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.

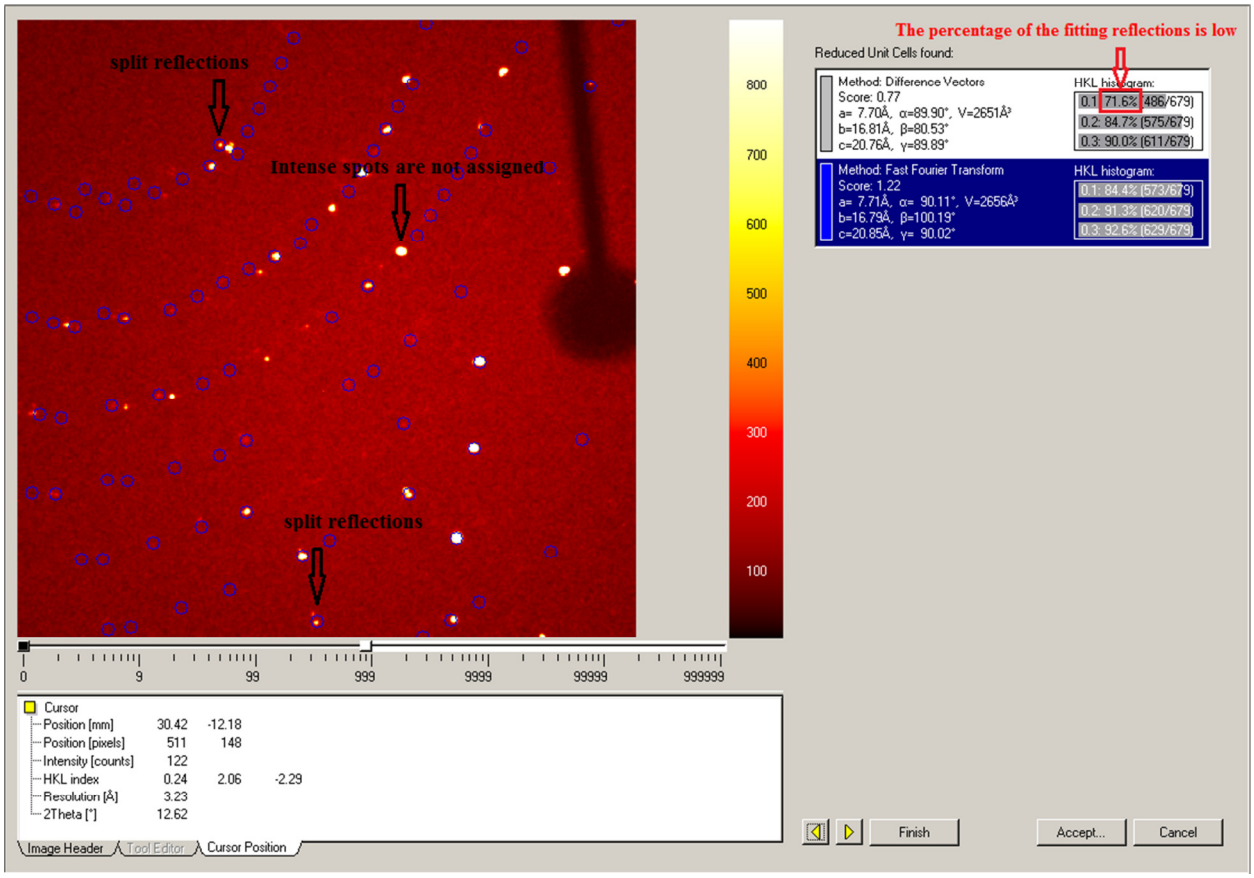


Figure 1. Unit cell indexing result window with easily recognizable warning signs for non-merohedral twinning.

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.

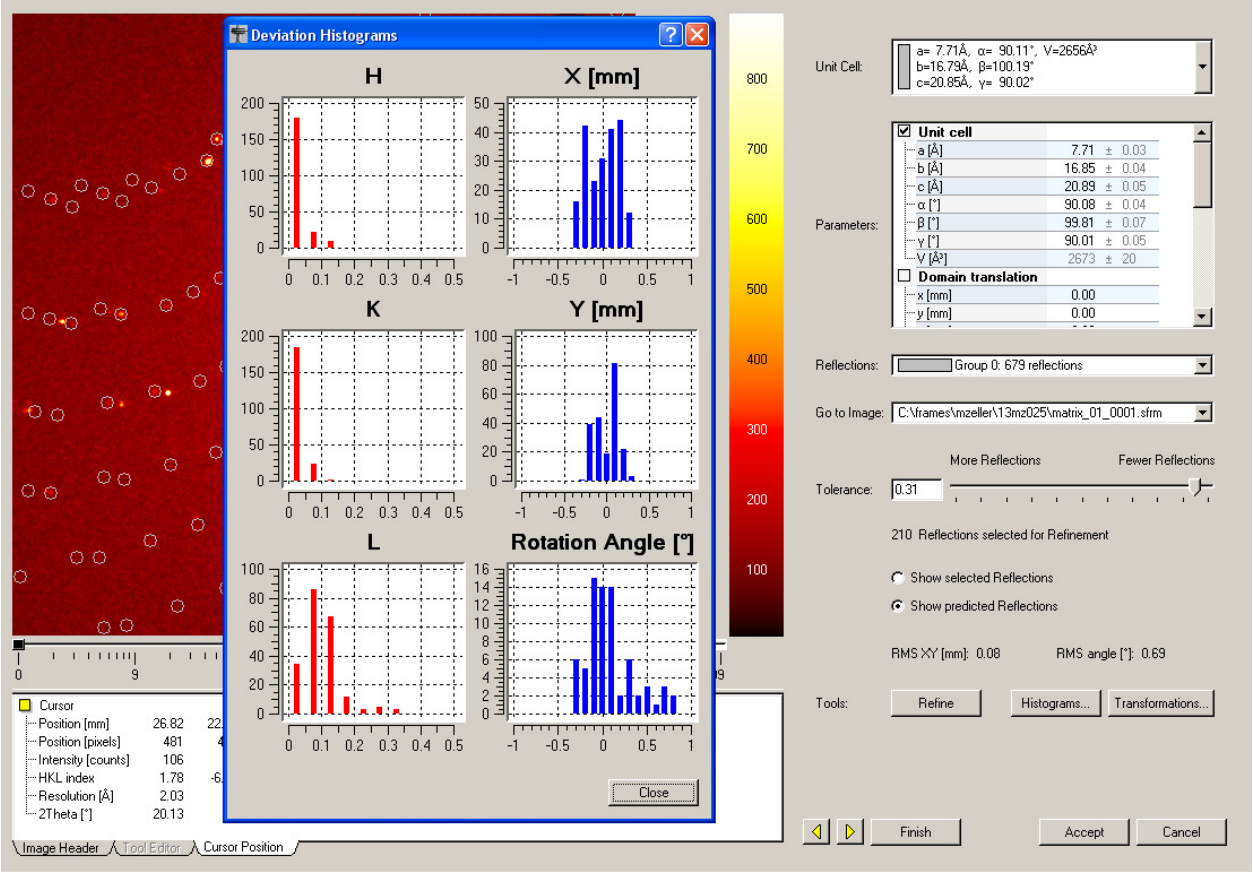


Figure. 2. The refinement window with <Histograms> tool

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>.

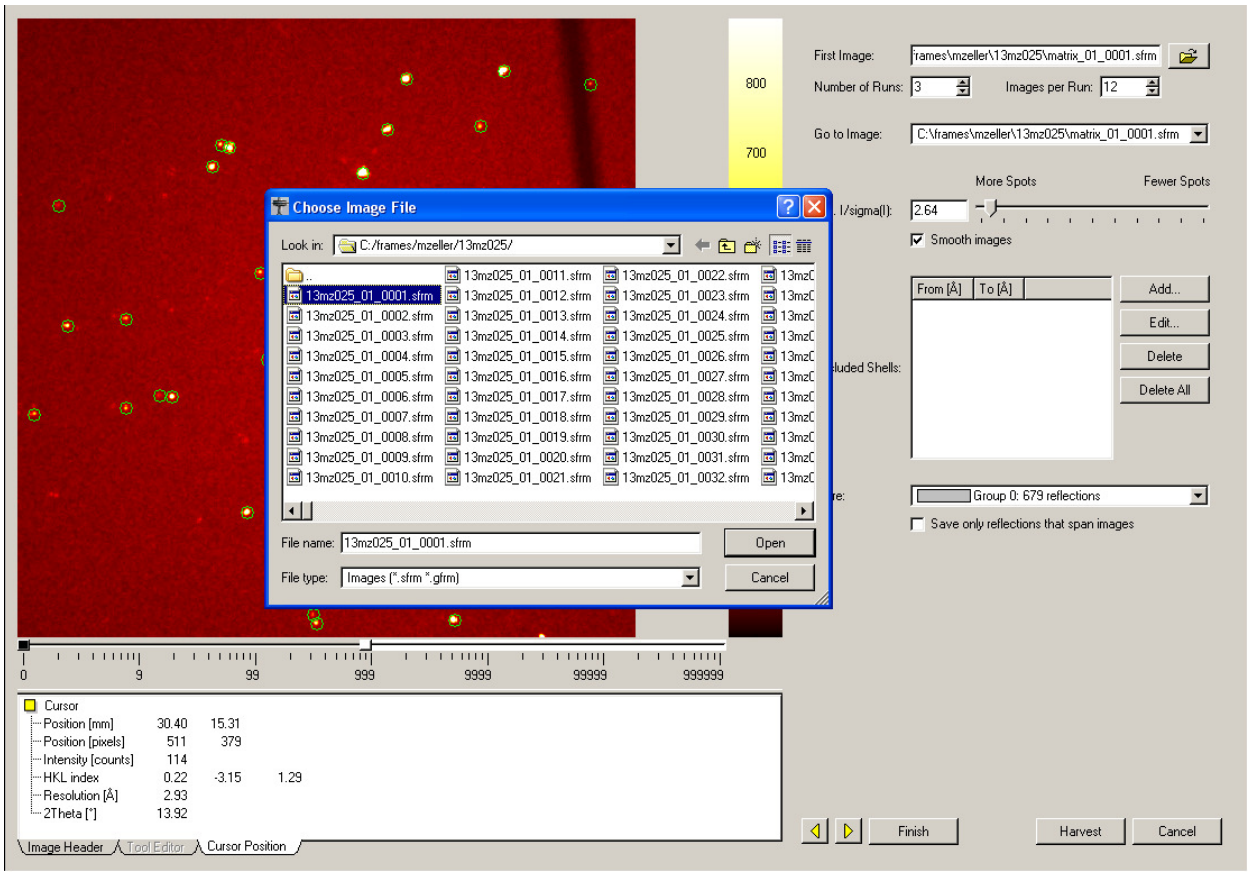


Figure. 3. Unit cell window.

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.

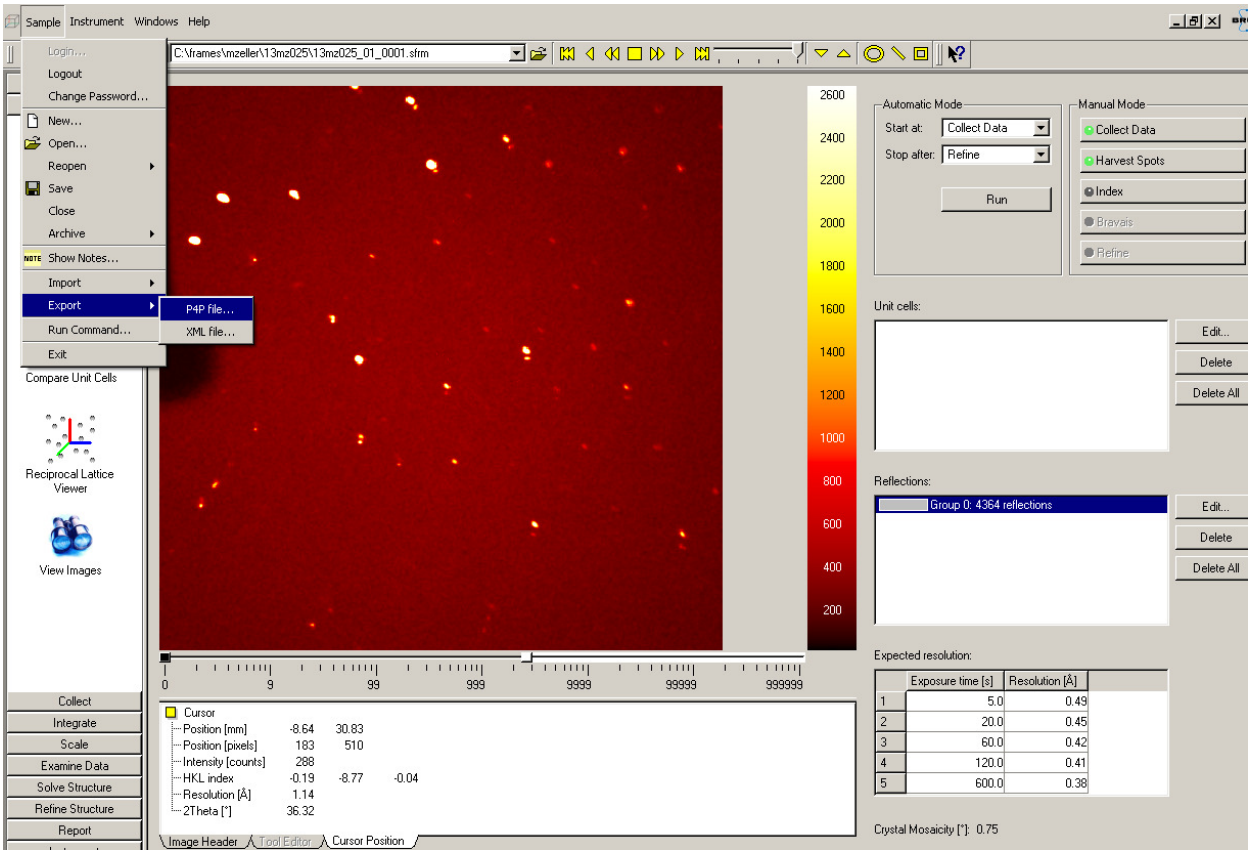


Figure. 4. Unit cell window.

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

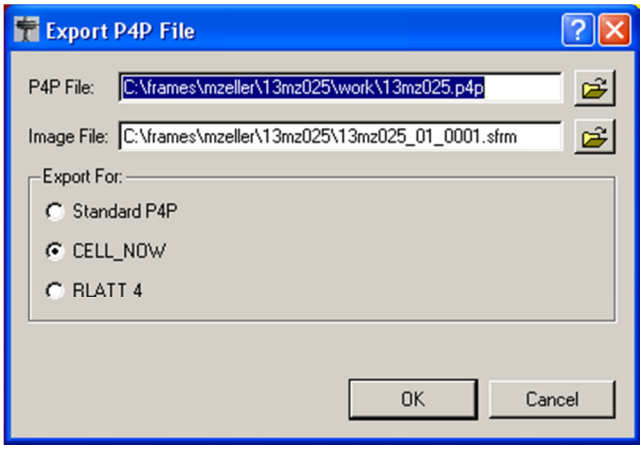


Figure. 5. The p4p file window.

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.

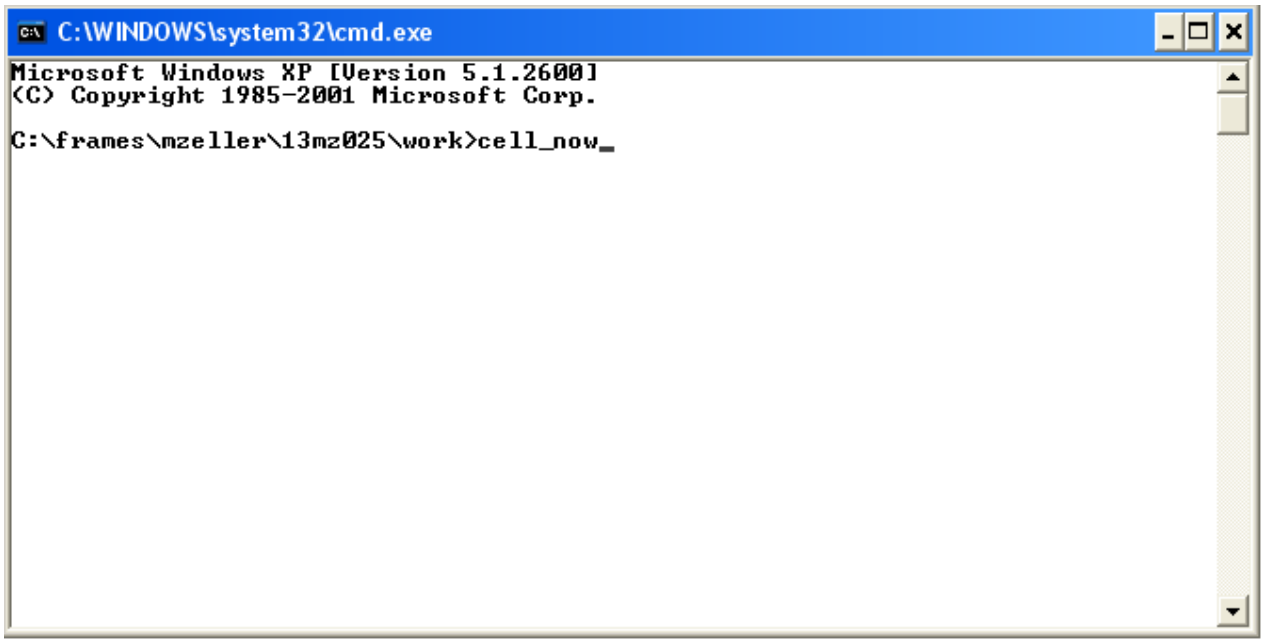


Figure. 6. The Run command window.

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

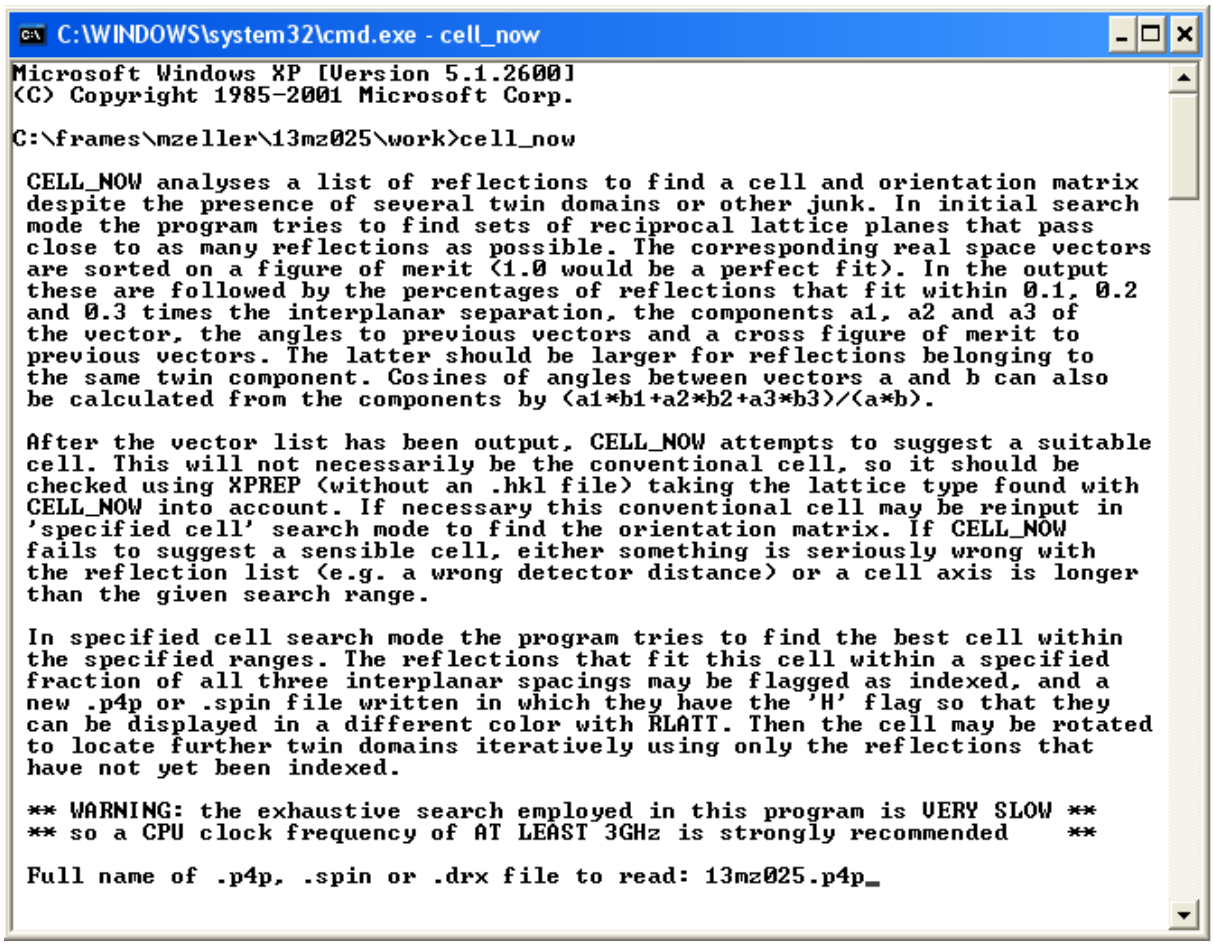


Figure. 7. The Run command window.

