

Structure Determination Using Jana2006

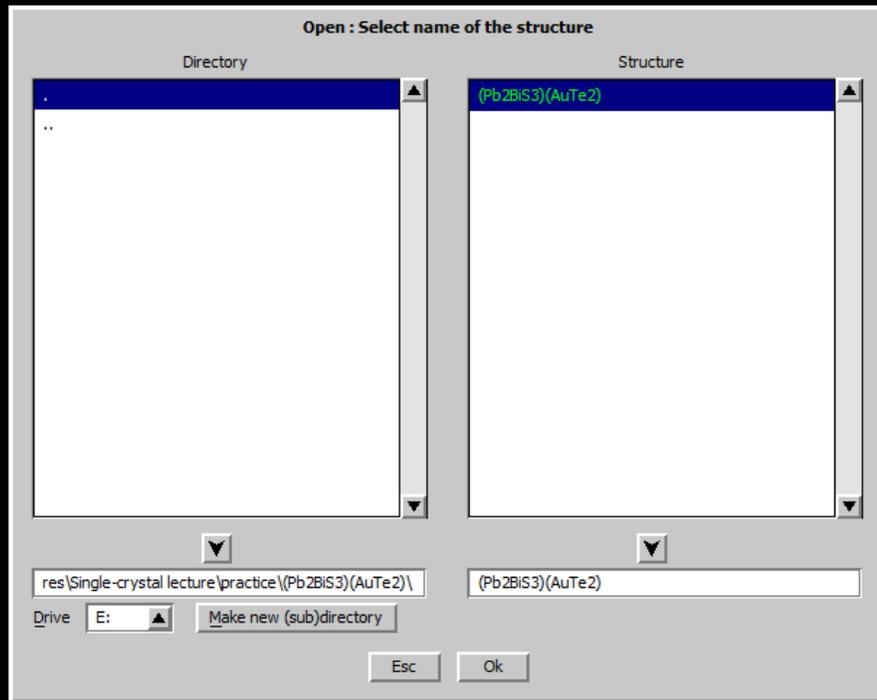
Structural Analysis from Single-Crystal
and Powder X-ray data

Import your data

Find your data from data reduction and place them in a separate folder

Start a new structure from the File -> Structure menu

Jana2006



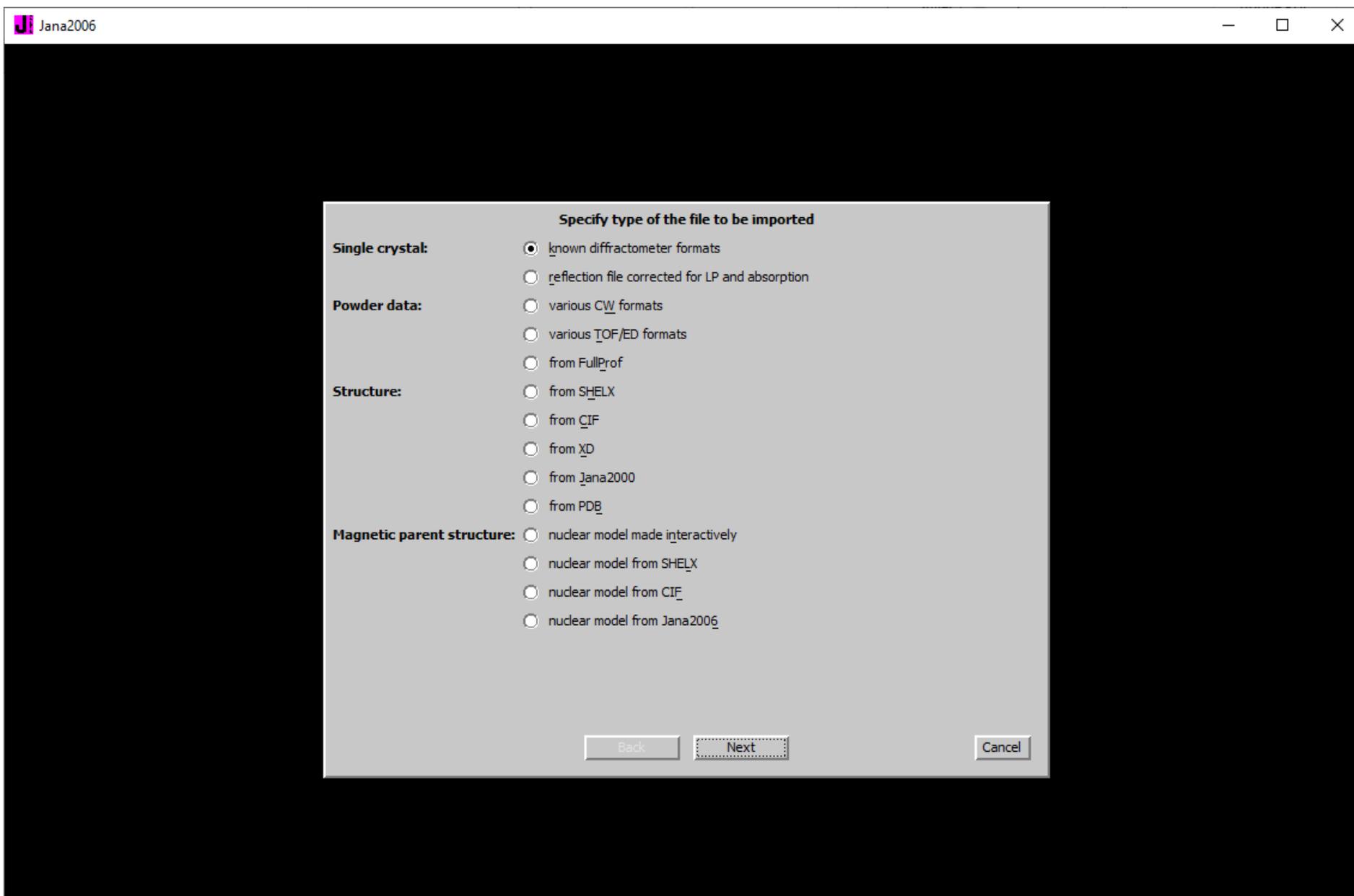
File formats:

- 1) hkl + sum files (STOE)
- 2) hkl + p4p files (Bruker)
- 3) hkl + cif_od files (Rigaku)

4) hkl from powder data...

... you should already be there
from pattern matching
(LeBail fitting)

Select single crystal with known diffractometer formats



Select the appropriate instrument/format

Jana2006

Data reduction file from:

File name: (Pb2BIS3)(AuTe2).hkl Browse

CAD4

Nonius-CCD

Siemens P4

Bruker-CCD

Bruker-CCD (raw)

Oxford Diffraction-CCD

Oxford Diffraction-PD

Rigaku-CCD

IPDS Stoe

D ϕ -ILL, D23 or Trics-Zebra

ILL-Vivaldi

ISIS SXD

TOPAZ

Koala at ANSTO

SCD-LANL

Hasylab E1

Hasylab HUBER

Hasylab XDS

6T2 LBB

Pets electron diffractometer

SENJU TOF

Polarized neutrons

SHELX on I - abs.correction needed

Back Next Cancel

Structure: E:\Crete\Teaching\UoC\ETY-453_Crystal Chemistry\2020 lectures\Single-crystal lecture\practice\((Pb2BIS3)(AuTe2))\((Pb2BIS3)(AuTe2)

Check that your data are properly imported

Unit cell, wavelength and temperature should be as in the experiment

Jana2006

Complete/correct experimental parameters

Cell parameters: 4.0997 9.3435 12.3722 90 90 90

Number of input indices: 3 Info about metrics parameters

1st modulation vector:

2nd modulation vector:

3rd modulation vector:

X-ray tube

Wave length: 0.71073

Temperature: 293

Polarization correction:

Circular polarization

Perpendicular setting Info

Parallel setting Info

Guinier camera

Linearly polarized beam

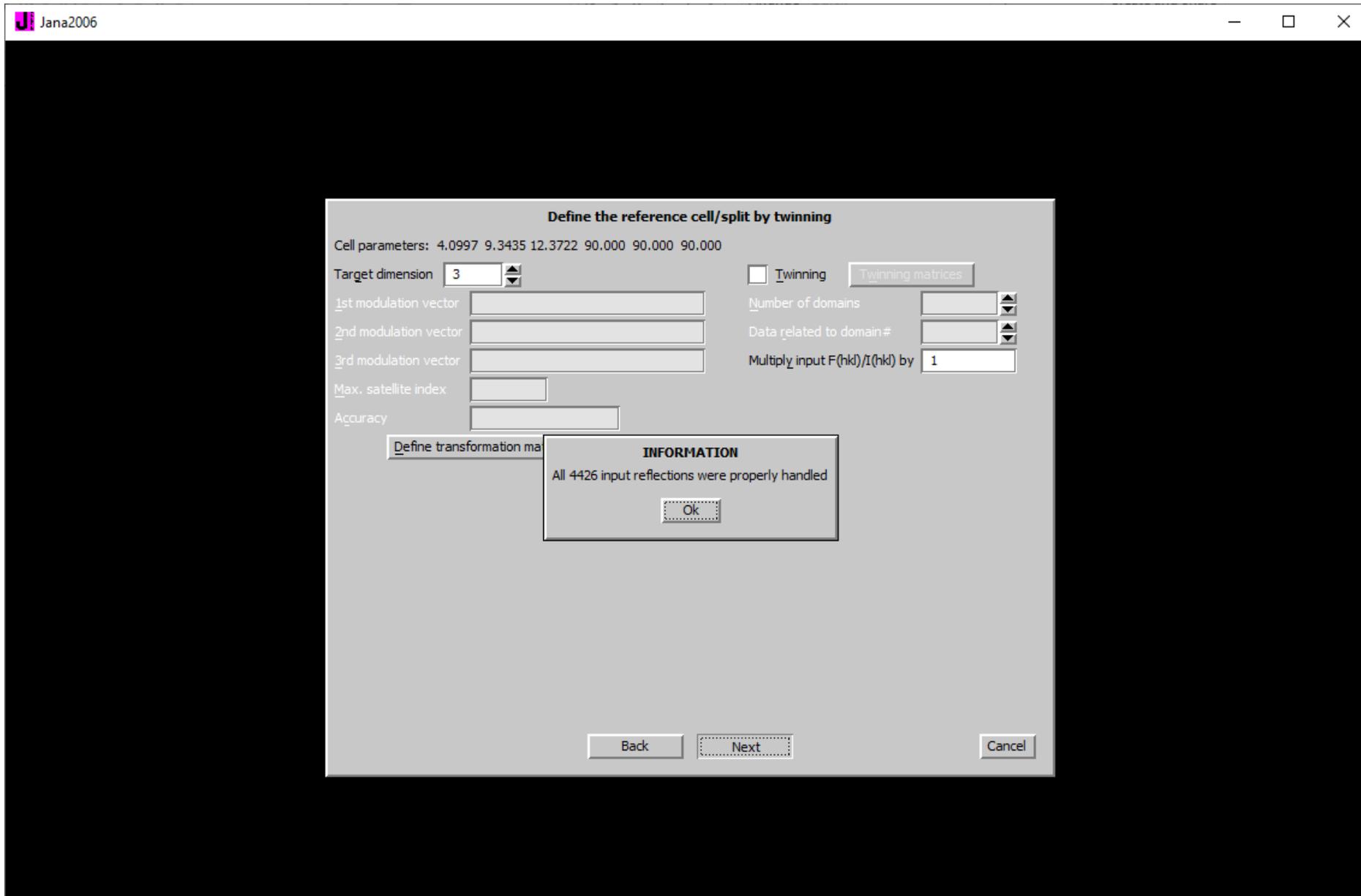
Monochromator parameters:

Perfectness: 0.5

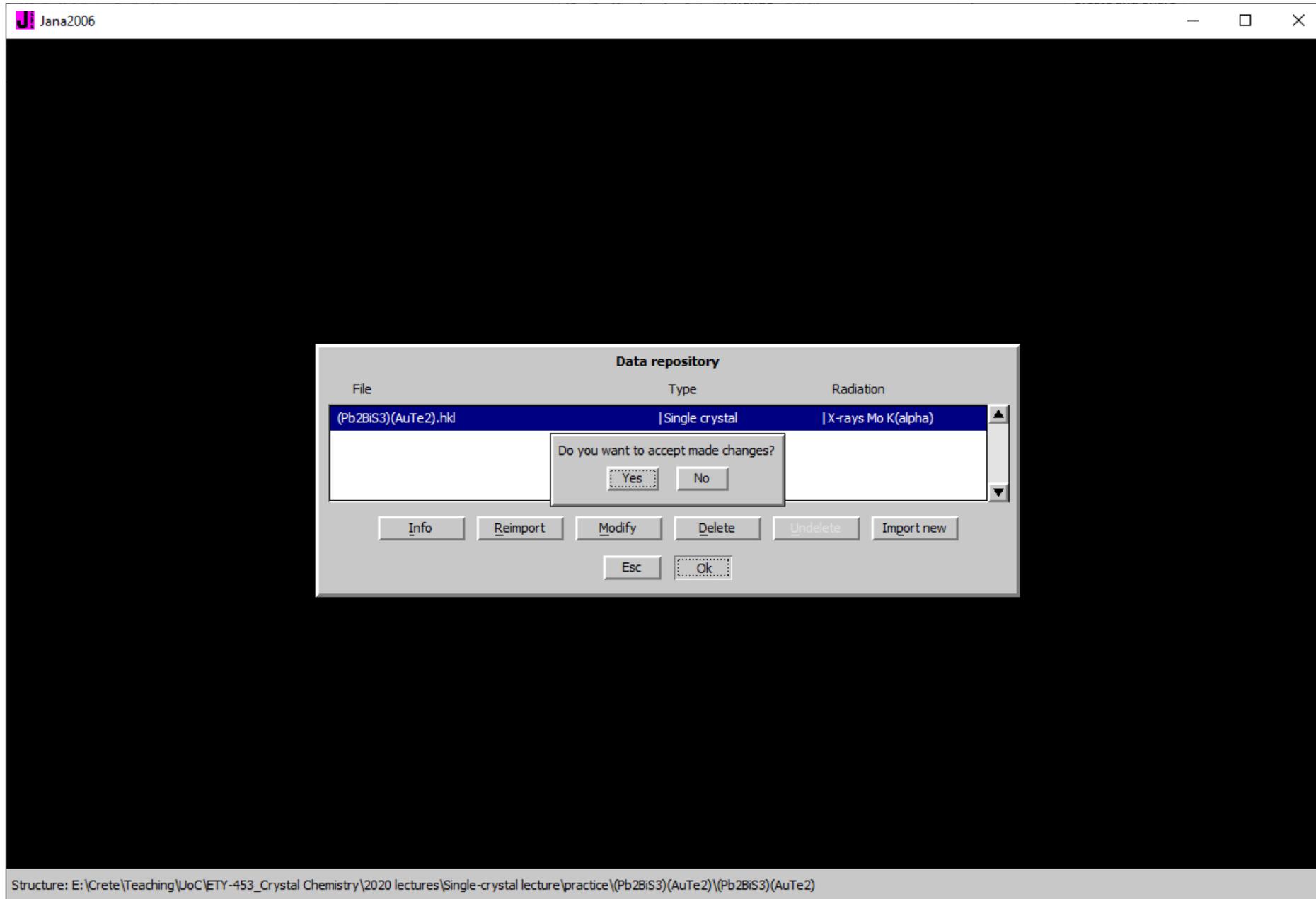
Glancing angle: 6.106711 Set glancing angle

Back Next Cancel

Import your reflection file (hkl) by clicking through to the menu below



Click through and accept your hkl file, if correct



You are now in the process of finding your space group.
Keep default commands

Jana2006

Tolerances for crystal system recognition:
Original cell parameters: 4.100 9.344 12.372 90.00 90.00 90.00
Maximal deviation for cell lengths in [Å]
Maximal deviation for cell angles in deg

Tolerances for space group recognition:
Maximal ave(I/sig(I)) for centering
Maximal ave(I/sig(I)) for extinctions

Search for higher symmetrical supercell (recommended)
 Introduce twin laws in case of subgroups
 Use old twin matrices in testing

Structure: E:\Crete\Teaching\UoC\ETY-453_Crystal Chemistry\2020 lectures\Single-crystal lecture\practice\(\Pb2BiS3)(AuTe2)\(\Pb2BiS3)(AuTe2)

Select the highest possible crystal system and point group, provided the Rint(obs/all) values are comparable. Rint will always be highest for the highest symmetry, so this requires some experience. You can return here at any time and select a different one.

The screenshot shows the 'Select Laue symmetry' dialog box in the Jana2006 software. The dialog contains a table with the following data:

Crystal system	Point group	Rint(obs/all)	#averaged	Redundancy
Tridinic	-1	8.12/10.86	866/1980	1.967
Monodinic-setting "a"	2/m	10.84/13.78	560/1123	3.467
Monodinic-setting "b"	2/m	10.37/13.26	550/1099	3.543
Monodinic-setting "c"	2/m	10.89/13.85	540/1104	3.527
Orthorhombic	mmm	12.01/14.74	367/646	6.028

Below the table, there are two radio buttons: 'ordered by Laue symmetry' (selected) and 'ordered by Rint'. A 'Details' button is located to the right of the radio buttons. At the bottom, there are 'Back', 'Next', and 'Cancel' buttons. The text 'Averages made from 1490/3894 reflections' is displayed above the 'Back' and 'Next' buttons.

Structure: E:\Crete\Teaching\JoC\ETY-453_Crystal Chemistry\2020 lectures\Single-crystal lecture\practice\(\Pb2BiS3)(AuTe2)\(\Pb2BiS3)(AuTe2)

Find your unit cell centering. Look for the zeroes (no systematic absence violations) with the highest symmetry

Select cell centering

Centering	obs/all	ave(I/sig(I))	
<input checked="" type="radio"/> P	0/0	0.000/0.000	Details
<input type="radio"/> A	756/1968	23.322/9.612	Details
<input type="radio"/> B	739/1949	23.604/9.590	Details
<input type="radio"/> C	565/1877	17.301/5.912	Details
<input type="radio"/> I	744/1878	23.473/9.918	Details
n.a. R-obverse	994/2603	25.279/10.292	Details
n.a. R-reverse	995/2604	25.537/10.396	Details
<input type="radio"/> F	1030/2897	21.772/8.406	Details

Warning: The cell centering need not be one you expect from collection as the program first transform the cell to the reduced form.
Moreover after your selection the program makes another transformation whenever the centring is not the standard one.

