

Example 13.5: B₁₈H₂₂

Last update 12.3.2021

| Data | Topic | Level |
|----------------------|--|--------------|
| Electron diffraction | Complete procedure from data reduction to structure refinement including microscope distortions using calibrated values. | Intermediate |

Introduction

In this example, one of the possible ways for obtaining the precise lattice parameters is shown. Because the B₁₈H₂₂ is a molecular crystal it is necessary to not only to count with the distortions of the diffraction data by the microscope optics but also with the beam damage induced changes of the lattice. Molecular crystals are significantly beam sensitive, which results in lattice parameters changes induced and intensified by the accumulated electron dose. The optical distortions induced by the microscope optics were calibrated using Lutetium aluminium garnet. These calibrations can then be used for correction the optical distortions in the data obtained on the molecular crystal materials. The stability of the distortions is relatively good so it can be done only once in a while. The problem of lattice parameters evolution stemming from the accumulated electron dose is a much less defined problem and can be mitigated by using symmetry restrictions following the crystal system. The unit cell refinement using these symmetry restrictions is implemented in PETS 2.0.

The example will also show you difference in kinematical and dynamical refinement. You will see that it is significantly easier to locate hydrogen atoms and the temperature factors are more reflecting the real situation in the material.

Input data

Data

Electron diffraction data were measured on a transmission electron microscope FEI Tecnai G² 20 (LaB₆) with a precession device (DigiStar). Accelerating voltage was 200 kV. The sample temperature was 100 K.

Input files

folder "dp-100": contains 64 measured diffraction patterns (16-bit TIF files)

B18H22.pts: input file for PETS

Calibrated_optical_distortions.txt: values of pincushion, spiral and elliptical distortions of the microscope and setting of the distortion refinement keys.

Additional information

Supplementary files for easier reproducibility:

folder "PETS_output_CIF": expected output of the data reduction

folder "JANA": intermediate states of the refinements. Thus, different steps can be evaluated without having to carry out the computationally expensive (i.e. time consuming) dynamical refinement steps

Software used:

Jana2020 (jana.fzu.cz)

PETS (pets.fzu.cz)

VESTA (jp-minerals.org/vesta/en/download.html)

References

For further information about the data processing in PETS2.0, see:

- L. Palatinus et al. Specifics of the data processing of precession electron diffraction tomography data and their implementation in the program PETS2.0. Acta Cryst. B 75: 512-522 (2019).
- PETS2.0 manual pets.fzu.cz/download/

For further information about the dynamical refinement and associated parameters:

- L. Palatinus et al. Structure refinement using precession electron diffraction tomography and dynamical diffraction: theory and implementation. Acta Cryst. A71: 235–244 (2015).
- L. Palatinus et al. Structure refinement using precession electron diffraction tomography and dynamical diffraction: tests on experimental data. Acta Crystallogr B71: 740–751 (2015)

Instructions

PART 1 - Data reduction using PETS 2.0

1. Data import

File B18H22.pts is the input for PETS. The file was generated automatically during data collection.

Open file B18H22.pts in any plain-text editor (Notepad, Notepad++, Vim, ... NOT MS Word).

Lines beginning with a hashtag are ignored by PETS. The relevant lines are the following:

```
lambda 0.0251
Aperpixel 0.001850
dstarmax 1.2
phi 1.00
omega 0.5
noiseparameters 3.5 38
reflectionsize 20
I/sigma 5
bin 2

imagelist
dp-100\001.tif -20.00 0.00
dp-100\002.tif -19.00 0.00
...
endimagelist
```

Lambda: relativistic wavelength in Å

Aperpixel: size of one pixel in Å⁻¹

phi: precession angle

omega: orientation of the tilt axis (alpha)

bin: binning factor by which the image resolution is reduced for image processing

reflectionsize: the reflection diameter.

Other parameters, especially concerning the detector specific noise parameters, are explained in more detail in the manual available at <http://pets.fzu.cz/>

Open file Calibrated_optical_distortions.txt and copy the whole text into the B18H22.pts anywhere in the text with the exception of the imagelist. The resulting B18H22.pts file may look like the following:

```

lambda 0.0251
Aperpixel 0.001850
dstarmax 1.2
phi 1.00
omega 0.5
noiseparameters 3.5 38
reflectionsize 20
I/sigma 5
bin 2

#If yes, center of distortions is assumed to be a constant offset from the
center of the diffraction pattern. Otherwise constant coordinates of the
distortion centers are assumed.
distcenterasoffset no

#Distortion parameters.
distortions
  1040.0000    990.0000    0.0000    0.0000
    0.000000    0.000000    1.100000    0.000000
    0.000000    0.000000    0.000000    0.000000
    0.500000    0.000000    0.000000    55.000000
    0.000000    0.000000    2.500000    0.000000
    0.000000    0.000000    0.000000    0.000000
    0.500000    0.000000    0.000000    10.000000
    0.000000    0.000000    0.000000    0.000000    0.000000    0.000000
enddistortions

#Refinement keys for the distortion parameters.
distortionskeys
  0 0 0 0
  0 0 0 0
  0 0 0 0
  0 0 0 0
  0 0 0 0
  0 0 0 0
  0 0 0 0
  0 0 0 0 0 0
Enddistortionskeys

imagelist
dp-100\001.tif -20.00  0.00
dp-100\002.tif -19.00  0.00
...
Endimagelist

```

The first two numbers in the first row of “distortions” are the coordinates of the optical axis of the microscope on the detector. The pincushion distortion represents the third number in the second row and the spiral distortion is the third number in the fifth row. Amplitude and phase of the elliptical distortion are determined in the rows four and seven by the first and fourth number, respectively. All values are in pixels. All distortion keys are zeroes, which means that the distortions will not be refined during the unit cell refinement.

[Keep your text editor opened, we will update some parameters later.](#)

2. **Run PETS 2.0**

[Start PETS by double clicking the executable](#)

[Go to File->Open in the upper menu](#)

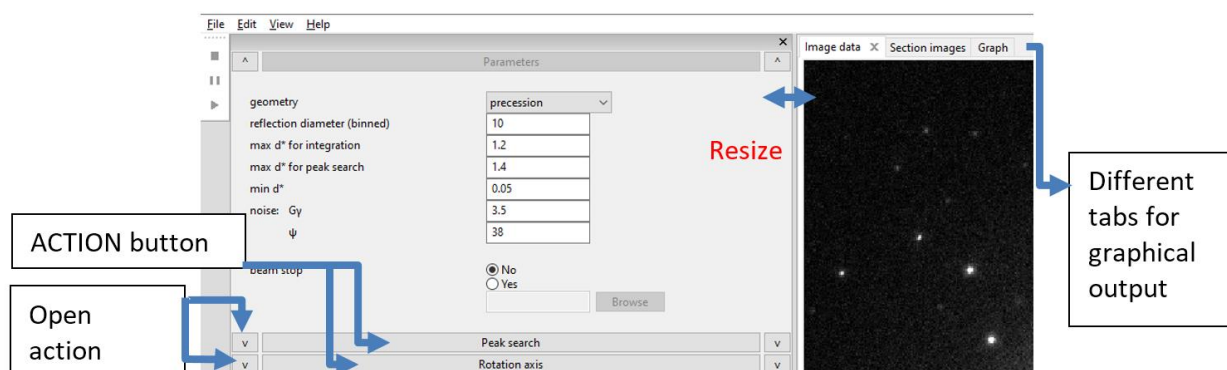
[Open the file B18H22.pts in the folder](#)

In the Main panel (Image data tab) you can view individual diffraction images (frames) using the arrows at the bottom of the panel. The contrast of the images can be adjusted by changing

the value of “Display cut off” at the bottom of the starting window showing the “Image data” panel. At e.g. 15000 only the primary beam and the strongest reflections are visible, at 10 we can clearly see the detector background noise. Values between 50 and 500 are useful to look at the relevant features, i.e. the reflections.

The arrows to the left of “Frame” (at the bottom controls below the diffraction pattern) and the value next to it control the image that is shown. The options “raw” and “processed” show you the raw diffraction pattern as recorded and the processed frame (some background correction etc.), respectively. “Simulated” frames will be available after frame orientation procedure.

3. Parameters



Data reduction steps are run by clicking the respective action button. The action options can be modified in the panel, which opens after clicking the small arrow next to the action button. The “Parameters” action button (first in the list of action buttons) is gray because there is no associated action. Note that you can adapt the size of the different window components (e.g. make the plot window more narrow or wider) and decouple the action bar or the console from the main window.

Click on the arrow (v) next to “Parameters” to check the following options:

In our input file .pts, we defined “reflections size 20” and “bin 2”, hence the binned reflection diameter is 10 pixel. The default parameters used for min d* of 0.05 Å⁻¹ is OK. Here it means that any reflection/peak that is less than 0.05*Aperpixel pixel away from the center is ignored. “Precession” geometry was set in the input file because the data set was measured using precession.

