
[S] Determine or input SPACE GROUP           [U] UNIT-CELL transformations
[A] Absorption, powder, SIR, SAD, MAD etc.   [T] Change TOLERANCES
[M] Test for MEROHEDRAL TWINNING             [O] Self-rotation function
[L] Reset LATTICE type of original cell      [Q] QUIT program

Select option [F]:

Output file name (without extension) [13mz025_0m]:

XM/SHELXD (M) or XS/SHELXS (S) format [S]:


File 13mz025_0m.ins set up as follows:

TITL 13mz025_0m in P2(1)/c
CELL 0.71073   7.6963  16.8048  10.4051  90.000  99.264  90.000
ZERR    2.00   0.0016   0.0036   0.0022   0.000   0.003   0.000
LATT  1
SYMM -X, 0.5+Y, 0.5-Z
SFAC C H N O CL CU BR
UNIT 40 40 8 2 4 2 4
TEMP 23.000
SIZE 0.27 0.41 0.55
TREF
HKLF 4
END

Do you wish to (over)write the intensity data file 13mz025_0m.hkl ? [Y]:
```

Figure. 47. XPREP window.

- Select XS on the toolbar.

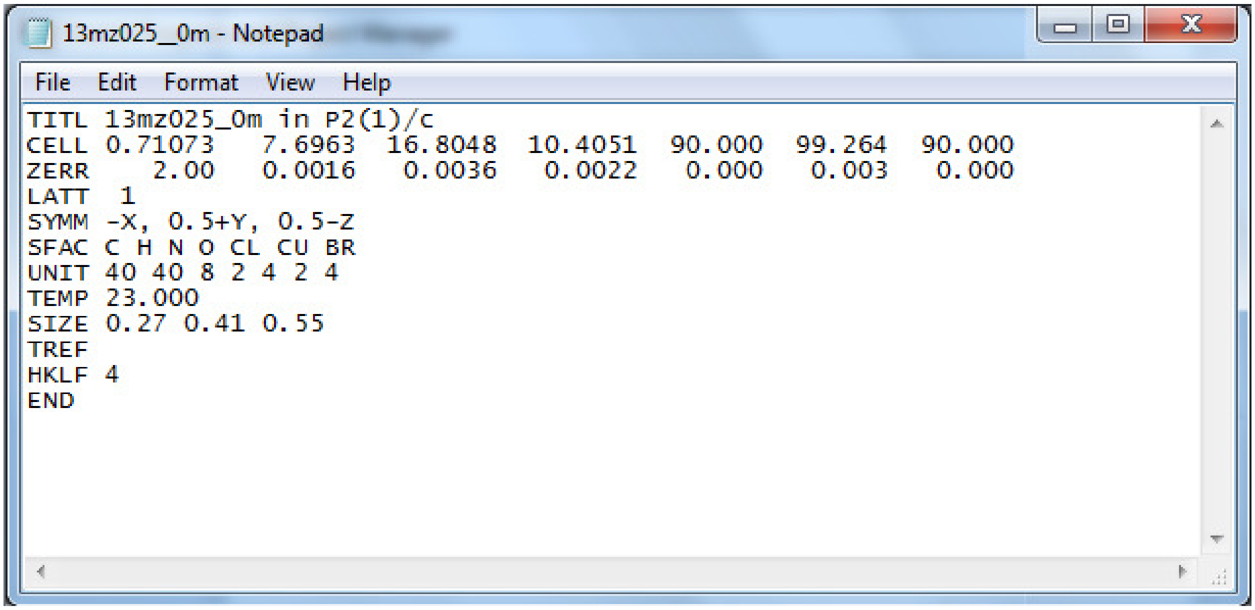


Project name: 13mz025_0m

Project path: C:\Users\VAIO\Desktop\report_twinning\12mz025_twin\13mz025_0m.*

```
    152 Reflections and   1992. unique TPR for phase annealing
    244 Phases refined using    6476. unique TPR
    389 Reflections and   12721. unique TPR for R(alpha)
    387 Unique negative quartets found,   387 used for phase refinement
     19 Unique NQR employed in phase annealing
    128 Parallel refinements,  highest memory =    5218 /   73305