Back Next Cancel

Structure: E:\Crete\Teaching\UoC\ETY-453_Crystal Chemistry\2020 lectures\Single-crystal lecture\practice\(\Pb2BiS3)(AuTe2)\(\Pb2BiS3)(AuTe2)

Select the space group with the highest possible symmetry

Jana2006

Select space group

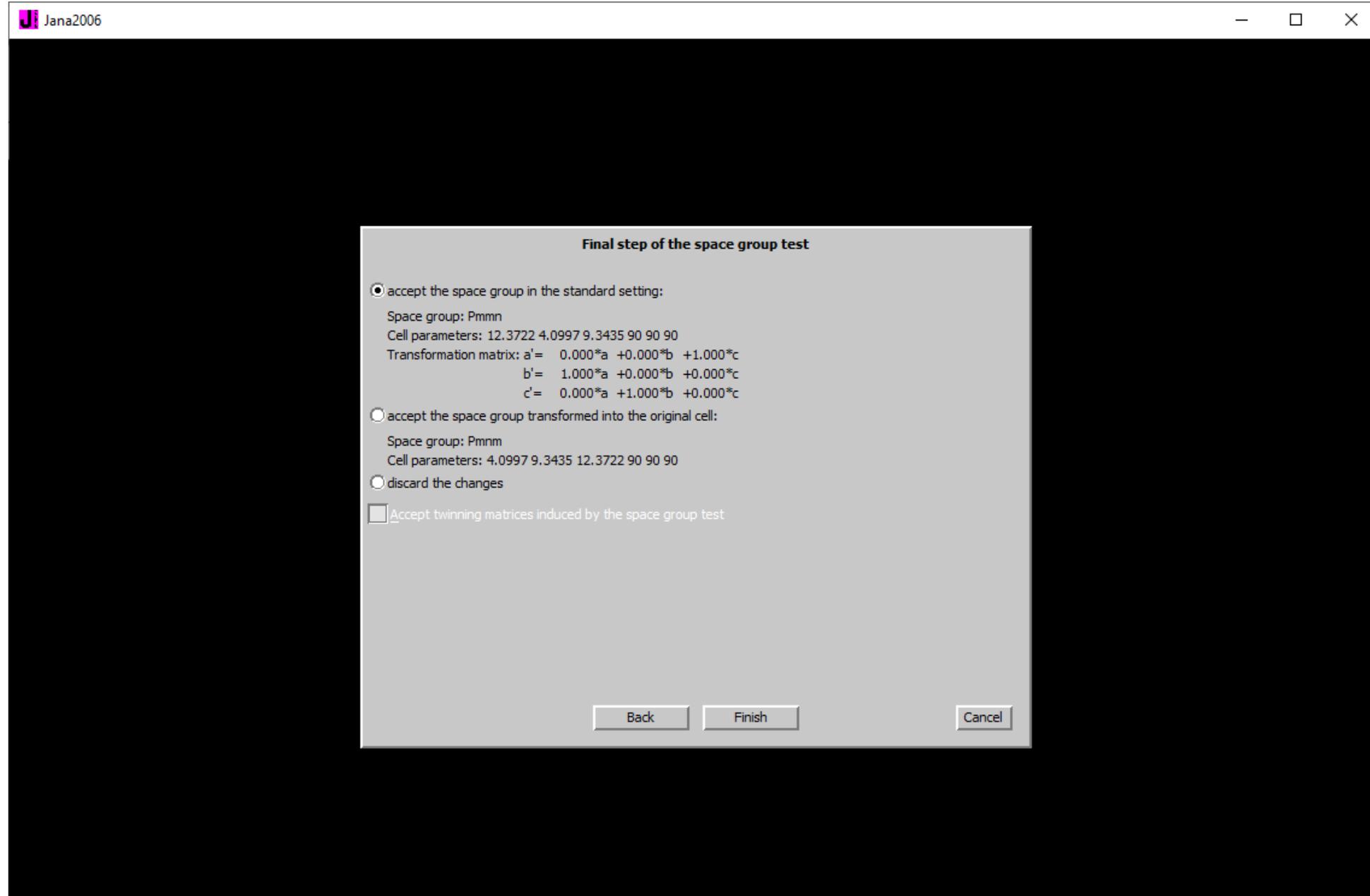
Space group	obs/all	ave(I/sig(I))	FOM
Pmnm	0/0	0.000/0.000	1.00000
Pmmm	0/0	0.000/0.000	1.00000
P21nm	0/0	0.000/0.000	1.00000
Pmn21	0/0	0.000/0.000	1.00000
Pm2m	0/0	0.000/0.000	1.00000
P2mm	0/0	0.000/0.000	1.00000
Pmm2	0/0	0.000/0.000	1.00000
P21221	0/0	0.000/0.000	1.00000
P2122	0/0	0.000/0.000	1.00000
P2221	0/0	0.000/0.000	1.00000
P222	0/0	0.000/0.000	1.00000

Details

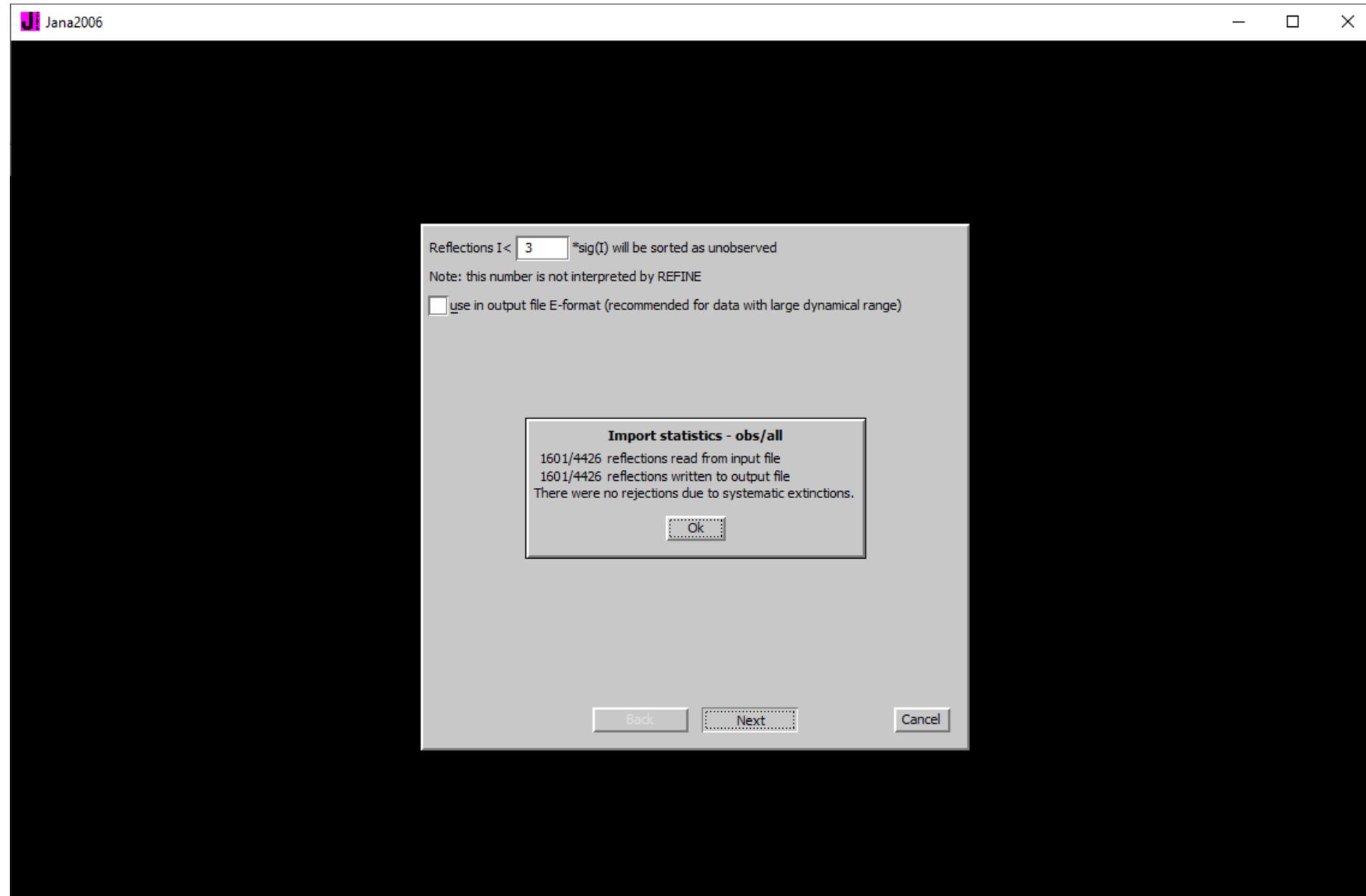
Back Next Cancel

Structure: E:\Crete\Teaching\JoC\ETY-453_Crystal Chemistry\2020 lectures\Single-crystal lecture\practice\(\Pb2BiS3)(AuTe2)\(\Pb2BiS3)(AuTe2)

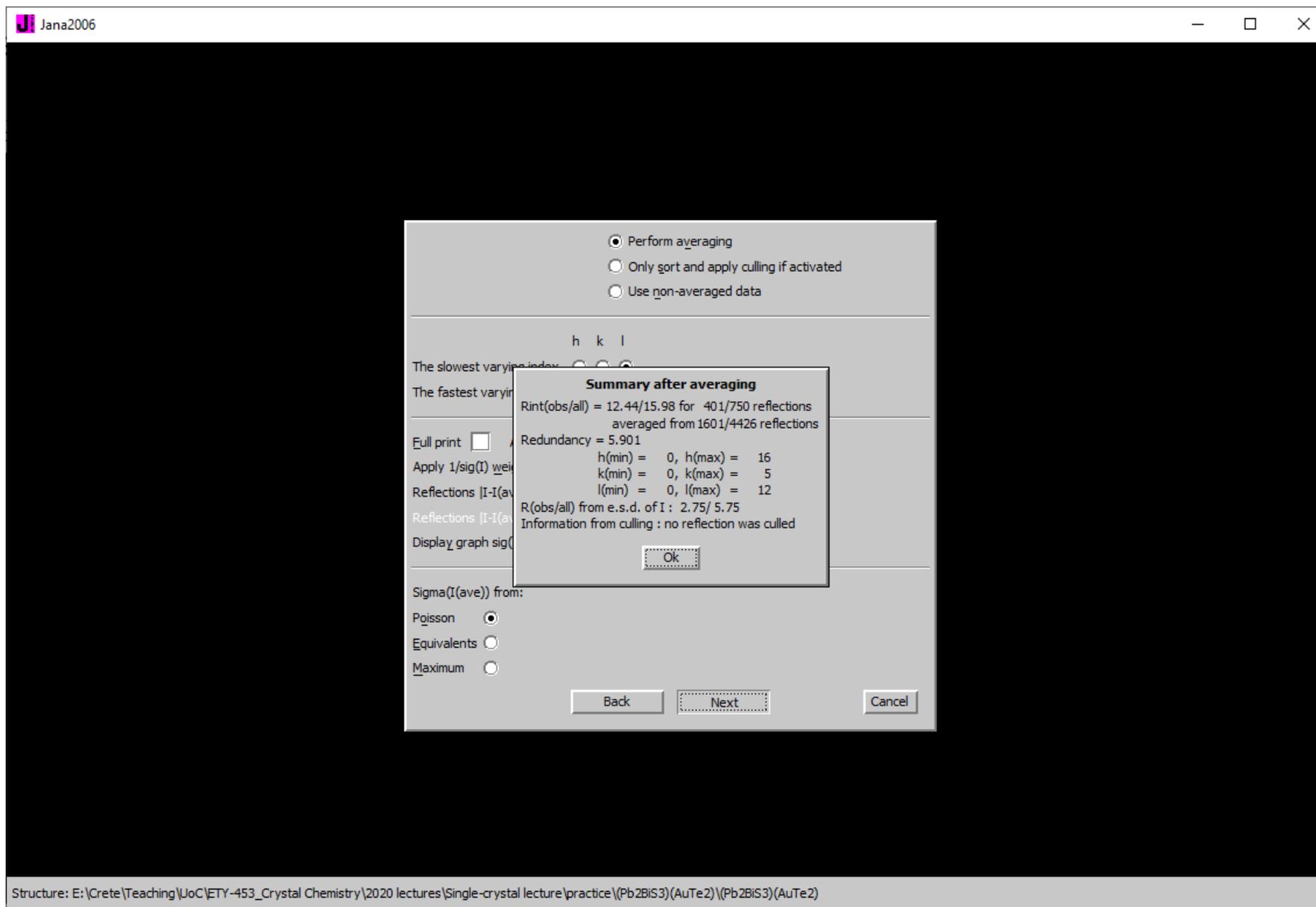
Accept you unit cell and proceed (standard or not is a matter of taste)



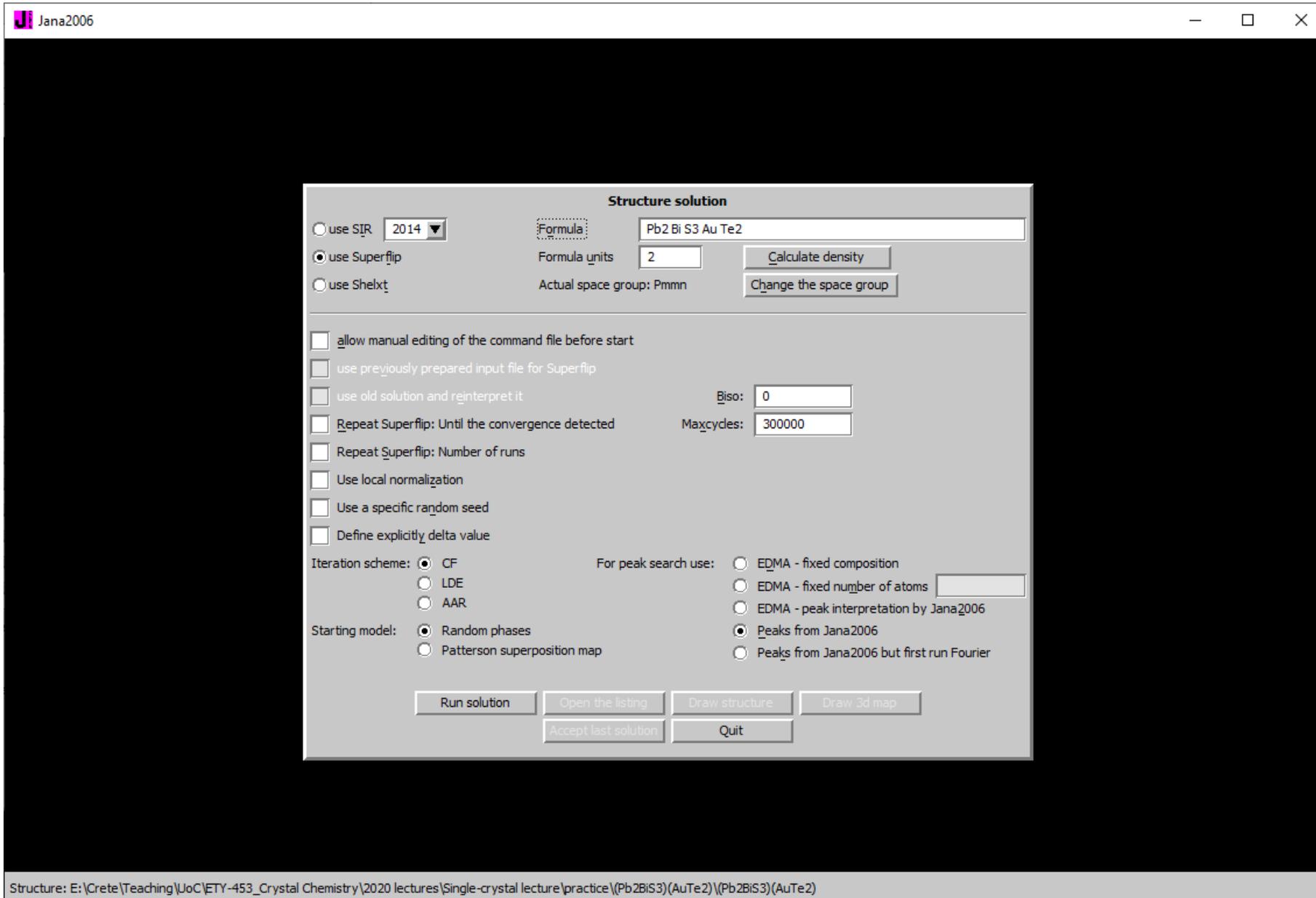
Read through the transformed hkl file. Keep default settings



Read and merge your (symmetry equivalent) reflections. Keep default settings.
Accept and proceed.



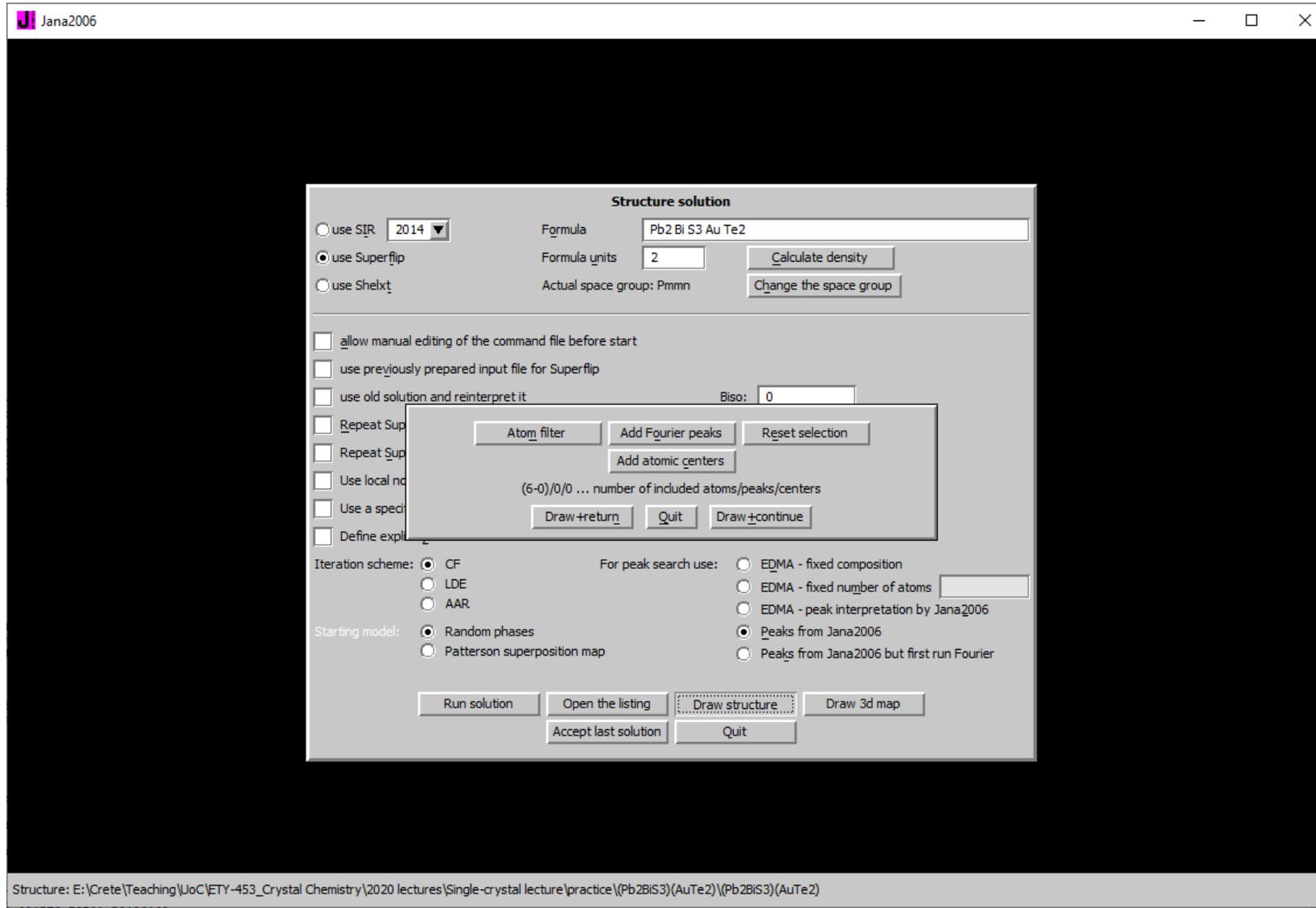
This is where you solve the structure. Type your formula and number of formula units as your best estimate. All elements present should be typed. Check density to make sense.