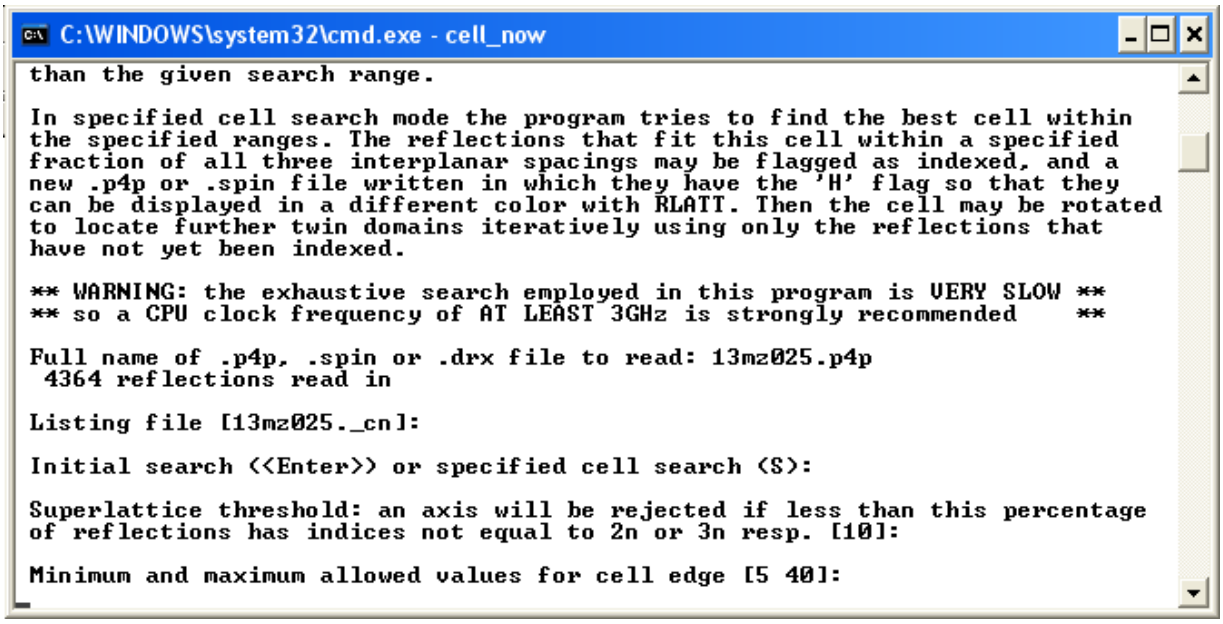


Figure. 8. The Run command window.

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.

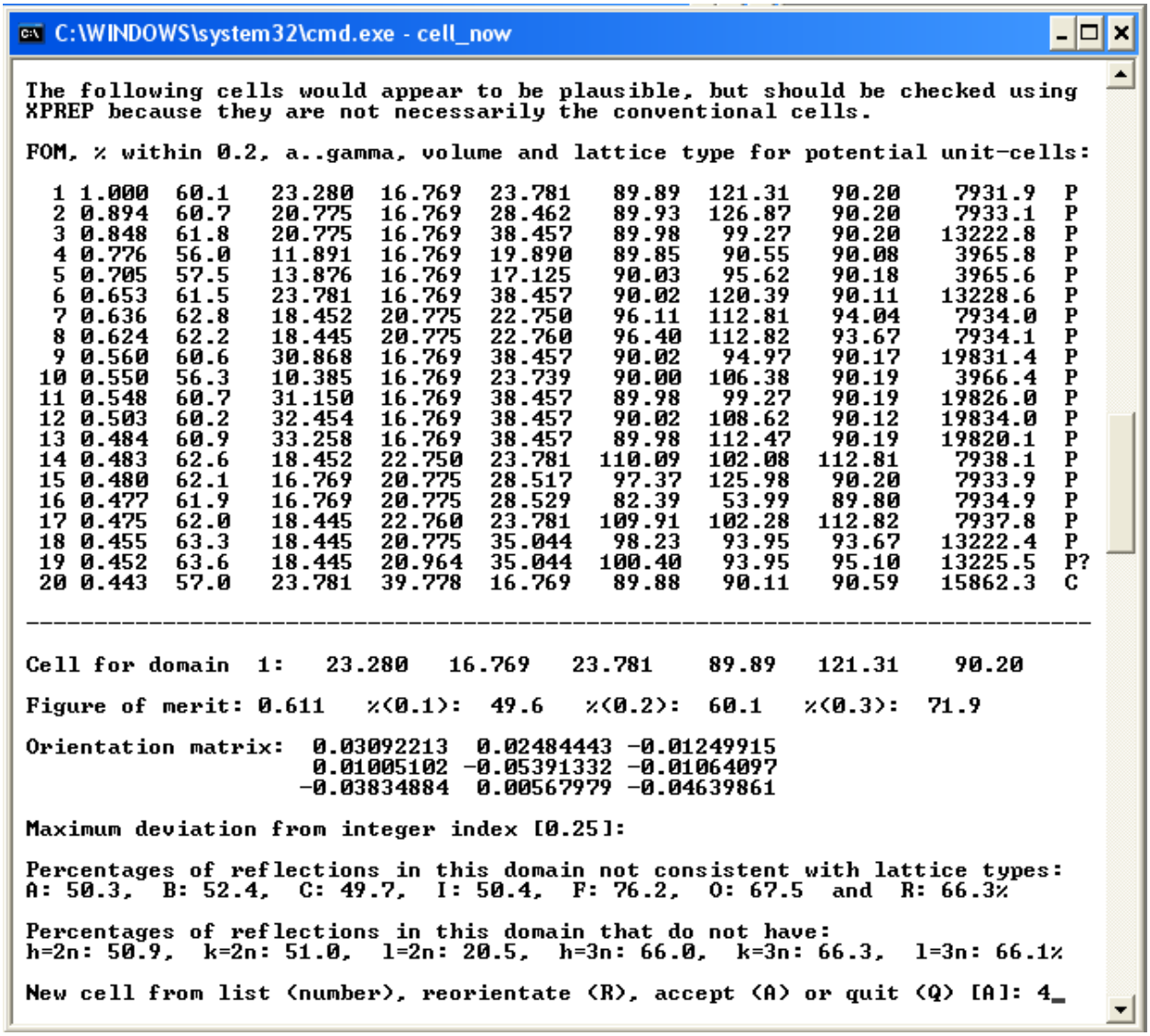


Figure. 9. The Run command window.

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.

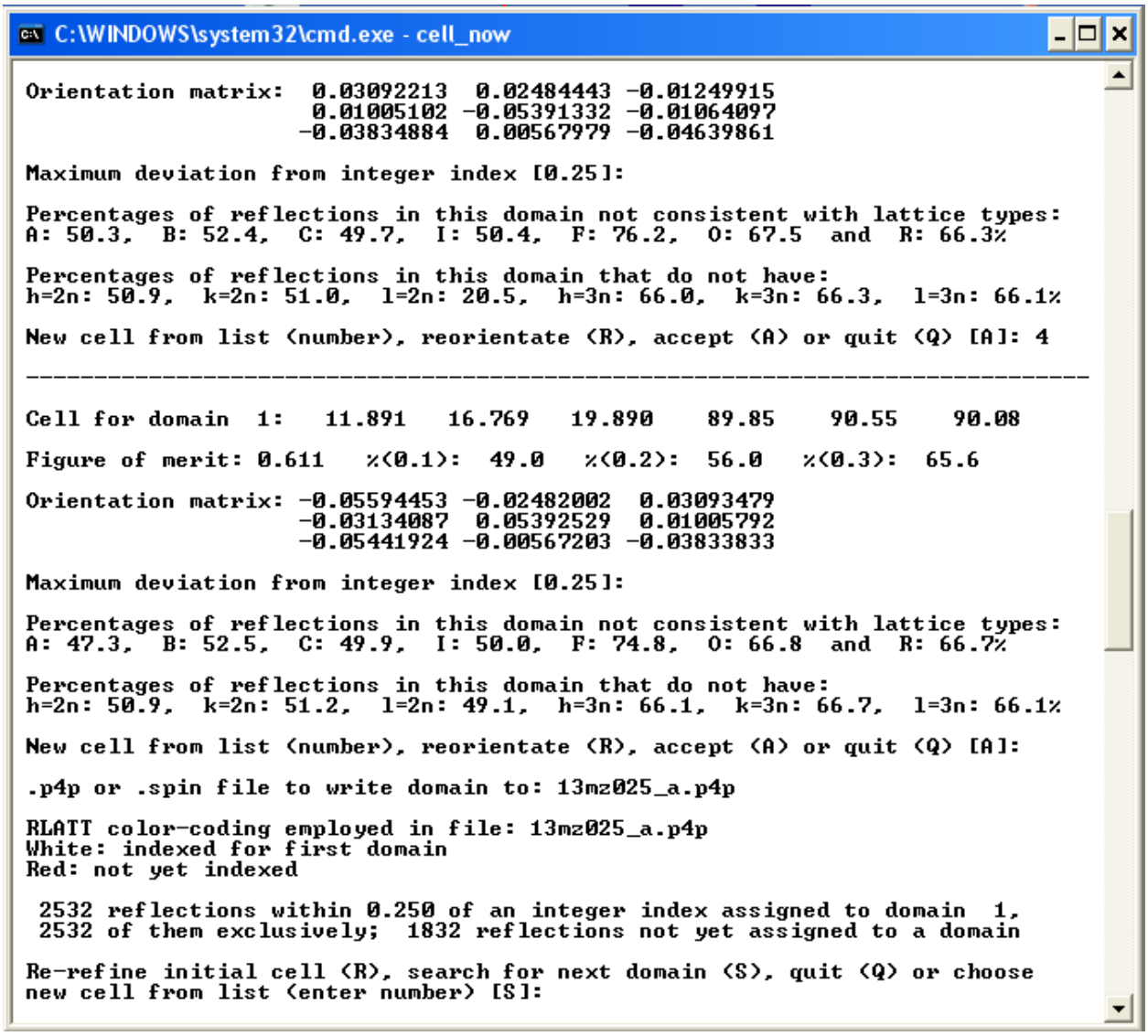


Figure. 10. The Run command window.

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).

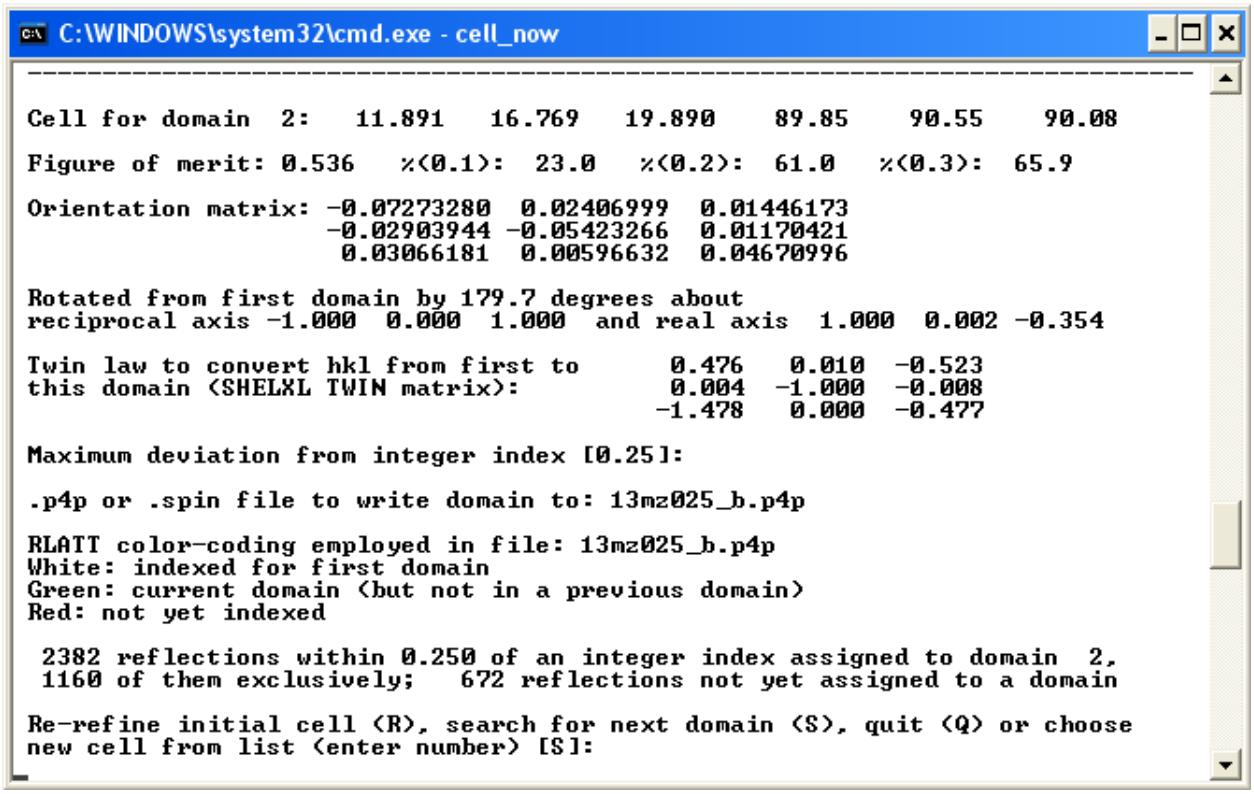


Figure. 11. The Run command window.

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

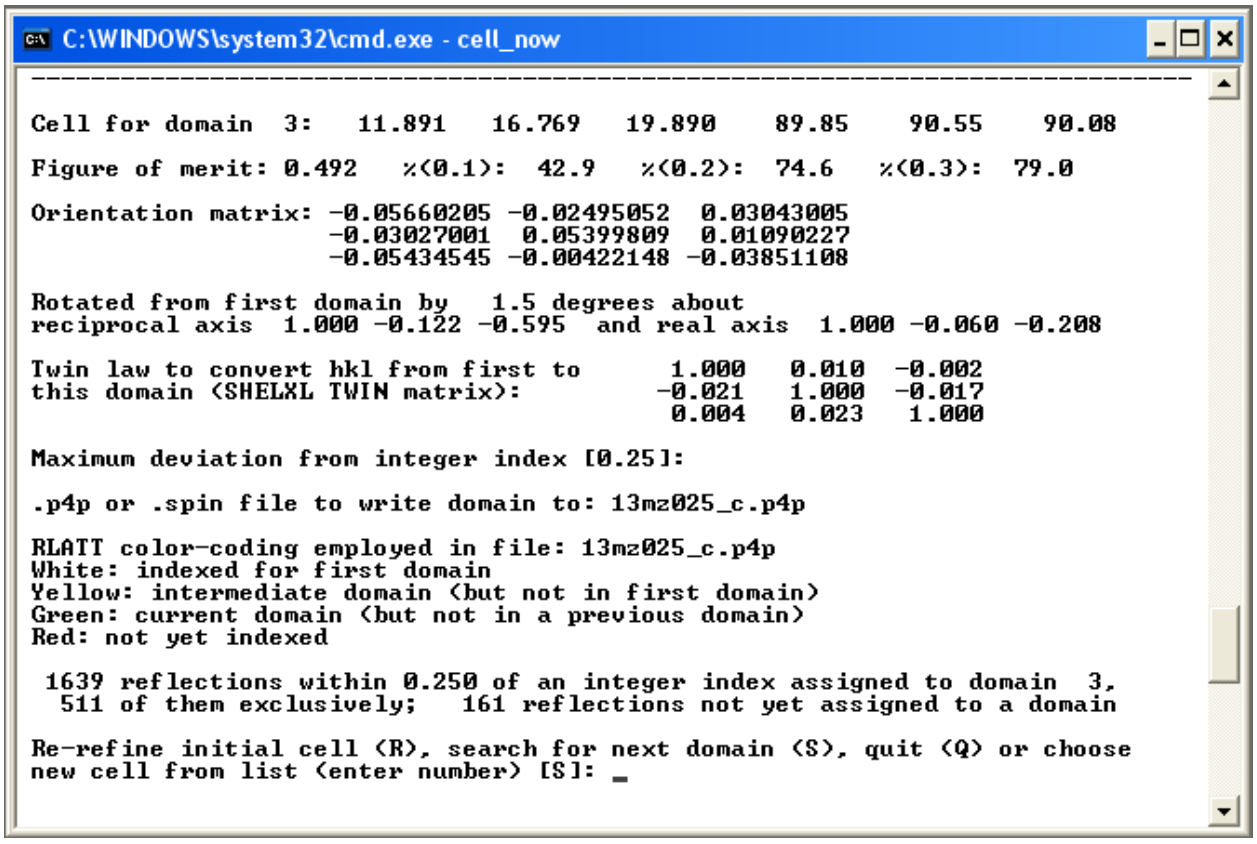


Figure. 12. The Run command window.

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.

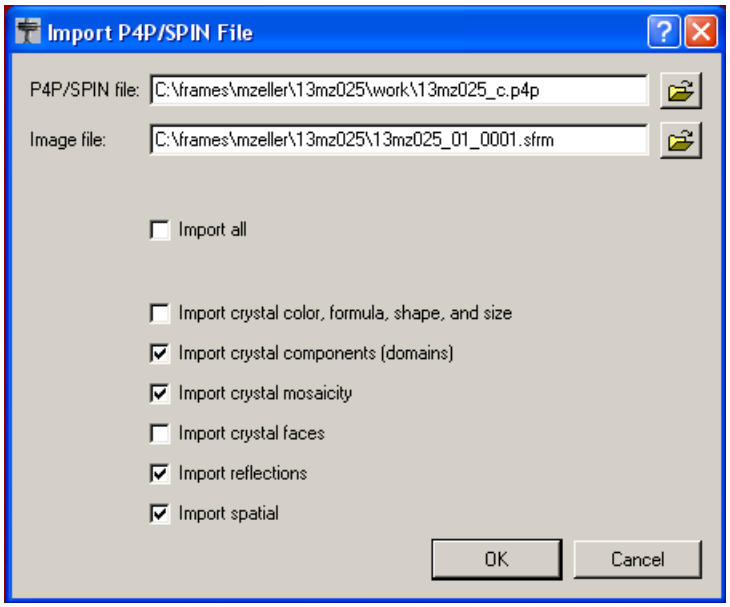


Figure. 13. The ImportP4P/SPIN File window.

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

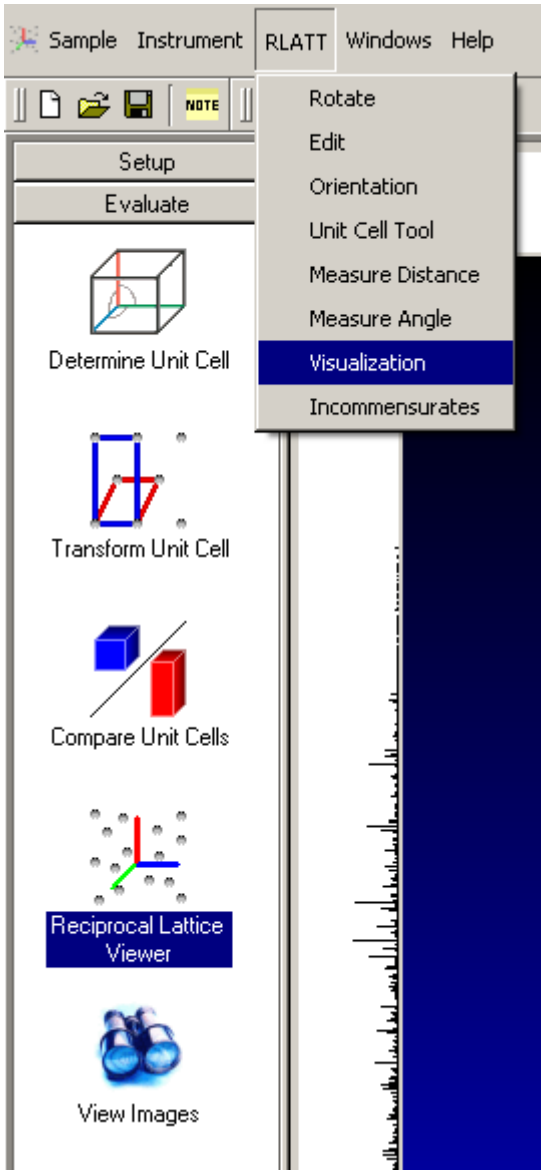


Figure. 14. Open <RLATT> tools.

- 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.