  Try    Ralpha Nqual Sigma-1 M(abs) CFOM   Seminvariants
 1887409. 0.052 -0.977  0.931  1.281  0.052* +++++ +++++ +++++ +++++ +++++
 Freq: 0 0 117 0 0 0 0 0 0 0 0 0 2 1 7 1 0 0 0 0 0 0 0 0 0 0 0 0 0 / 128
 2023385. 0.052 -0.977  0.931  1.281  0.052  +++++ +++++ +++++ +++++ +++++
 Freq: 0 0 228 0 0 0 0 0 0 0 0 0 4 1 20 2 0 0 1 0 0 0 0 0 0 0 0 0 0 / 256

     256. Phase sets refined - best is code  1887409.  with CFOM =  0.0523

 Fourier and peaksearch
 RE = 0.259 for  18 atoms and  1171 E-values
 Fourier and peaksearch
 RE = 0.257 for  18 atoms and  1171 E-values
 Fourier and peaksearch

 +++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++
 +  13mz025_0m    finished at 14:21:16   Total elapsed time:       0.3 secs  +
 +++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++
Press any key to continue . . .
```

Figure. 48. Shelxtl Program with process of structure solution by classical direct methods.

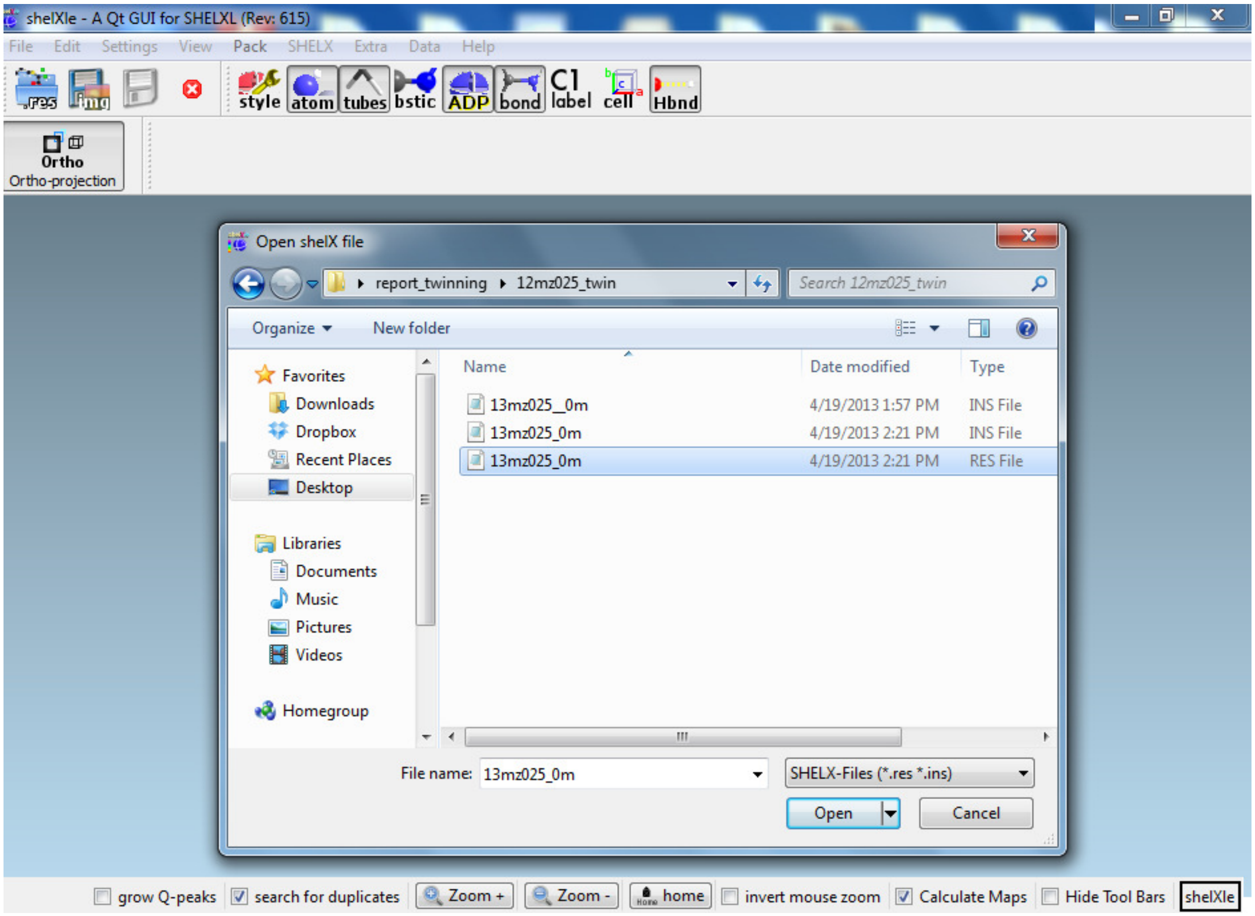