- Make sure
- Superflip (the program),
 - CF (the method)
 - random phases (the starting model) are selected.

Run solution

Draw your structure with your visualizer (VESTA, DIAMOND, MERCURY or anything else) to make sure you are happy (Draw + return). You need to set the software path in JANA2006 (Tools-> Programs-> . Accept solution if you are happy with it, else try a different model.



Inspect the tabs to make sure everything is correct

Jana2006

File Edit/View Run Wizards Parameters Tools

EditM50 Edit atoms Edit profile

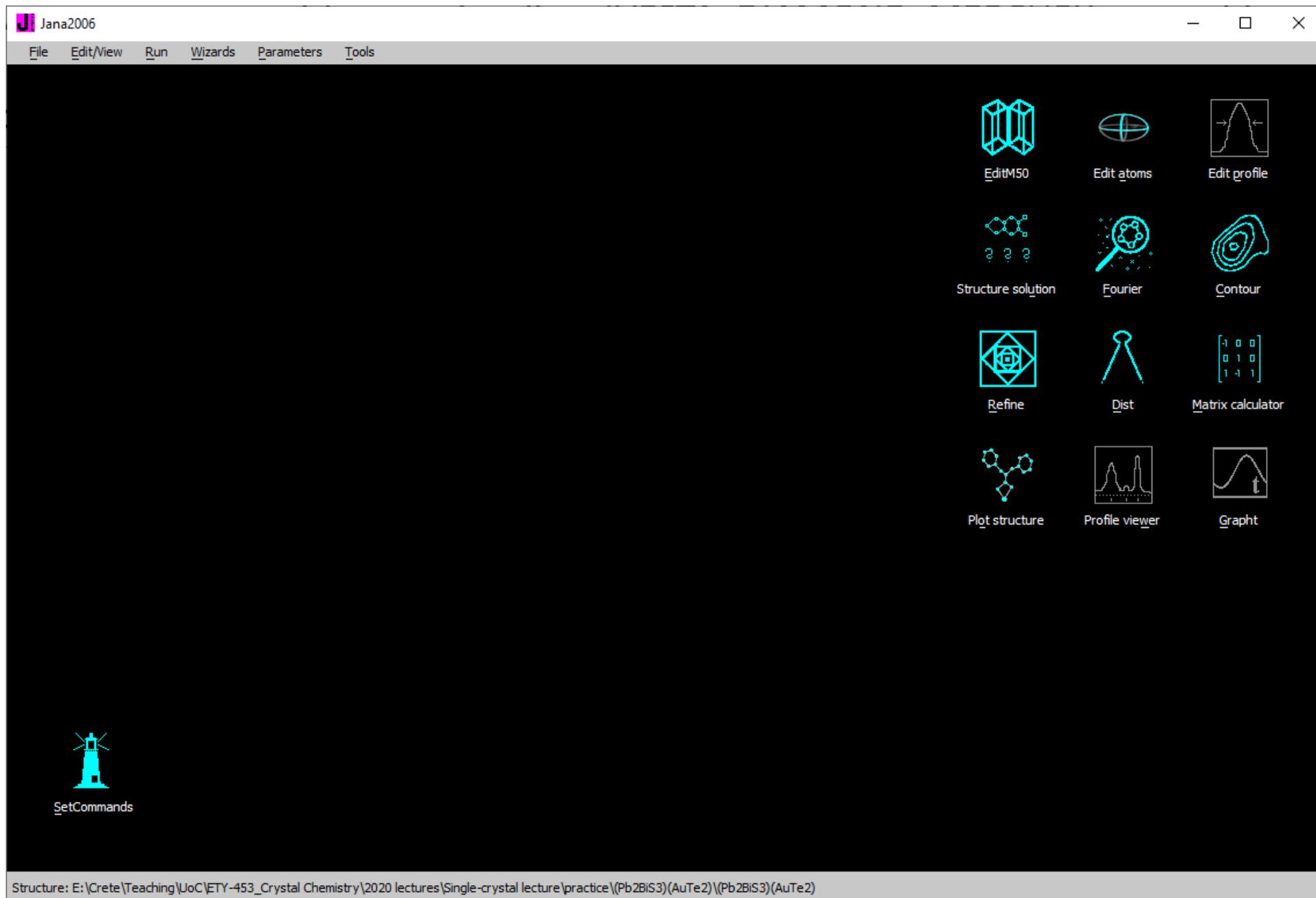
Structure solution Fourier Contour

Refine Dist Matrix calculator

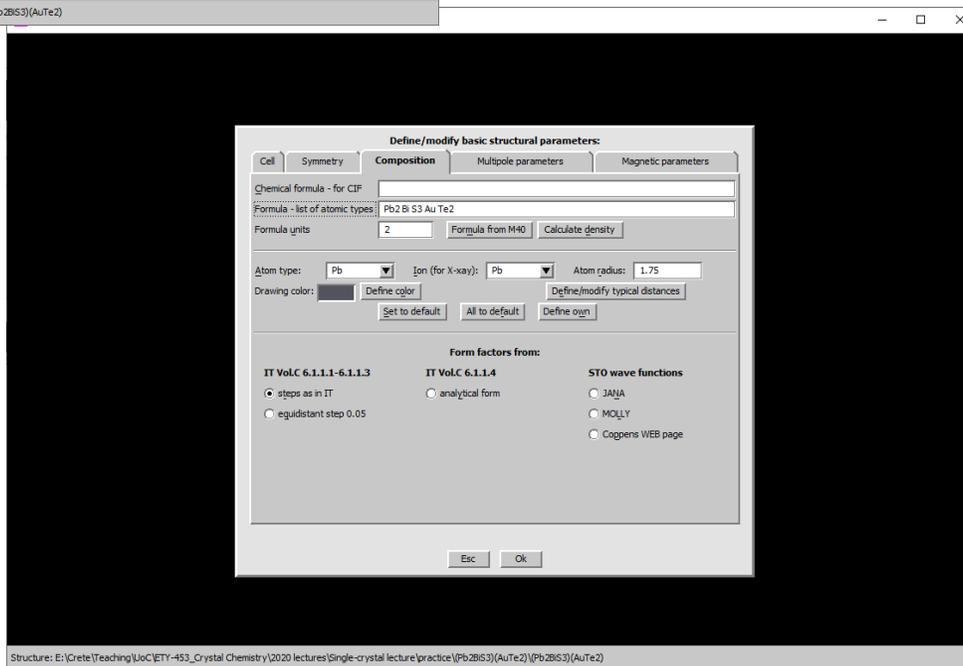
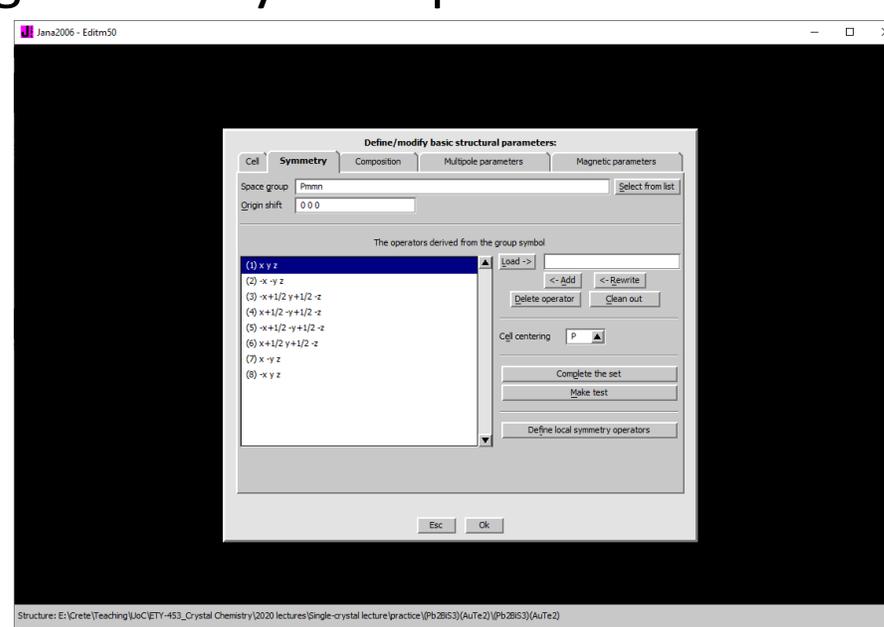
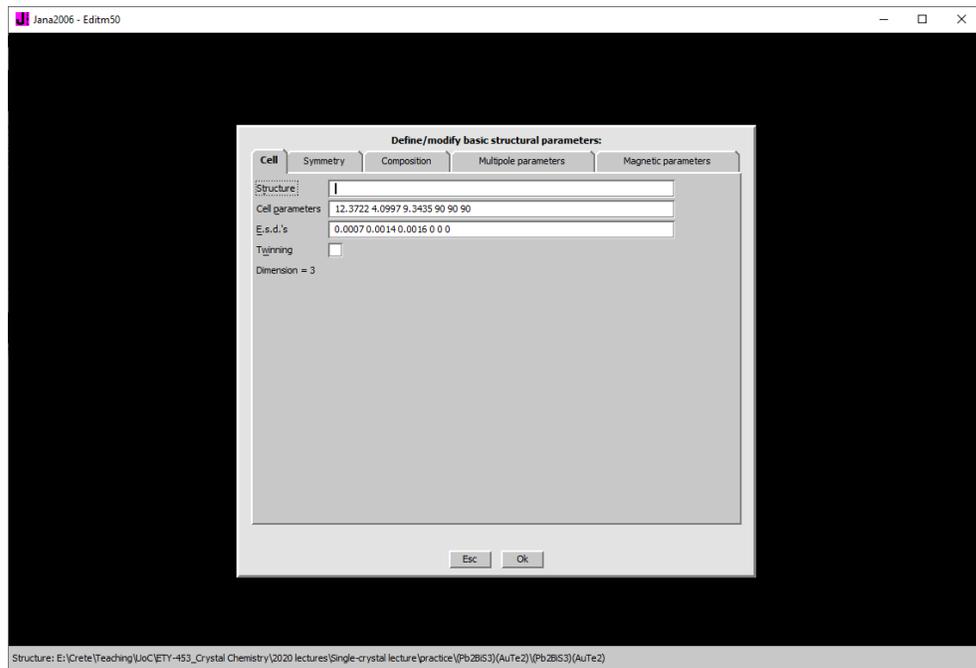
Plot structure Profile viewer Graph

SetCommands

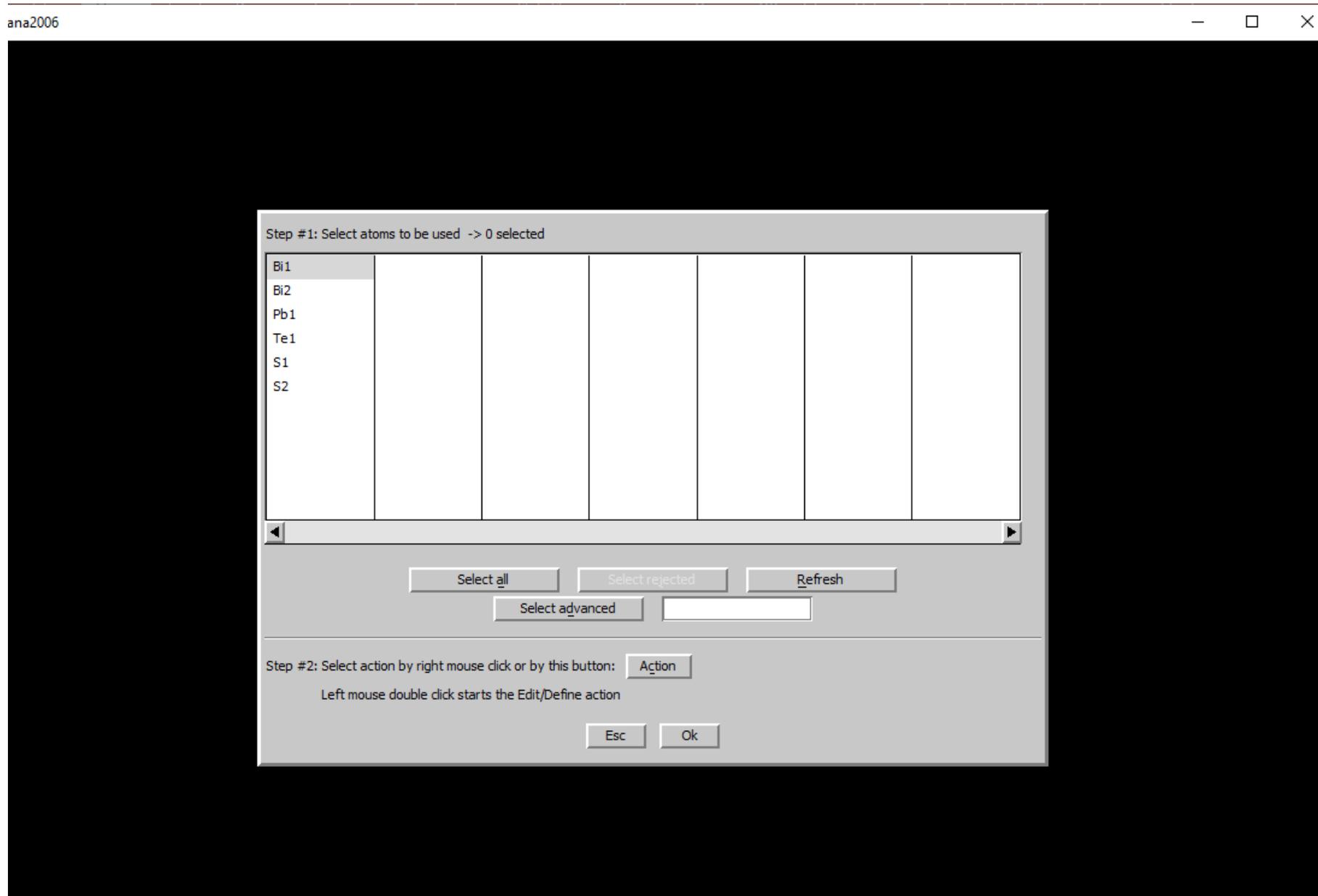
Structure: E:\Crete\Teaching\UoC\ETY-453_Crystal Chemistry\2020 lectures\Single-crystal lecture\practice\(\Pb2BiS3)(AuTe2)\(\Pb2BiS3)(AuTe2)

The image shows a screenshot of the Jana2006 software interface. At the top, there is a title bar with the text 'Jana2006' and standard window control buttons (minimize, maximize, close). Below the title bar is a menu bar with the following items: 'File', 'Edit/View', 'Run', 'Wizards', 'Parameters', and 'Tools'. The main area of the window is a dark grey grid of 12 icons, each with a label below it. The icons are arranged in four rows and three columns. The first row contains 'EditM50' (a 3D unit cell diagram), 'Edit atoms' (a sphere with a grid), and 'Edit profile' (a peak with a vertical line). The second row contains 'Structure solution' (a molecular structure), 'Fourier' (a magnifying glass over a structure), and 'Contour' (a contour plot). The third row contains 'Refine' (a unit cell with a structure inside), 'Dist' (a pair of compasses), and 'Matrix calculator' (a 3x3 matrix). The fourth row contains 'Plot structure' (a molecular structure), 'Profile viewer' (a plot with peaks), and 'Graph' (a smooth curve). In the bottom-left corner, there is a small icon of a lighthouse and the text 'SetCommands'. At the very bottom of the window, there is a status bar with the text 'Structure: E:\Crete\Teaching\UoC\ETY-453_Crystal Chemistry\2020 lectures\Single-crystal lecture\practice\(\Pb2BiS3)(AuTe2)\(\Pb2BiS3)(AuTe2)'. The text in the status bar is partially obscured by a grey bar at the bottom of the image.

Check M50. The information should agree with your input



Check Edit atoms. This is what results from structure solution.