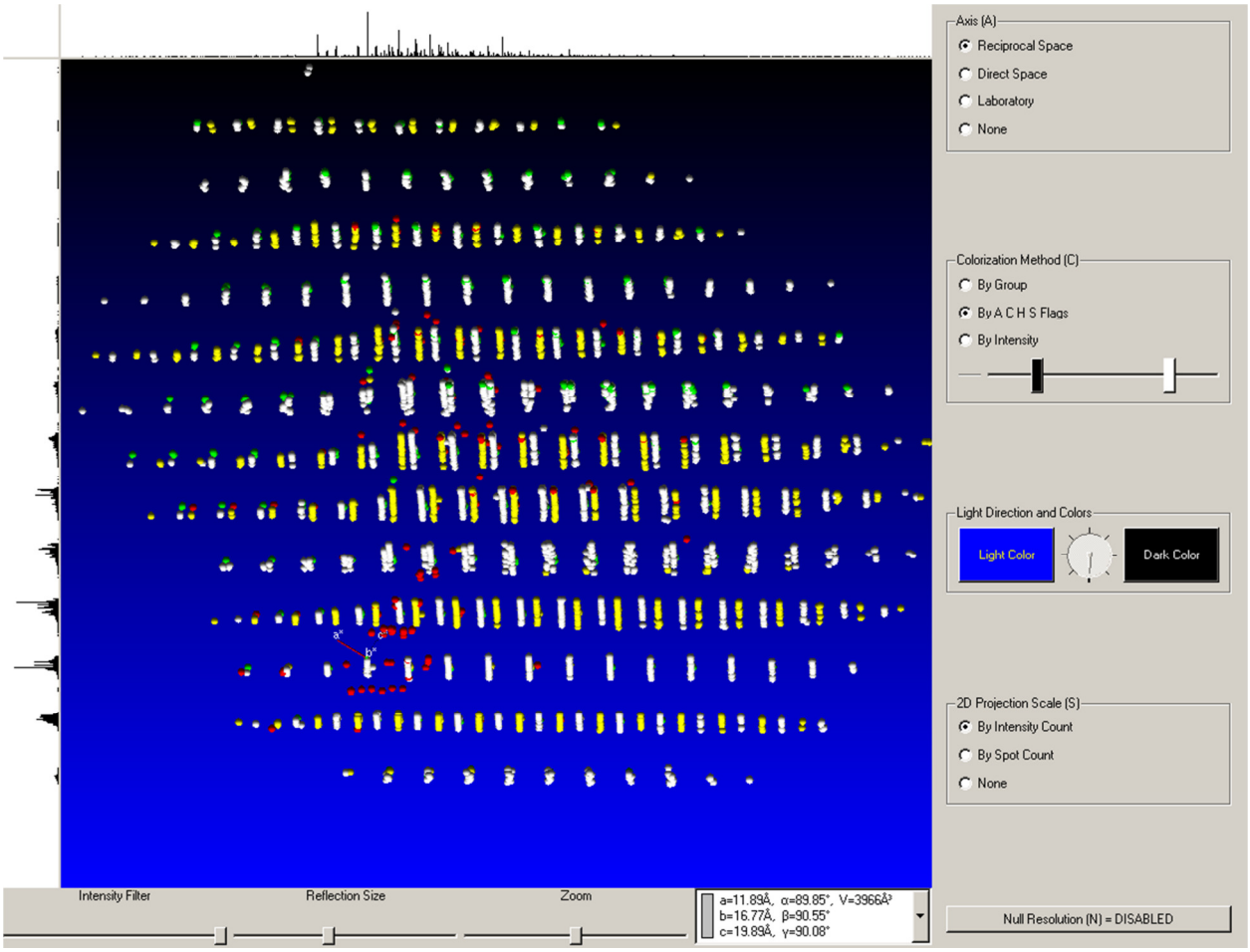


Figure. 15. A typical reciprocal lattice view of a non-merohedrally twinned crystal. Two lattices are clearly visible (white and yellow), with reflections overlapping in some rows, and displaying separate moieties in other rows.

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).

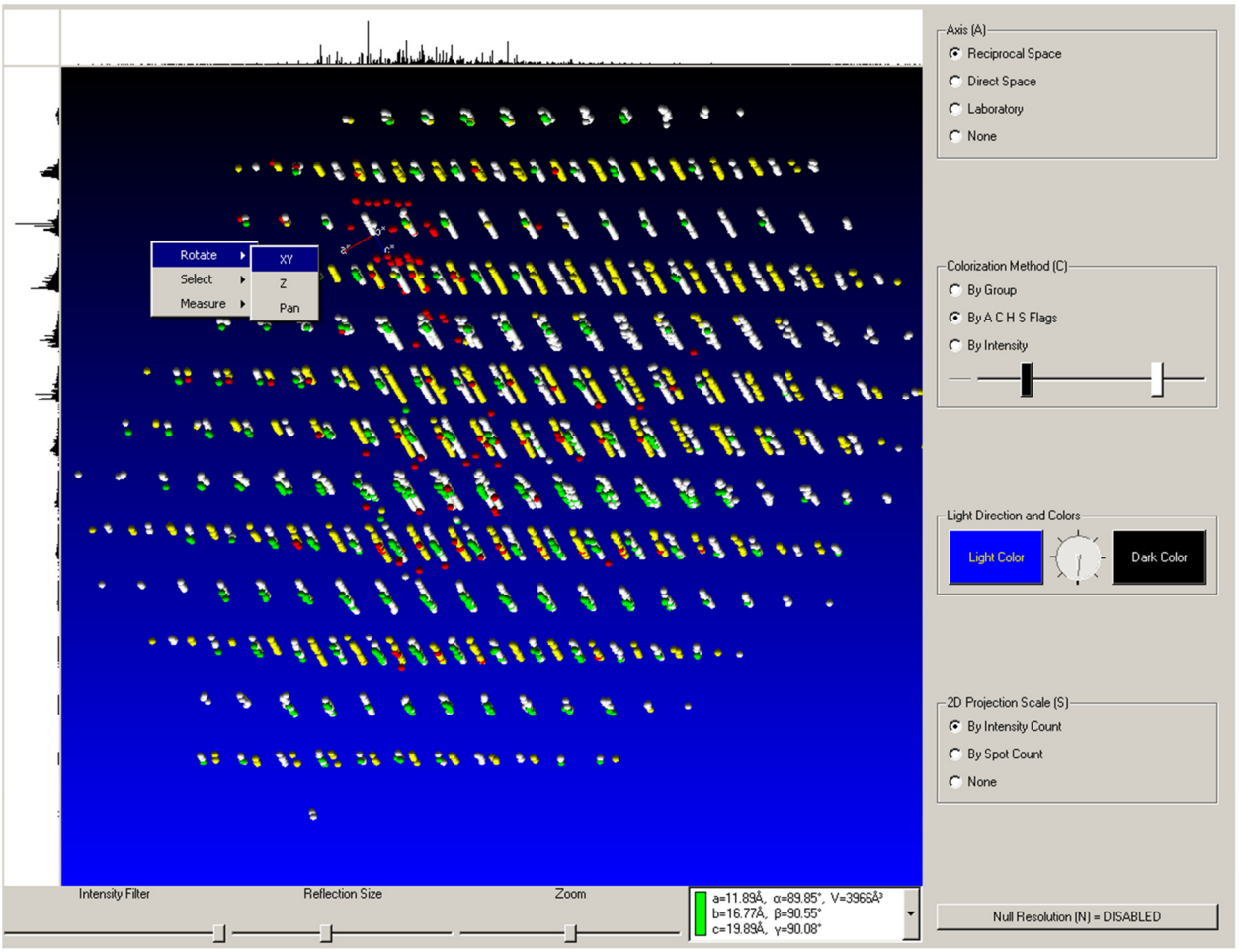


Figure. 16. Above shows a typical reciprocal lattice view of a non-merohedrally twinned crystal.

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.

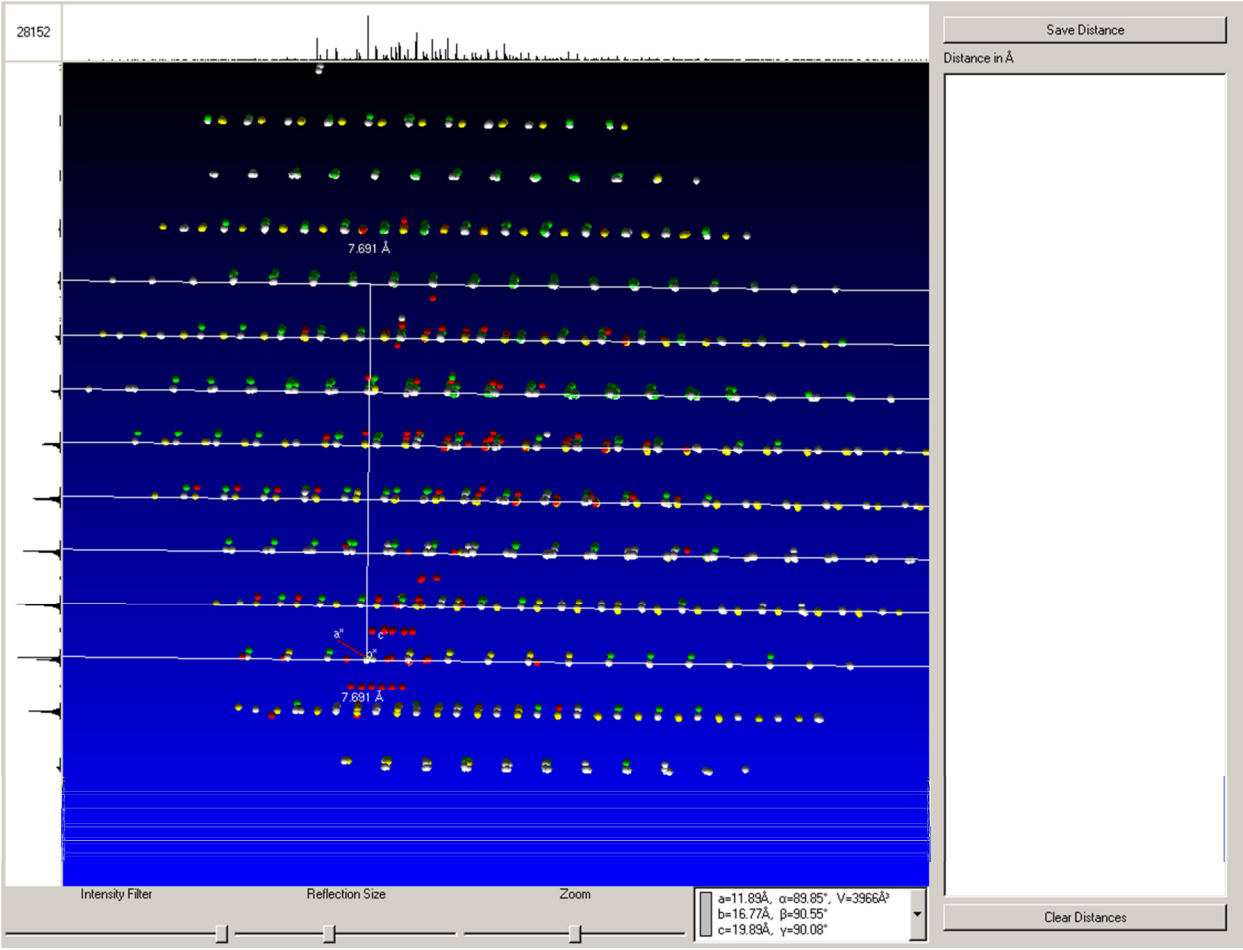


Figure. 17. The Reciprocal Lattice window with lattice parameter measurement, view down the b-axis from Cell Now. Note the disagreement of the directions of the a- and b-axes with the observed patterns, and the measured lattice constant value of 7.691 Å which is not found in the unit cell from Cell Now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

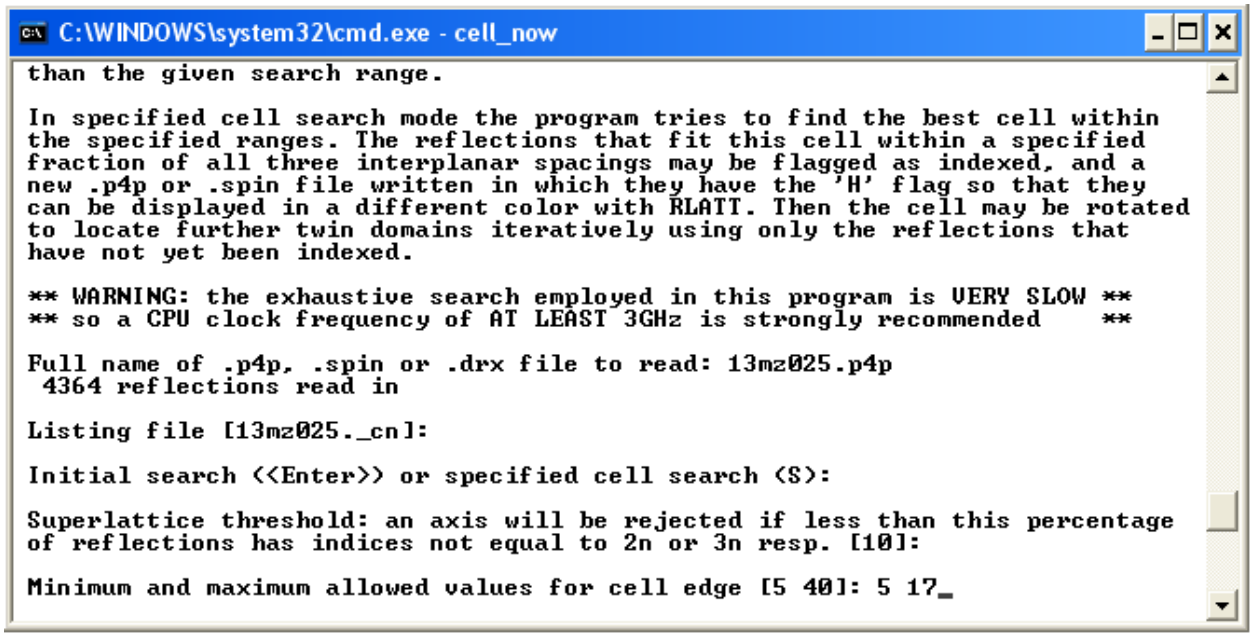


Figure. 18. The Run command window.

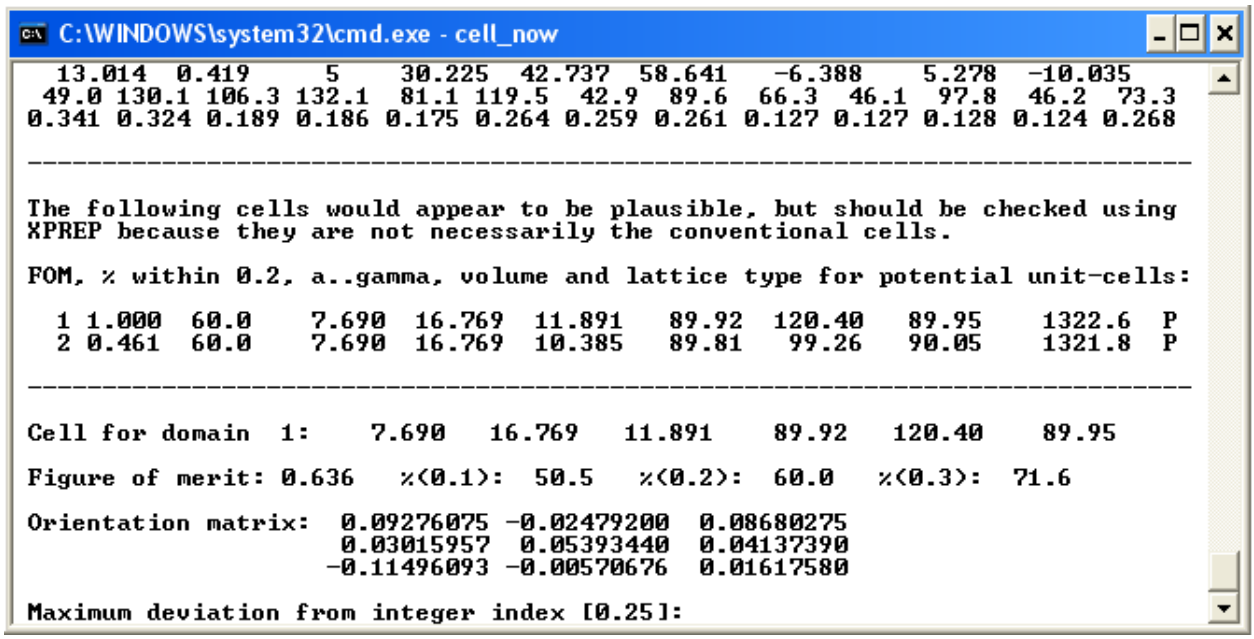


Figure. 19. The Run command window.

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).

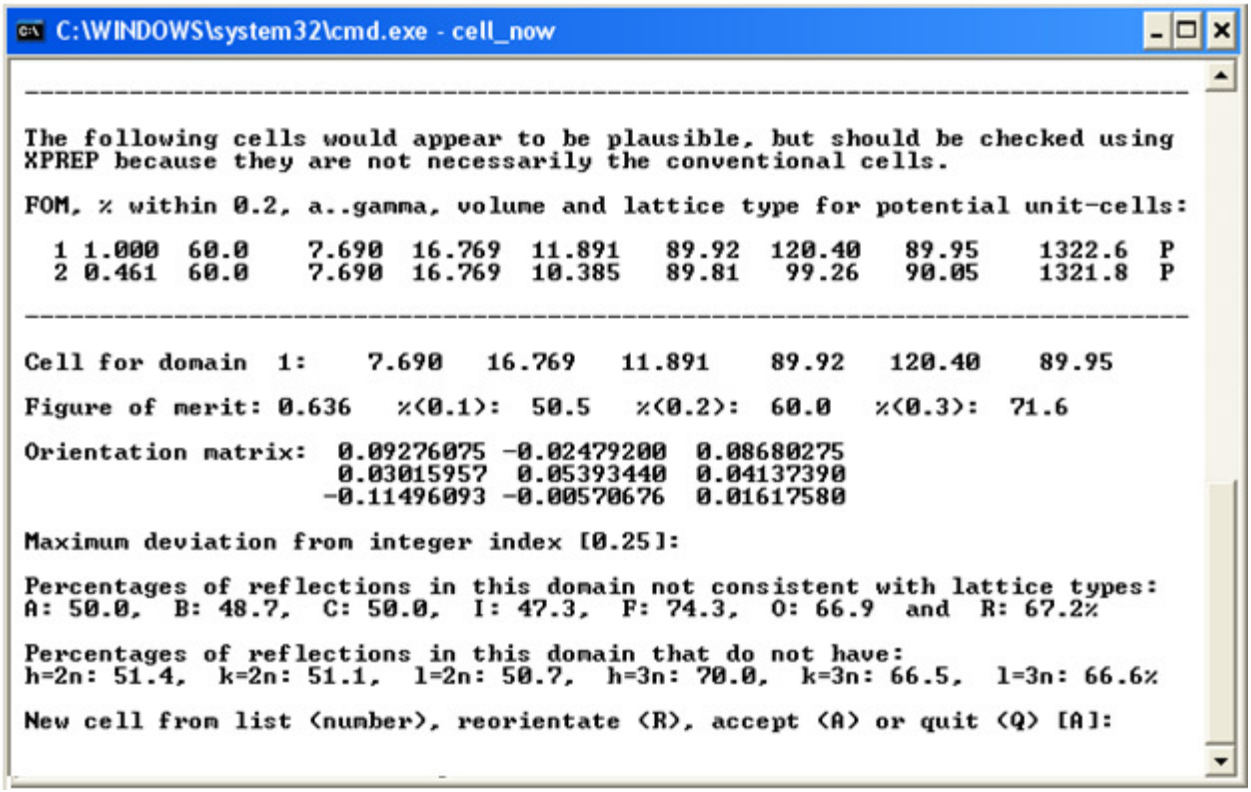


Figure. 20. The Run command window.

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).

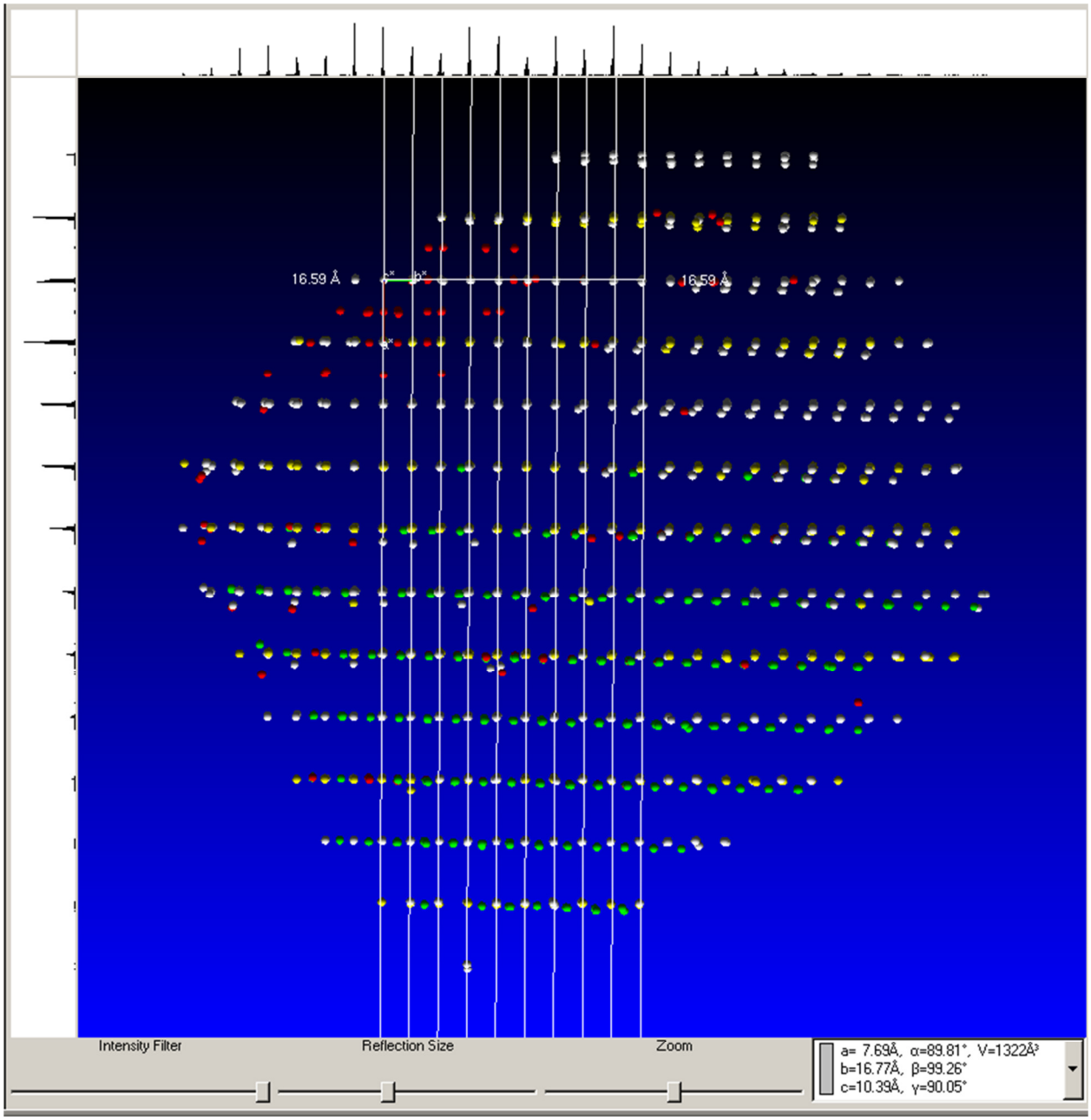


Figure. 22. The Reciprocal Lattice window with a non-merohedrally twinned crystal.