4. Peak search

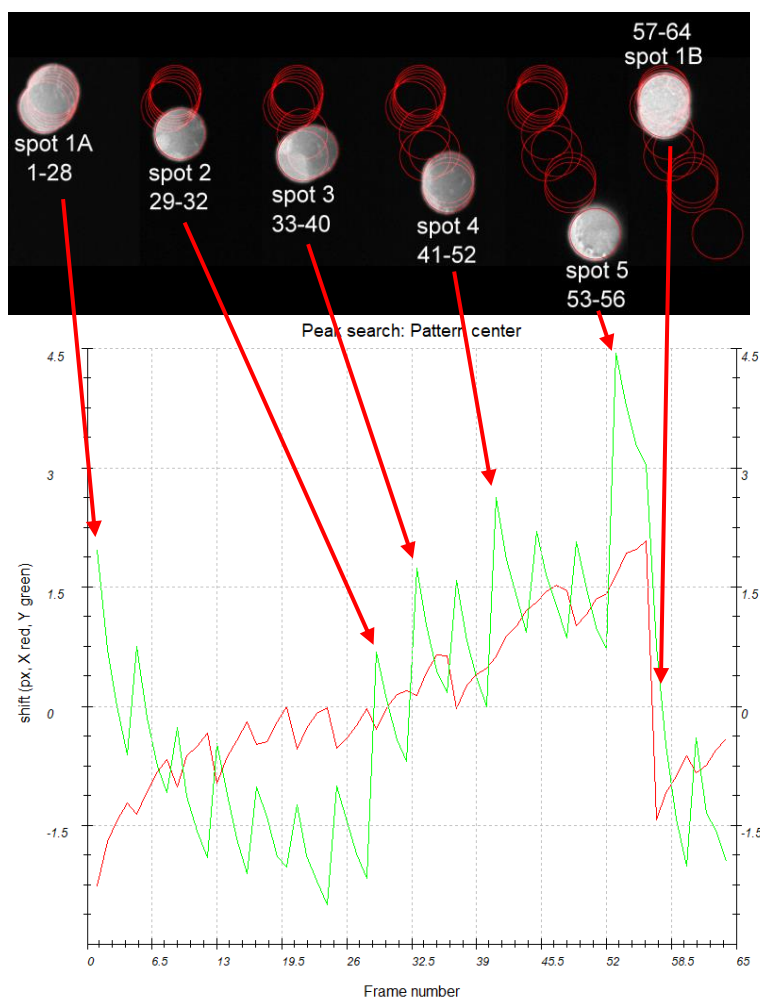
Click on the arrow (v) next to “Peak search”, keep all setting and run “Peak search” by clicking on the action button.

The console output reports that 4178 peaks were written to the raw peak list (saved as B18H22.rpl). During peak search the determined pixel coordinates of the center of each diffraction pattern is determined and saved as the file B18H22.cenloc.

Click on “Graph” (next to “Image data” and “Section images”).

Red line: deviation of the x coordinate of the center from its initial value as a function of the frame number. Green line: deviation of the y coordinate. This should be checked because in the peak search options we used “Always accept center”, which might fail. You can see a sawtooth shape of the lines, which is a result of a crystal shift within the convergent beam caused by the goniometer tilt, which is corrected every fourth frame. On top of that, after especially frames 28, 52 and 56 there are visible jumps of the center. These jumps are real and they are a result of

shifting the beam to the different (fresh) crystals spots as it is marked at the top of the figure. Last eight frames are taken from the same spot as the first 28 frames (marked as spot 1B). At the current stage, we assume that all pattern centers were correctly determined.



5. Rotation axis (ω)

The next step after peak search is the refinement of the angle ω between the horizontal axis (x axis of the diffraction image) and the projection of the rotation axis. This angle depends on the experimental setup, and needs to be refined for each data set.

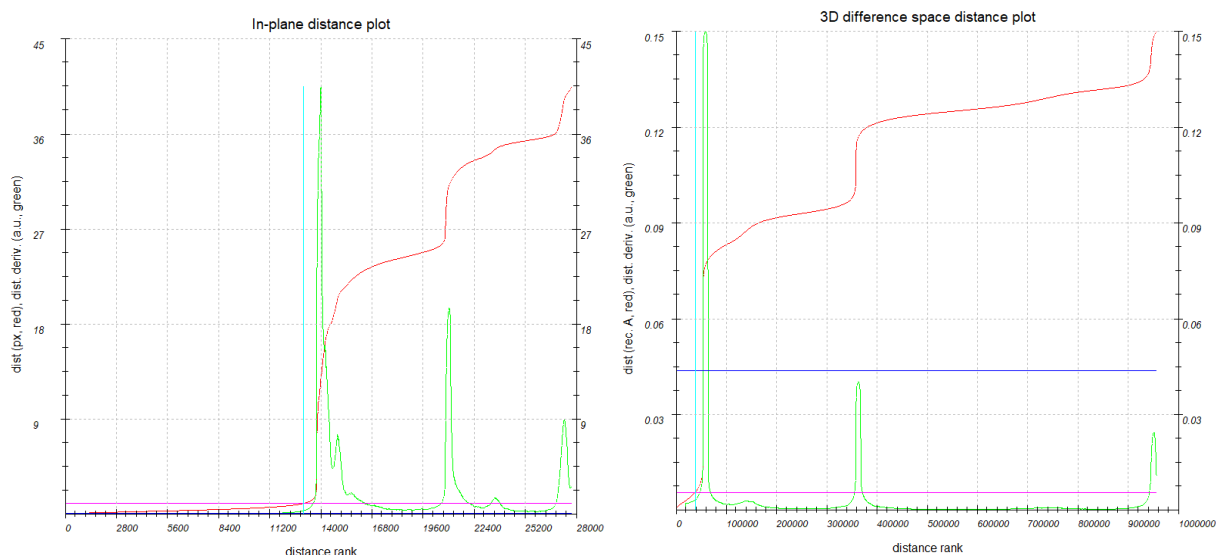
[Click the action button "Rotation axis" to start the procedure.](#)

In the plot window "Cylindric projection", the difference space of the extracted peak positions is represented as a cylindric projection. In the ideal case, it consists of well-defined spots on sinusoidal curves and no elongated features. Here, the starting angle (defined in the input file) is 0.5° and it refines to 0.1562° .

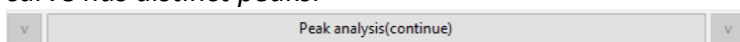
6. Peak analysis

[Click the action button "Peak analysis".](#)

PETS starts the analysis of the peak positions obtained in the peak hunting procedure. In the first step, distance distribution between peaks in the image plane is analyzed. A sorted plot of inter-peak distances (left screenshot) is displayed in the Processing tab (red curve) with its derivative (green):



For good quality data sets, the red curve contains distinct steps, and, consequently, the green curve has distinct peaks.



[Click "Peak analysis \(continue\)"](#)

Difference peaks are saved to B18H22.diff

In the next step, an autoconvolution of the diffraction pattern (difference space) is analyzed, and the groups of peaks in the autoconvolution (clusters) are replaced by the cluster centers. Again, a distance distribution is displayed (right screenshot), and clear-cut steps on the curve indicate a well-defined lattice.

[Click "Peak analysis \(continue\)"](#)

Cluster map file is written to B18H22.clust

Now the first step is finished and PETS prepared the files needed for the determination of the orientation matrix and the unit cell parameters.

7. Find unit cell and orientation matrix

[Click "Find unit cell and orientation matrix"](#)

The main menu on the left is replaced by a new set of buttons to determine, refine and modify the orientation matrix, which in turn defines the unit cell parameters. On the right side, a new tab "3D panel" opens, where it displays a projection of the peaks determined from the peak search and peak analysis. Note that the view can be enlarged by scrolling the wheel of your mouse, and rotated by holding the left mouse button. By default, the "Data used for indexing" is "xyz", i.e. the points in B18H22.xyz are used for the plot and for the orientation matrix determination.

Click “Find possible cells automatically”

The screenshot shows a software interface for indexing. At the top, a dropdown menu is set to 'xyz'. Below it, a box labeled 'Cell 1' is empty. To its right, the following statistics are displayed: indexed/all: 1381 / 1499, indexed: 92.13 %, volume: 1390.57, and Bravais class: tP. Below these are buttons for 'New cell', 'Delete cell', 'Undo', and 'Redo'. A table of cell parameters is shown:

| | a | b | c | α | β | γ |
|-------|---------|---------|---------|----------|---------|----------|
| cell: | 10.7474 | 10.7790 | 12.0037 | 89.929 | 89.824 | 89.877 |
| s.u.: | - | - | - | - | - | - |

Below the table is an 'Orientation matrix' section with a 3x3 grid of values:

| | | |
|-----------|-----------|-----------|
| -0.052996 | 0.076360 | 0.001051 |
| 0.046448 | 0.033054 | -0.065925 |
| -0.060759 | -0.041030 | -0.050922 |

The bottom panel, titled 'Find possible cells automatically', has two radio buttons: 'from difference space' (selected) and 'from triplets'. It contains several input fields: 'Maximal d* difference for indexing (rec.Å):' with value 0.018, 'Angular tolerance for rec. direction (deg):' with value 2, 'Maximal volume (Å^3):' with value 5000, 'Symmetry search: tolerance for lengths (%):' with value 3, and 'tolerance for angles (deg):' with value 0.6. At the bottom is a table of results:

| | a | b | c | α | β | γ | V | Bravais | ind/all |
|---|--------|--------|--------|----------|---------|----------|----------|---------|-----------|
| 1 | 10.747 | 10.779 | 12.004 | 89.929 | 89.824 | 89.877 | 1390.574 | tP | 2436/2436 |

Open the details by clicking on the arrow next to the action button. You see the parameters used in the procedure as well as the possible unit cells found by PETS (only one is found with the default settings). The first one in the list (see screenshot) is automatically used. The cell angles are very close to 90 degrees, a and b are almost equal. Therefore, PETS determined that the Bravais class is tetragonal primitive (tP). Based on the initial orientation matrix, reciprocal space sections can be generated to investigate visually the symmetry in reciprocal space. As a part of the automatic indexing procedure the lattice parameters and orientation matrix are refined. We are happy with the result and thus no additional cell refinement is needed at this moment.