```
TITL 13mz025_0m in P2(1)/c
CELL 0.71073   7.6963  16.8048  10.4051  90.000  99.264  90.000
ZERR    2.00   0.0016   0.0036   0.0022   0.000   0.003   0.000
LATT  1
SYMM -X, 0.5+Y, 0.5-Z
SFAC C H N O CL CU BR
UNIT 40 40 8 2 4 2 4
TEMP 23.000
SIZE 0.27 0.41 0.55
TREF
HKLF 4
END
```

Figure. 49. Typical initial INS file.

Structure refinement

- Open shelXle program
- Open file 13mz025_0m.res.



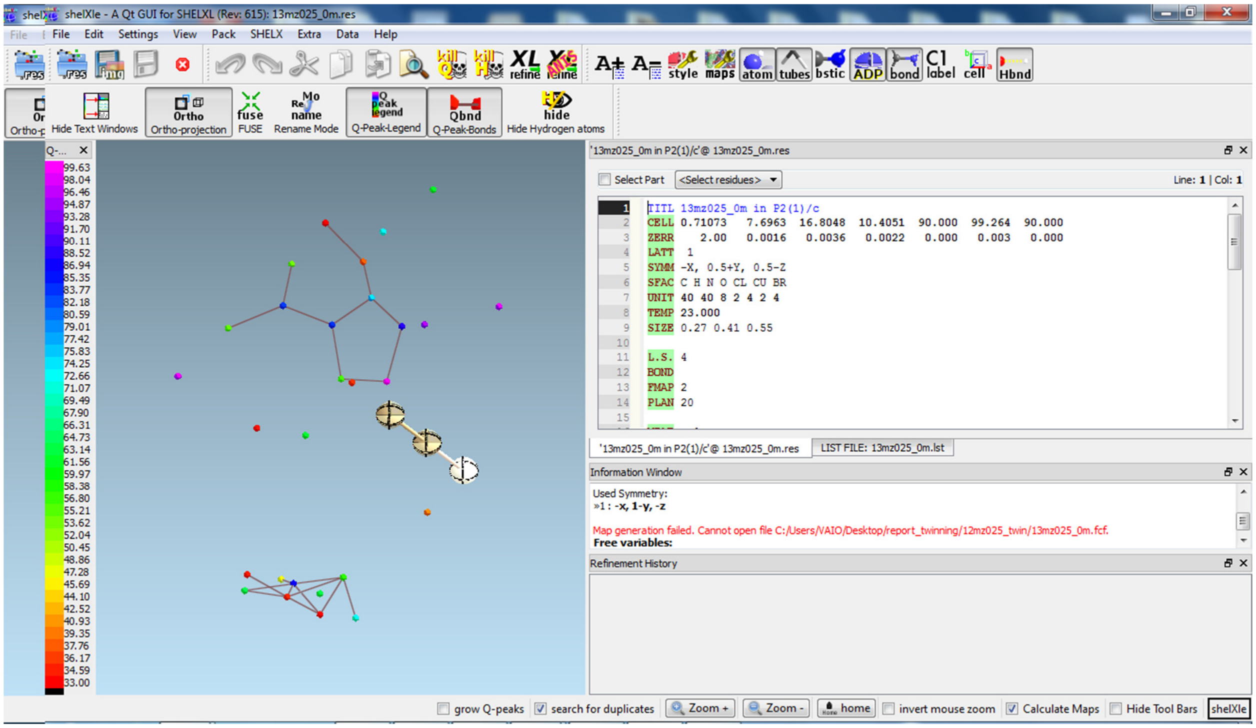
Name	Date modified	Type
13mz025__0m	4/19/2013 1:57 PM	INS File
13mz025_0m	4/19/2013 2:21 PM	INS File