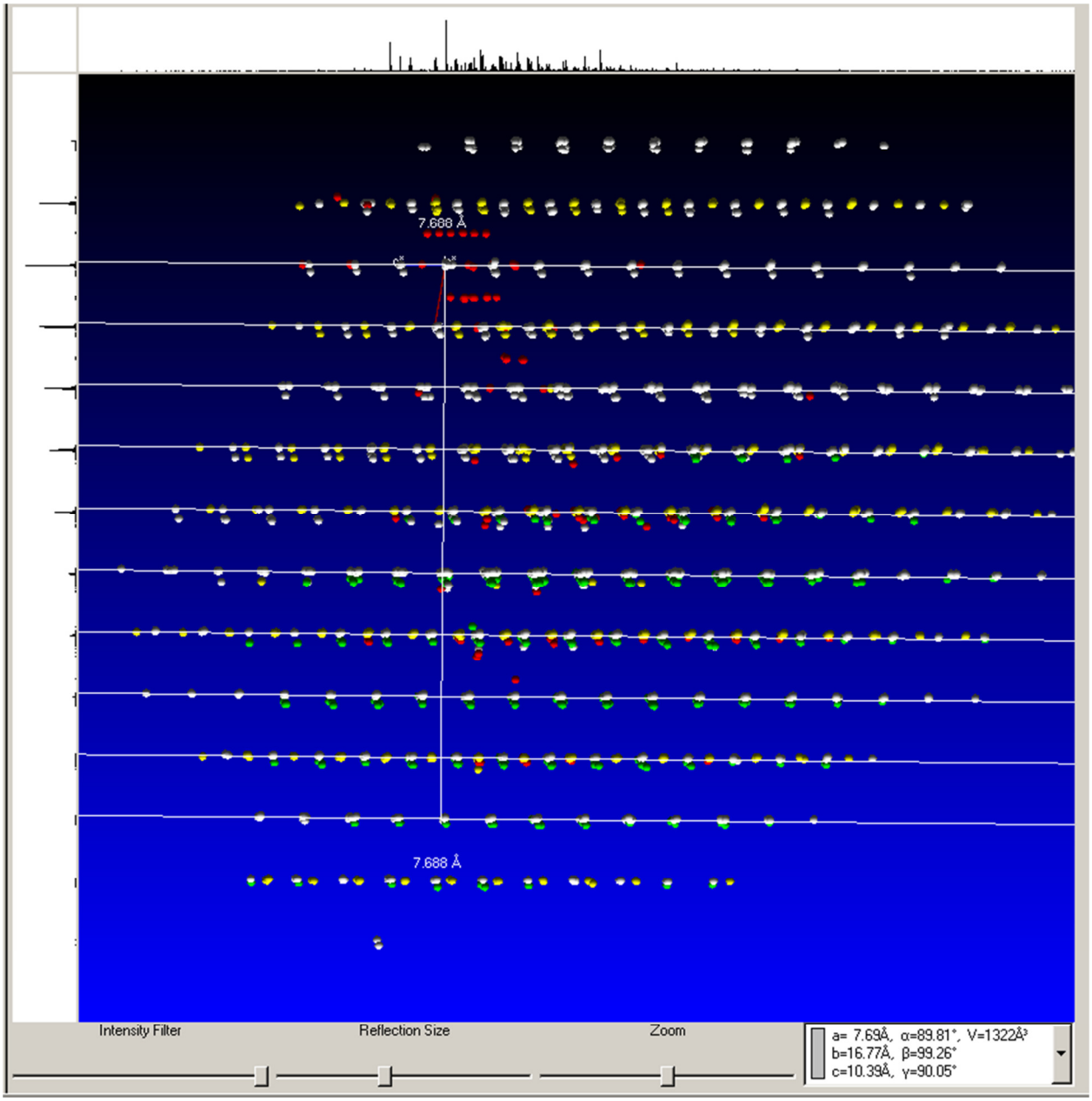


Figure. 23. The Reciprocal Lattice window with a non-merohedrally twinned crystal.

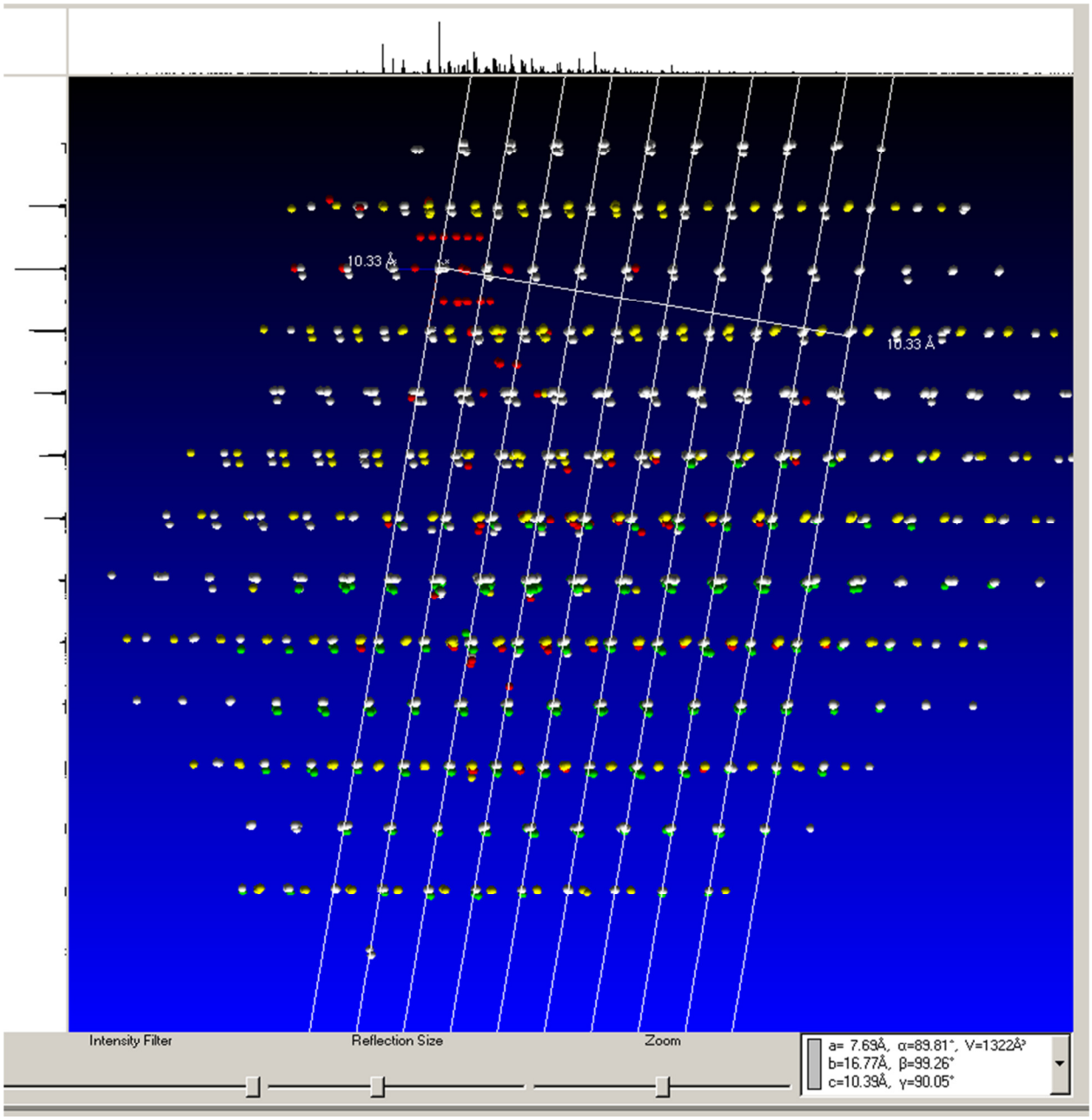


Figure. 24. The Reciprocal Lattice window with a non-merohedrally twinned crystal.

- 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.

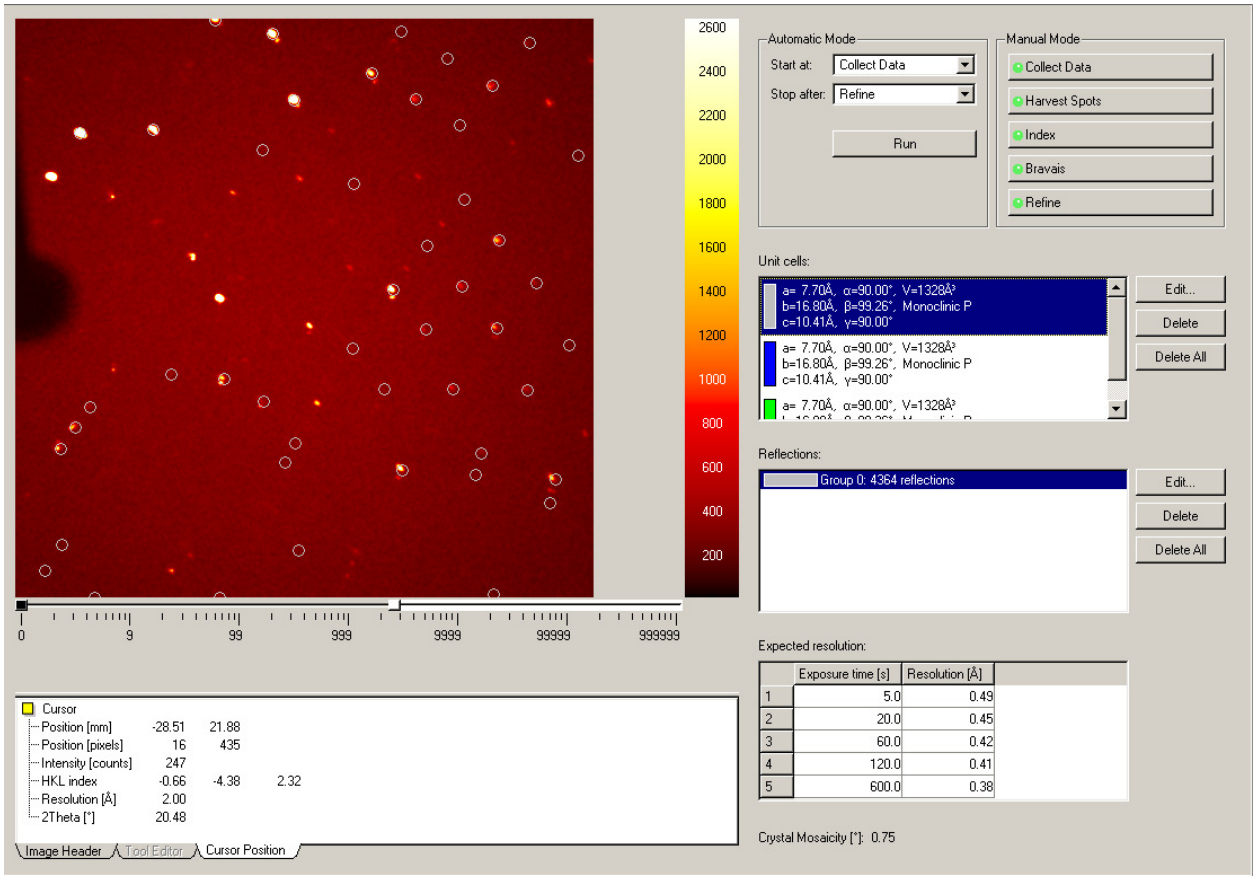
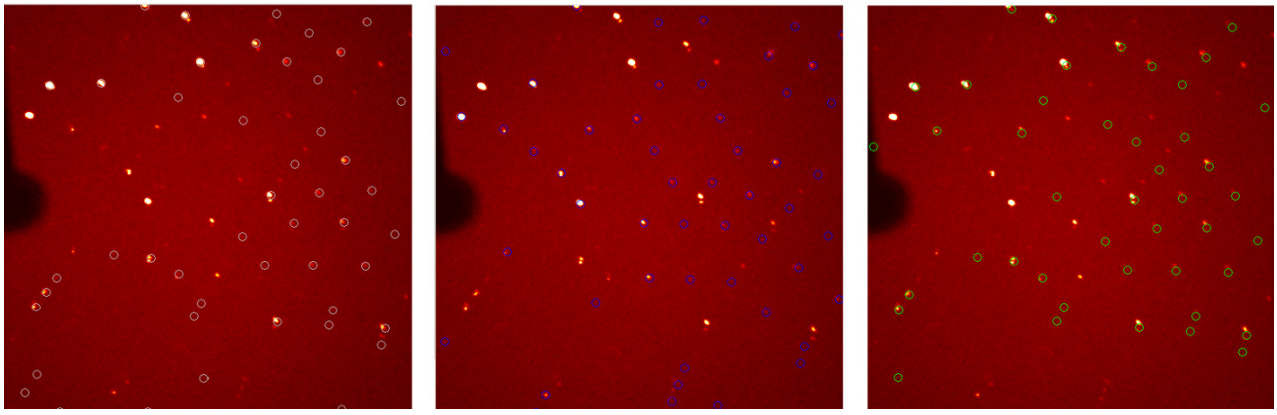


Figure. 25. Unit cell window. Predicted spot positions for moiety 1 shown.

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).

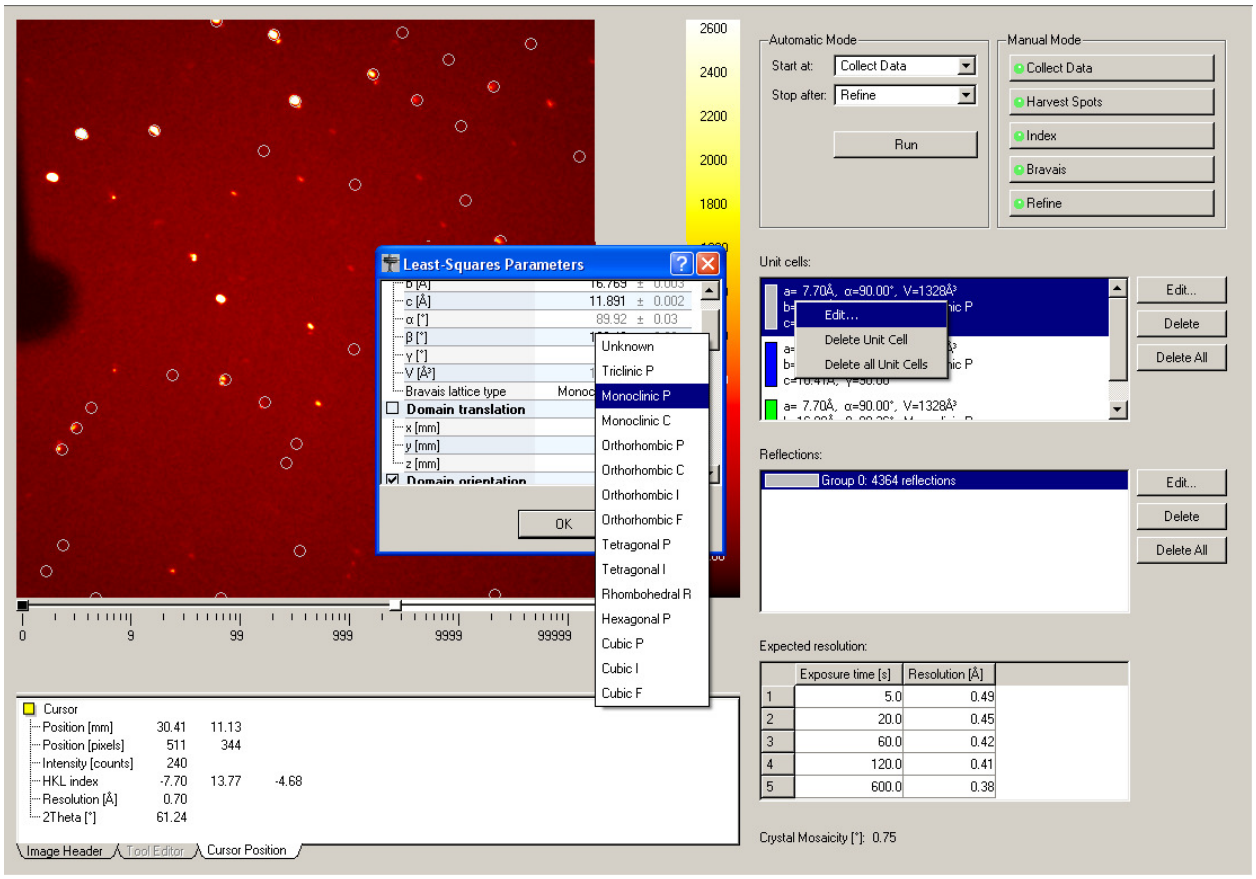


Figure. 27. Unit cell window.

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>.

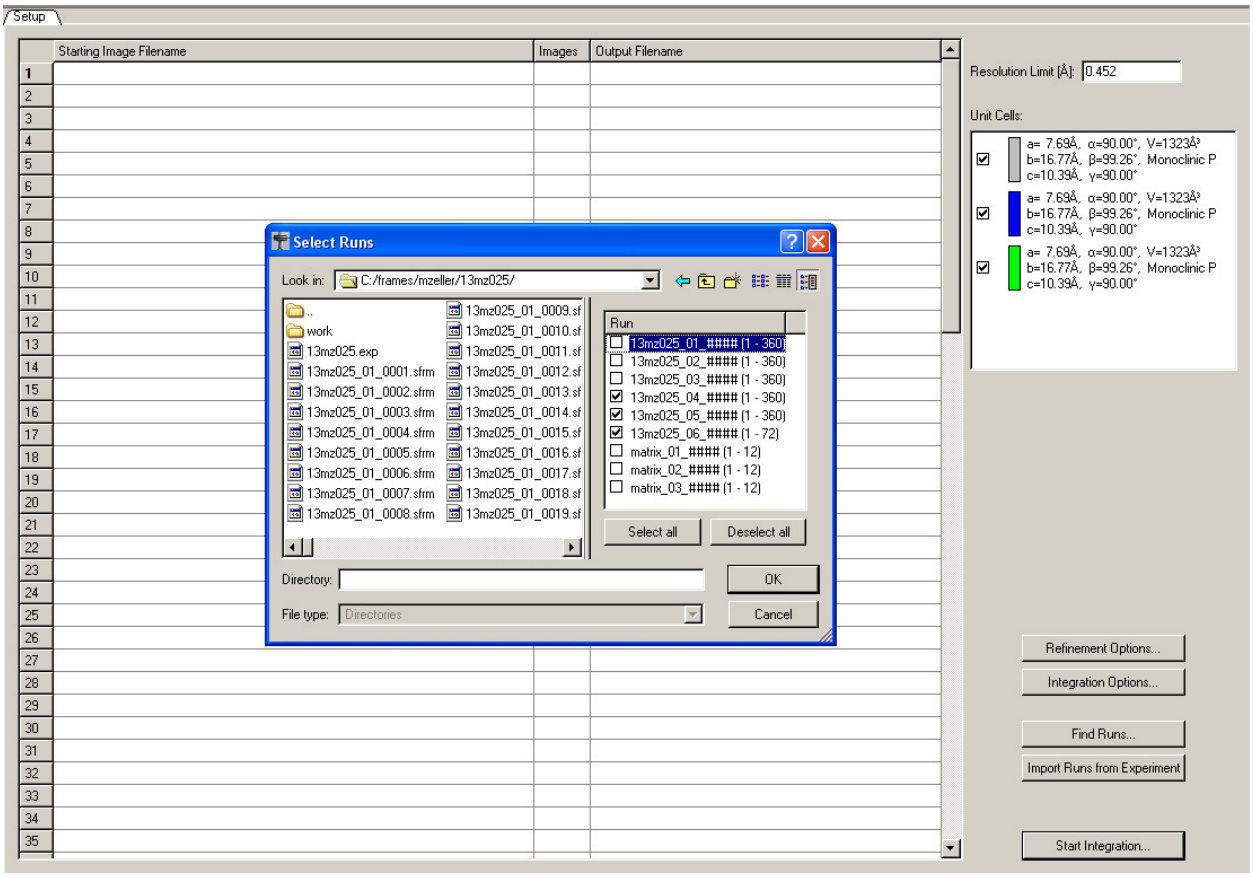


Figure. 28. Integration Start Window with Fine Runs window open.