Inspect your atoms (double click)

Jana2006

Step #1: Select atoms to be used -> 1 selected

Atom edit

Define | **Edit**

Name: Bi1 | Type: Bi **atom type (JANA's choice)**

ADP parameter(s):

- isotropic **atom name (your choice)**
- harmonic (anisotropic)
- anharmonic
- Use TLS

Esc | Ok

Structure: E:\Crete\Teaching\JoC\ETY-453_Crystal Chemistry\2020 lectures\Single-crystal lecture\practice\{Pb2BiS3}(AuTe2)\{Pb2BiS3}(AuTe2)

Atomic model

Jana2006

Step #1: Select atoms to be used -> 1 selected

Atom edit

Define | **Edit**

Occupancy: 1 | Name: Bi1 | Type: Bi

ax: 0.25 | x: 0 | y: 0.5 | z: 0.313 **coordinates**

Uiso: 0.037995 **thermal parameter**

Refine all | Fix all | Reset

Show/reset site occupancy | Apply site symmetry | Show symmetry restrictions

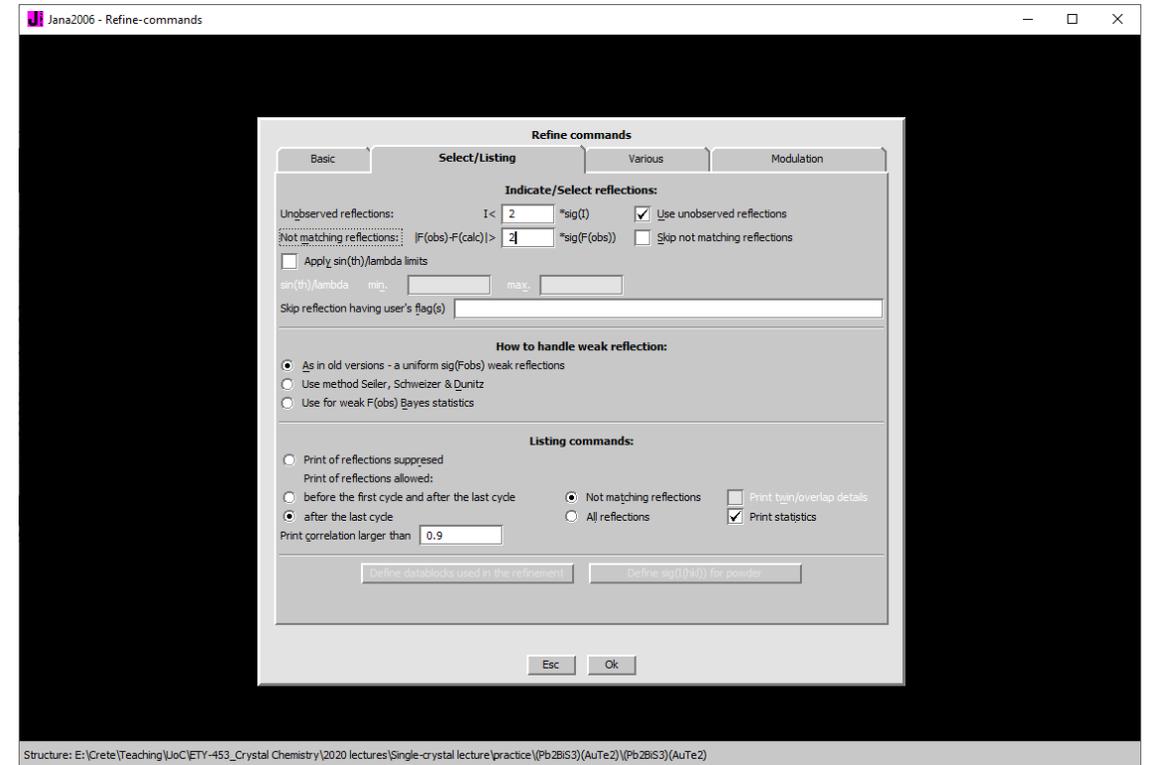
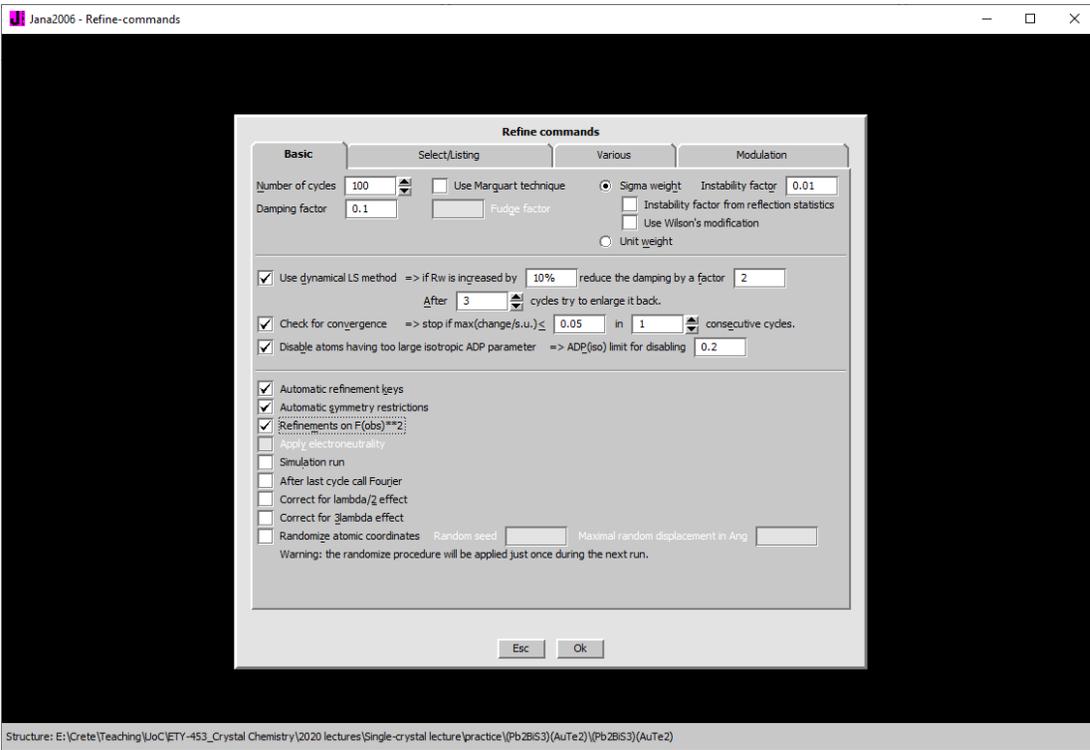
Edit special parameters: ADP

Esc | Ok

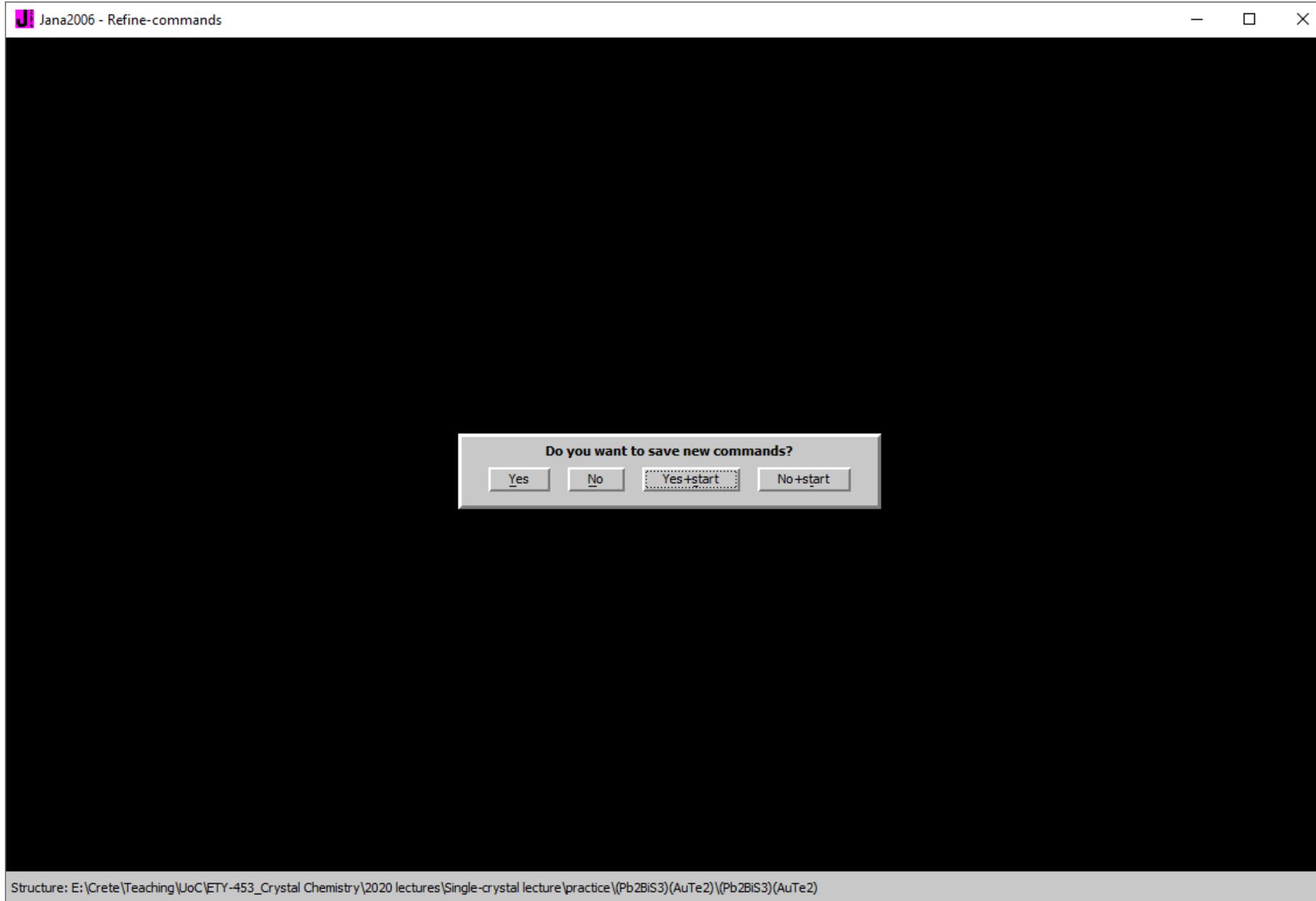
Structure: E:\Crete\Teaching\JoC\ETY-453_Crystal Chemistry\2020 lectures\Single-crystal lecture\practice\{Pb2BiS3}(AuTe2)\{Pb2BiS3}(AuTe2)

occupancy (multiplicity)

Setup the refinement process (right click)



All set (Save and Start)



This is the result. GOF should be close to 1.
R(obs) (structure quality) should be as low as possible < 10%
Refinement stops once Maximum change < 0.05 or when cycles run out

The screenshot shows the 'Jana2006 - Refine' window. The main area is black with a white text box in the center containing the following statistics:

```
R factors : [750=485+265/16], Damping factor: 0.1000  
GOF(obs)= 6.39 GOF(all)= 5.29  
R(obs)= 17.91 wR2(obs)= 31.30 R(all)= 24.10 wR2(all)= 32.42  
Last wR2(all): 32.43 32.43 32.43 32.43 32.42 32.42 32.42 32.42  
Maximum change/s.u. : 0.0483 for Uiso[Te1]
```

Below the statistics is a dialog box titled 'Regular end of REFINE program'. It asks 'Open the listing?' and has 'Yes' and 'No' buttons. At the bottom of the dialog, it says 'It remains 7 sec - press space for pause'.

At the bottom of the window, the file path is displayed: Structure: E:\Crete\Teaching\JoC\ETY-453_Crystal Chemistry\2020 lectures\Single-crystal lecture\practice\{Pb2BIS3}(AuTe2)\{Pb2BIS3}(AuTe2)

Inspect your atoms again

Thermal parameters and some coordinates do refine. Coordinates at special lattice positions do not refine. They are fixed due to symmetry

Step #1: Select atoms to be used -> 0 selected

Atom edit

Define Edit

#	Name	B1	Type	Bi
1	ai	0.25	x 0.5	y 0 z 0.310641

Uiso 0.026325

Refine all Fix all Reset

Show/reset site occupancy Apply site symmetry Show symmetry restrictions

Edit special parameters: ADP

Esc Ok

Esc Ok

Structure: E:\Crete\Teaching\UoC\ETY-453_Crystal Chemistry\2020 lectures\Single-crystal lecture\practice\{(Pb2BIS3)(AuTe2)\(Pb2BIS3)(AuTe2)

This is your structure so far. Some atoms look ok, but some atoms are mislabeled

(Pb2BiS3)(AuTe2)_tmp.cif - VESTA

File Edit View Objects Utilities Help

a b c a* b* c* Step (°): 45.0 Step (px): 10 Step (%): 10

Tools Style Objects (Pb2BiS3)(AuTe2)_tmp.cif

Structural models
 Show models
 Show dot surface

Style
 Ball-and-stick
 Space-filling
 Polyhedral
 Wireframe
 Stick

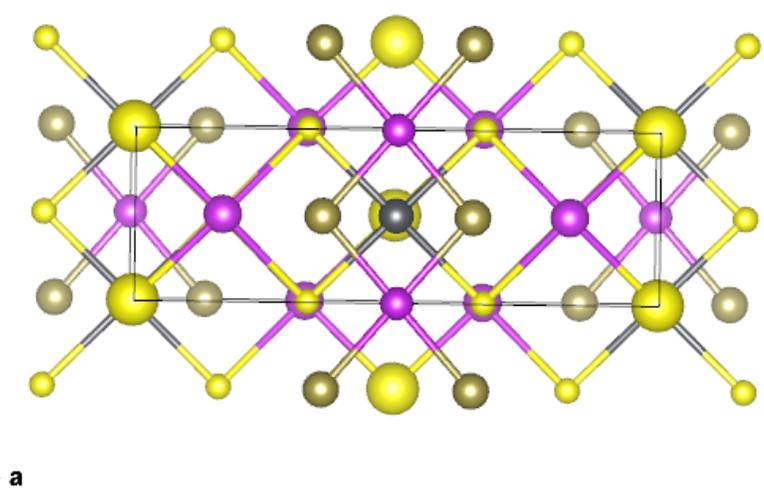
Volumetric data
 Show sections
 Show isosurfaces
 Surface coloring

Style
 Smooth shading
 Wireframe
 Dot surface

Crystal shapes
 Show shapes

Style
 Unicolor
 Custom color
 Wireframe

Properties...
Boundary... Orientation...

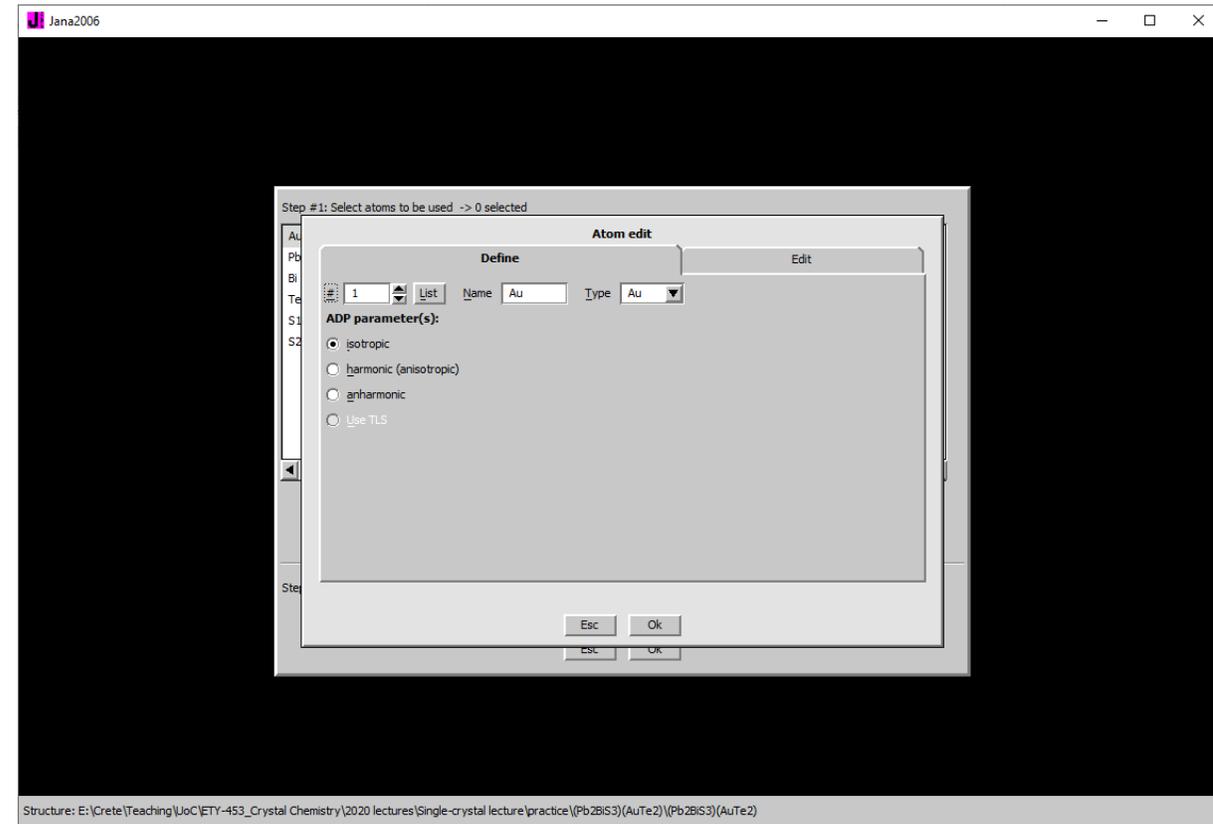
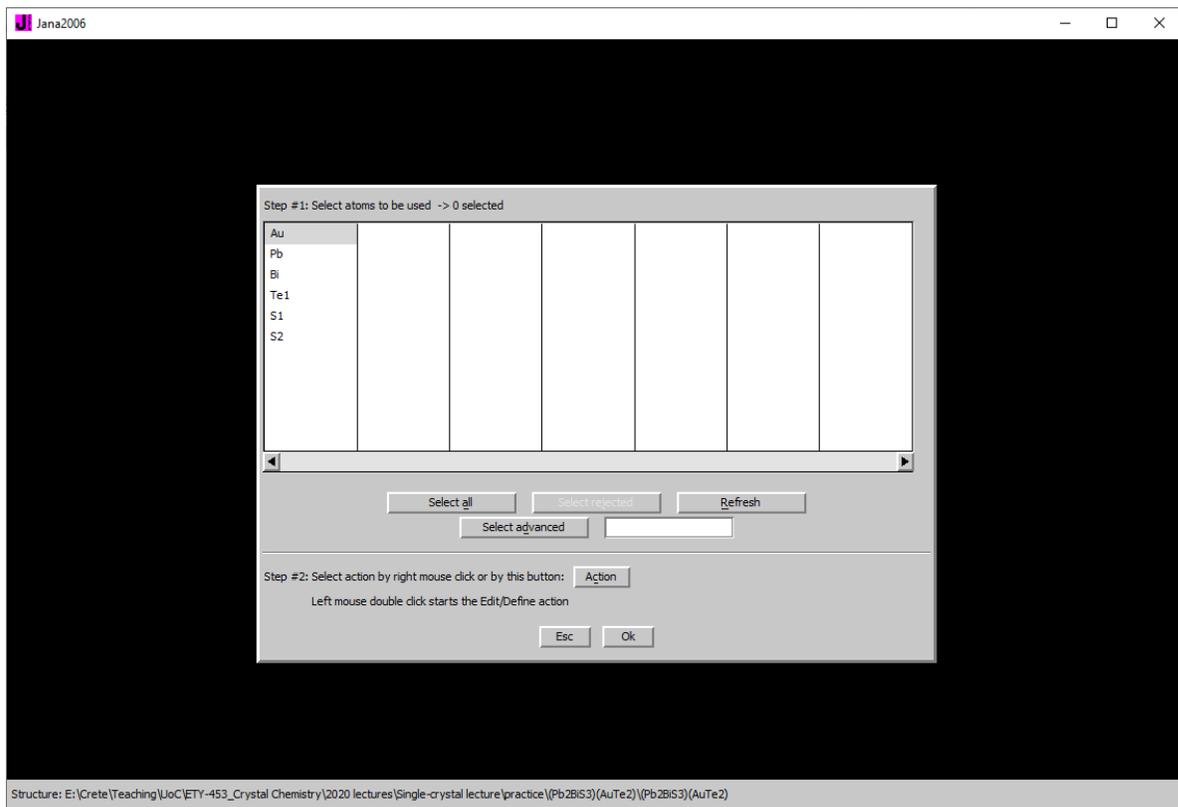


		x	y	z	Occ.	U	Site	Sym.	
1	Bi	Bi1	1.00000	0.50000	0.00213	1.000	0.026	2b	mm2
2	Bi	Bi2	0.33055	1.00000	0.31755	1.000	0.034	4f	.m.
3	Pb	Pb1	0.50000	0.50000	0.64818	1.000	0.029	2a	mm2
4	Te	Te1	0.85709	1.00000	0.00432	1.000	0.031	4f	.m.
5	S	S1	0.50000	0.50000	0.36647	1.000	0.064	2a	mm2
6	S	S2	0.33228	1.00000	0.62124	1.000	0.015	4f	.m.

Number of polygons and unique vertices on isosurface = 0 (0)
52 atoms, 71 bonds, 15 polyhedra; CPU time = 2 ms

Output Summary Comment

Change the atoms in a way it seems appropriate.