13mz025_0m	4/19/2013 2:21 PM	RES File

File name: 13mz025_0m

SHELX-Files (*.res *.ins)

☐ grow Q-peaks ☑ search for duplicates ☐ invert mouse zoom ☑ Calculate Maps ☐ Hide Tool Bars

Figure. 50. shelXle window.



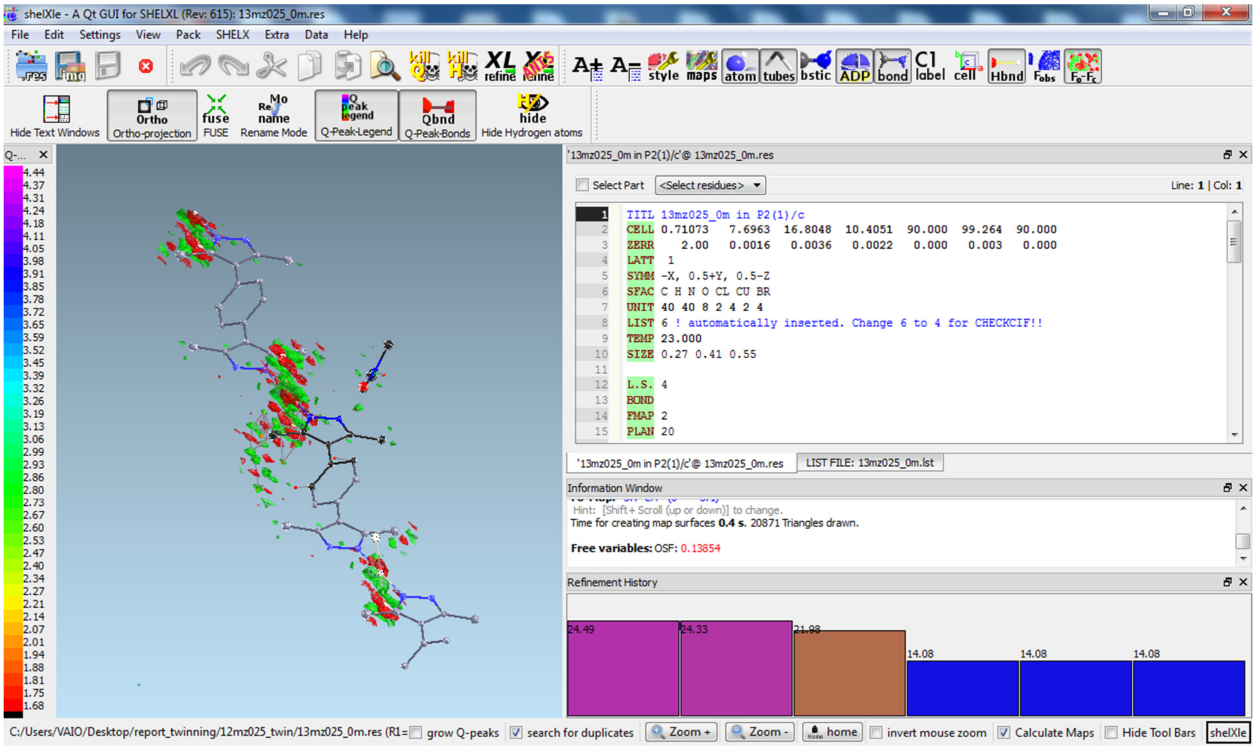
```
TITL 13mz025_0m in P2(1)/c
CELL 0.71073   7.6963  16.8048  10.4051  90.000  99.264  90.000
ZERR    2.00   0.0016   0.0036   0.0022   0.000   0.003   0.000
LATT  1
SYMM -X, 0.5+Y, 0.5-Z
SFAC C H N O CL CU BR
UNIT 40 40 8 2 4 2 4
TEMP 23.000
SIZE 0.27 0.41 0.55