[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.

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

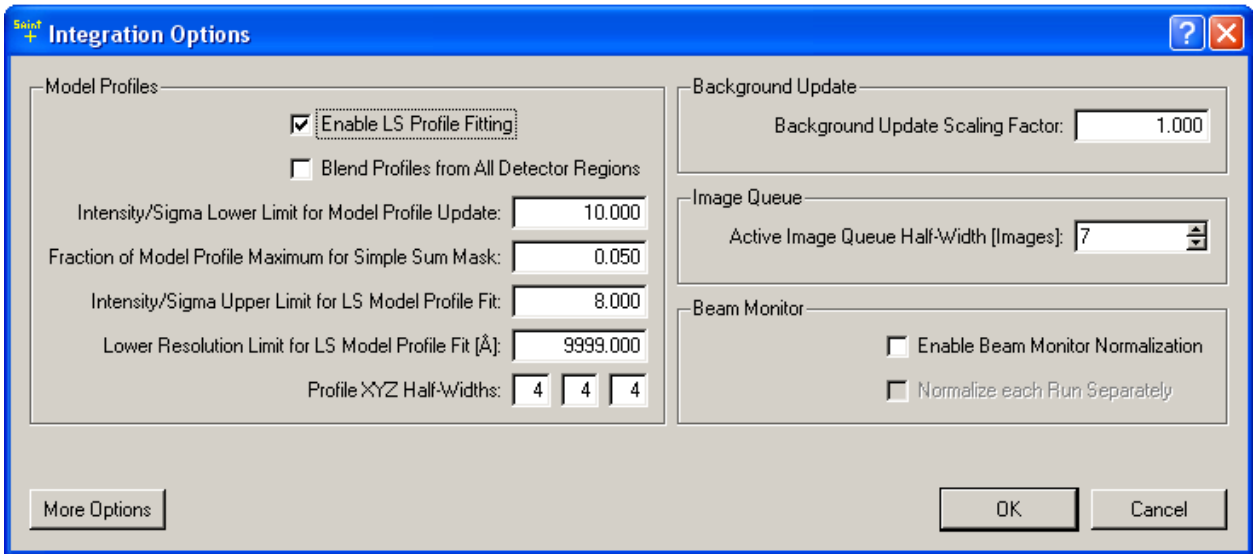


Figure. 29. Integration Option window.

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

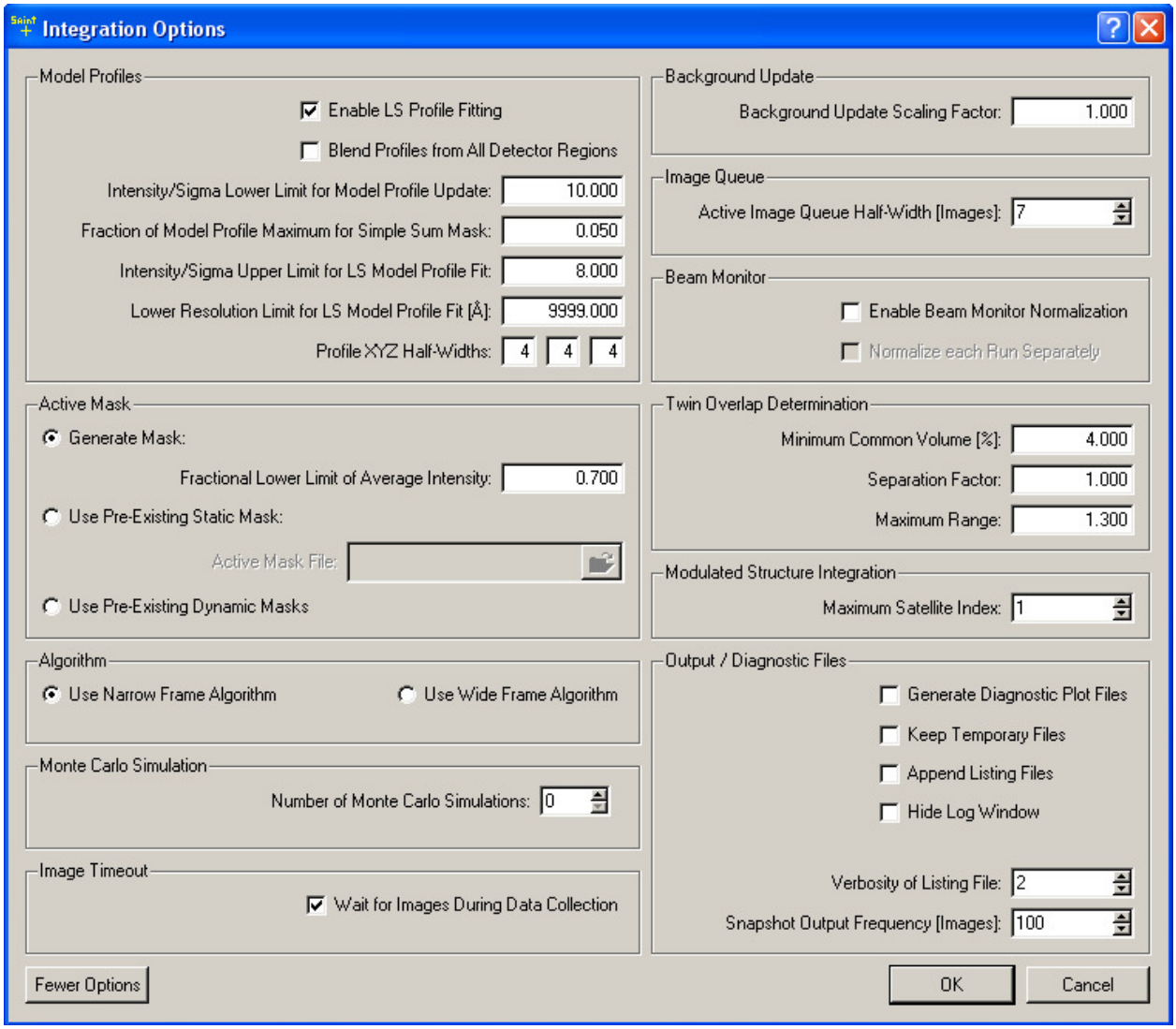


Figure. 30. Integration Option window.

- Click on <Start integration> button.

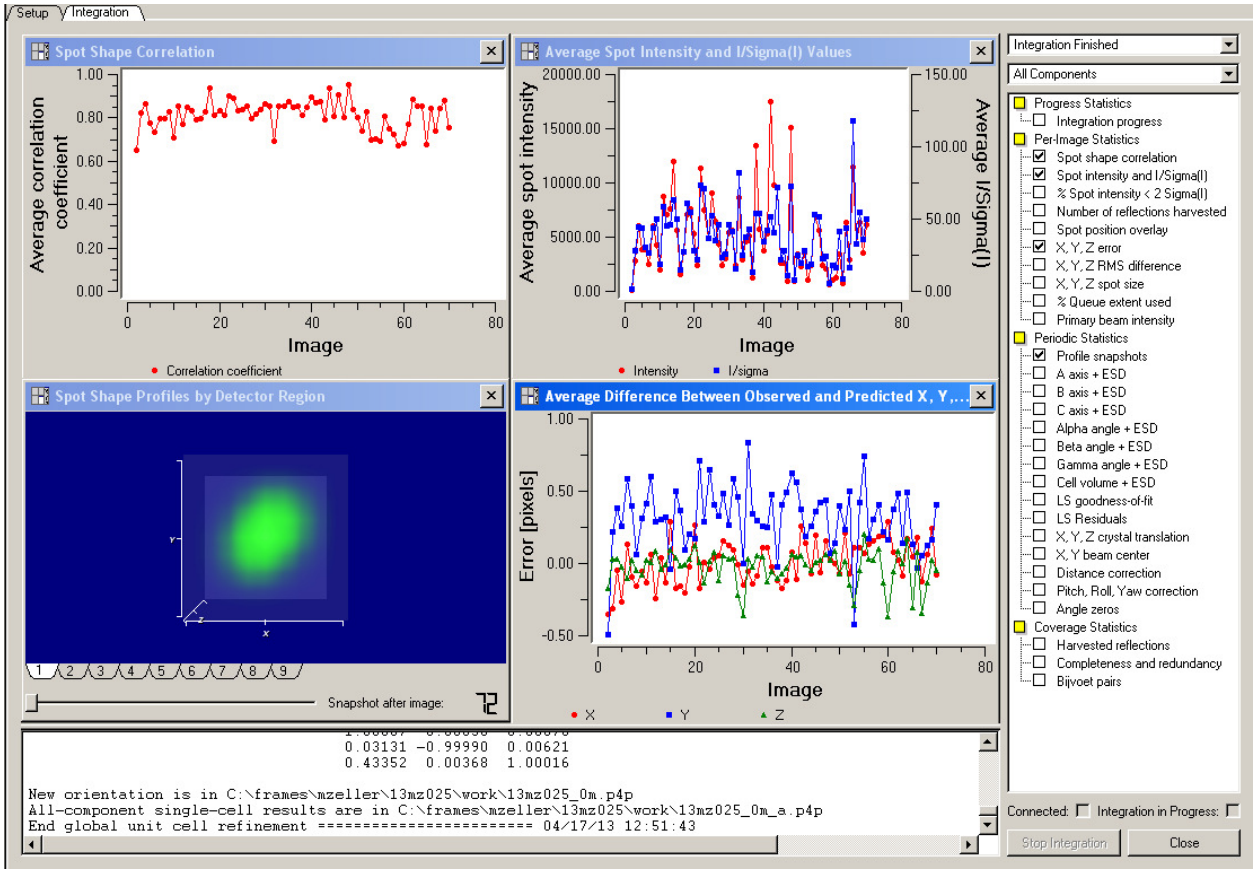


Figure. 31. Integration Option window with active Integration running.

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

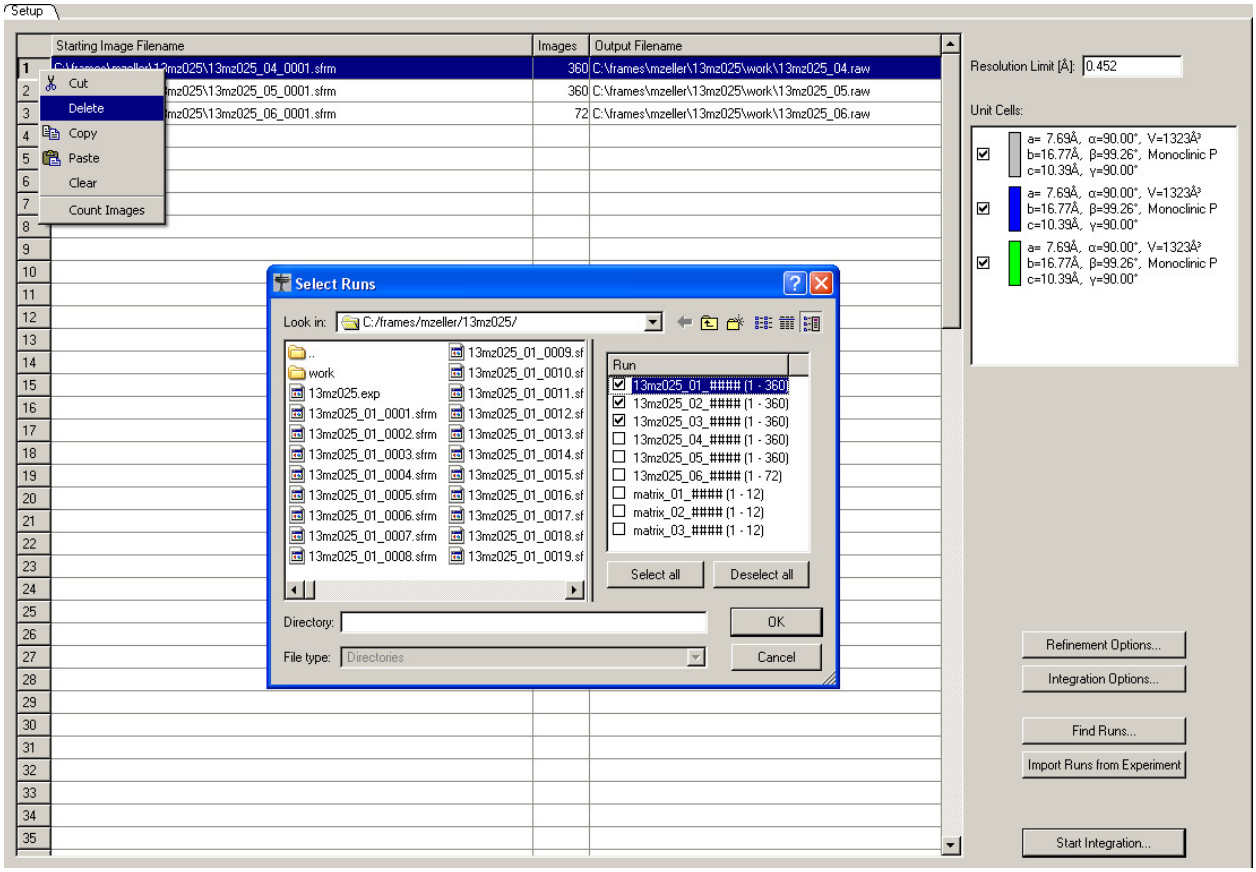


Figure. 32. Integration Start Window with Fine Runs window open.

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

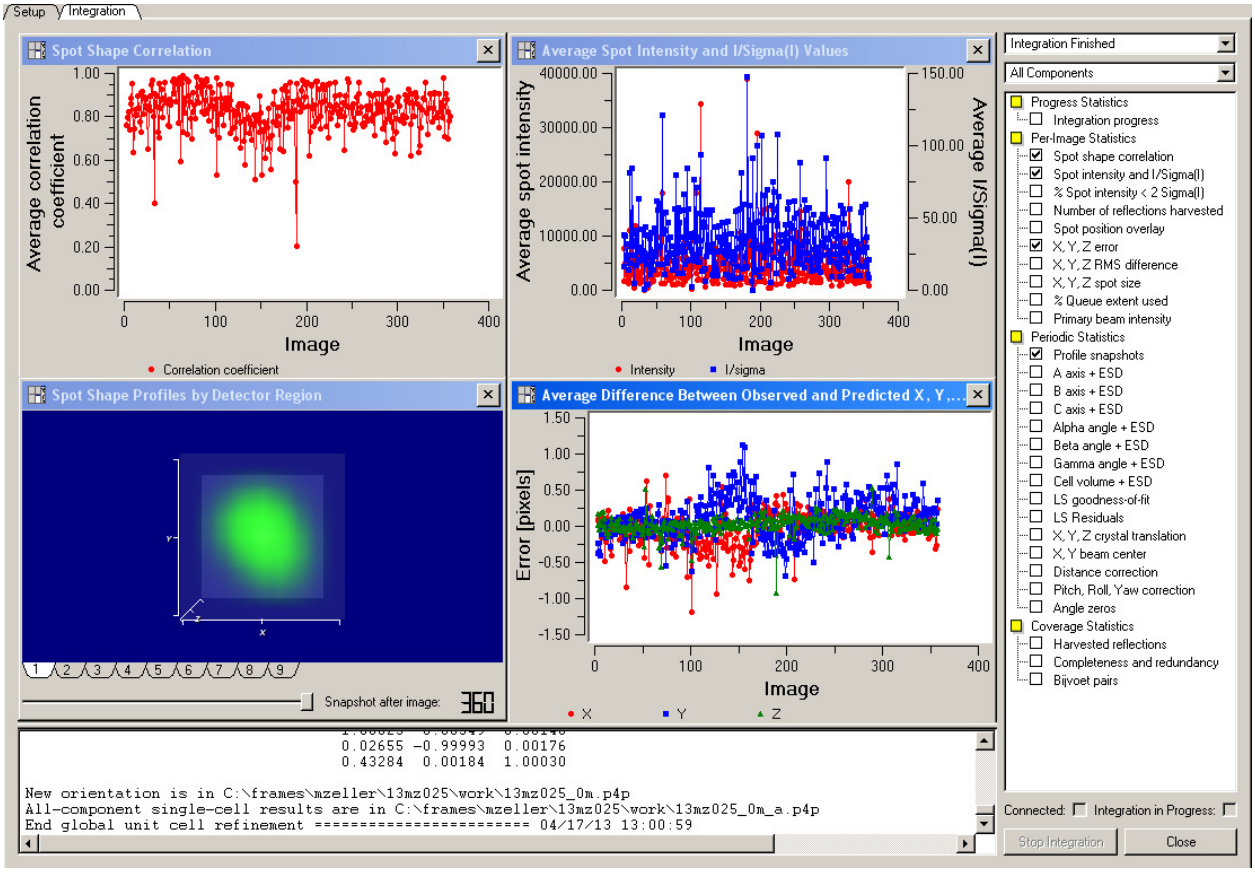


Figure. 33. Integration Option window with active Integration running.

- 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.

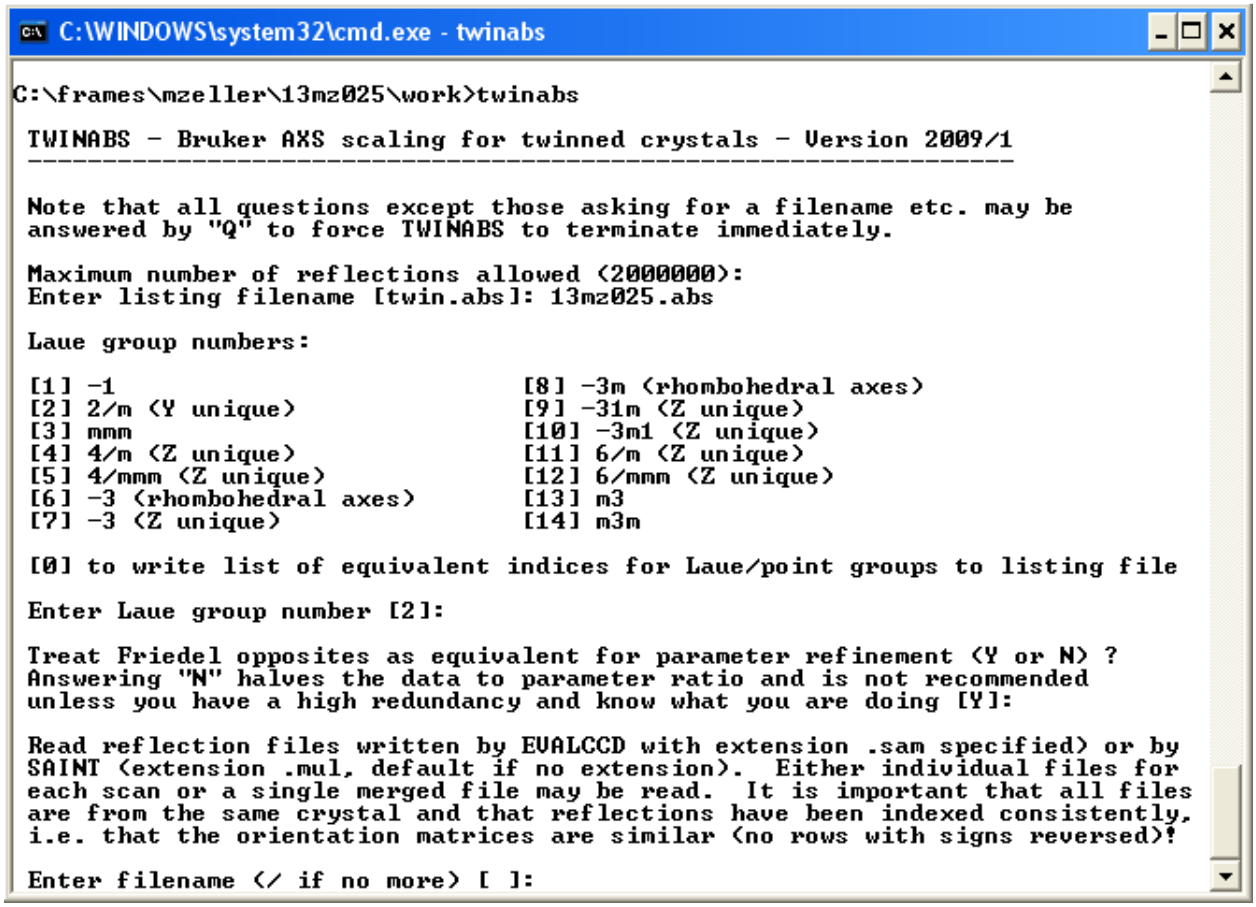


Figure. 34. The Run command window.

- 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.