Leave the indexing panel by clicking “Finish”

8. Create reciprocal-space sections

In the parameters (arrow next to action button “Reciprocal-space section”), you can change the size of pixels in the reconstruction images and the thickness of the slab that is projected into the section in \AA^{-1} . Too small numbers may result in “holey” reconstructions. We will proceed with the default values.

Click “Reciprocal-space sections”; OK (use the standard set)

PETS reconstructs sections through reciprocal space at planes $hk0$, $hk1$, $hk2$, $h0l$, $h1l$, $h2l$, $0kl$, $1kl$, $2kl$ from the diffraction patterns. These sections are used as an aid to determine the space group. The generation of the sections takes about 2 minutes.

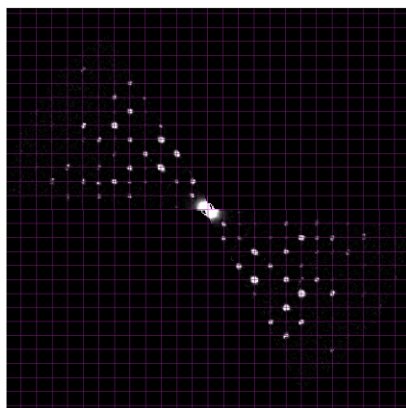
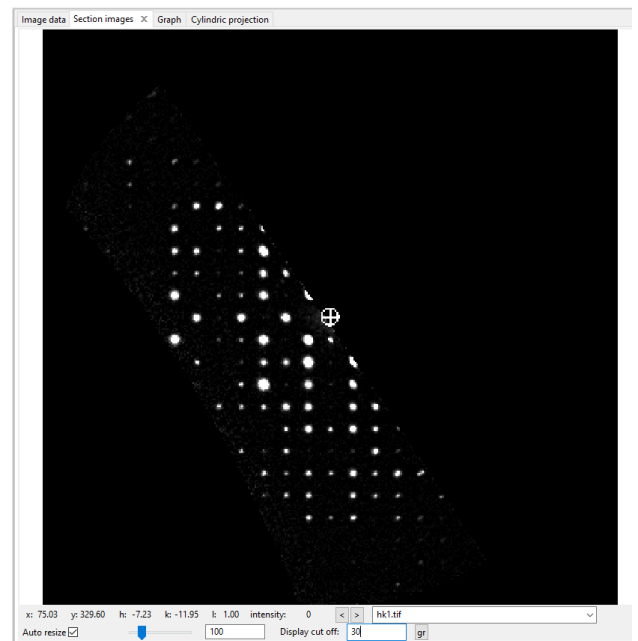
Change to the tab “Section images”

Choose “hk1.tif”

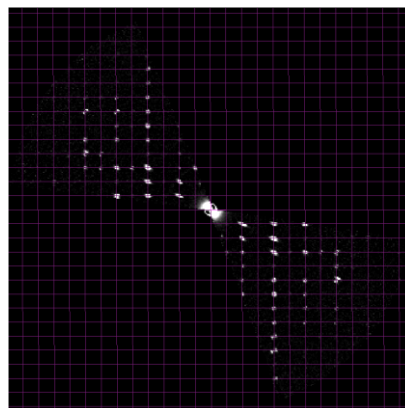
Set the Display cut off to 30

The $hk1$ section, though very incomplete, clearly violates tetragonal symmetry. The point group of this section seems to be $2mm$. Note that the white circle with a cross in the middle of the image indicates the center of the section, in this case the expected position of the 001 reflection. In many cases, the reliable determination of the Laue class of the tetragonal or cubic crystal systems requires checking sections that are not included in the standard set, e.g. sections perpendicular to $\langle 110 \rangle$.

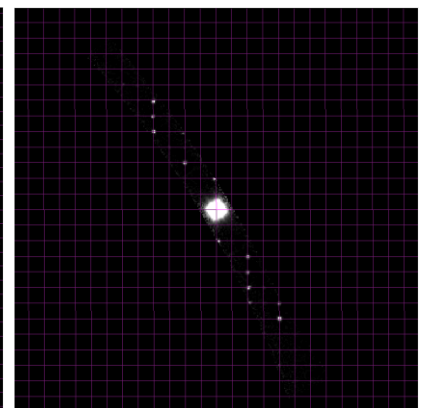
The sections $hk0$, $h0l$ and $0kl$ may give indications on the presence of glide planes and/or 2_1 screw axes. For your convenience you may turn on the grid, which will show the positions of the expected diffraction maxima by clicking on "gr" button next to the Display cut off window. The $0kl$ section suggests the presence of an n -glide plane as reflections with $k+l = 2n+1$ are mostly absent or very weak. The $h0l$ section suggests the presence of a a -glide plane (h odd are either absent or very weak) perpendicular to the b -axis. Only a small fraction of the $hk0$ section was measured. However, this is sufficient to recognize that reflections with h odd are absent, suggesting a a -glide plane perpendicular to the c -axis. From this quick analysis we derive that the space group $Pnaa$ most likely describes the symmetry of the crystal structure. The standard setting of this space group (#57) is $Pccn$ and we should therefore adapt the orientation matrix.



$0kl$



$h0l$



$hk0$

9. Change orientation matrix

Click "Find unit cell and orientation matrix"

Click on "..." next to the Bravais Class

Change the Crystal system to "o" (orthorhombic); OK

The Bravais class is now "oP"

Open the parameters of "Modify cell" (click on the arrow)

Data used for indexing: xyz

Cell 1

indexed/all: 1381 / 1499
 indexed: 92.13 %
 volume: 1390.57
 Bravais class: oP

New cell Delete cell
 Undo Redo

| | a | b | c | α | β | γ |
|-------|---------|---------|---------|----------|---------|----------|
| cell: | 10.7474 | 10.7790 | 12.0037 | 89.929 | 89.824 | 89.877 |
| s.u.: | - | - | - | - | - | - |

Orientation matrix

| | | |
|-----------|-----------|-----------|
| -0.052996 | 0.076360 | 0.001051 |
| 0.046448 | 0.033054 | -0.065925 |
| -0.060759 | -0.041030 | -0.050922 |

Modify cell

Reduce cell Check centering Go to supercell

Transformation matrix:
☐ direct space ☒ reciprocal space

$a^* = 0 \cdot a^* + 1 \cdot b^* + 0 \cdot c^*$
 $b^* = 0 \cdot a^* + 0 \cdot b^* + 1 \cdot c^*$
 $c^* = 1 \cdot a^* + 0 \cdot b^* + 0 \cdot c^*$

☐ transform centering for changed cell volume

Transform by matrix Reset to unit matrix

Enter the transformation matrix (right screenshot) 0 1 0; 0 0 1; 1 0 0;

Click on "Transform by matrix"

The new lattice parameters are 10.7790 12.0037 10.7474 89.824 89.877 89.929.

Important! Now the unit cell will be refined and for the best result it is necessary to switch the data from .xyz to .cor, which contain the searched peaks aligned using the diffraction centers. Using .cor file, the refinement will be done against the data, which were not mathematically processed in the step "Peak analysis".

Change "Data using for indexing" to .cor.

Open the parameters of "Refine cell" (click on the arrow).

Select "refine cell and distortions", check "refine cell". Leave all other settings default.

Refine cell

☐ refine UB + cell
☐ refine cell from d
☒ refine cell and distortions

Maximal d* difference for indexing(Invc(A)): 0.018

☒ symmetry triclinic
☐ fixed cell parameters 10 10 10 90 90 90

☐ fixed up to a scale factor

☒ refine cell ☐ refine distortions

☒ Distortions

Center: x 1040 y 990

| | rad | lin | quad | cub | phase |
|---|-----|-----|------|-----|-------|
| 0 | 0 | 0 | 0 | 1.1 | |
| 1 | 0 | 0 | 0 | 0 | 0 |
| 2 | 0.5 | 0 | 0 | 0 | 55 |

sg terms 0 0 0

| | tan | lin | quad | cub | phase |
|---|-----|-----|------|-----|-------|
| 0 | 0 | 0 | 0 | 2.5 | |
| 1 | 0 | 0 | 0 | 0 | 0 |
| 2 | 0.5 | 0 | 0 | 0 | 10 |

sg terms 0 0 0

☐ tie elliptical distortion

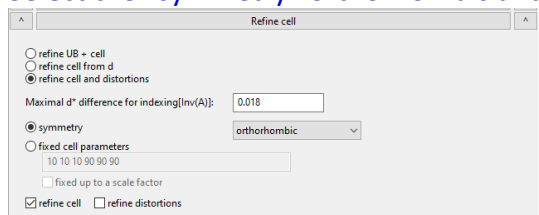
The distortion parameters, which were copied at the beginning to the B18H22.pts can be seen by clicking on the "(v) Distortions" button.

Click on "Refine cell"

| | a | b | c | α | β | γ |
|-------|---------|---------|---------|----------|---------|----------|
| cell: | 10.7826 | 12.0118 | 10.7441 | 89.831 | 89.885 | 89.869 |
| s.u.: | 0.0009 | 0.0028 | 0.0016 | 0.023 | 0.010 | 0.016 |

The resulting unit cell angles are all by more than 0.1° off the expected 90°. This deviation is caused solely by a deterioration of the crystal, which results in a gradual change of the lattice parameters with the accumulated electron dose as we will see in the part dedicated to frame-by-frame distortions refinement. To mitigate this problem, we will use the symmetry restrictions of the unit cell during the refinement.