L.S. 4
BOND
FMAP 2
PLAN 20
```

Used Symmetry:
»1 : -x, 1-y, -z

Map generation failed. Cannot open file C:/Users/VAIO/Desktop/report_twinning/12mz025_twin/13mz025_0m.fcf.
Free variables:

Figure. 51. shelXle window with initial result from XS.

Start by solved and refined the structure using the HKLF 4 data set in the normal way.



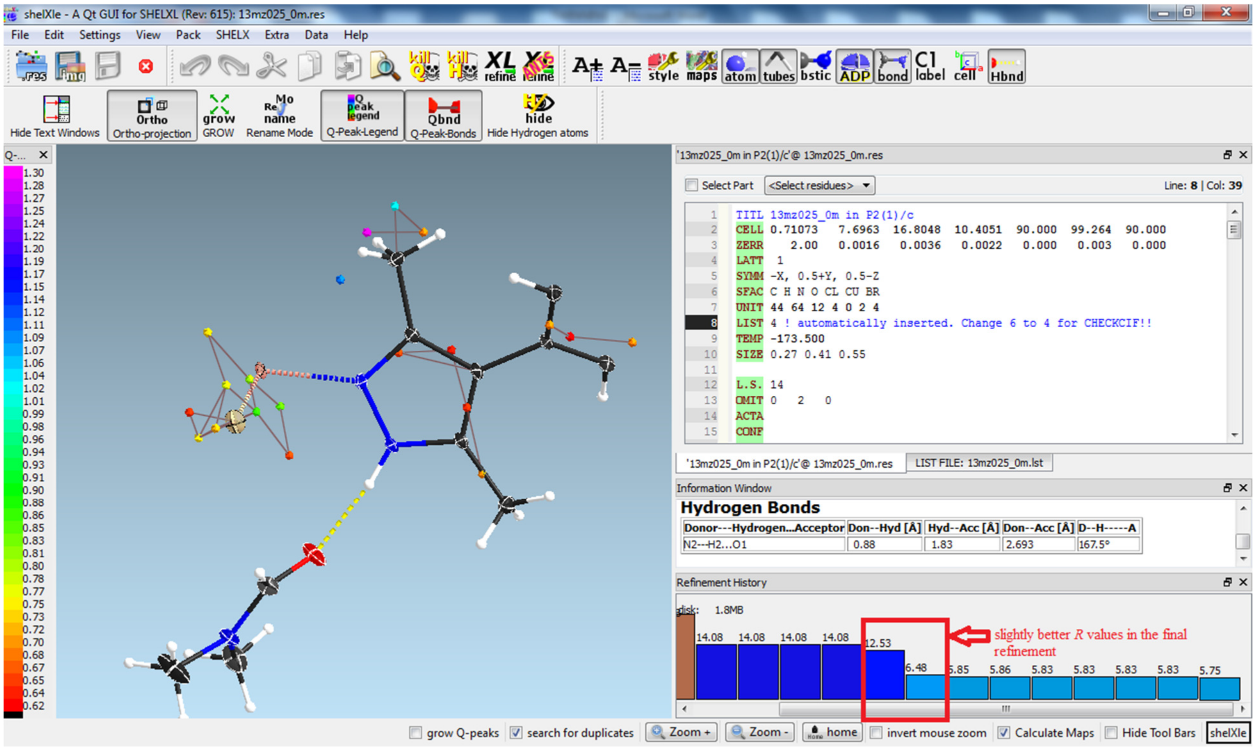
```
TITL 13mz025_0m in P2(1)/c
CELL 0.71073   7.6963  16.8048  10.4051  90.000  99.264  90.000
ZERR    2.00   0.0016   0.0036   0.0022   0.000   0.003   0.000
LATT  1
SYMM -X, 0.5+Y, 0.5-Z
SFAC C H N O CL CU BR
UNIT 40 40 8 2 4 2 4
LIST 6 ! automatically inserted. Change 6 to 4 for CHECKCIF!!
TEMP 23.000
SIZE 0.27 0.41 0.55