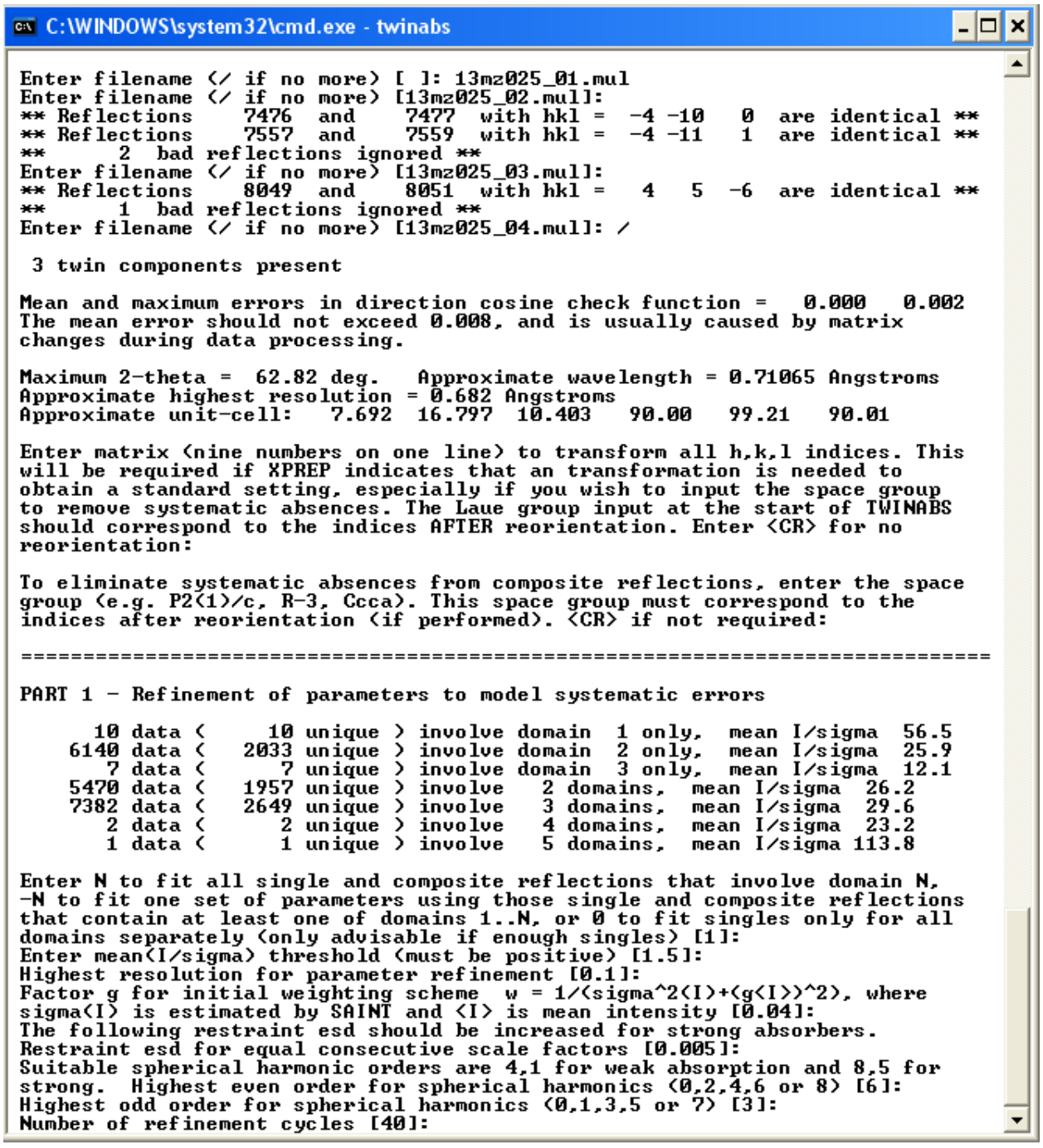


Figure. 35. The Run command window.

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

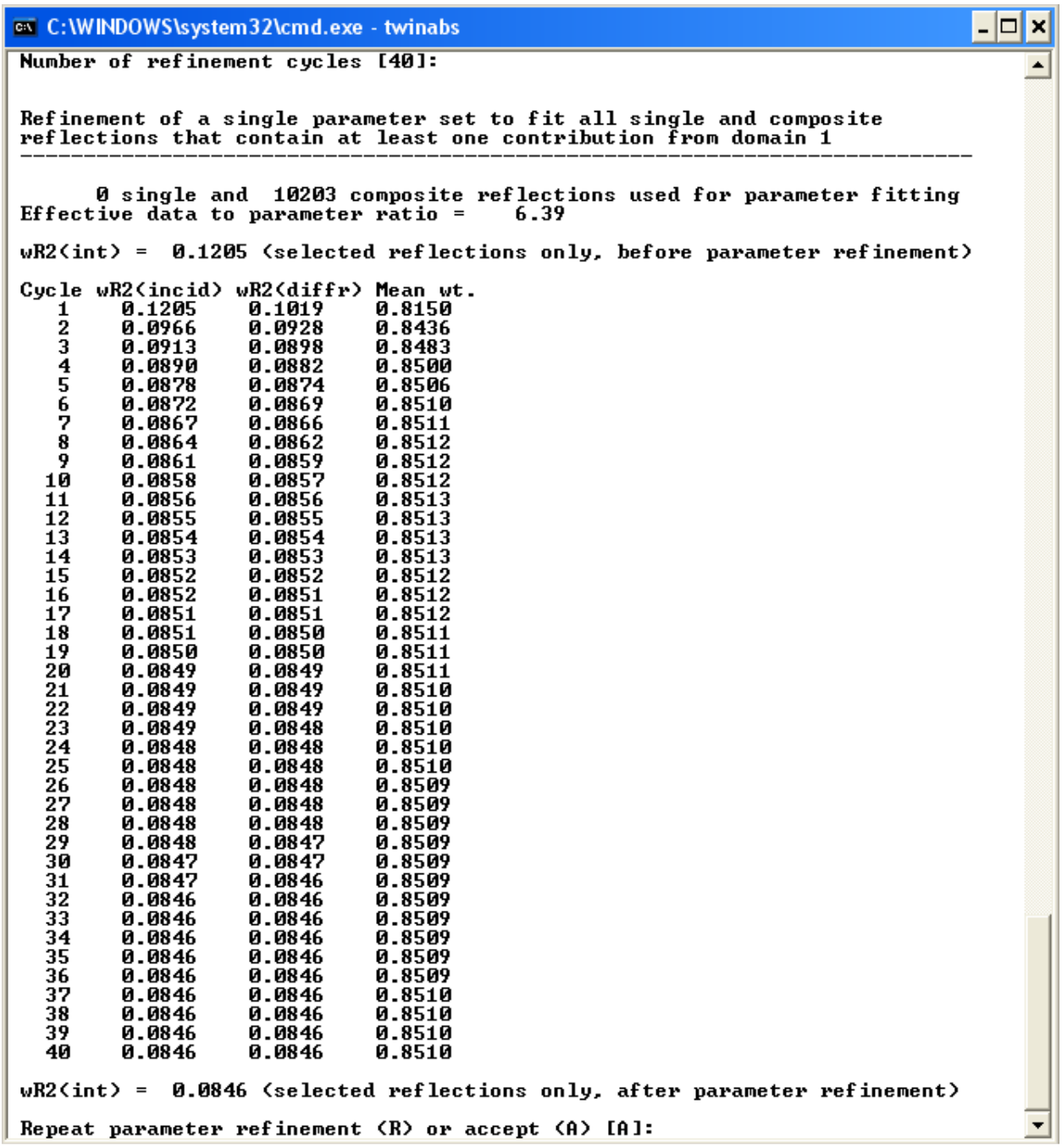


Figure. 36. The Run command window.

Go to the outlier rejection step.

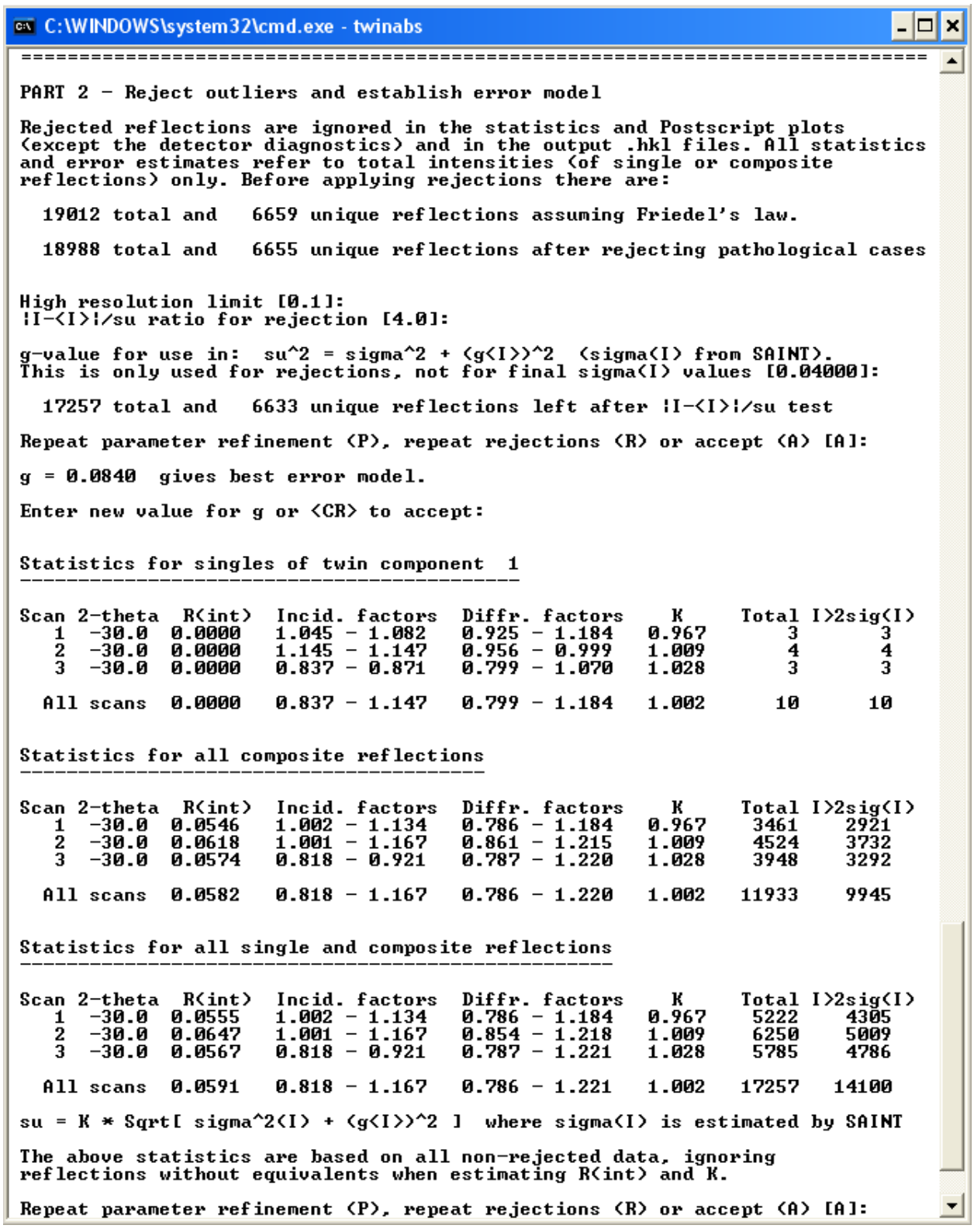


Figure. 37. The Run command window.

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.

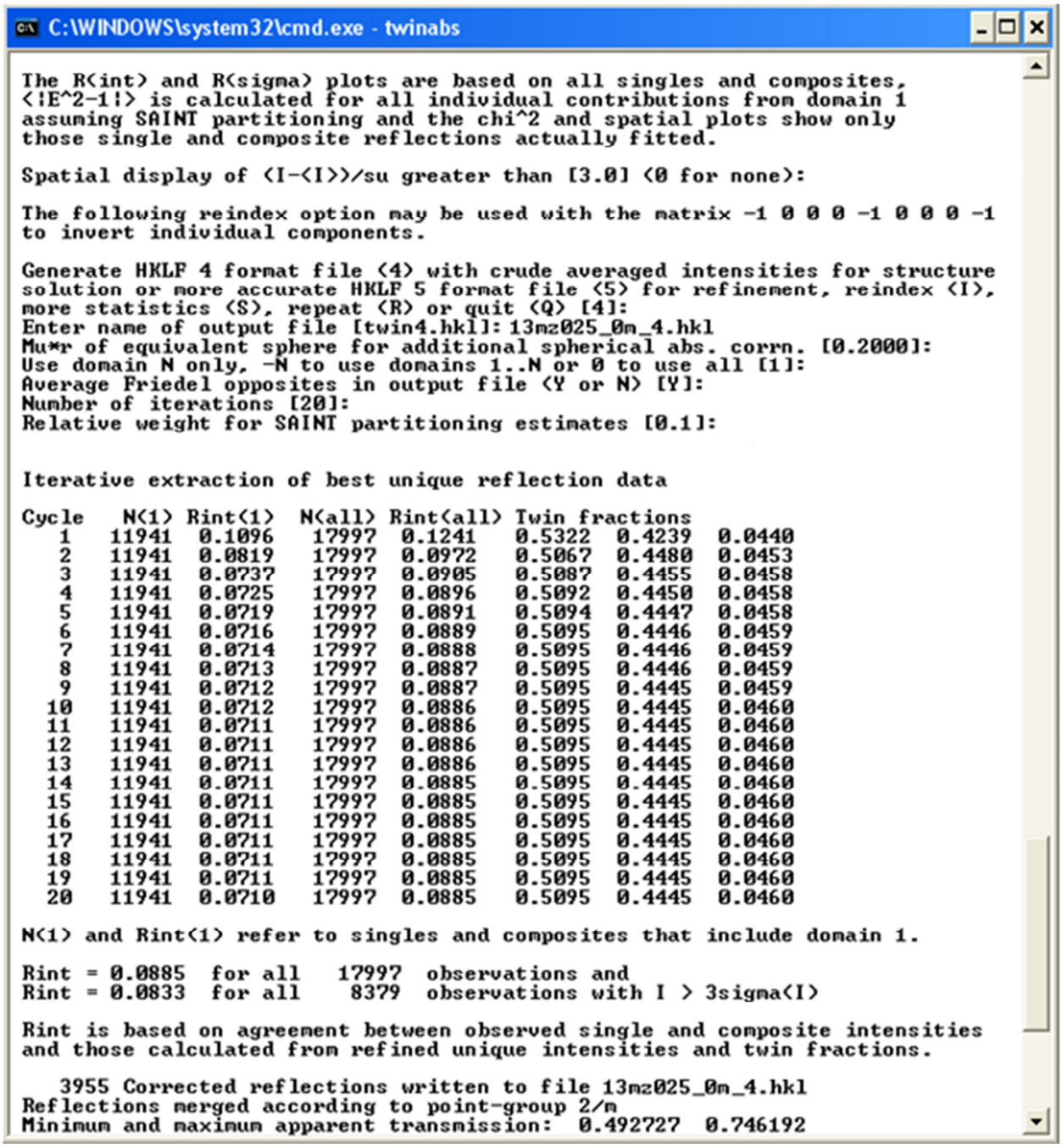


Figure. 38. The Run command window.

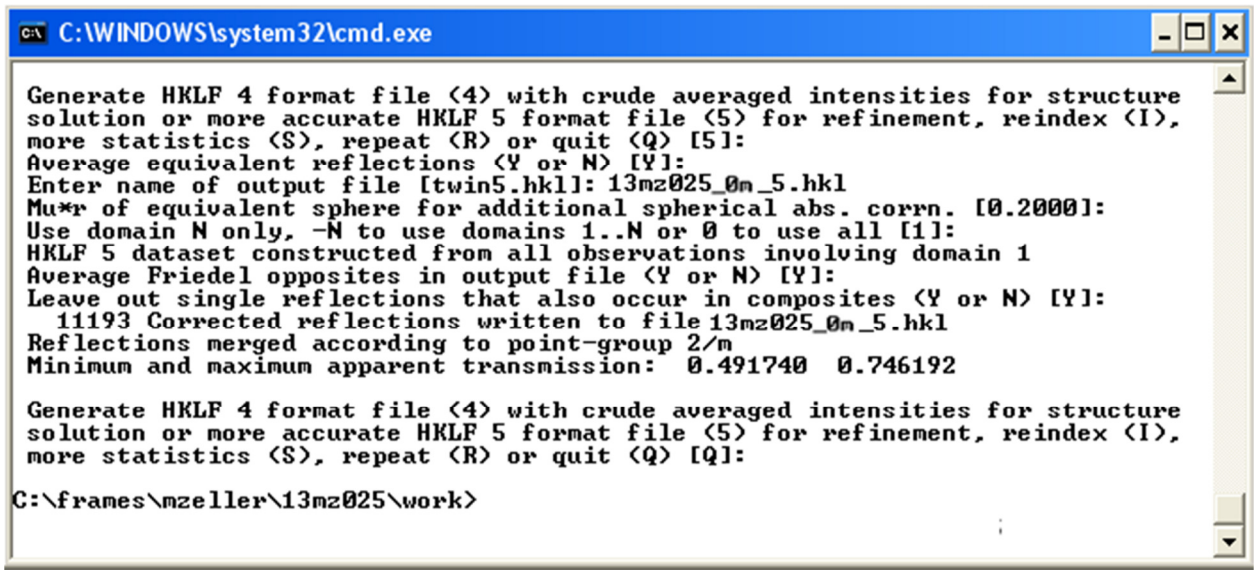


Figure. 39. The Run command window.

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.

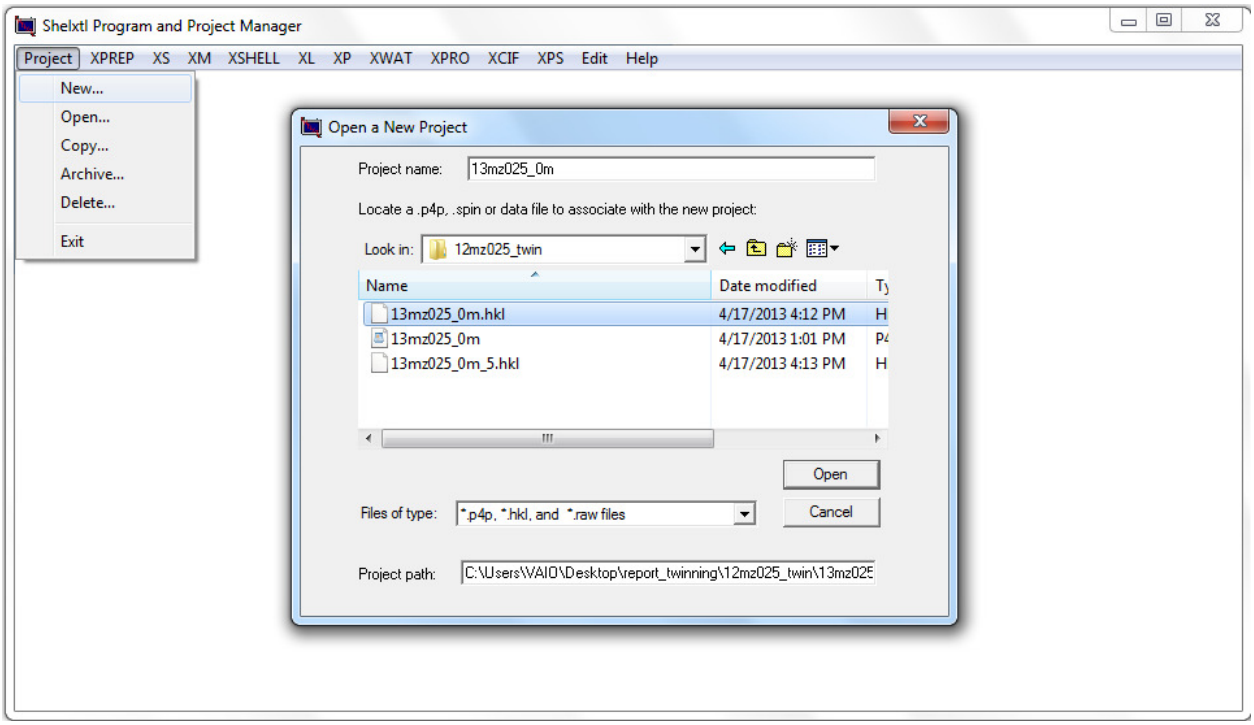


Figure. 40. Shelxtl Program and project manager window.