Select the “symmetry” orthorhombic and click on “Refine cell”.



The resulting lattice parameters are 10.7826 12.0118 10.7441 90.000 90.000 90.000

Click on “Finish”

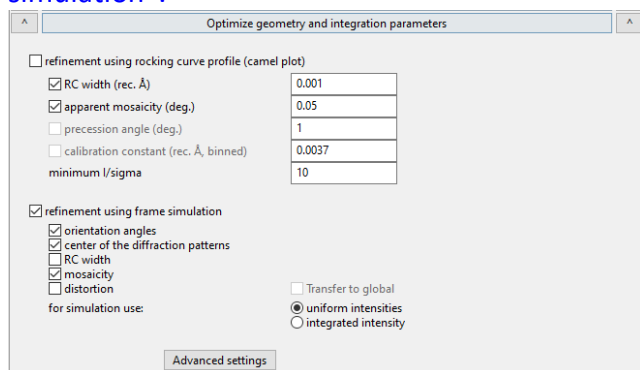
The orientation matrix is written to B18H22.celllist. Note that the reciprocal space sections were not changed and thus do not correspond to the new orientation matrix. You should generate new sections.

Click “Reciprocal-space sections”; OK (use the standard set)

10. Frame orientation optimization

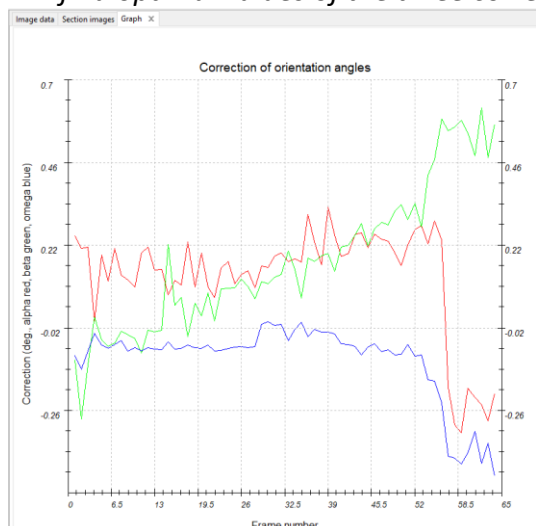
Open the options of “Optimize geometry and integration parameters”.

Uncheck the “refinement using rocking curve profile” and check “refinement using frame simulation”.



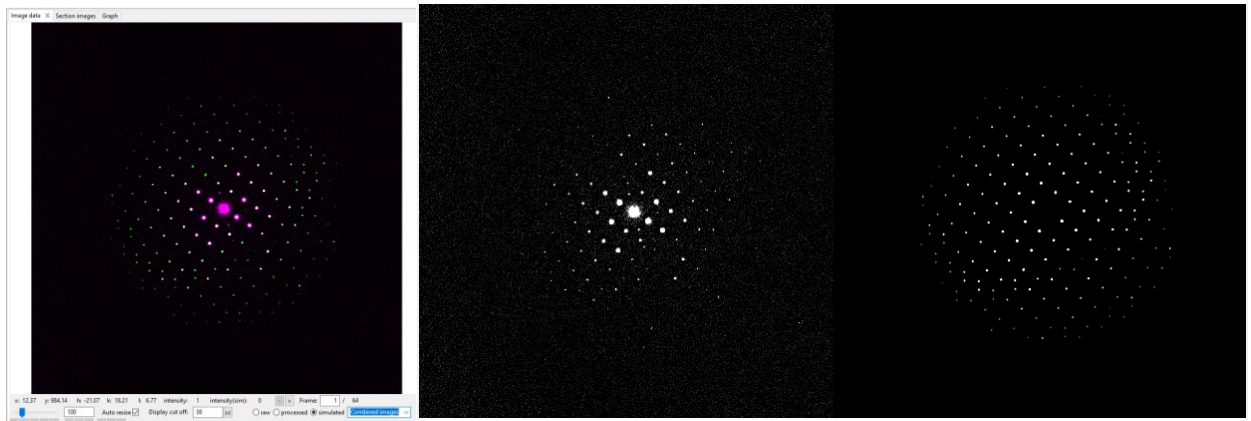
Click on “Optimize geometry and integration parameters”.

The procedure will use default uniformly decreasing intensities with increasing resolution. PETS will find optimal values of the three correction angles alpha, beta and omega.



You can check the process in “Image data” card by switching the radiobutton to “simulated”.

Then you can choose if you want to see the data and the simulation in parallel or if you want to see the combination of the two images (purple – data, green – simulation). Or if you want to see just the simulation.



Check the “distortion”.

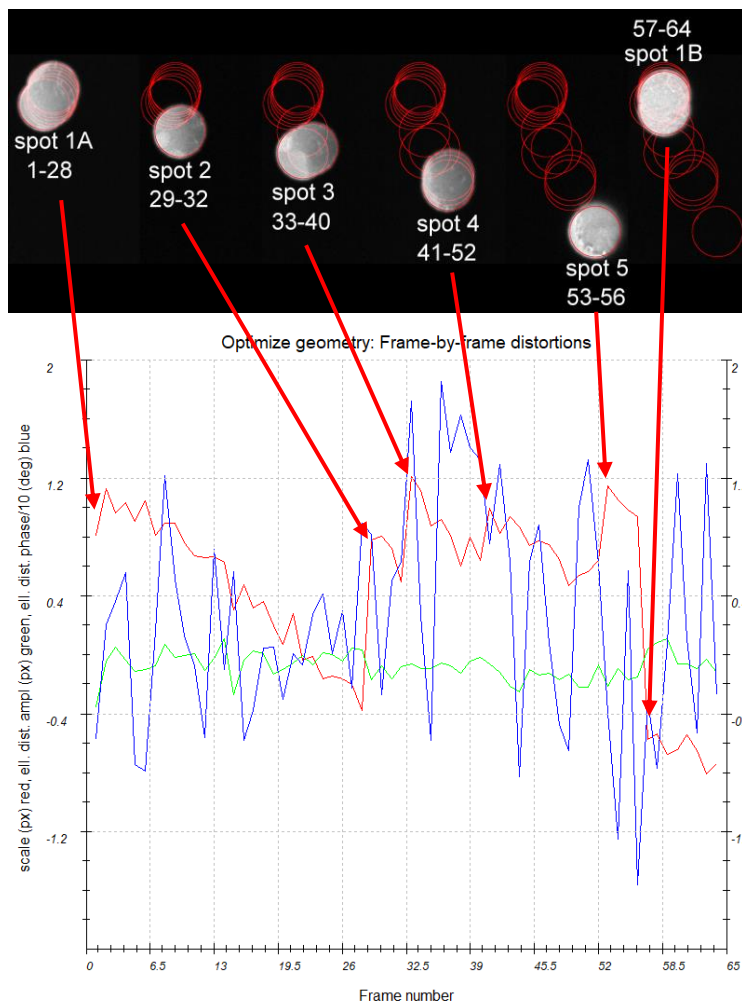
This will allow PETS to refine individual frame-by-frame magnification and amplitude and phase of the elliptical distortion.

Click on “Optimize geometry and integration parameters”.

The correction angles are now much smaller.

Choose “Frame-by-frame” distortions in the Graph tab.

There you will see the correction of the apparent magnification (red curve), amplitude and phase of the elliptical distortion (green and blue curve, respectively). The elliptical distortion is constant, which is normal for the microscope setting, which was used during this data collection. In terms of the lattice parameters stability this indicates that the lattice parameter ratios and angles remain constant. This should be the case also for the apparent magnification (red curve), if the unit cell volume would be constant. But there is an obvious trend observable in the graph which is strongly correlated with the accumulated doses in the particular measured spots. The apparent magnification of the reciprocal space is decreasing, which means that the actual volume of the unit cell increases as a result of the accumulated beam damage.



11. Integrate intensities

Click on “Process frames for integration” and uncheck “shift integration box to maximum intensity”.

All the significant distortions in the data were corrected so the integration box shift is no more necessary during the integration. In the “Image data” tab, diffraction images with circles (integration boxes) drawn at positions predicted from the orientation matrix are displayed. Green circle: observed reflection with $I > 3\sigma(I)$. Brown circle: reflection with $I < 3\sigma(I)$. Diamond: reflection expected either on upcoming or on previous frame(s). In the parameters the “max d^* for integration” was set to 1.2 \AA^{-1} . The main reason for this is that a reduced resolution used for the refinement will reduce the time needed for dynamical refinement cycles later. The curious user is encouraged to set the “max d^* for integration” limit to 1.4 in the options of “Parameters”.

In the “Graph” tab, choose the “Rocking curve”

From these plots the RC width and the apparent mosaicity can be derived.

Open the options of “Optimize geometry and integration parameters”

Note the default options: RC (rocking curve) width = -0.001 \AA^{-1} , apparent mosaicity = 0.05° .

Check the “refinement using rocking curve profile” and uncheck “refinement using frame simulation”.