Refine again. This is the new structure

(Pb2BiS3)(AuTe2)_tmp.cif - VESTA

File Edit View Objects Utilities Help

a b c a* b* c* Step (°): 45.0 Step (px): 10 Step (%): 10

Tools Style Objects

Structural models

- Show models
- Show dot surface

Style

- Ball-and-stick
- Space-filling
- Polyhedral
- Wireframe
- Stick

Volumetric data

- Show sections
- Show isosurfaces
- Surface coloring

Style

- Smooth shading
- Wireframe
- Dot surface

Crystal shapes

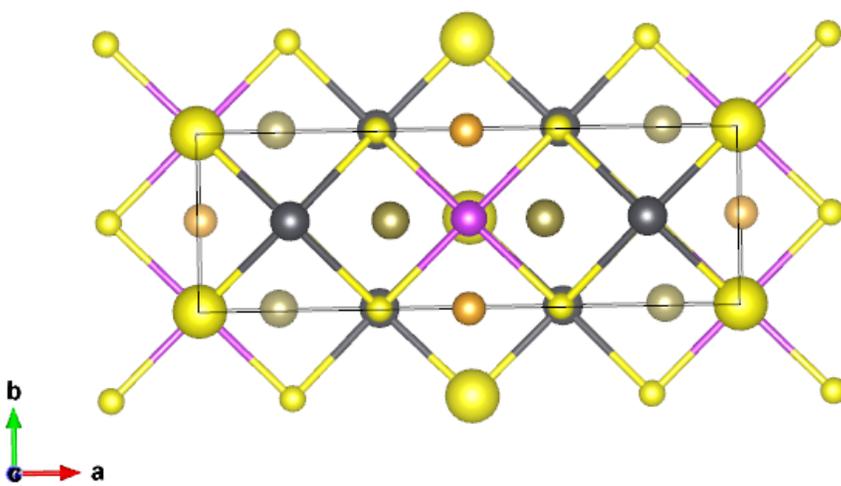
- Show shapes

Style

- Unicolor
- Custom color
- Wireframe

Properties...

Boundary... Orientation...



		x	y	z	Occ.	U	Site	Sym.	
1	Au	Au	1.00000	0.50000	0.00210	1.000	0.025	2b	mm2
2	Pb	Pb	0.33057	1.00000	0.31755	1.000	0.034	4f	.m.
3	Bi	Bi	0.50000	0.50000	0.64813	1.000	0.029	2a	mm2
4	Te	Te1	0.85723	1.00000	0.00430	1.000	0.031	4f	.m.
5	S	S1	0.50000	0.50000	0.36699	1.000	0.064	2a	mm2
6	S	S2	0.33224	1.00000	0.62129	1.000	0.015	4f	.m.

Number of polygons and unique vertices on isosurface = 0 (0)
44 atoms, 55 bonds, 11 polyhedra; CPU time = 2 ms

Output Summary Comment

Check the formula in M50. Is the unit cell contents complete?

Jana2006 - Editm50

Define/modify basic structural parameters:

Cell | Symmetry | **Composition** | Multipole parameters | Magnetic parameters

Chemical formula - for CIF:

Formula - list of atomic types:

Formula units:

Atom type: Ion (for X-ray): Atom radius:

Drawing color:

INFORMATION

Formula from M40 : Au Bi Pb2 S3 Te2
Molecular weight = 1171.71
Calculated density = 8.2109 g.cm⁻³
Absorption coefficient $\mu(\text{Mo-K}\alpha) = 75.968 \text{ mm}^{-1}$

IT Vol.C 6.1.1.1-6.1.1.3

steps as in IT

equidistant step 0.05

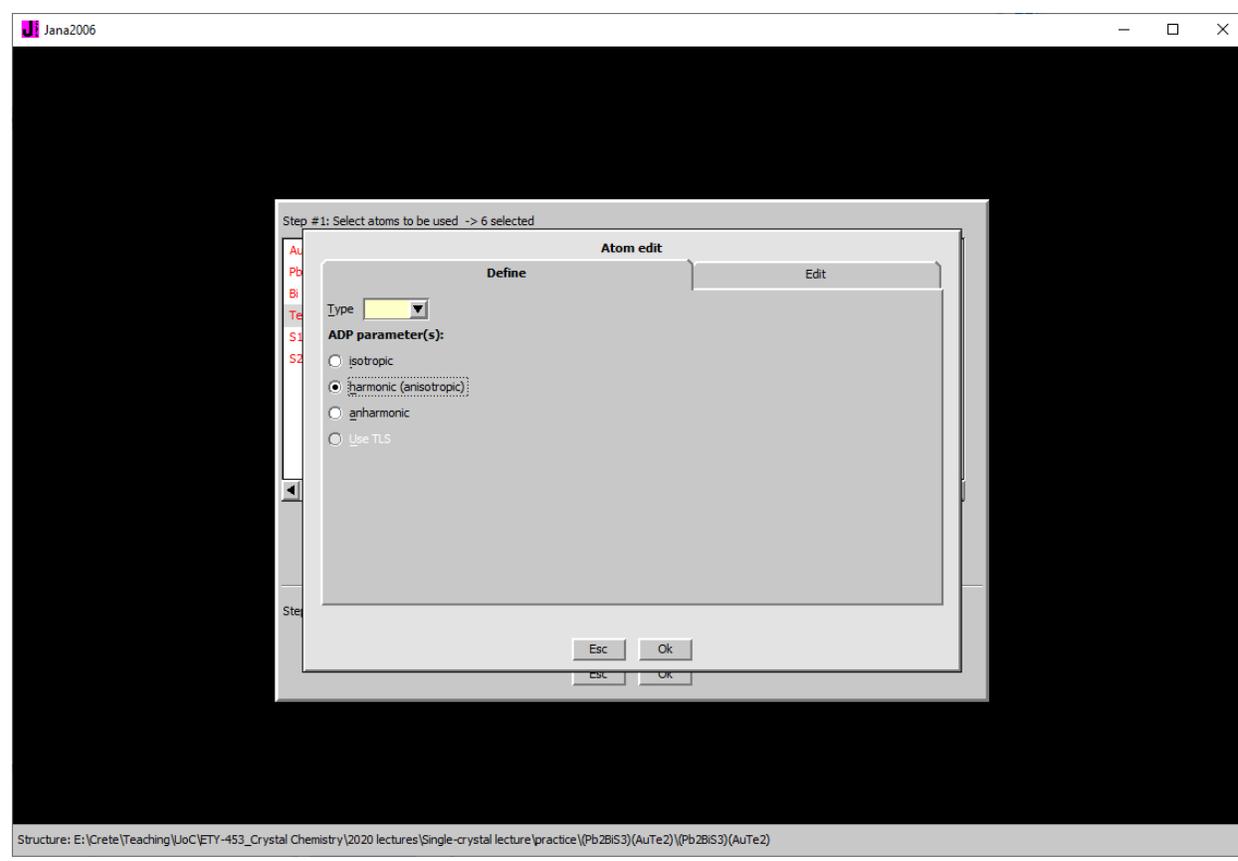
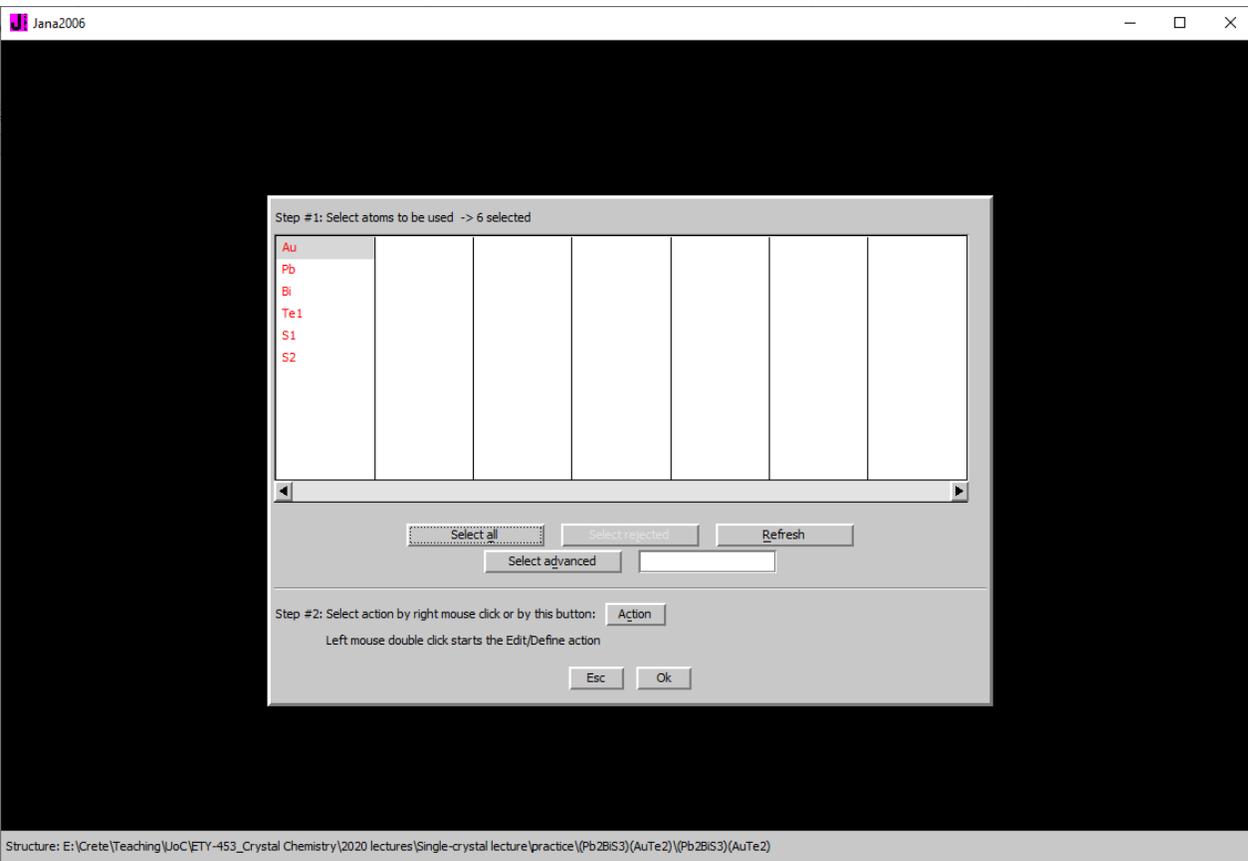
NA

MOLLY

Coppens WEB page

everything looks good
all atoms assigned

Once we have the structure and we are happy with the atoms, we start refining the thermal motions. This is very important when heavy atoms are present.



Refine again. Notice how the statistics improve drastically. With $R < 10\%$ and $wR < 20\%$, you are about to complete the refinement. Hard part is done!

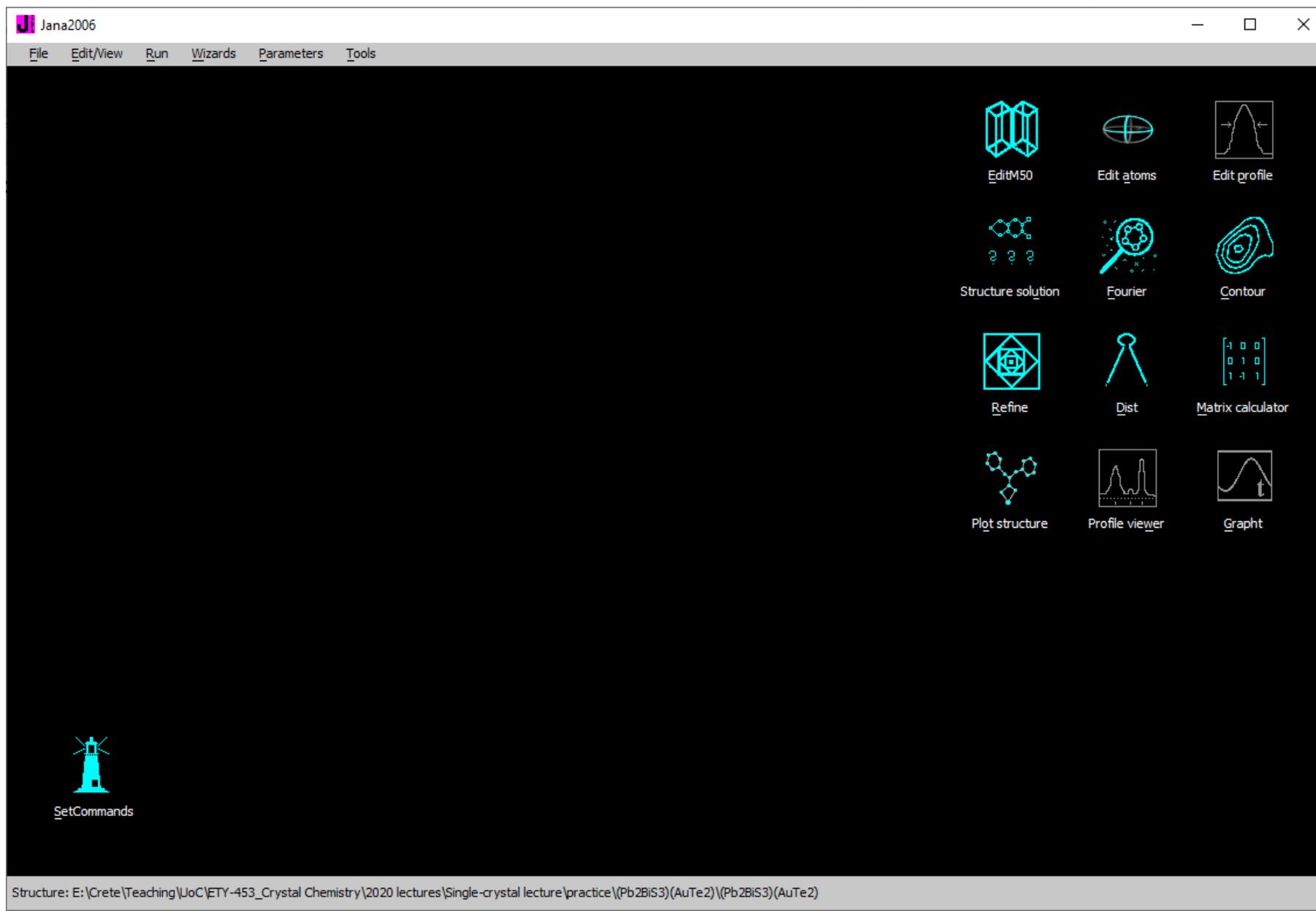
The screenshot shows the 'Jana2006 - Refine' window. The main area is black with a white text box in the center displaying the following statistics:

```
R factors : [750=401+349/31], Damping factor: 1.0000  
GOF(obs)= 2.95 GOF(all)= 2.28  
R(obs)= 7.02 wR(obs)= 6.43 R(all)= 13.33 wR(all)= 6.93  
Last wR(all): 13.90 7.19 6.93 6.93 6.93  
Maximum change/s.u. : -0.0102 for U33[S1]
```

Below the statistics, a dialog box titled 'Regular end of REFINE program' is displayed. It asks 'Open the listing?' and has 'Yes' and 'No' buttons. The 'No' button is selected. At the bottom of the dialog, it says 'It remains 8 sec - press space for pause'.

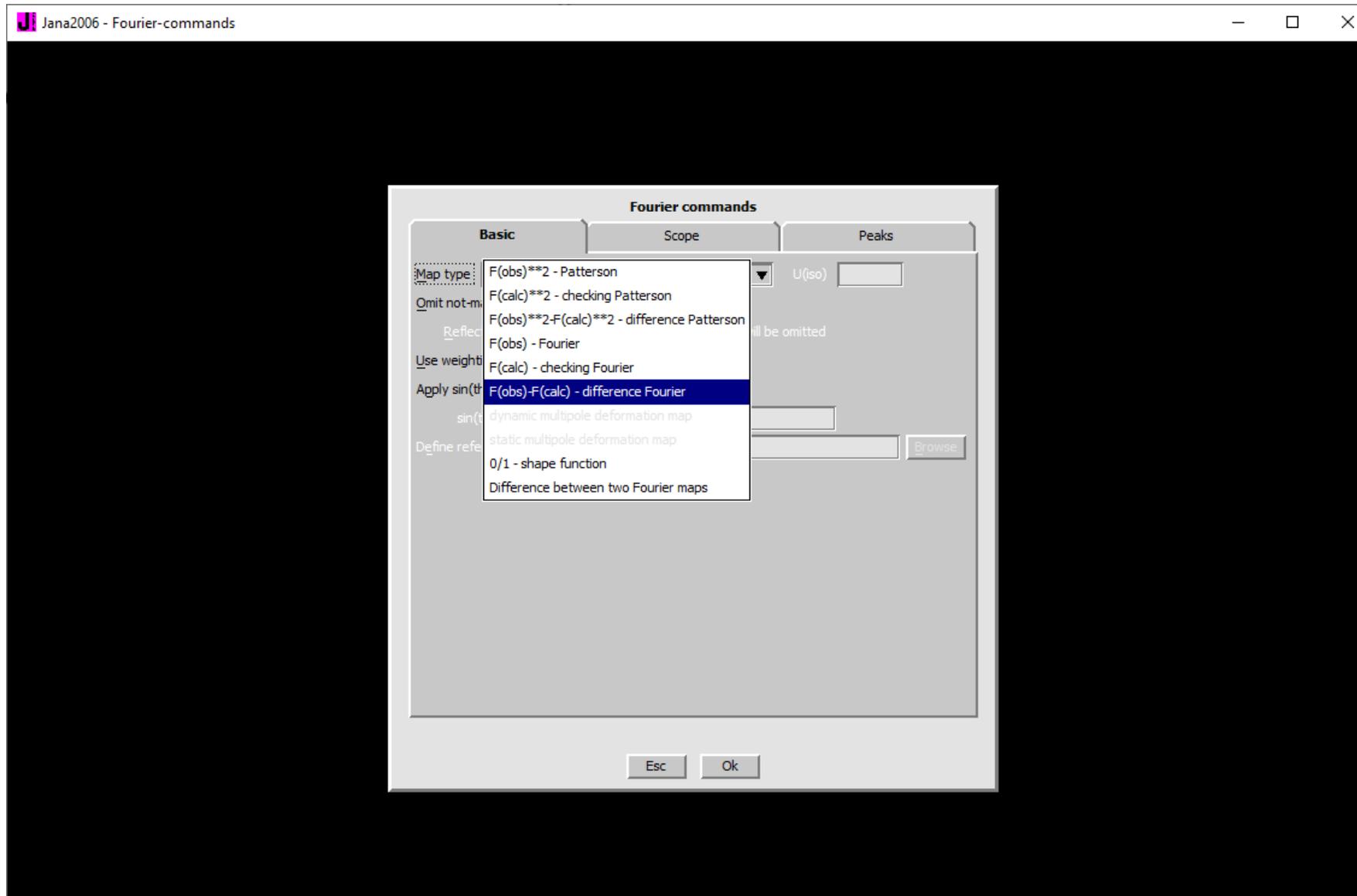
At the bottom of the window, the structure path is shown: Structure: E:\Crete\Teaching\JoC\ETY-453_Crystal Chemistry\2020 lectures\Single-crystal lecture\practice\(\Pb2BiS3)(AuTe2)\(\Pb2BiS3)(AuTe2)

Before you finish. Make sure you check the details and right your files properly. First part is Fourier, followed by Dist