L.S. 4
BOND
FMAP 2
PLAN 20
```

Hint: [Shift+ Scroll (up or down)] to change.
Time for creating map surfaces **0.4 s.** 20871 Triangles drawn.

Free variables: OSF: 0.13854

Figure. 50. shelXle window.

In this case insert the line ***BASF 0.4 0.2*** *before the FVAR line and change the HKLF 4 line to HKLF 5 and then refine the file with the 13mz025_0m_5.hkl data. R1 should all decrease.*



```
TITL 13mz025_0m in P2(1)/c
CELL 0.71073   7.6963  16.8048  10.4051  90.000  99.264  90.000
ZERR    2.00   0.0016   0.0036   0.0022   0.000   0.003   0.000
LATT  1
SYMM -X, 0.5+Y, 0.5-Z
SFAC C H N O CL CU BR
UNIT 44 64 12 4 0 2 4
LIST 4 ! automatically inserted. Change 6 to 4 for CHECKCIF!!
TEMP -173.500
SIZE 0.27 0.41 0.55

L.S. 14
OMIT 0   2   0
ACTA
CONF
```

Hydrogen Bonds

Donor---Hydrogen...Acceptor	Don--Hyd [Å]	Hyd--Acc [Å]	Don--Acc [Å]	D--H-----A
N2---H2...O1	0.88	1.83	2.693	167.5°

slightly better *R* values in the final refinement

Figure. 51. shelXle window.

Run refinement cycle until no changes are observed for R1 and Goof, and until Max. dU and Maximum are basically zero.

Creating and Validating the CIF File

- Open 13mz025_0m.cif
  Add information

```
data_13mz025_0m

_audit_creation_method           SHELXL-2012
_chemical_name_systematic
;
 ?
;
_chemical_name_common            ?
_chemical_melting_point          ?
_chemical_formula_moiety        'C16 H18 Br2 Cu N4, 2(C3 H7 N O)' Add
_chemical_formula_sum       'C22 H32 Br2 Cu N6 O2'
_chemical_formula_weight        635.89
_cell_measurement_reflns_used    4749         Add*
_cell_measurement_theta_min      2.6815       Add*
 _cell_measurement_theta_max      31.3544      Add*


_exptl_crystal_description       block        Add*
_exptl_crystal_colour            black        Add*
```

**This part add information from the *.p4p file here*

```
_exptl_absorpt_correction_type    'multi-scan'  Add
_exptl_absorpt_correction_T_min   0.479534      Add**
_exptl_absorpt_correction_T_max   0.746217      Add**
_exptl_absorpt_process_details    'TWINABS (Sheldrick, 2009)' Add
```

***This part add information from the *.abs file here*

```
_diffrn_source                    'fine focus sealed tube'   Add
_diffrn_measurement_device_type   'Bruker AXS SMART APEX CCD diffractometer'   Add
_diffrn_measurement_method        'omega and phi scans'      Add

_computing_data_collection        'Apex2 v2012.4-3 (Bruker, 2012)' Add
_computing_cell_refinement        'SAINT V8.18C (Bruker, 2012)' Add
_computing_data_reduction         'SAINT V8.18C (Bruker, 2012)' Add
_computing_structure_solution     'SHELXS-97 (Sheldrick, 2008)' Add
_computing_structure_refinement
;
SHELXL-2012 (Sheldrick, 2012),
SHELXLE Rev609 (H\"ubschle et al., 2011) Add
;
```

_computing_molecular_graphics 'Shelxtl (Bruker, 2003)' Add
_computing_publication_material 'Shelxtl (Bruker, 2003)' Add

_atom_sites_solution_primary direct Add
_atom_sites_solution_secondary difmap Add

_exptl_special_details
;
Reflections 0 2 0 was affected by the beam stop and were omitted from the refinement.
The crystal under investigation was found to be non-merohedrally
twinned. The orientation matrices for the three components were
identified using the program Cell_Now, with the three components
being related by a 180 degree rotati on around the real and reciprocal
axis a ans b. The three components were integrated using Saint,
resulting in the following statistics:

```
  10 data (  10 unique) involve domain  1 only,  mean I/sigma  56.5
 6140 data (2033 unique) involve domain  2 only,  mean I/sigma  25.9
   7 data (   7 unique) involve domain  3 only,  mean I/sigma  12.1
 5470 data (1957 unique) involve   2 domains,  mean I/sigma  26.2
 7382 data (2649 unique) involve   3 domains,  mean I/sigma  29.6
   2 data (   2 unique) involve   4 domains,  mean I/sigma  23.2
   1 data (   1 unique) involve   5 domains,  mean I/sigma 113.8
```

The exact twin matrix identified by the integration program was program was found to be (1.000 0.001 0.001 / 0.001 -1.000 -0.008 / -0.434 0.003 -1.000) and (-0.999 0.010 0.003 / 0.051 1.000 -0.009 / -0.006 -0.002 -1.001).

The data were corrected for absorption using twinabs, and
the structure was solved using direct methods with only the
non-overlapping reflections of component 1. The structure was
refined using the hklf 5 routine with all reflections of component
1 (including the overlapping ones), resulting in a BASF value of
0.457(2) to 0.107(29).

The Rint value given is for all reflections and is based on
agreement between observed single and composite intensities
and those calculated from refined unique intensities and twin
fractions (TWINABS (Sheldrick, 2009)).
; Add

----------------------------*The end*-------------------------------------