Optimize geometry and integration parameters

☒ refinement using rocking curve profile (camel plot)

☒ RC width (rec. Å) 0.001

☒ apparent mosaicity (deg.) 0.05

☐ precession angle (deg.) 1

☐ calibration constant (rec. Å, binned) 0.0037

minimum I/sigma 10

☐ refinement using frame simulation

☒ orientation angles

☒ center of the diffraction patterns

☐ RC width

☒ mosaicity

☒ distortion

for simulation use:

☐ Transfer to global

☒ uniform intensities

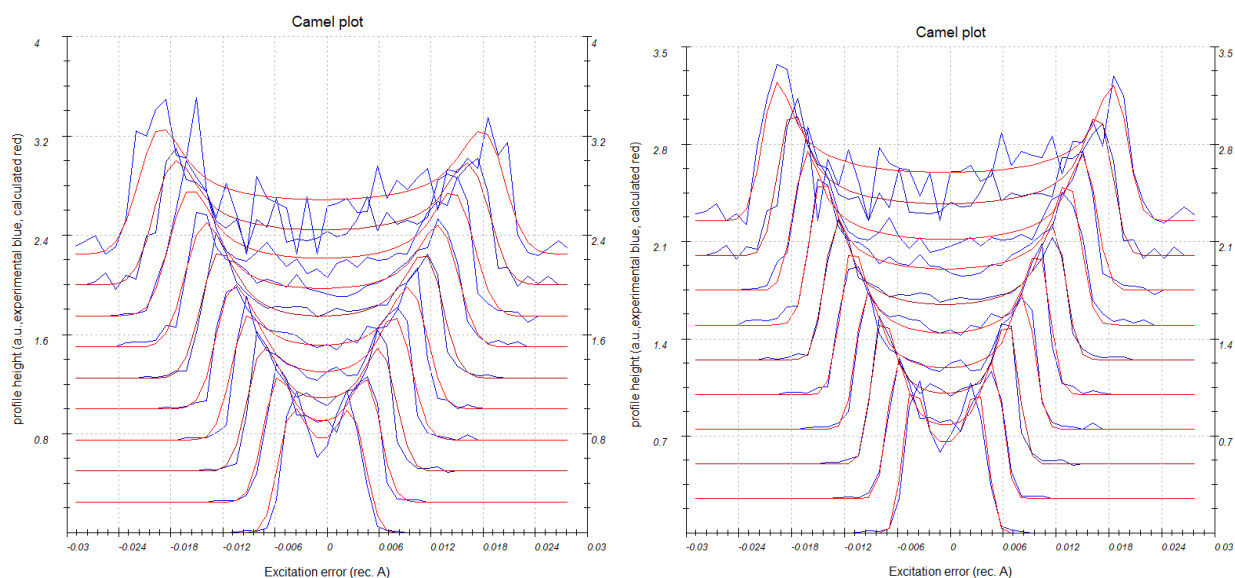
☐ integrated intensity

Advanced settings

Click on “Optimize geometry and integration parameters”.

Note how the parameters change once the parameters are refined. The predicted camel plot (red curve) fits now better to the observed profiles (blue curves).

Left screenshot: before optimized geometry. Right screenshot: with optimized geometry. The determined RC width is 0.0006 Å^{-1} and the apparent mosaicity 0.032° .



12. Intensity integration

Open the parameters for “Finalize integration”.

Check that both “kinematical” and “dynamical” integrations are checked.

Activate “Frame scaling”.

Set the Laue class to “mmm” (screenshot).

Finalize integration

☒ kinematical

intensity estimation:

☐ fit profile

☒ integrate profile

☐ maximum intensity

☐ Bragg position

integration parameters: 1 min. number of reflection measurements

1 reflection width multiplier

☒ frame scaling

Laue class for scaling: mmm

Interframe correlation range: 0

Interframe correlation weight: 0

☒ dynamical

virtual frames: number of frames: 1

step between frames: 1

Click on “Finalize integration”

In the console, the Rint is shown for different Laue classes. If we finalize integration with two different “intensity estimation” settings (“fit profile” and “integrate profile”), we end up with the following statistics for the Laue class mmm:

| Intensity estimation | Rint(obs) | Rint(all) | Nobs/Nall |
|----------------------|-----------|-----------|-----------|
| Integrate profile | 20.39 | 21.39 | 830/1251 |
| Fit profile | 12.50 | 13.91 | 729/1242 |

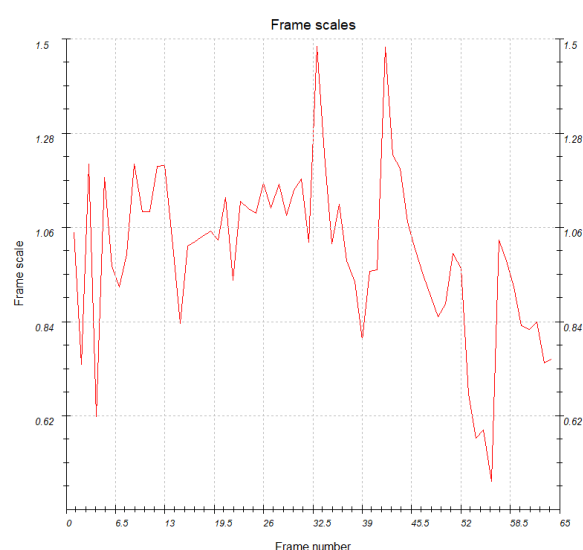
Default value for intensity estimation is “integrate profile”, which is the more robust technique and works well even for poorer data. However, for good data the “fit profile” option gives better results.

Select the radio button “fit profile” for the “intensity estimation”

Click on “Finalize integration”

Two files were saved in the main folder: B18H22.cif_pets and B18H22_dyn.cif_pets.

Look at the “Frame scales” of the Graph tab



13. Unit cell refinement after the removal of the effect of the beam damage

Click on “Find unit cell and orientation matrix”.

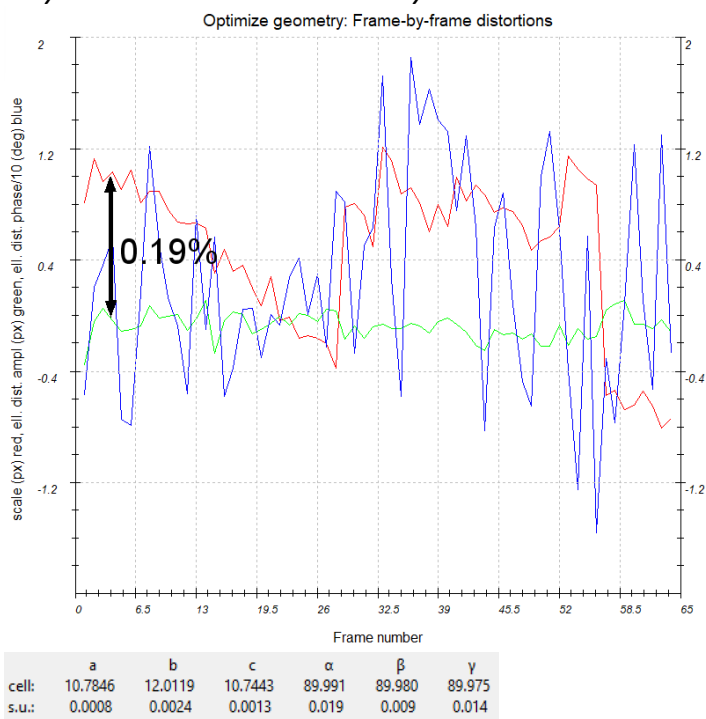
Open “Refine cell” options.

Set “Maximal d* difference for indexing to 0.01 Å⁻¹ and “symmetry” to triclinic.

Click on “Refine cell”.

The resulting lattice parameters are 10.785(1) 12.012(2) 10.744(1) 89.99(2) 89.98(1) 89.98(1). The lattice constants obtained from XRPD data measured at the same temperature were 10.7566(3) 11.9796(4) 10.7142(3) 90 90 90. The a, b, and c lattice parameters from electron diffraction are 0.26, 0.27, and 0.28% longer than those from the XRPD. The source of this discrepancy is twofold. The first is the unit cell expansion, which is dependent on the accumulated dose. From the image below, you can see that the first four frames taken on the fresh spots have on average the apparent magnification by 1.0 pixels larger than the magnification represented by the refined unit cell. Therefore, the unit cell parameters on these frames are 0.19% shorter than the parameters of the unit cell obtained in step 9. The resting ~0.08% difference comes from the inevitable fluctuation of the magnification, which according to our experience is about 0.2% for the excitation of the diffraction lens close to the eucentric focus. The fluctuation is not reflected in the e.s.d.s. In conclusion, using calibrated microscope

distortions and beam damage effects suppression it is possible to arrive to the lattice parameters very similar to those obtained by the XRPD.



Click on “Finish”.

You can close PETS now.

Answer “Yes” to the question about saving the project

The current state of the project is saved in the file B18H22.pts2. Opening this file with PETS next time allows you to continue from the current state of processing. If you open B18H22.pts, you must start from scratch again, because B18H22.pts is not overwritten by PETS and still stores the initial settings.

PART 2: Structure solution and kinematical refinement in Jana2020

1. Create new structure

Create a new folder with the name “Jana_kinematical”

Important! The data-processing procedure is almost never perfectly reproducible. Small differences in the indexing and cell refinement procedure may result in small differences of integrated intensities. If you want to be sure that you can reproduce the following part of the tutorial, do not use the file B18H22.cif_pets that you just created, but use the file B18H22.cif_pets from the folder PETS_output_CIF. Using your own cif_pets file is also possible, but your results may slightly differ from the results described in this tutorial.