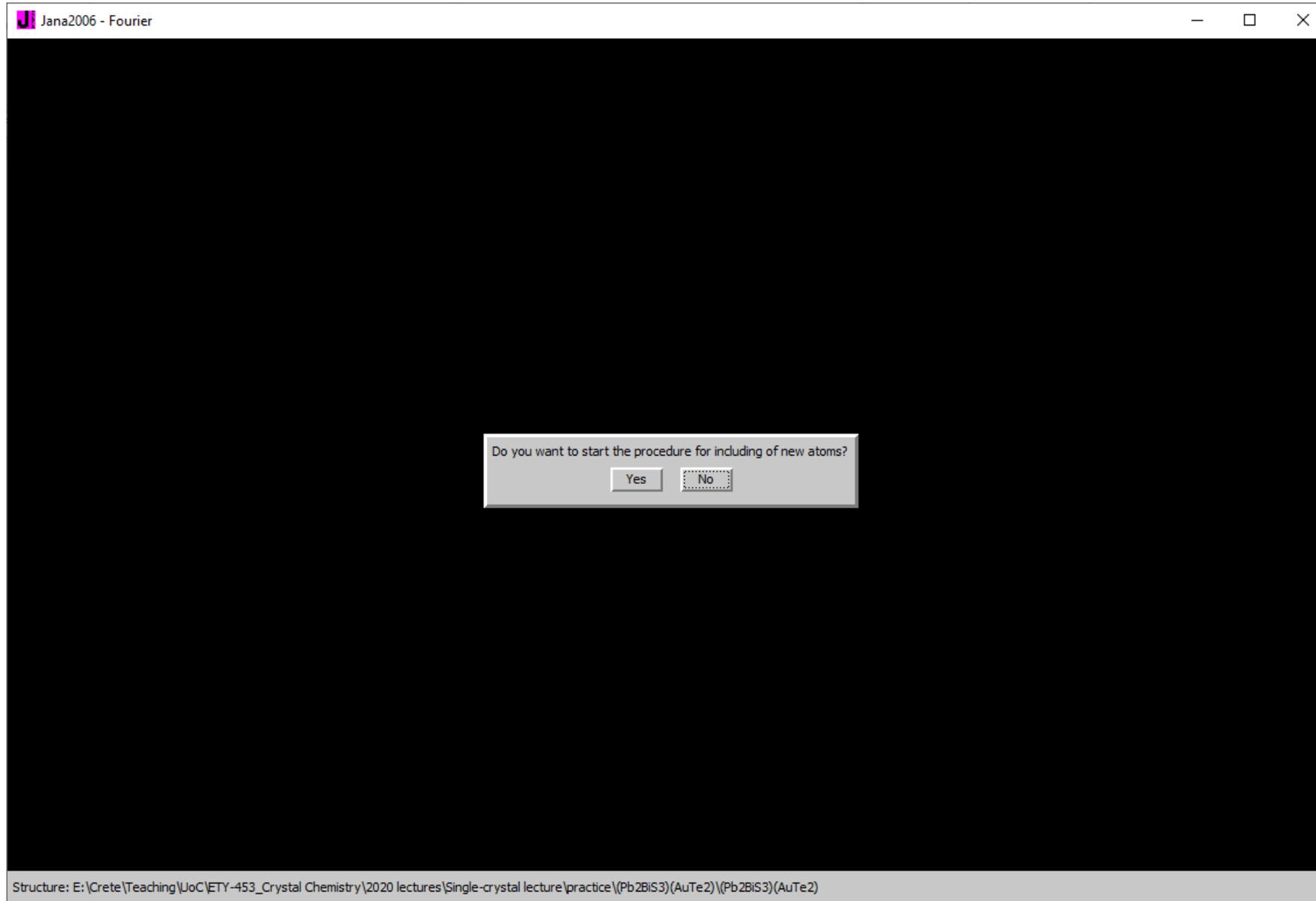


Run Fourier (right click)

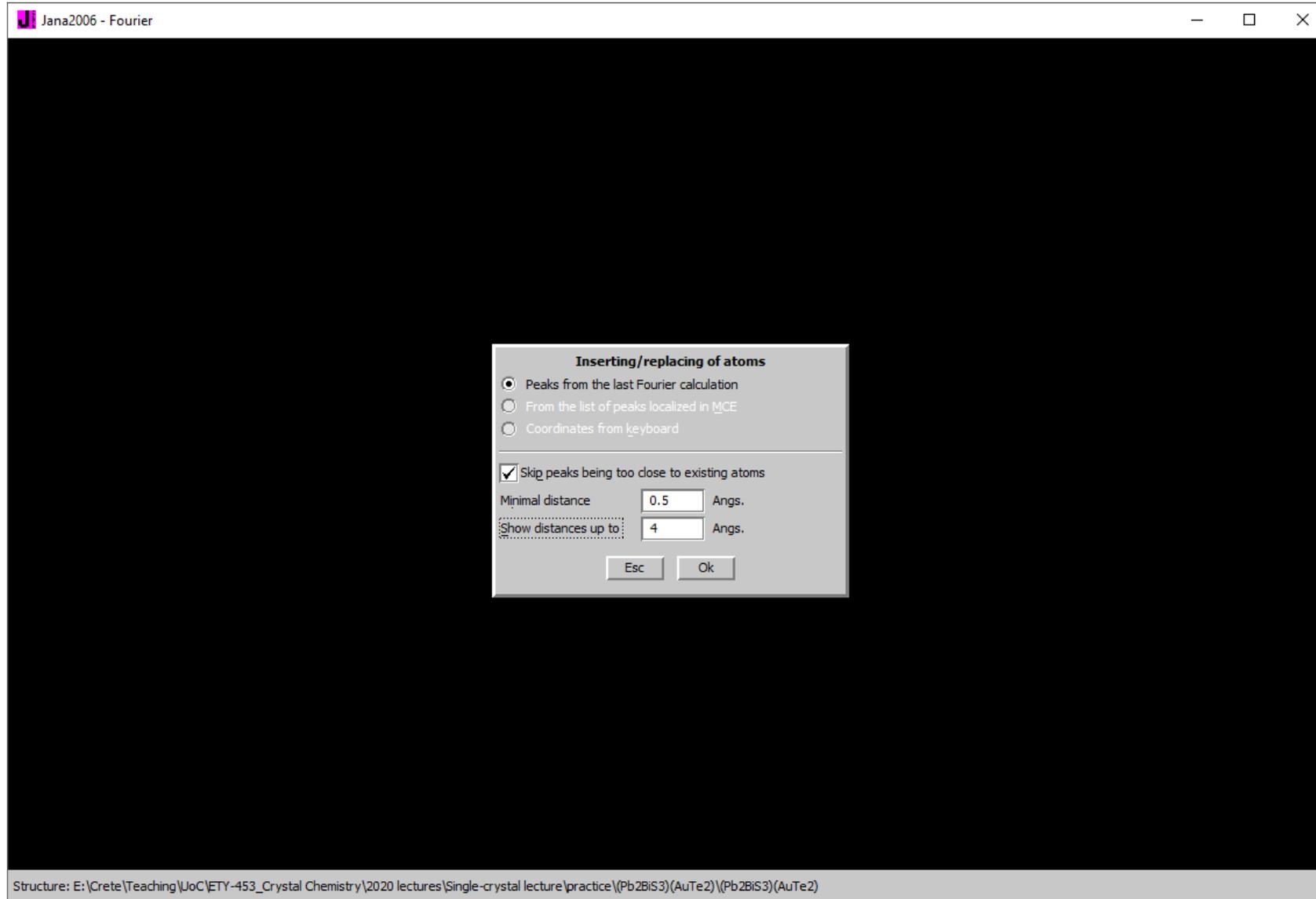
Select difference Fourier to assign unaccounted electron density to new atoms. Ok and start.



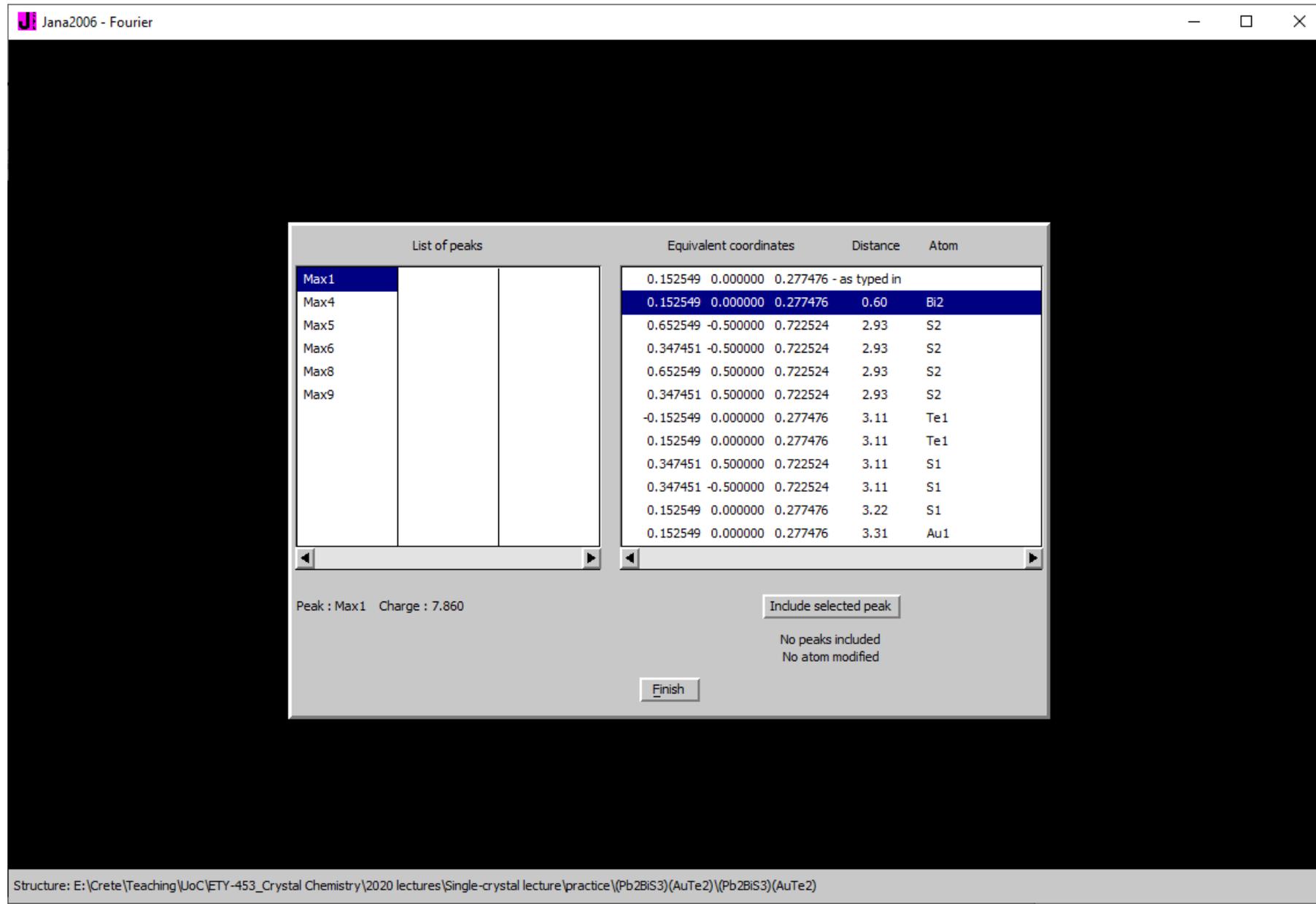
Select No. All the atoms are present.



If there are atoms missing, you click yes on this menu. Choose to look for new atoms and select reasonable distances for new atoms. Not too close and slightly above what you expect for bonding distance



Include and name the new atoms. In this case, there is not appreciable density close to the atoms. Seems that all atoms have been found. No additions needed



The screenshot shows the 'Jana2006 - Fourier' window. It features a 'List of peaks' table on the left and an 'Equivalent coordinates' table on the right. The 'List of peaks' table has columns for peak name, intensity, and charge. The 'Equivalent coordinates' table has columns for x, y, z coordinates, distance, and atom name. The 'List of peaks' table shows a list of peaks from Max1 to Max9. The 'Equivalent coordinates' table shows a list of coordinates for atoms Bi2, S2, Te1, and Au1. The 'Include selected peak' button is highlighted, and the 'Finish' button is visible at the bottom.

Peak	Intensity	Charge
Max1		
Max4		
Max5		
Max6		
Max8		
Max9		

x	y	z	Distance	Atom
0.152549	0.000000	0.277476	- as typed in	
0.152549	0.000000	0.277476	0.60	Bi2
0.652549	-0.500000	0.722524	2.93	S2
0.347451	-0.500000	0.722524	2.93	S2
0.652549	0.500000	0.722524	2.93	S2
0.347451	0.500000	0.722524	2.93	S2
-0.152549	0.000000	0.277476	3.11	Te1
0.152549	0.000000	0.277476	3.11	Te1
0.347451	0.500000	0.722524	3.11	S1
0.347451	-0.500000	0.722524	3.11	S1
0.152549	0.000000	0.277476	3.22	S1
0.152549	0.000000	0.277476	3.31	Au1

Peak : Max1 Charge : 7.860

Include selected peak

No peaks included
No atom modified

Finish

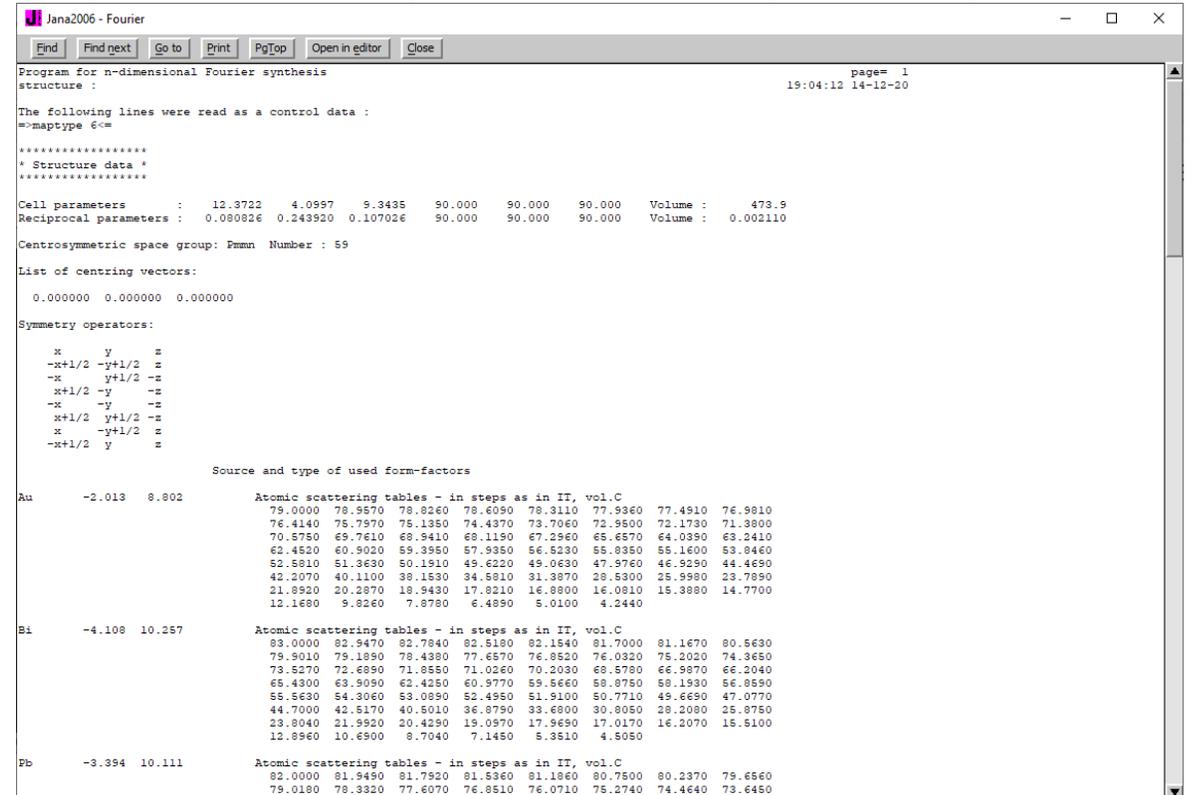
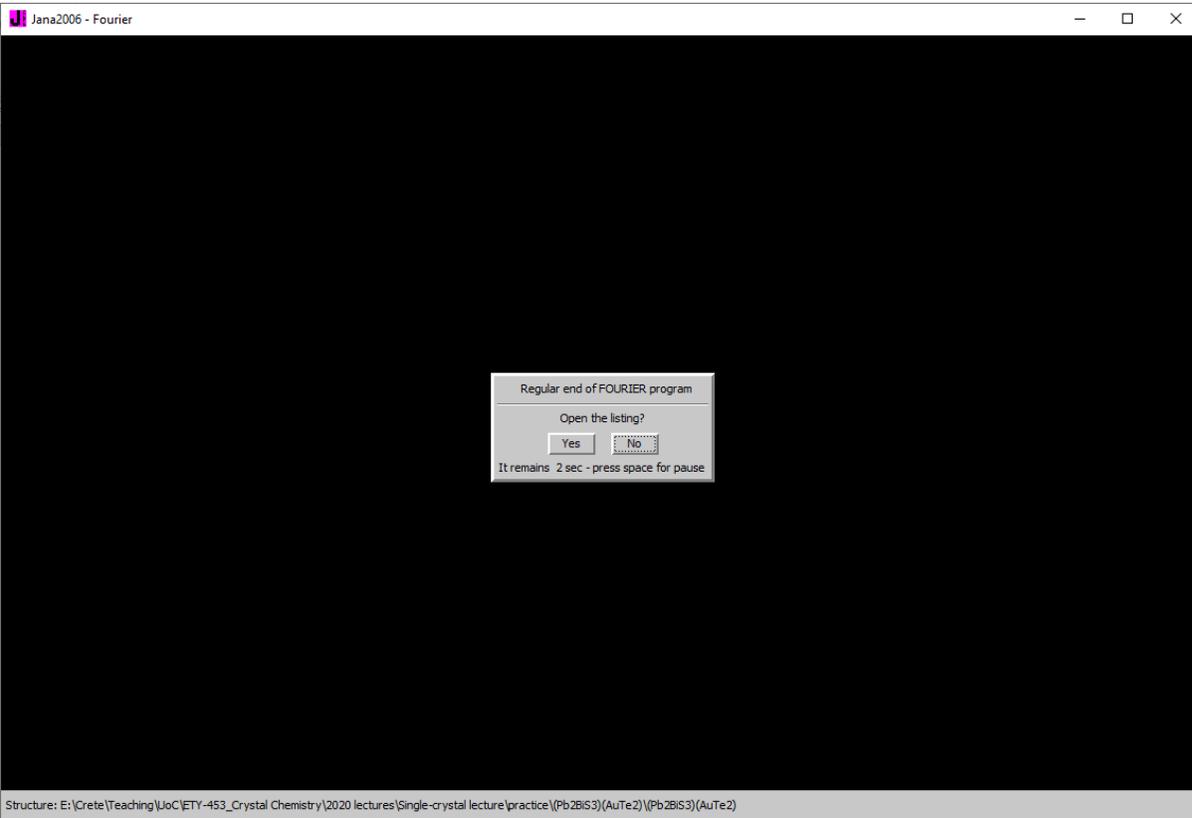
Structure: E:\Crete\Teaching\UoC\ETY-453_Crystal Chemistry\2020 lectures\Single-crystal lecture\practice\{Pb2BiS3}(AuTe2)\{Pb2BiS3}(AuTe2)

Now this is your new atom list. Modify, then, run refine again. And so on and so forth, until all atoms are present.