XPREP

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

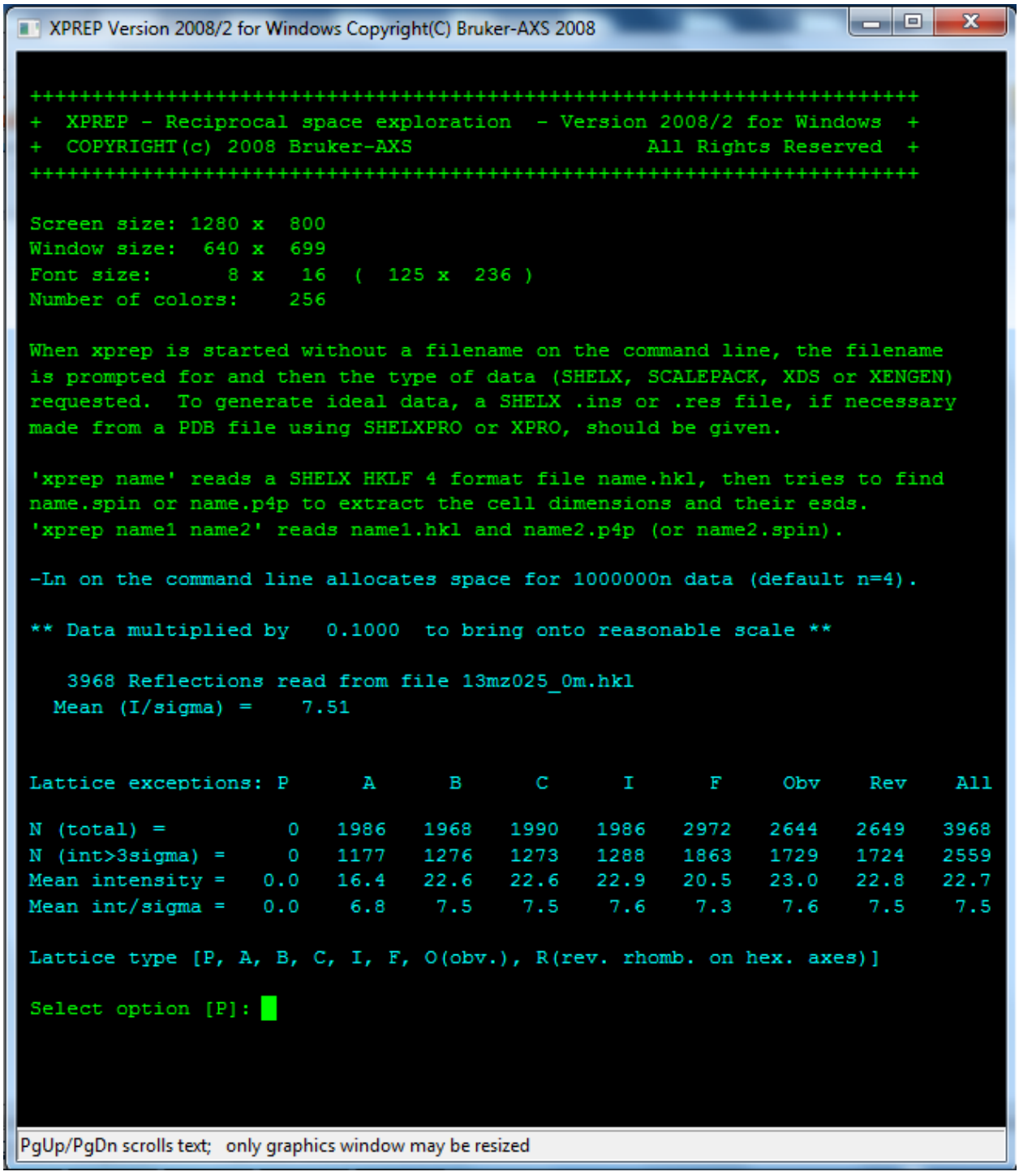


Figure. 41. XPREP window.

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

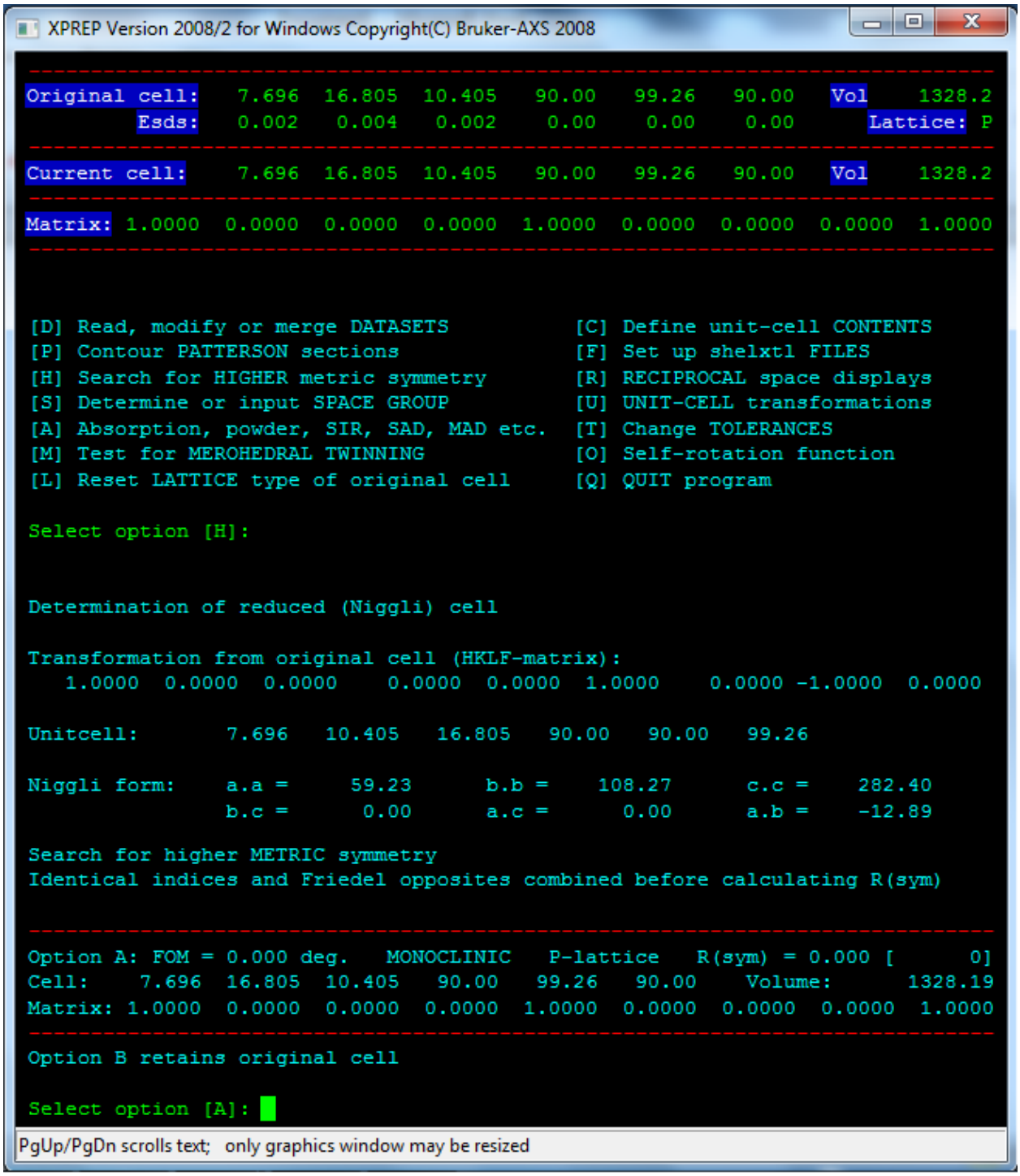


Figure. 42. XPREP window.

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

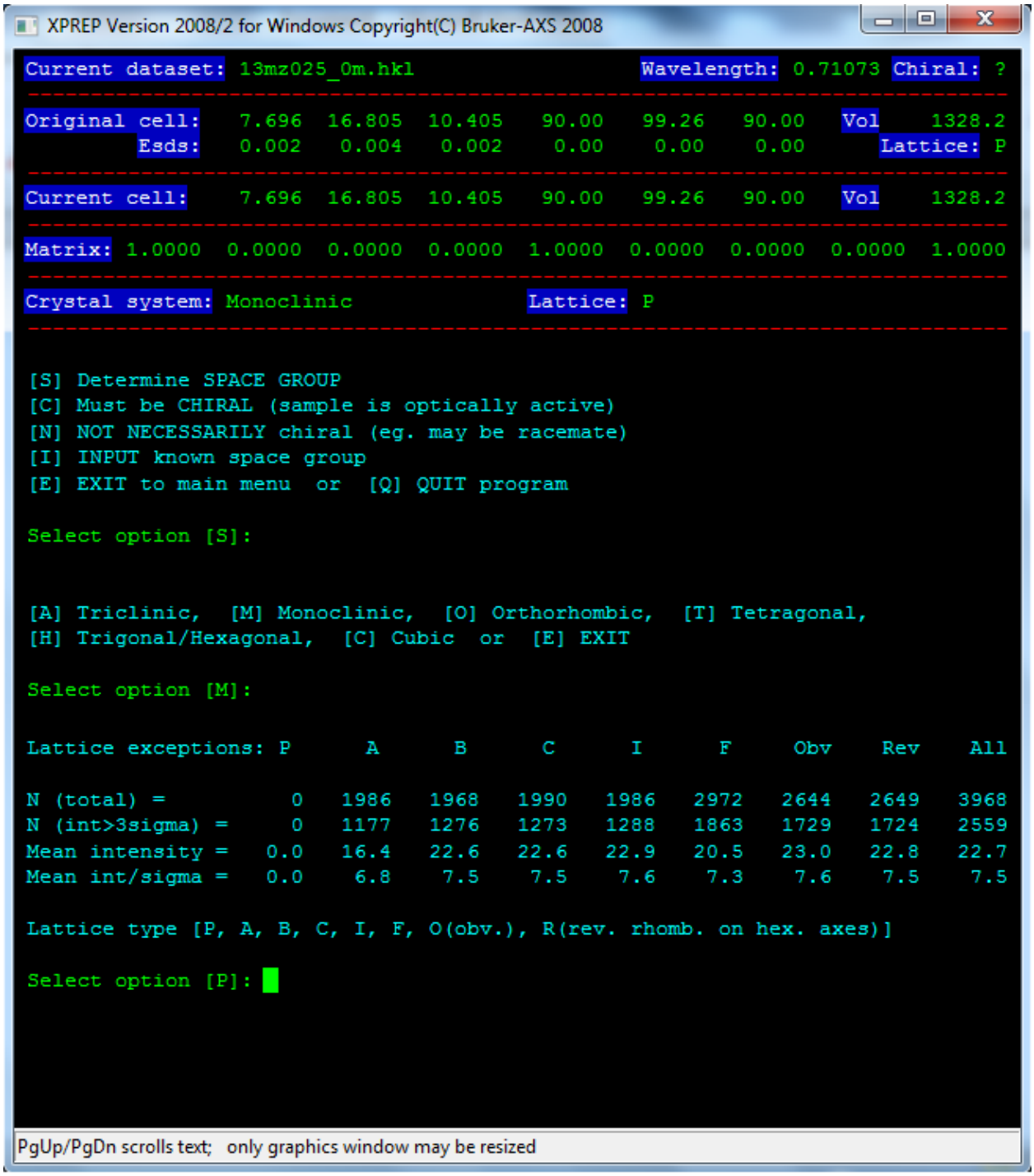


Figure. 43. XPREP window.

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

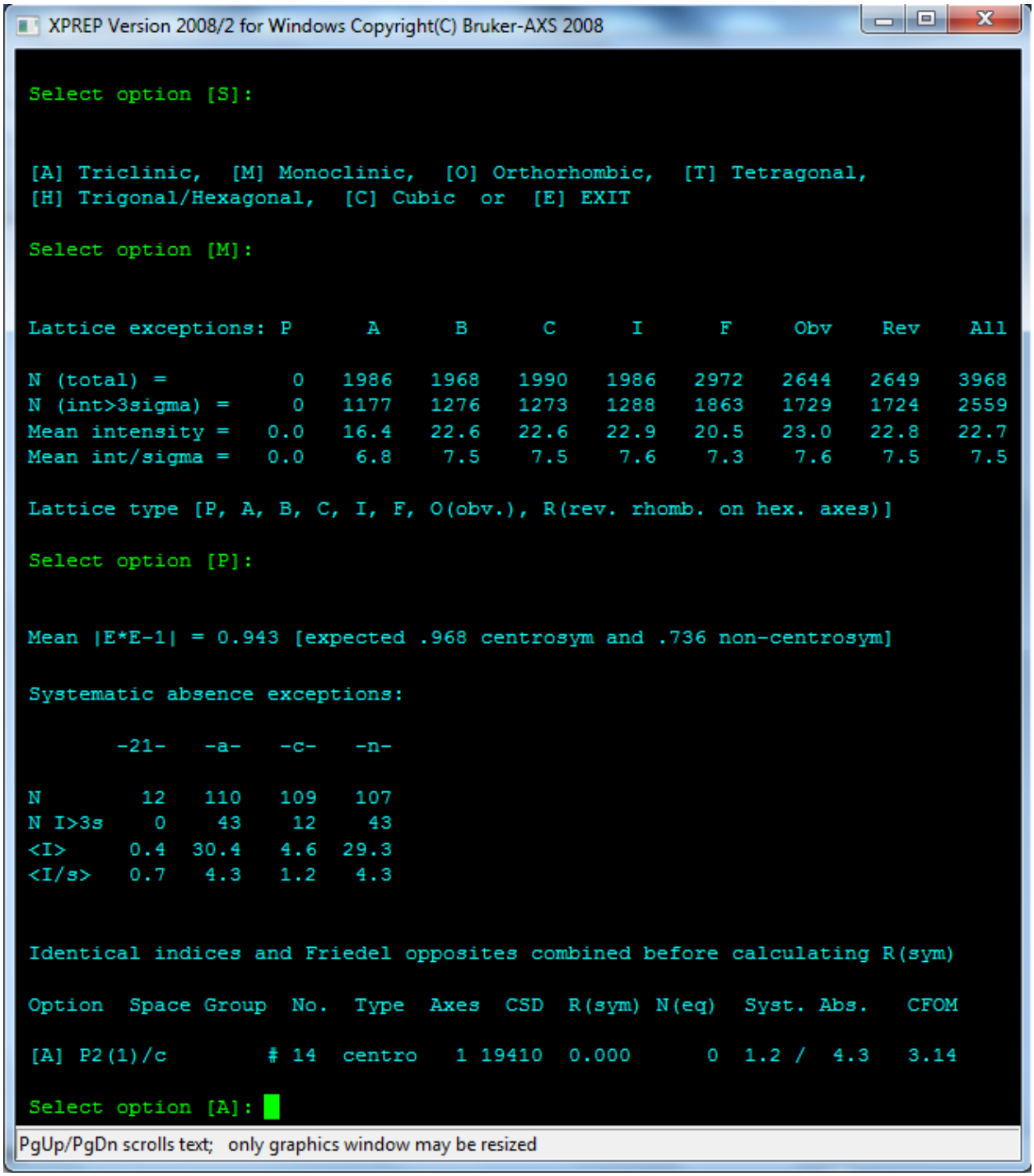


Figure. 44. XPREP window.

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

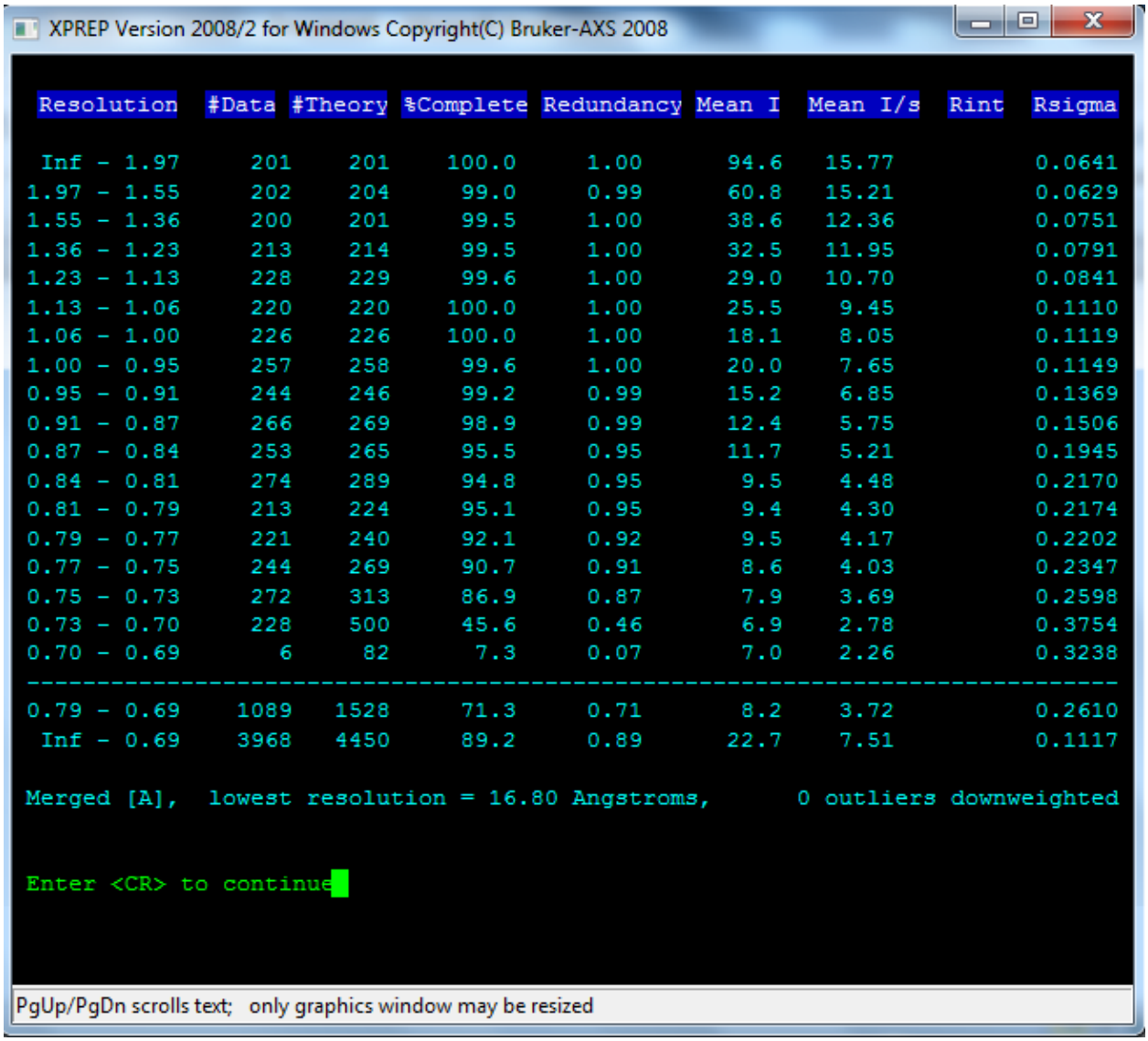


Figure. 45. XPREP window.

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

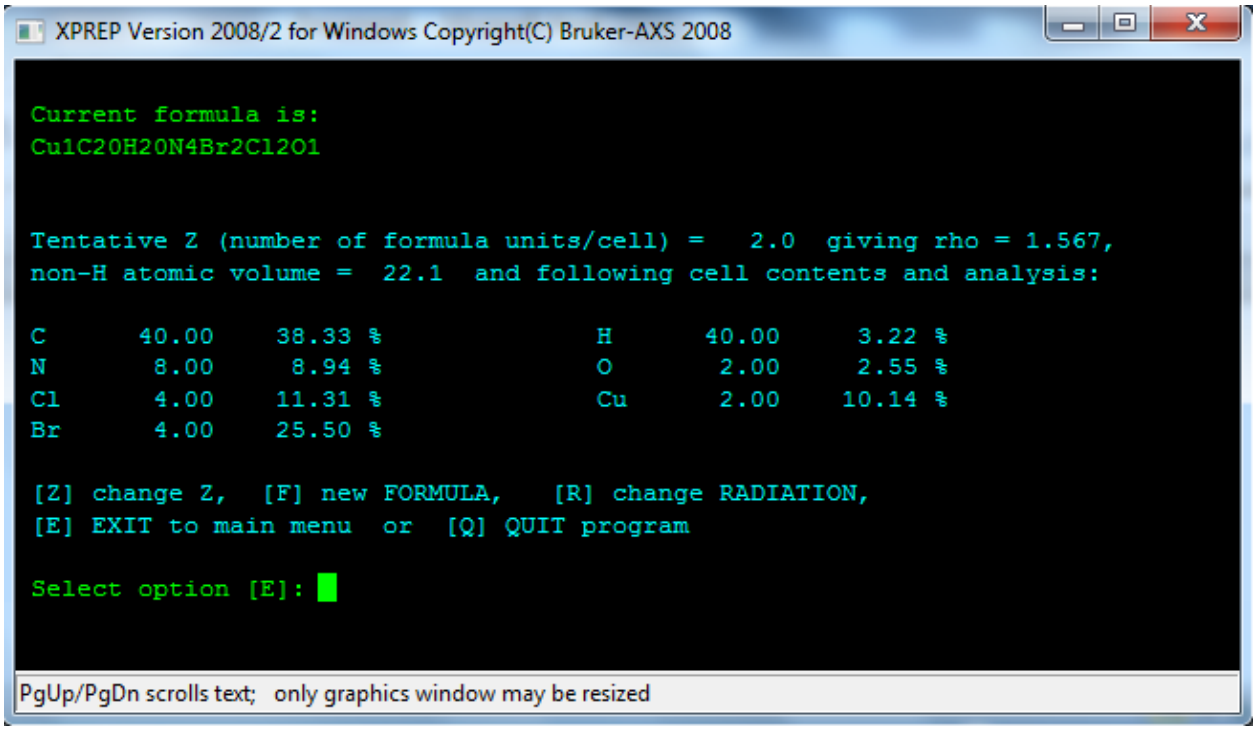


Figure. 46. XPREP window.