Copy the file B18H22.cif_pets to the folder “Jana_kinematical”

Start Jana2020

In the Main menu bar, use “Structure → New” and open new structure “B18H22” in directory Jana_kinematical

2. Import Wizard

[On the screen: Specify type of the file to be imported]

Select “Single crystal: Known diffractometer formats”; NEXT

[On the screen: Data reduction file from]

Select "Pets electron diffractometer" (Shortcut ALT + P)

File name has automatically changed to B18H22.cif_pets.

NEXT

Set temperature to 100; NEXT

Note the wavelength of 200 kV electrons, which is 0.0251 Å.

Leave all settings unchanged (no twinning, no modulation); NEXT

The program reads 3690 reflections from the file

OK

For absorption correction select "None or done before importing"; NEXT

FINISH; OK to accept made changes, i.e. accept the new data set

3. Symmetry Wizard

NEXT to close the information window and start the symmetry wizard.

Symmetry Wizard can be started separately by expanding "Reflection file" in the Command tree and selecting "Make space group test". The default settings are prepared for x-ray diffraction.

For electron diffraction we have to increase the tolerances.

Set "Maximal $\text{ave}(I/\sigma(I))$ for systematic extinctions induced by other symmetry" to 25 (default is 10)

Thus, less space groups will be excluded even if there are many intense reflections in the data set that are forbidden by the reflection conditions of the respective space group.

NEXT;OK

Select Orthorhombic Laue symmetry (mmm)

NEXT

The statistics clearly exclude a centered cell. The choice of a primitive cell is also in agreement with our visual inspection of the reciprocal space sections.

NEXT

The reciprocal space sections suggest space group Pccn. The intensity statistics indicate that 41 observed reflections with an average $I/\sigma(I)$ of 11.487 contradict the reflection conditions of Pccn. For electron diffraction experiments, this may be acceptable and a careful analysis of the reciprocal space sections often results in a correct determination of the space group. In our case, we have good indications for the n glide plane perpendicular to c^ and the c glide plane perpendicular to a^* . Pccn is the most probable space group complying with these observations.*

Choose Pccn; NEXT

Accept the space group in the standard setting; FINISH

Symmetry is saved in file B18H22.m50.

4. Creating refinement reflection file

In this step, the program creates the file B18H22.m90 containing the data set merged by symmetry and with discarded forbidden reflections. This file will be used for refinement.


Proceed with the wizard, leaving all settings default.

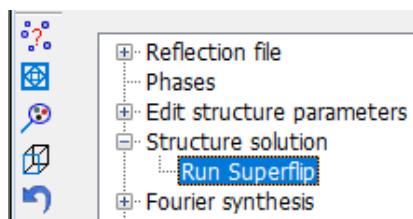
The summary after averaging indicates an internal R-factor of 12.34 for 699 observed reflections.

OK; FINISH

5. Structure Solution Wizard

OK to close the information window and start the solution wizard.

Structure solution wizard can be executed separately using the quick button,  or from the Command tree through “Structure solution->Run Superflip”.



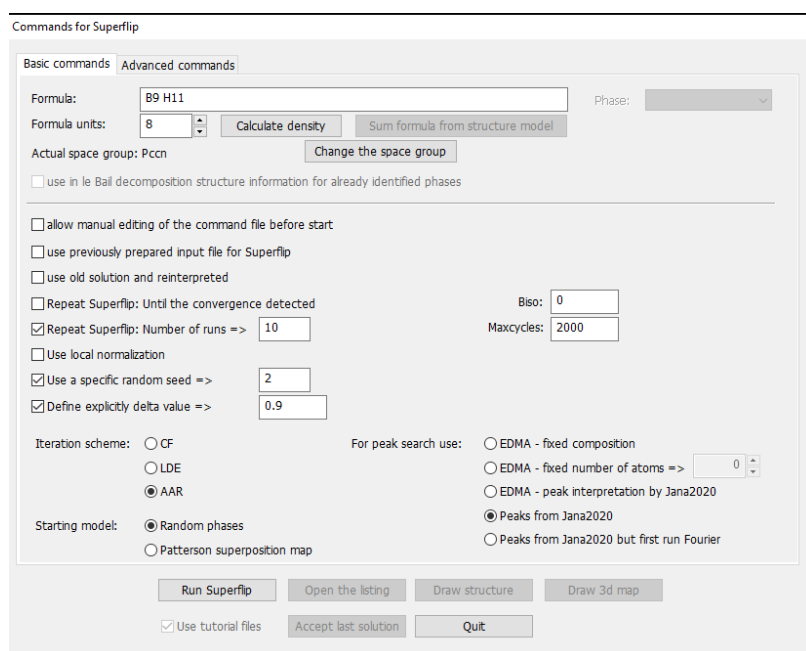
In “Formula” textbox type: B9 H11

In “Formula units” type 8

Check “Use a specific random seed” and enter the value 2.

Switch iteration scheme to AAR.

“Run Superflip”.



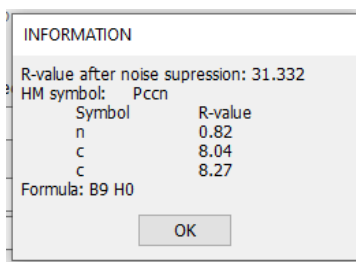
The specific random seed is used just to guarantee reproducibility of the results. This also requires that the imported data set (B18H22.cif_pets) is identical to the one used for this tutorial.

Window of Superflip appears with iteration running. It finishes after 10 runs.

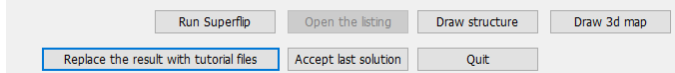
Check “Use tutorial files”

This option ensures the reproducibility of the structure solution step. Jana2020 will replace the job. Files by job.tut_ files, which were generated in the same way as it is described in this manual. However, due small random effects, which occur during the structure solution, small differences were frequently found in between the solutions. Therefore, we have decided to provide the solution files to ensure the reproducibility. You can of course use your own solutions, but the agreement between your results and the results in this manual may slightly differ.

Information about the match between the found density symmetry and the suggested space group is shown after the end of the search.



OK; for reproducibility reasons click on “Replace the result with tutorial files”; OK. You can also go back to your original result using the same button.



Press “Open the listing”

Superflip performed ten solution attempts, and picked the best one. Its characteristics are displayed near the bottom of the listing:

At the very bottom the most reliable solution is described after the line “Properties of the saved density”:

| Run | Rvalue | Peaks | Symm. | Der. SG |
|-----|--------|-------|-------|---------|
| 1 | 30.54 | 3.00 | 8.67 | Pccn |

The space group proposed by Superflip is the same as our initial guess. By going through the complete log file we can see that for all solutions out of the 10 attempts the space group was Pccn, further supporting our initial analysis of the space group symmetry. We can now close the log file. Note that it is saved B18H22.sflog, so you can also open it with a text editor.

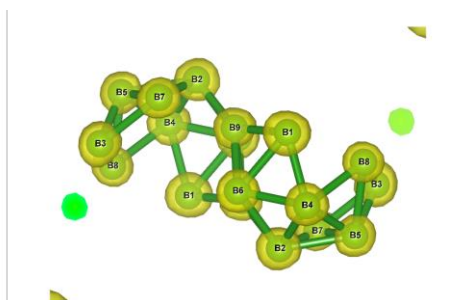
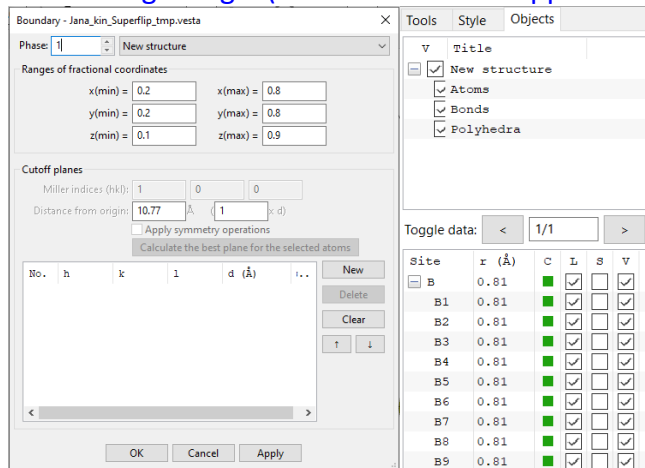
Close the listing

Click on “Draw 3d map”

VESTA opens with a density shown as isosurfaces (in the default settings the shown isosurface level corresponds to the 2σ level based on the statistics of the 3D electrostatic potential map). The map is shown together with an interpretation of it as a model of boron atoms (green spheres). Note that if you used a different B18H22.cif_pets file, most likely your solution will look different. The shape of the blobs will actually not differ much, but an origin shift (most likely $1/2a$, $1/2b$, $1/2c$ or a combination of these) will make a direct comparison more challenging. The solution puts the molecule in the middle of the unit cell. You can limit the 3D map by setting the boundaries on the card “style” to the limits shown in the figure.

Tools (from the menu bar) -> Boundary (CTRL+Shift+B); change the range of x, y, z as in the screenshot; APPLY; OK

Set viewing along b (click on “b” in the upper left corner).