The screenshot shows the Jana2006 software window with a dialog box titled "Step #1: Select atoms to be used -> 0 selected". The dialog box contains a table with 13 rows of atom labels and 7 empty columns. Below the table are buttons for "Select all", "Select rejected", "Refresh", and "Select advanced" (with an adjacent empty text box). Below these buttons is a section for "Step #2: Select action by right mouse click or by this button:" with an "Action" button. A note states "Left mouse double click starts the Edit/Define action". At the bottom of the dialog are "Esc" and "Ok" buttons. The status bar at the bottom of the window displays the file path: "Structure: E:\Crete\Teaching\JoC\ETY-453_Crystal Chemistry\2020 lectures\Single-crystal lecture\practice\{(Pb2BiS3)(AuTe2)\}(Pb2BiS3)(AuTe2)".

Atom Label	Column 1	Column 2	Column 3	Column 4	Column 5	Column 6	Column 7
Bi1							
Bi2							
Au1							
Te1							
S1							
S2							
Bi3							
S3							
Bi4							
Te2							
Pb2							

HOWEVER, in our case we have selected No. This is what comes out of Fourier.



HOWEVER, in our case we have selected No. This is what comes out of Fourier.

Jana2006 - Fourier

Find Find next Go to Print PgTop Open in editor Close

	x	y	z	charge	rho	rel		x	y	z	charge	rho	rel
1.	0.2500	0.2500	0.0289	2.84	7.05	999	6.	0.5753	0.2500	0.7889	1.65	5.92	649
2.	0.5714	0.2500	0.5713	1.86	4.05	732	7.	0.4049	0.2500	0.0997	1.58	6.13	621
3.	0.5601	0.2500	0.9760	1.85	6.51	728	8.	0.7500	0.2500	0.8991	1.52	8.98	598
4.	0.5990	0.2500	0.0435	1.78	6.04	700	9.	0.2708	0.2500	0.7508	1.43	4.91	562
5.	0.2500	0.2500	0.5124	1.73	3.98	680	10.	0.5923	0.2500	0.9138	1.42	3.98	559

This is what you are looking for. Unaccounted electron density (in $e/\text{\AA}^3$ units). It is positive for electron deficit

...and negative for electron excess (list below)

Program for n-dimensional Fourier synthesis structure : page= 4
19:04:12 14-12-20

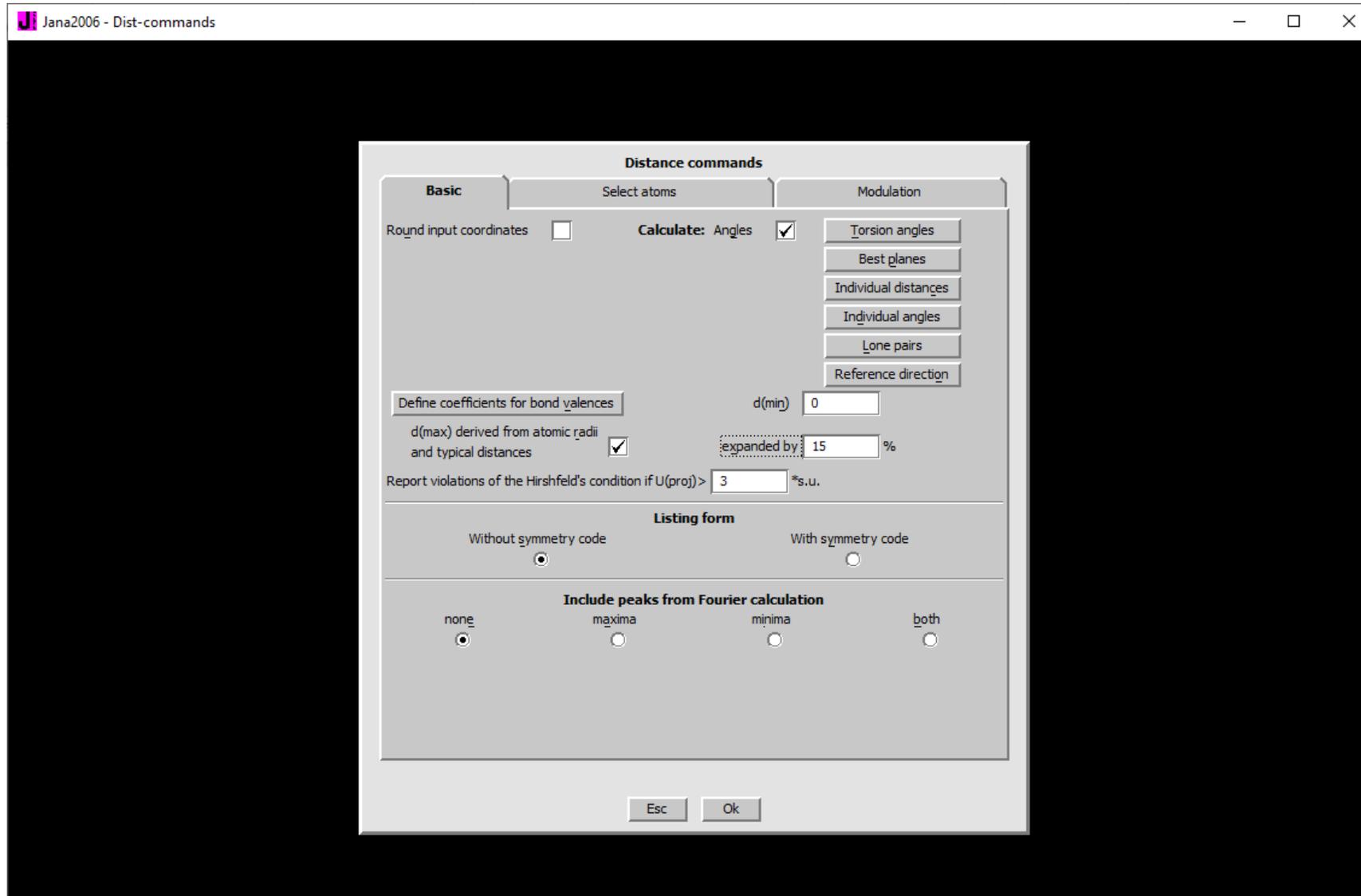
Searching for negative peaks - maximum number of peaks to be found : 10

The list of negative peaks written to the m40 file

Run Dist (right click)

Select all the parameters that you want to be written in your final .cif file.

Click ok and start the program.



This is the Dist output file. Bond angles and bond distances are included.

```
Jana2006 - Dist
Find Find_next Go to Print PgTop Open in editor Close
Distances and angles will be calculated from d(min)= 0.000 to specific maximal distances as listed below expanded by 15%:
Au-Au 2.880 R    Au-Bi 3.260 R    Au-Pb 3.190 R    Au-S 2.341 T    Au-Te 2.810 R
Bi-Bi 3.640 R    Bi-Pb 3.570 R    Bi-S 2.631 T    Bi-Te 3.190 R
Pb-Pb 3.500 R    Pb-S 2.744 T    Pb-Te 3.120 R
S-S 2.080 R     S-Te 2.410 R
Te-Te 2.740 R

T ... typical distances taken either from the file distributed with Jana program or defined by user
R ... distance as derived from atomic radii:

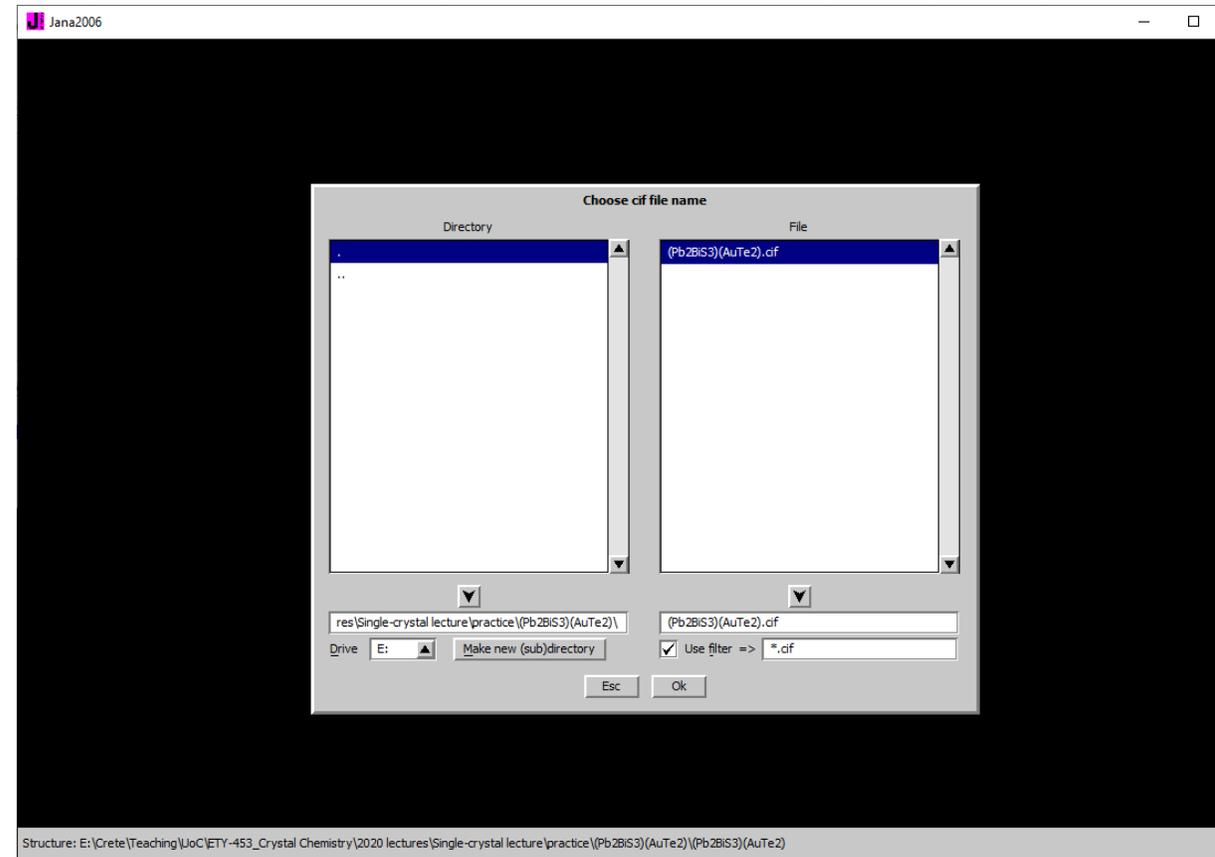
Atom type      Atom radius
  Au           1.440
  Bi           1.820
  Pb           1.750
  S            1.040
  Te           1.370

*****
* atom Au      *
*****
Tel.....2.7150(19)  S1.....2.974(4)  S2.....80.9(4)  S2.....134.9(2)  S2.....84.0(2)
Tel.....98.05(6)   S2.....80.9(4)  S1.....93.99(6)  S2.....134.9(2)  S2.....84.0(2)
Tel.....81.94(6)   S2.....160.5(5) S2.....134.9(2) S2.....134.9(2) S2.....2.906(10)
Tel.....179.17(18) S2.....89.1(2)  S2.....46.2(2)  S2.....167.9(4)  S2.....89.7(3)
Tel.....2.7150(19) S2.....2.794(12) S2.....46.2(2)  S2.....89.0(3)  S2.....167.9(4)
Tel.....179.17(18) S2.....79.6(3)  S2.....46.2(2)  S2.....89.0(3)  S2.....2.906(10)
Tel.....81.94(6)   S2.....79.6(3)  Bi.....4.0997(14) S2.....2.906(10)
Tel.....2.7150(19) S2.....2.947(11) Bi.....**      S2.....89.7(3)
Tel.....98.05(6)   S2.....88.1(3)  S1.....90.00(7)  S2.....2.906(10)
Tel.....2.7150(19) S2.....2.947(11) S2.....45.1(2)  *
*          *      S2.....134.9(2)  *
*****          *
* atom Pb      * * atom Bi      *
*****          *
Bi.....4.081(3)   Pb.....4.081(3)  Bi.....4.0997(14) Au.....2.7150(19)
S1.....134.42(10) Pb.....172.02(10) S1.....90.00(7)  Au.....98.05(9)
S1.....134.42(10) Bi.....90.000(5)  S2.....134.9(2) Au.....2.7150(19)
S2.....87.6(3)   Bi.....90.000(5)  S2.....45.1(2)  *
S2.....45.4(2)  S1.....93.99(6)  S2.....134.9(2)  *
S2.....45.4(2)  S2.....46.2(2)  S2.....45.1(2)  * atom S1      *
S1.....2.974(4) S2.....46.2(2)  S1.....2.69(2)  *****
S1.....87.13(11) S2.....134.9(2) S2.....84.0(2)  Pb.....2.974(4)
S2.....84.0(2)  S2.....84.0(2)  S2.....84.0(2)  Pb.....87.13(12)

Calculating of distances, angles, torsion angles and best planes
structure :
Pb.....90.68(12)
Pb.....164.1(8)
Bi.....97.9(4)
Pb.....2.974(4)
Pb.....164.1(8)
Pb.....90.68(12)
Bi.....97.9(4)
Pb.....2.974(4)
Pb.....87.13(12)

page= 4
19:57:01 14-12-20
```

Finalize the structure by making a .cif file. All your work is written in this single file.



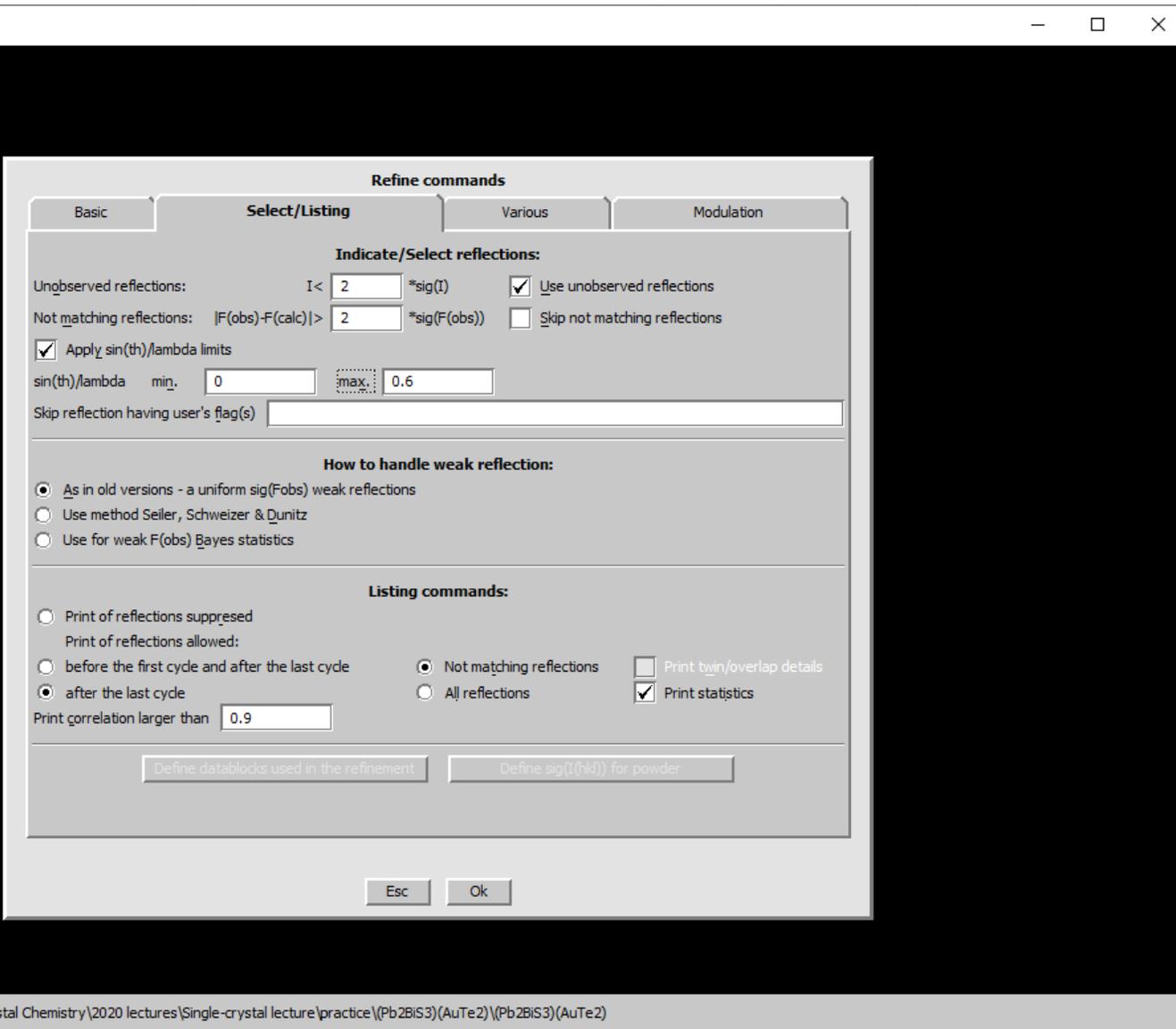
This is how your folder should look like once you are done.

File Explorer window showing the folder `(Pb2BiS3)(AuTe2)`. The folder contains 21 items, including files for data, output, and visualization.

Name	Date modified	Type	Size
<code>(Pb2BiS3)(AuTe2).cif</code>	2/9/2014 1:57 AM	Mercury File	10 KB
<code>(Pb2BiS3)(AuTe2).dis</code>	12/14/2020 7:57 PM	DIS File	20 KB
<code>(Pb2BiS3)(AuTe2).fou</code>	12/14/2020 7:04 PM	FOU File	9 KB
<code>(Pb2BiS3)(AuTe2).hkl</code>	2/9/2014 12:45 AM	HKL File	147 KB
<code>(Pb2BiS3)(AuTe2).inflip</code>	12/14/2020 6:34 PM	INFLIP File	43 KB
<code>(Pb2BiS3)(AuTe2).l51</code>	12/14/2020 8:02 PM	L51 File	1 KB
<code>(Pb2BiS3)(AuTe2).m40</code>	12/14/2020 7:04 PM	M40 File	5 KB
<code>(Pb2BiS3)(AuTe2).m50</code>	12/14/2020 7:57 PM	M50 File	1 KB
<code>(Pb2BiS3)(AuTe2).m70</code>	12/14/2020 7:59 PM	M70 File	6 KB
<code>(Pb2BiS3)(AuTe2).m80</code>	12/14/2020 6:53 PM	M80 File	128 KB
<code>(Pb2BiS3)(AuTe2).m81</code>	12/14/2020 7:04 PM	M81 File	366 KB
<code>(Pb2BiS3)(AuTe2).m83</code>	12/14/2020 6:53 PM	M83 File	111 KB
<code>(Pb2BiS3)(AuTe2).m85</code>	12/14/2020 6:53 PM	M85 File	3 KB
<code>(Pb2BiS3)(AuTe2).m89</code>	12/9/2020 10:17 PM	M89 File	34 KB
<code>(Pb2BiS3)(AuTe2).m90</code>	12/9/2020 10:18 PM	M90 File	39 KB
<code>(Pb2BiS3)(AuTe2).m95</code>	12/9/2020 10:18 PM	M95 File	978 KB
<code>(Pb2BiS3)(AuTe2).ref</code>	12/14/2020 6:53 PM	REF File	39 KB
<code>(Pb2BiS3)(AuTe2).rre</code>	12/9/2020 10:18 PM	RRE File	25 KB
<code>(Pb2BiS3)(AuTe2).s40</code>	12/14/2020 6:53 PM	S40 File	12 KB
<code>(Pb2BiS3)(AuTe2).sum</code>	2/9/2014 12:42 AM	SUM File	239 KB
<code>(Pb2BiS3)(AuTe2).usd</code>	12/14/2020 8:02 PM	USD File	1 KB

All files can be opened with notepad and contain information on what you did so far.