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

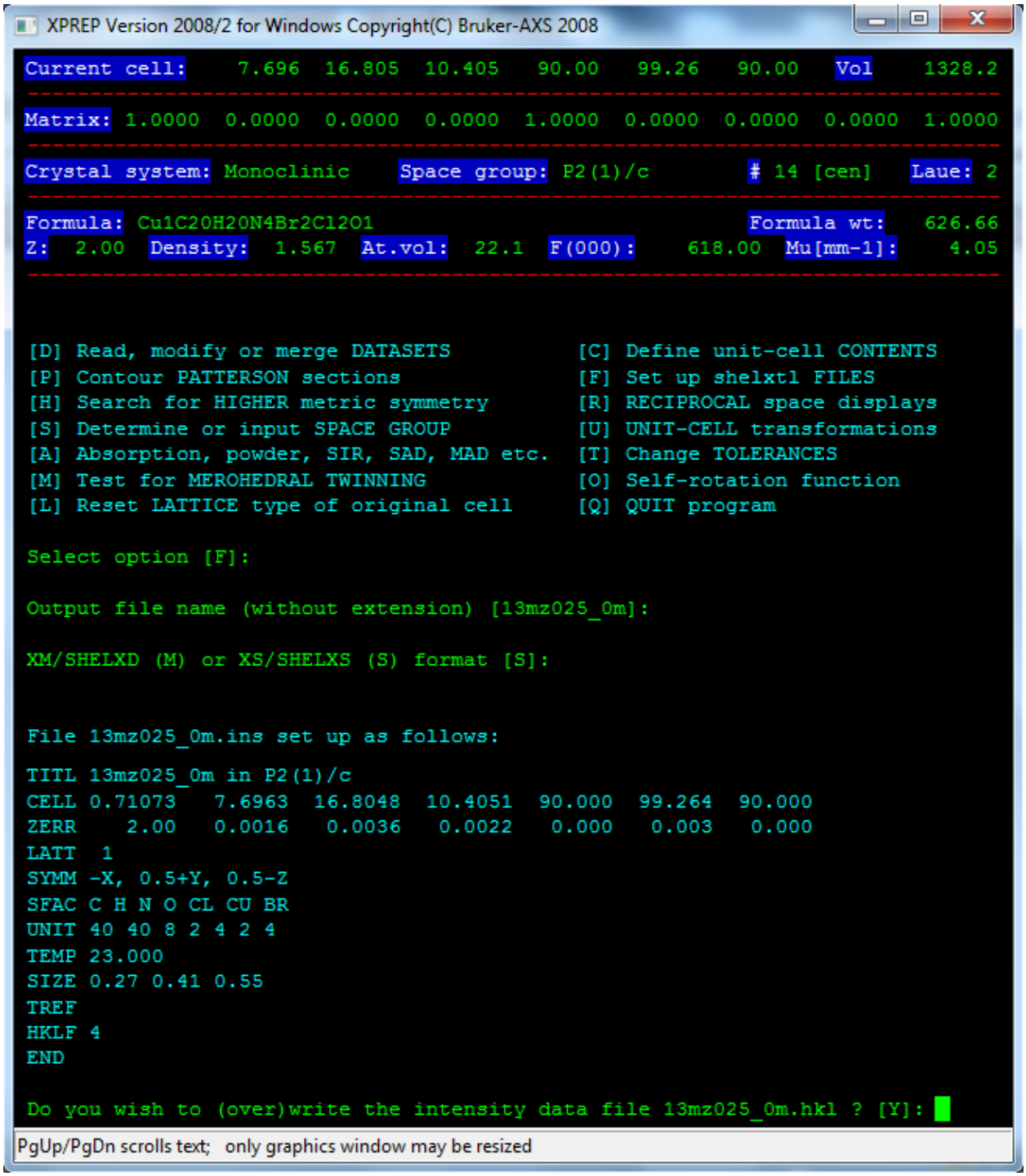


Figure. 47. XPREP window.

- Select XS on the toolbar.

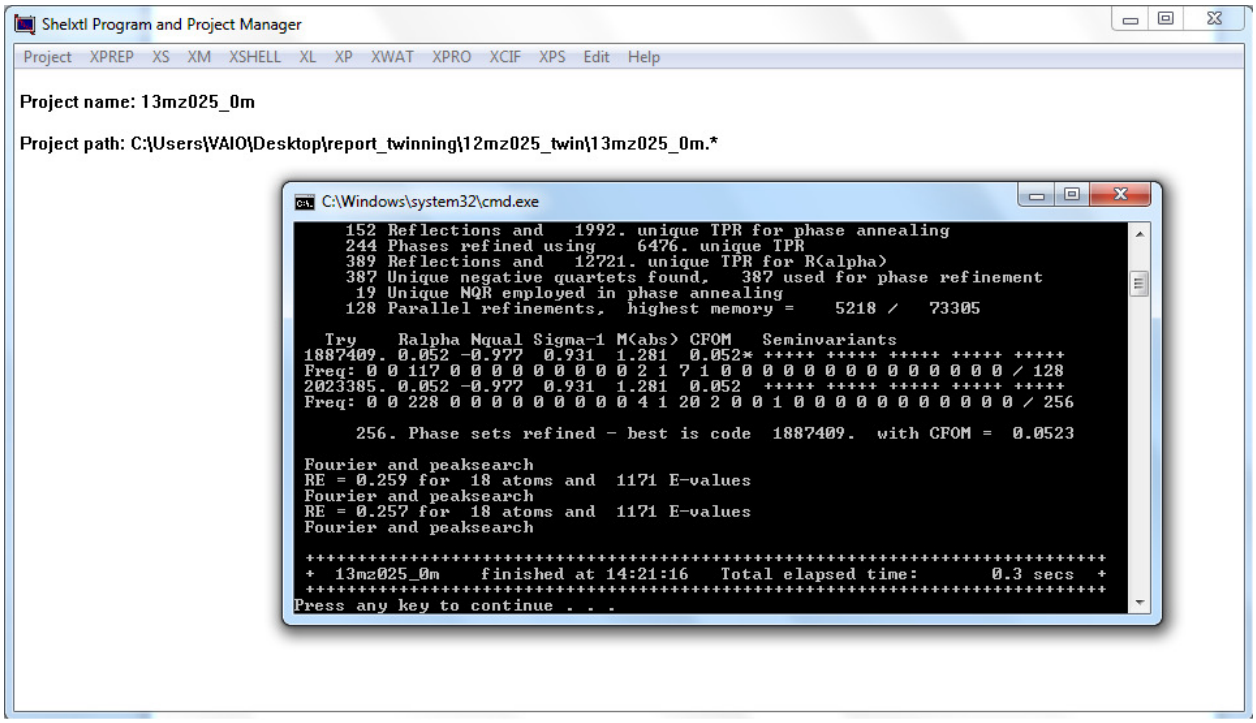


Figure. 48. Shelxtl Program with process of structure solution by classical direct methods.

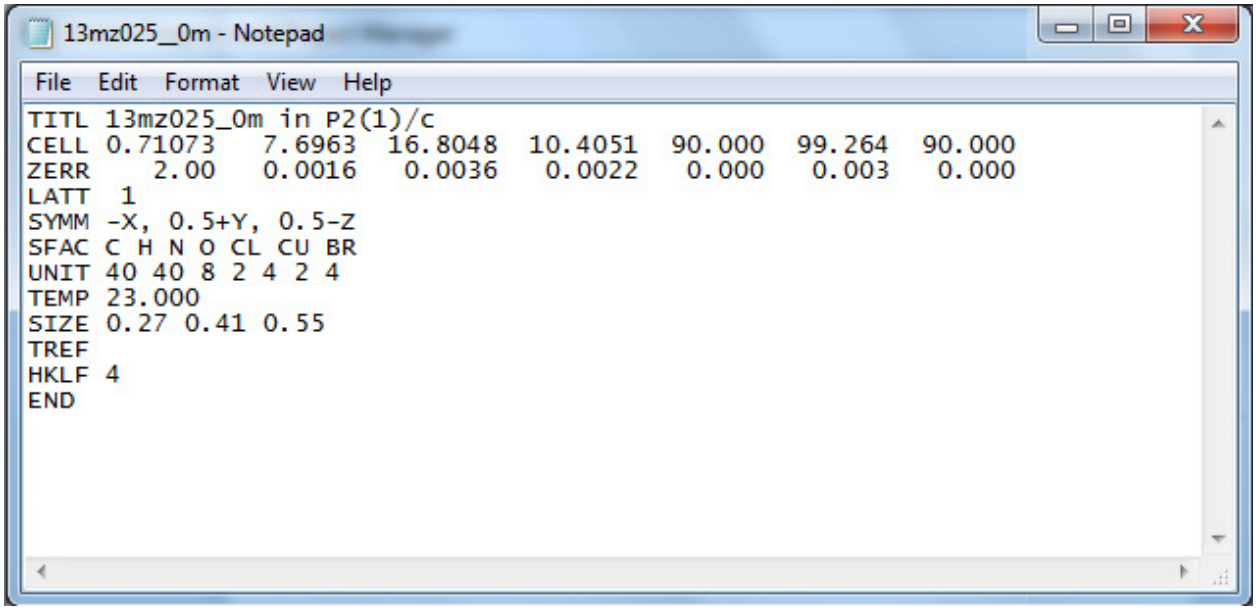


Figure. 49. Typical initial INS file.

Structure refinement

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

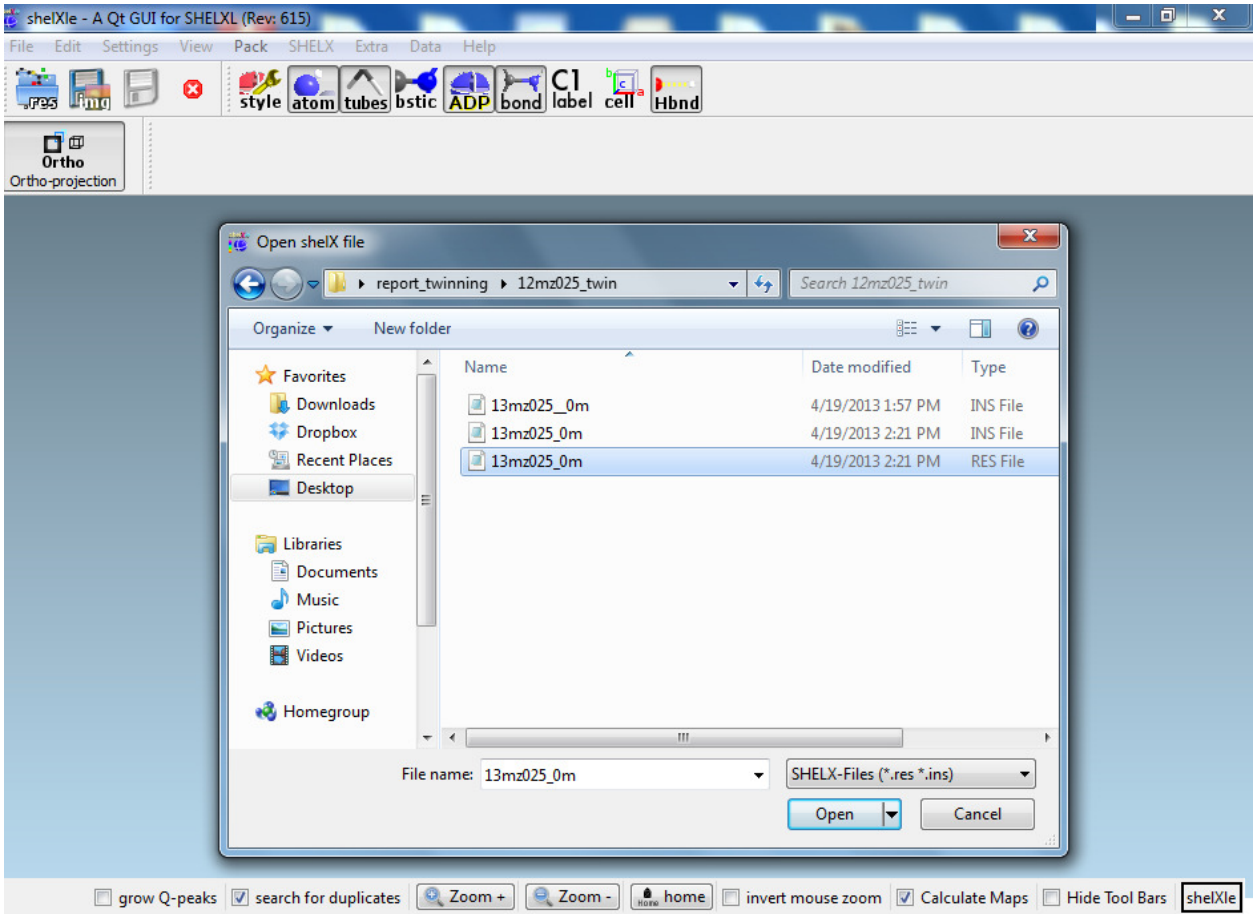


Figure. 50. shelXle window.

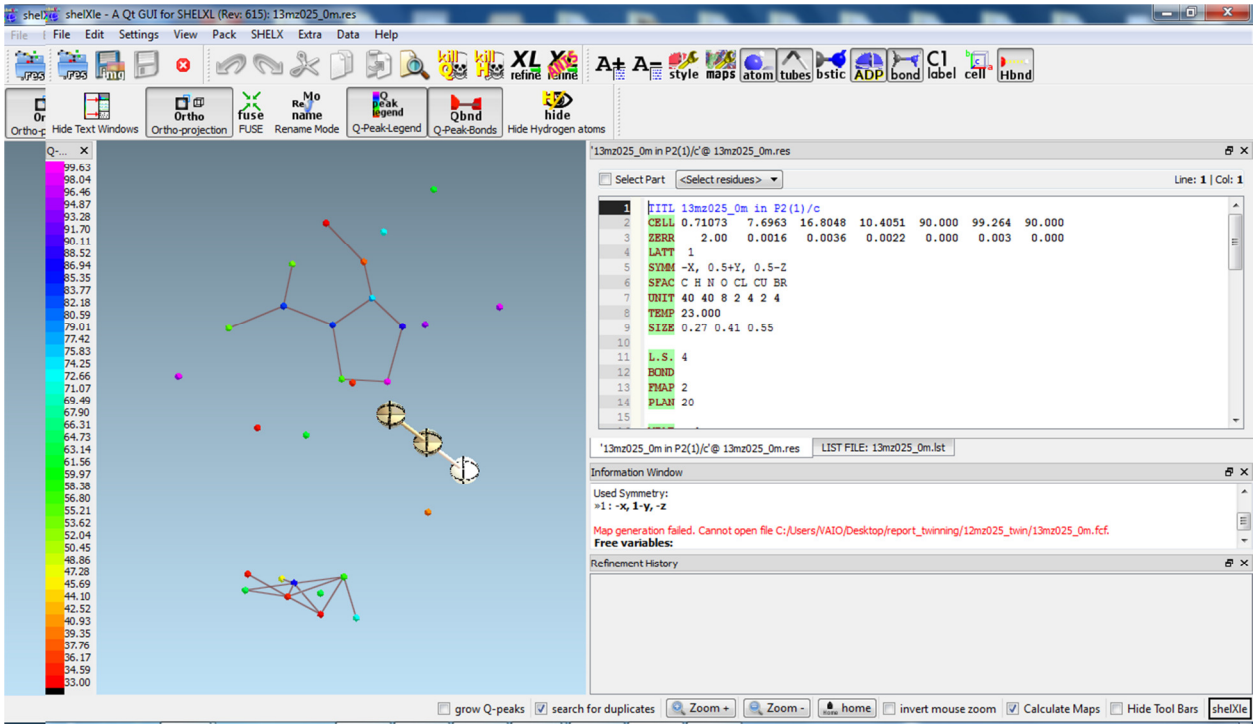


Figure. 51. shelXle window with initial result from XS.

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

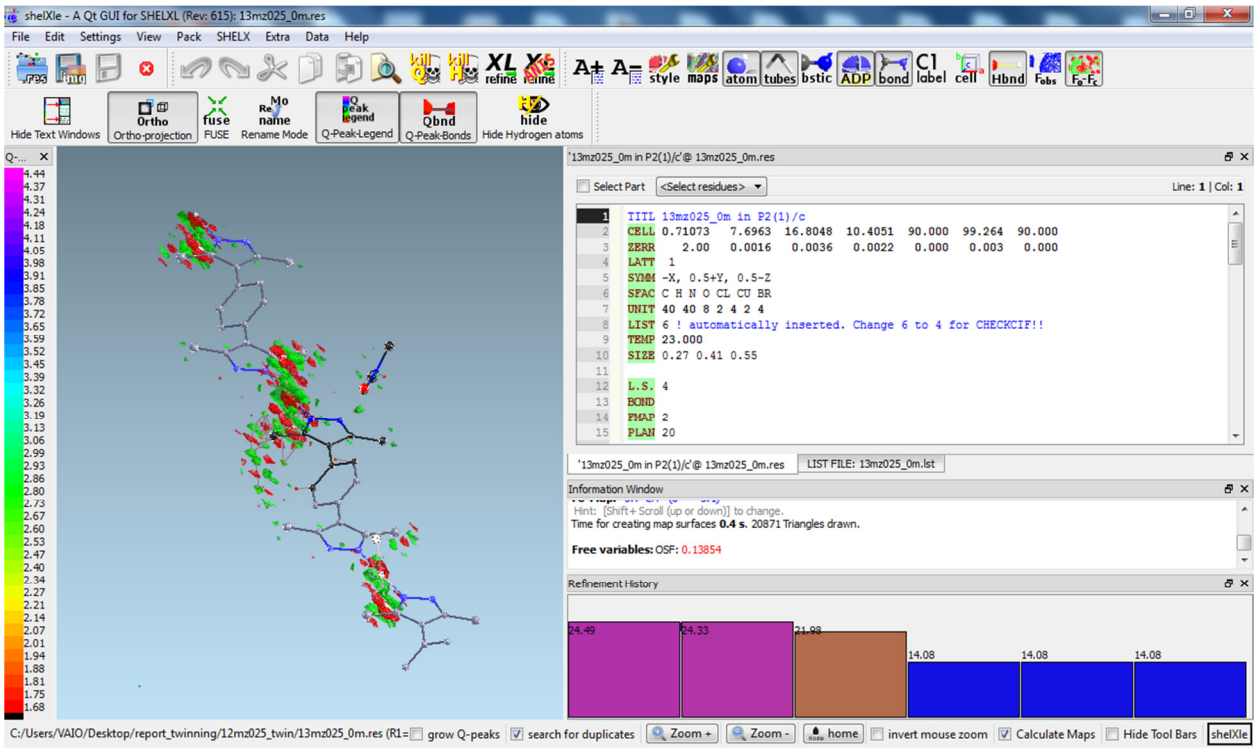


Figure. 50. shelXle window.

*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.*

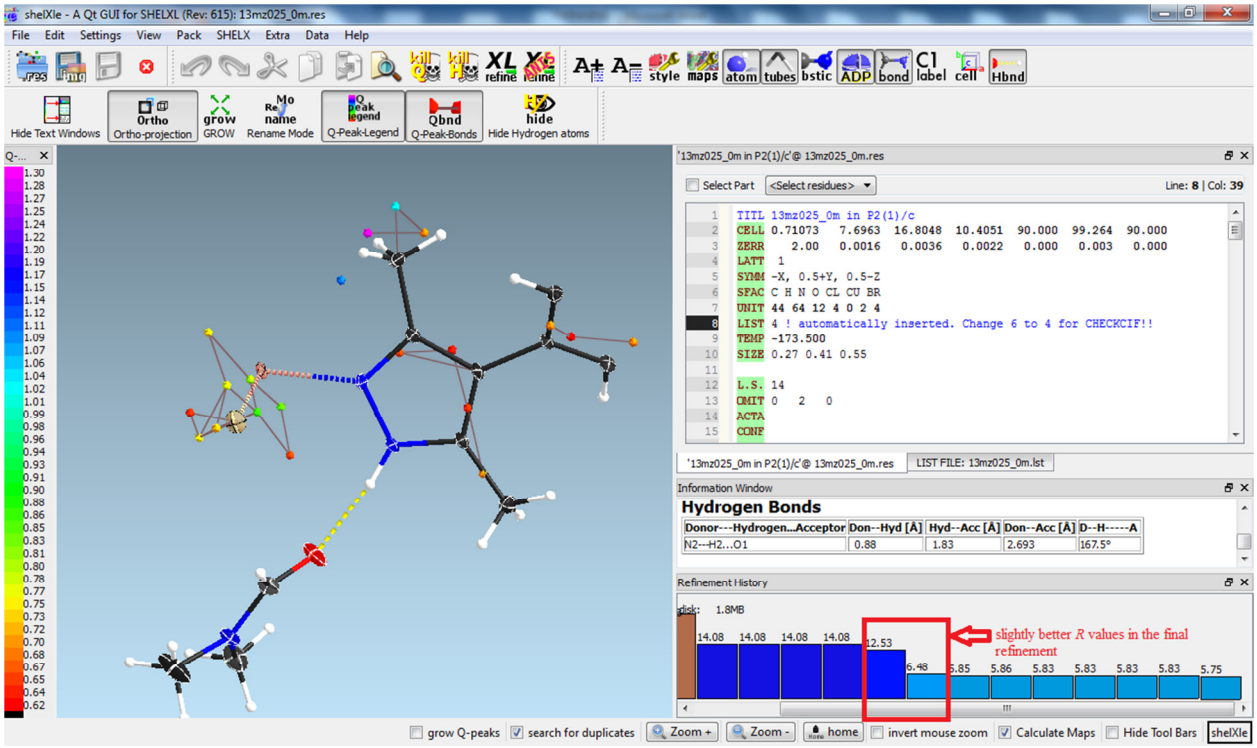


Figure. 51. shelXle window.

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*----------