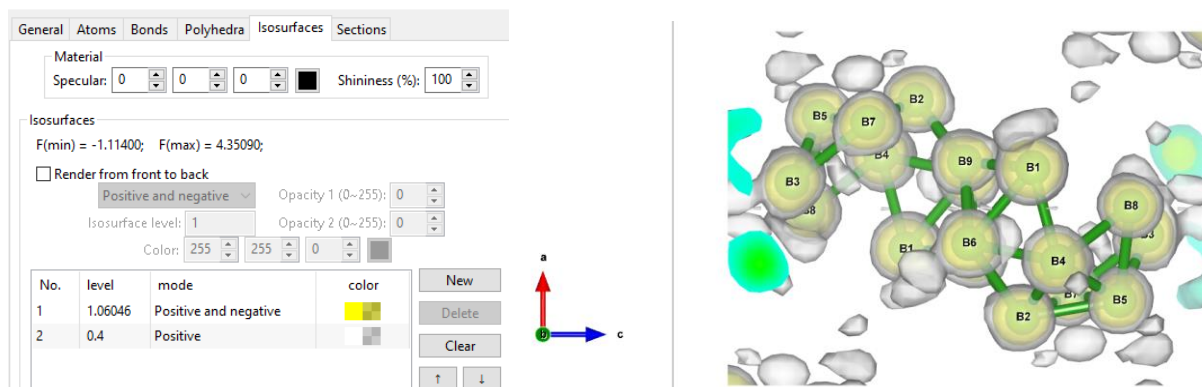


6. Hydrogen atoms in the structure solution

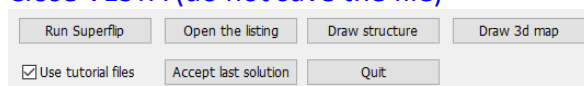
The last view in VESTA shows no signs of any of the 11 hydrogen atoms. However, changing the isosurface level to 0.4 (approximately 0.75σ) reveals new local maxima in the density map.

In VESTA:

Tools->Properties->Isosurfaces (CTRL+Shift+P); click on "New"; change the isosurface level to 0.5; mode "Positive"; change the color to white; OK



Most of the new local maxima visible at the lower isosurface level are close to the identified boron positions. Actually, 10 of them correspond to the hydrogen positions (11 total) we are looking for. Close VESTA (do not save the file)



In Jana: keep checked the "Use tutorialfiles" and click on "Accept last solution".

The program creates file B18H22.m40 with 9 B atoms.

The state of the Jana files after clicking on "Accept last solution" can be found in the folder "JANA/0-solution/" You can work with the files in this folder if you prefer. In that folder, the B18H22.sfllog is the log file of the structure solution and the M40 file contains the resulting structural model (9 B atoms, list of 72 local maxima). Opening this job with Jana you can open the electrostatic potential map with Run Contour->Use old maps->draw maps as calculated.

7. Kinematical refinement and identification of the hydrogen atoms

[On the screen: basic window of Jana]

Double click on "Refinement" -> "Run refinement"

The refinement converges after 7 cycles to R(obs) 24.30. At the end of the refinement Jana notifies you that there are serious warnings, which are related to (physically meaningless) negative displacement parameter of the B2 atom. We now want to look at the difference Fourier map to identify the hydrogen atoms.

The screenshot shows the 'R-factors overview' window in Jana, displaying the following data:

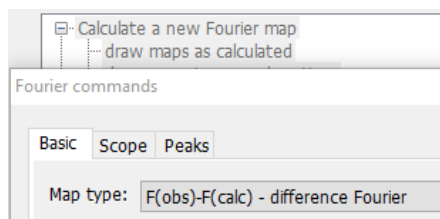
R factors : [1127=703+424/37], Damping factor: 1.0000
GOF(obs)= 14.14 GOF(all)= 11.11
R(obs)= 24.30 wR2(obs)= 51.74 R(all)= 28.12 wR2(all)= 51.96
Last wR2(all): 59.13 56.74 52.01 51.95 51.96 51.96 51.96 51.96
Maximum change/s.u. : -0.0284 for y[B5]

Below this is a 'Question' dialog box with the text: 'Serious warnings in the listing! Regular end of REFIN program'. It asks 'Open the listing?' with 'Yes' and 'No' buttons. At the bottom, it says 'It remains 5 sec - press this button for pause'.

Double click on "Run Contour"

“Calculate a new Fourier map” -> “draw maps as calculated”

In the card “Basic”, select “Map type:” “F(obs)-F(calc) – difference Fourier”

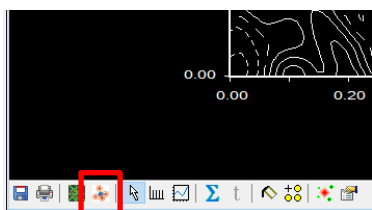


In the page “Scope”, select “Whole cell”; OK

A contour map is shown, which covers the coordinate range $0 \leq x \leq 1$, $0 \leq y \leq 1$, $z = 0$

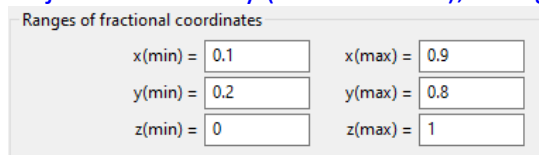
However, the identification of relevant peaks in the Difference Fourier map is easier with a program supporting a 3D view.

In the menu bar, use “Run->Vesta”



Alternatively, VESTA can be started from the lower toolbar

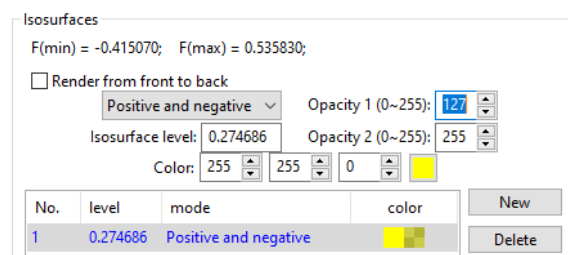
Objects->Boundary (CTRL+Shift+B); change the range of x, y, z as in the screenshot; APPLY; OK



Objects->Properties->Isosurfaces (CTRL+Shift+P), Isosurfaces

Click on the first entry in the list (1, 0.220, Positive and negative)

Enter in the input field of “Isosurface level”: 0.220×1.25 (This will set the isosurface level to 2.5σ)



All the 11 hydrogen atoms are clearly visible.

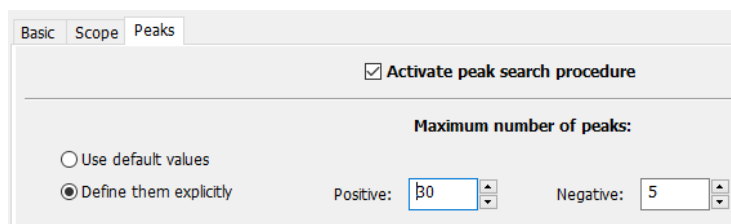
Close Vesta

“Quit” the Contour program in Jana

Right click on the quick button “Run Fourier synthesis”

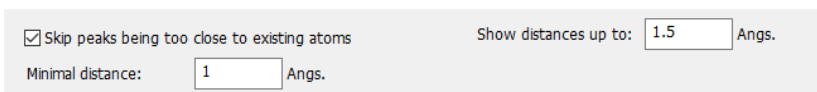
Check that the Map type is “F(obs)-F(calc) – difference Fourier”

In the section “Peaks”: Maximum number of peaks, select “Define them explicitly”, Positive: 30



OK; “Yes+Start” to start the Fourier calculation.

YES to start the procedure of including of new atoms. Set the minimal and maximal distance as on the screenshot.



The peak list is shown with the option to “Include the peak at the specified position” as new atom.

Include Max1 as H1b3b8

Include Max2 as H1b3

Include Max4 as H1b2

Include Max7 as H1b7

Include Max8 as H1b4

Include Max10 as H1b3b7

Include Max12 as H1b8

Include Max13 as H1b5

Include Max15 as H1b1b9

Include Max24 as H1b6

Include Max33 as H1b1

We have now 9 B atoms and 11 H atoms, i.e. all expected atoms were identified. We can now start the final refinement.

Note: to verify the proper number of atoms, start the quick button “Edit basic parameters”, go to the page “Composition” and click “Sum formula from structure model”. It will show B₉ H₁₁ for eight formula units.

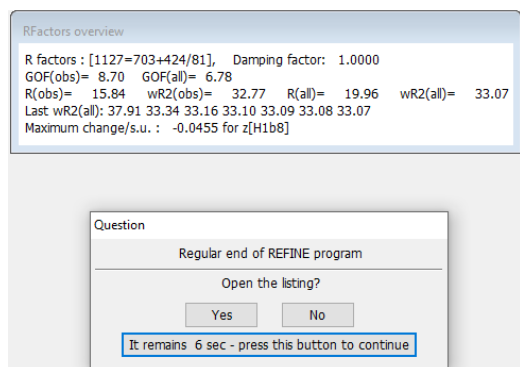
Finish; YES.

Close listing of Fourier calculation.

Double click on “Run Contour” -> “Use old Fourier map” -> “draw map as calculated” and run Vesta from the lower tool bar. Set isosurface level to 2.5σ and boundaries as in the screenshot. Check the positions of the introduced hydrogen atoms.

Close Vesta, close Contour.

Double click on “Refinement -> Run refinement” to start the refinement.

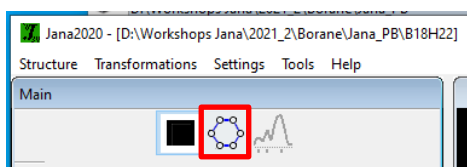




The refinement converges after 7 cycles.


Refinement -> View Refinement listing -> Go to (ALT+g) -> BOTTOM and scroll up.