Tweaks and Tricks



The screenshot shows the 'Refine commands' dialog box in the Jana2006 software. The 'Select/Listing' tab is active. The 'Indicate/Select reflections' section includes the following options:

- Unobserved reflections: $I < 2 * \text{sig}(I)$ Use unobserved reflections
- Not matching reflections: $|F(\text{obs}) - F(\text{calc})| > 2 * \text{sig}(F(\text{obs}))$ Skip not matching reflections
- Apply $\sin(\theta)/\lambda$ limits
- $\sin(\theta)/\lambda$ min.: 0 max.: 0.6
- Skip reflection having user's flag(s):

The 'How to handle weak reflection' section has three radio button options:

- As in old versions - a uniform $\text{sig}(F(\text{obs}))$ weak reflections
- Use method Seiler, Schweizer & Dunitz
- Use for weak $F(\text{obs})$ Bayes statistics

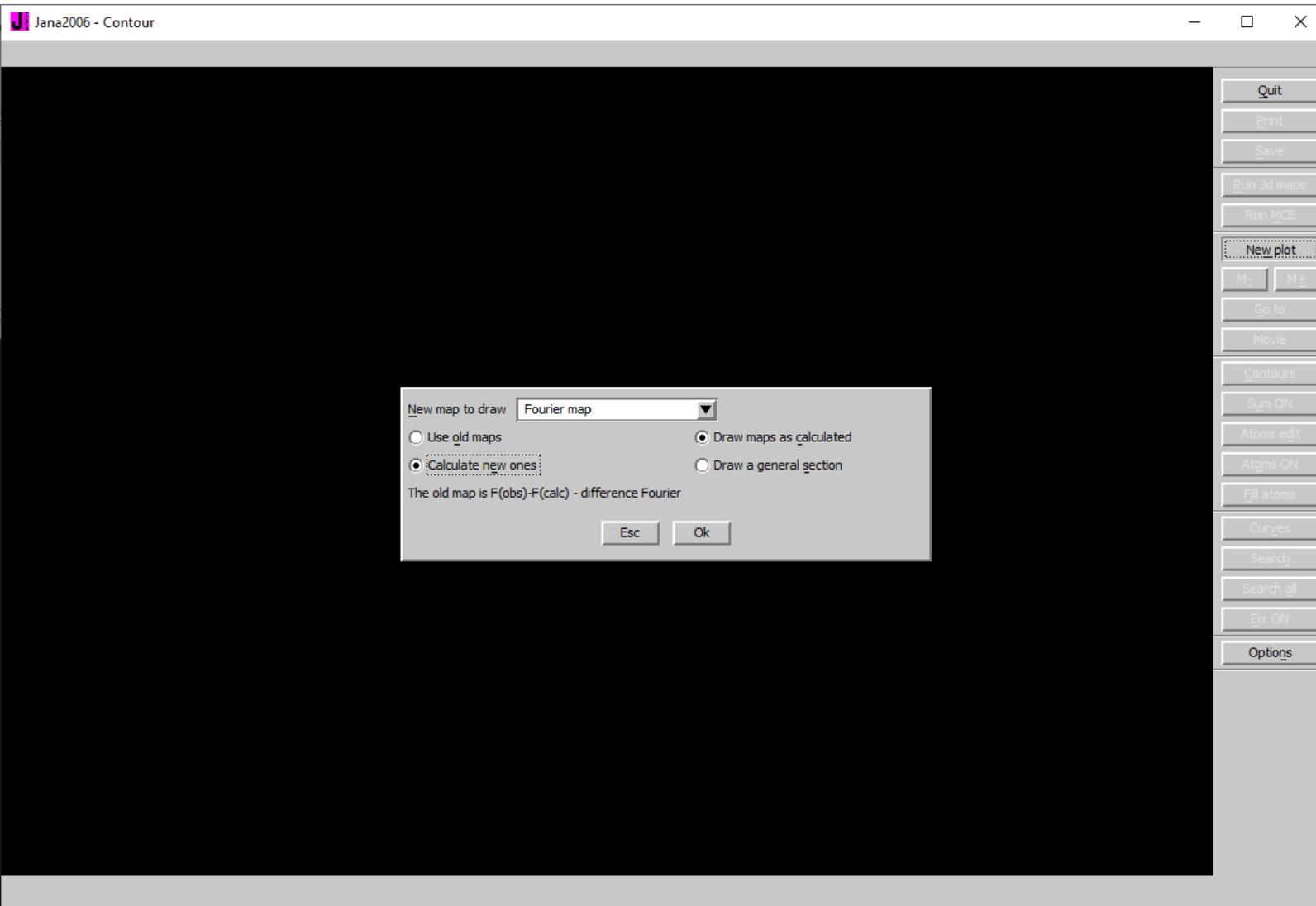
The 'Listing commands' section includes:

- Print of reflections suppressed
- Print of reflections allowed:
 - before the first cycle and after the last cycle
 - after the last cycle
- Print correlation larger than: 0.9
- Not matching reflections
- All reflections
- Print twin/overlap details
- Print statistics

Buttons at the bottom include 'Define datablocks used in the refinement', 'Define $\text{sig}(I(\text{hk}))$ for powder', 'Esc', and 'Ok'.

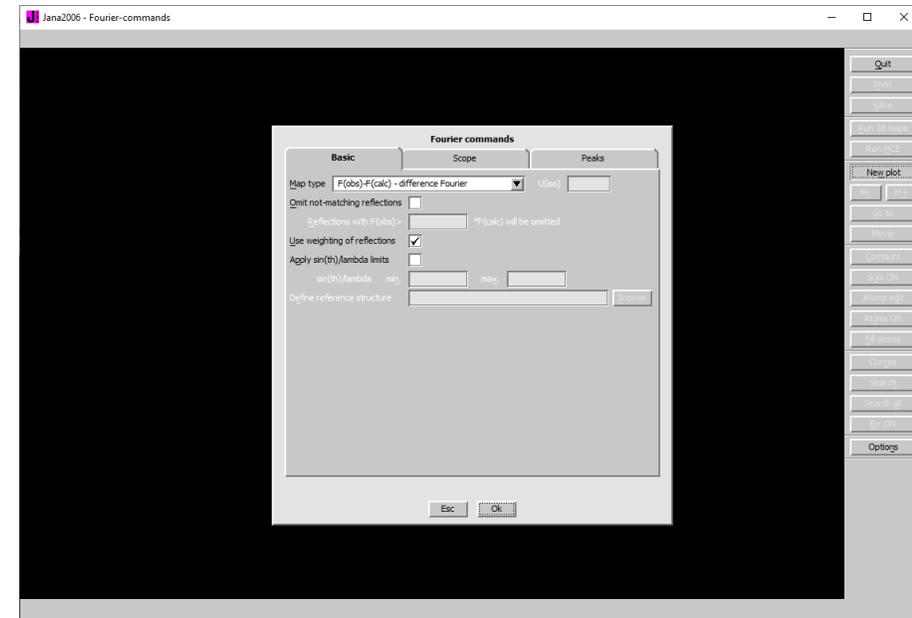
If your data are bad, or there is no intensity at high 2θ angles, then you can eliminate these data. You do this by making the $\sin(\theta)/\lambda$ term to correspond to $2\theta \sim 50^\circ$ ($\theta = 25^\circ$) for the wavelength you are using. This will immediately omit all data above this angle.

The Fourier map. Contour plot command

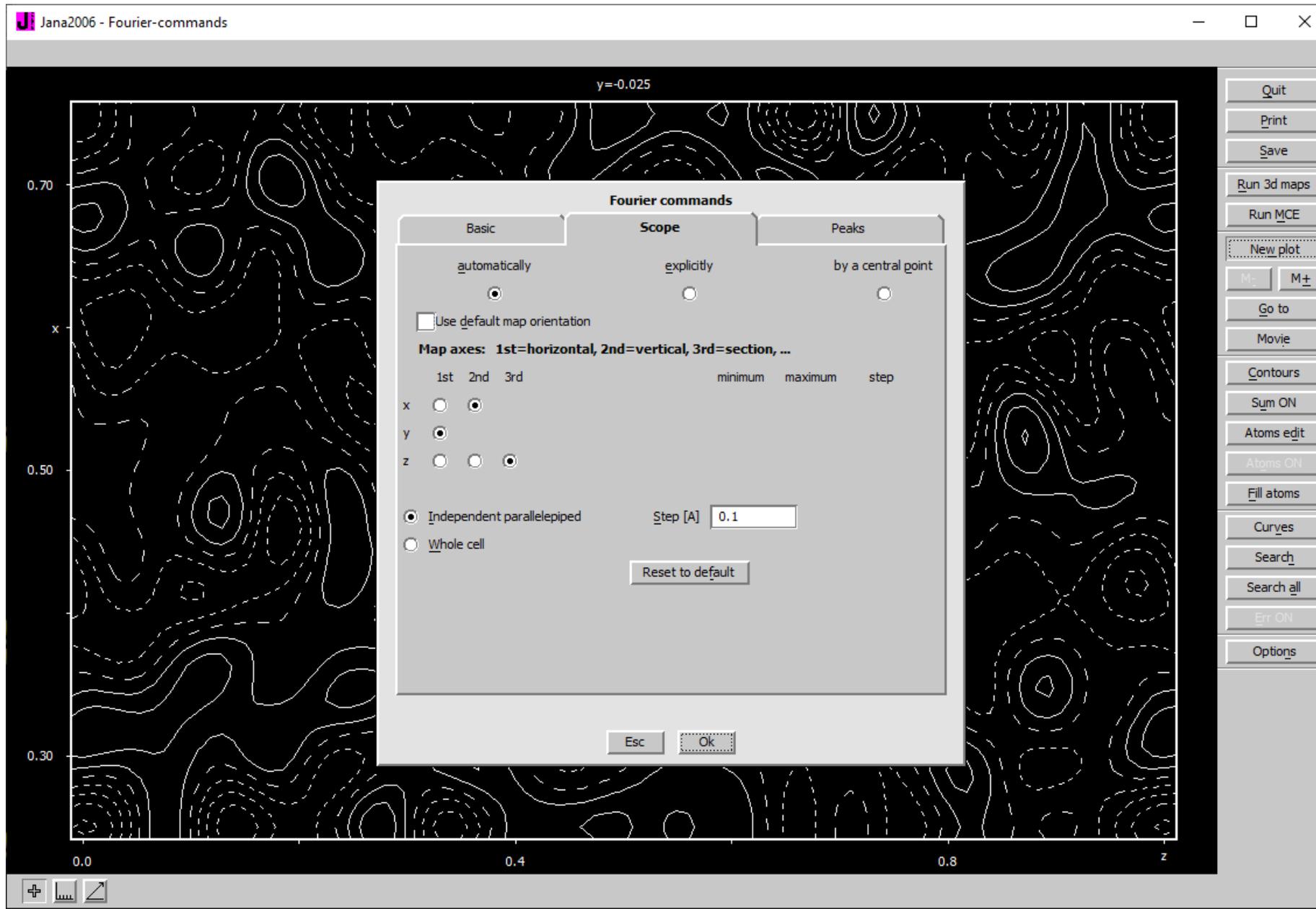


Click the contour icon and select calculate new ones in the next menu.

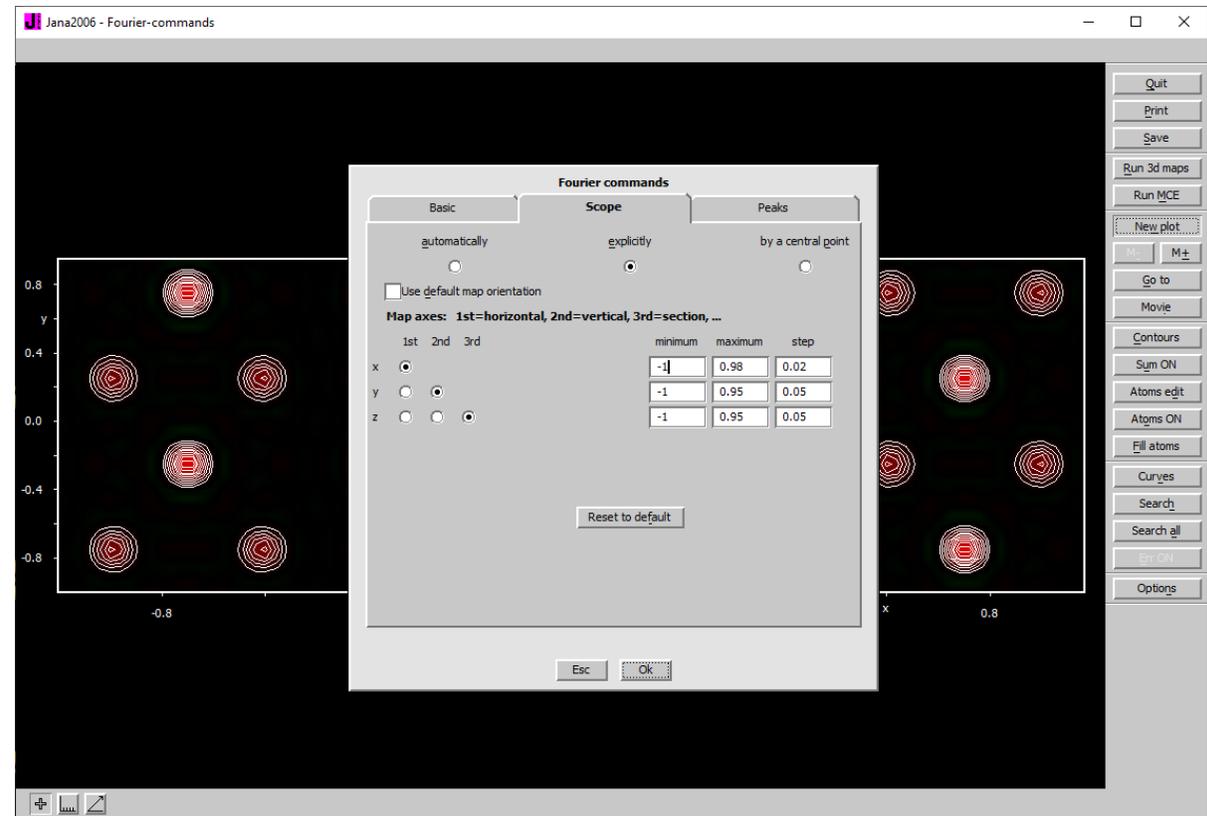
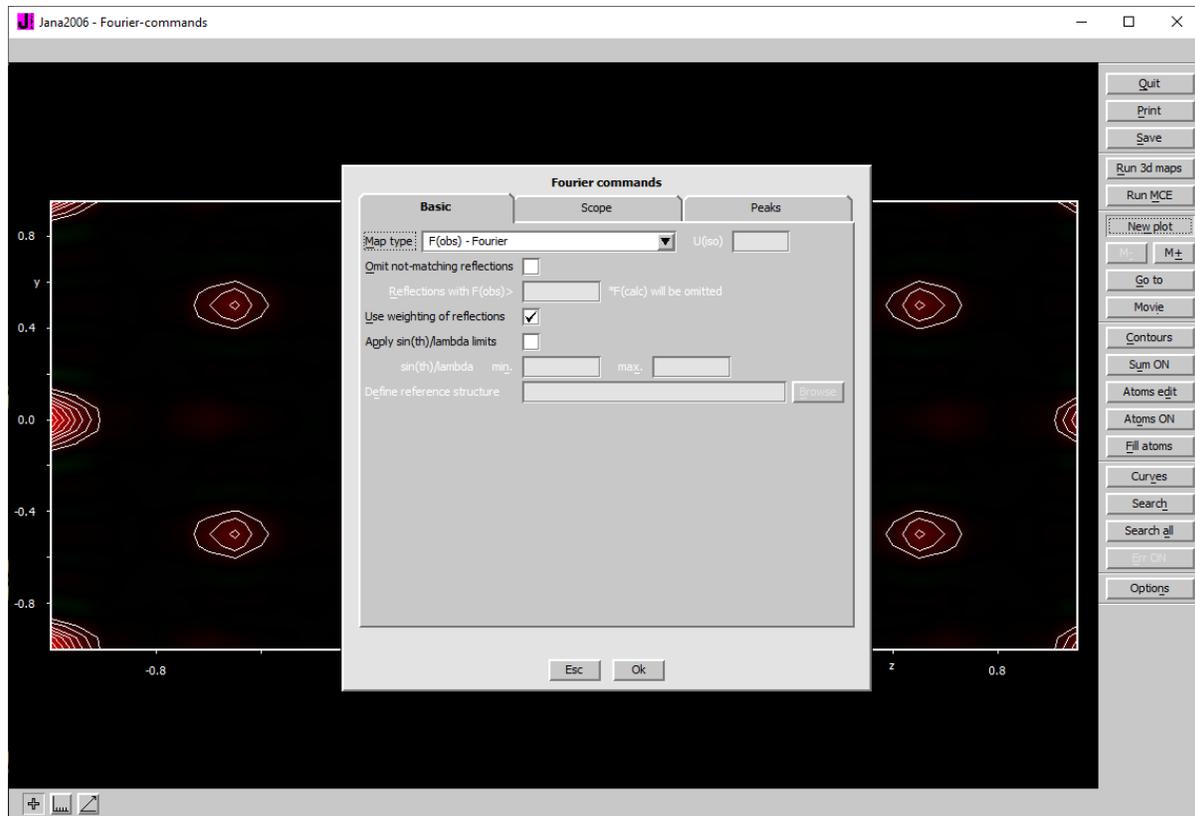
Accept the changes in the menu after that.



You can open the menu again using the new plot command. Choose the scope to orient the unit cell in the direction of your choice. You want to see the unit cell along the a-axis in this example



Run a difference Fourier map and select a 2x2x2 supercell viewed along the a-axis



This is the electron density map of the $[\text{AuTe}_2]^-$ layer

Fourier map (electron density on the atoms)

Difference Fourier map (electron density on not on the atoms)