There are no “Serious warnings” so the displacement parameters of all atoms are positive.

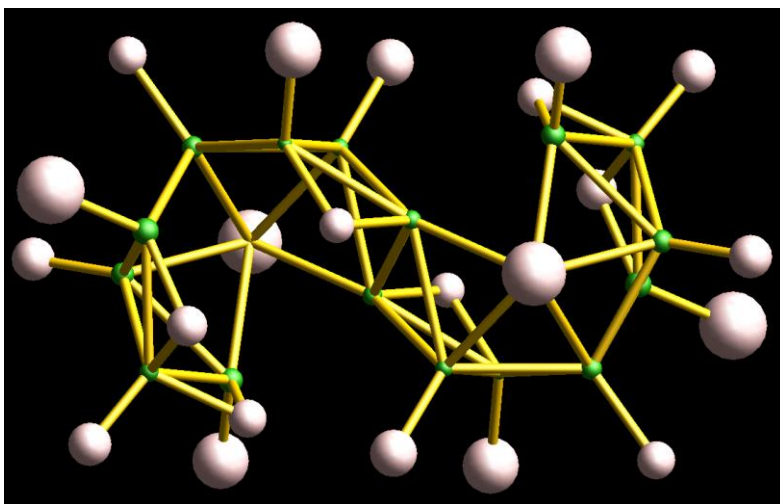
Close listing.



Click on “JanaDraw” and in the lower toolbar on “options”  and set the “Bonds between atoms...expanded by:” to 20% and then click on “Build molecule” .

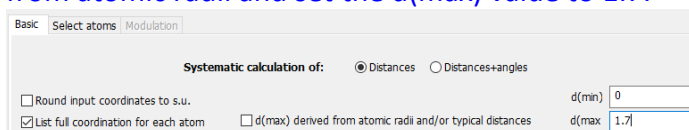
The displacement parameters can be visualized in JanaDraw by clicking the icon  (Draw ellipsoids).

The structure looks alright and displacement parameters seem reasonable. However, the ADPs cover rather large range of values. As a rule of thumb, the same atoms in a similar environment should have a similar (equivalent) isotropic displacement parameter.



Close JanaDraw.

Double click on “Distance calculation” and “Distance commands”. Uncheck “d(max) derived from atomic radii and set the d(max) value to 1.7.



OK;Yes+start;Yes. Check the B-H distances in the listing.

H1b1b9, H1b3b7 and H1b3b8 form 3-center 2-electron bonds and their bond lengths range from 1.21 to 1.38 Å. For the other hydrogens the bond lengths range from 1.16 to 1.46 Å.

The uncertainties are between 0.04 and 0.05 Å. We can now move on to the dynamical refinement.

The Jana files at the end of the kinematical refinement can be found in the folder “JANA/1-kinematical” for comparison. The following part of the tutorial is based on the files from this refinement.

PART 3: Dynamical refinement in Jana2020

1. Preparation of reflection file for the dynamical refinement

During the integration procedure in PETS, an input file for dynamical refinement was created. The integration for the dynamical refinement considers each diffraction image separately. The result is a list with the hkl indices, the measured intensity with the respective sigma and the number of the frame in which the intensity is present. PETS creates the file B18H22_dyn.cif_pets with all this information, which will be used in Jana2020.

Create a new folder "Jana_dynamical"

If you want to be sure that you can exactly reproduce the following part of the tutorial, use the file B18H22_dyn.cif_pets from the folder PETS_output_CIF provided with the example files.

Copy the PETS output file "B18H22_dyn.cif_pets" into the folder Jana_dynamical

[On the screen: basic window of Jana after finishing the kinematical refinement]

"Structure→Save as"

Locate the folder "Jana_dynamical" and save a new structure B18H22_dyn

Continue with the new structure.

"Reflection file→Import/modify reflection file→Reimport"

Select the B18H22_dyn.cif_pets as the new reflection file.

☒ Make the reflection file for dynamical refinement

Check "Make the reflection file for dynamical refinement; NEXT.

Set temperature to 100; NEXT; NEXT.

Jana reads 16707 reflections.

OK; NEXT; FINISH; OK.

Process the reflection by clicking on NEXT.

Notice that 4956 reflections are observed out of 16707 reflections read. 79 observed reflections were rejected because they violate the systematic extinctions.

OK;OK;FINISH.

"Edit structure parameters" → "Edit parameters for electron diffraction"

Set "Maximal diffraction vector g(max)" to 1.25

Set "Number of integration steps" to 200

Set "Limit on RSg" to 0.8 (this increases the number of reflections used for the refinement)

Select "For Fourier rescale to Fcalc"

Set "Number of threads" to the number of cores of your processor.

Activate "For Fourier rescale to Fcalc".

Orientation matrix: U11 0.07634 U12 0.00119 U13 -0.05284
U21 0.03309 U22 -0.06585 U23 0.04627
U31 -0.04094 U32 -0.05091 U33 -0.06107

Maximal diffraction vector g(max): 1.25 Number of integration steps: 200 Geometry: ☒ precession
Maximal excitation error (Matrx): 0.01 Use dynamic approach: ☒ ☐ rotation
Maximal excitation error (Refine): 0.1 Use twin version: ☐
RSg(max): 0.8 Apply correction for crystal tilt: ☒
OSg(min): 0 For Fourier rescale to Fcalc: ☒
Number of threads: 24
Dyngo commands:

Select zones for refinement Define zones of equal thicknesses

Run optimizations except of scale, optimize also: ☐ Thickness ☐ Orientation Show thickness plots

Zone#: 1 uvw: (-0.677, -0.351, -1.000) R(all)= Select zones for editing

Precession angle 1 Alpha -19.752 Beta -0.114
EDScale 1 EDThick 400 EDPhi 0 EDTheta 0

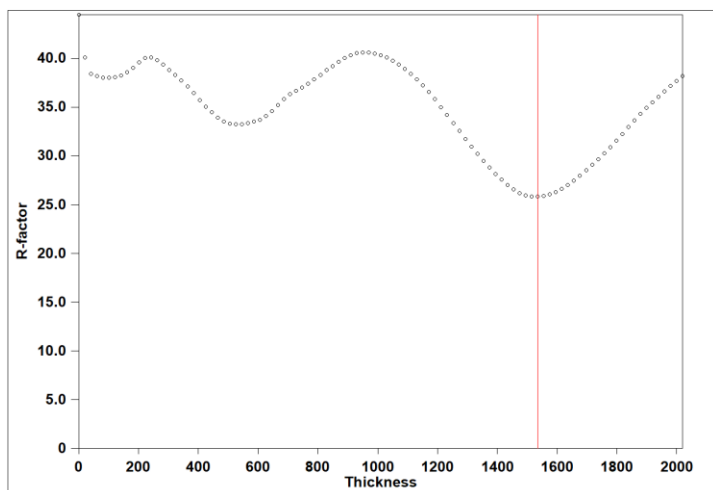
Esc OK

2. Determination of the thickness

In “Edit parameters for electron diffraction” check “Thickness” and then click on “Run optimizations”

“Show thickness plots”

For Zone#1, the best R factor is observed for a thickness of 840 Å. But for Zone#3, there are two minima about 500 and 1500 Å. Look at the rest of the frames. There are two frequent best thicknesses – one about 1000 and the other about 1500 Å. It will turn out during the refinement that the better choice is 1500 Å by 0.7 percent point of $R(\text{obs})$. If the thickness is larger than 1000 Å and the number of integration steps is about 100 you should consider to increase it to at least to 150. Thicker crystals have narrower diffraction maxima, which can be missed by a too low number of integration steps.



Close the plot.

Click on “Select zones for editing”, “Select all”, OK.

The EDThick box is yellow and locked because the parameter is not the same for all selected zones.

Click EDThick box; click Unlock and change the value to 1500.

This changes EDThick for all zones to 1500 Å.

Click on “Select zones for editing”; “Refresh”; OK

By changing number of zone by “Zone#” textbox, we can see that all zones have EDThick 1500 Å

Uncheck the checkbox “Thickness”

Click on “Run optimizations”

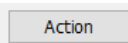
This will optimize the scale factor for each frame based on a thickness of 1500 Å. The optimization runs for about 2 minutes.

OK; YES to save m42 file.

The current state of the Jana files (before clicking “Refine”) is available in the tutorial folder: JANA/2-dynamic/B18H22_dyn_0-setup

3. Hydrogen localization

Click on “Edit atoms” icon . Select all hydrogen atoms and delete them by clicking on



and selecting delete atoms.

Yes to all; OK; Yes for rewriting m40.

Run refinement. It converges after 5 cycles.

```
-----  
| R factors : [8449=3154+5295/101],      Damping factor: 1.0000  
| GOF(obs)= 5.93      GOF(all)= 3.76  
| R(obs)= 16.35      wR2(obs)= 32.99      R(all)= 25.00      wR2(all)= 34.21  
| Last wR2(all): 40.04 34.36 34.22 34.21 34.21 34.21  
| Maximum change/s.u. : -0.0153 for x[B1]  
-----
```

The resulting state of the Jana files is available in the tutorial folder as JANA/2-dynamic/B18H22_1-hydrogens. You may use these files to save time, look at the results or simply for comparison purposes.

Double click on “Fourier synthesis” to unfold the options.

Double click on “Calculate Fourier maps”; No;No.

Click on “No”

Double click on “Run Contour”; double click on “Use old Fourier maps” and “Draw maps as calculated”.

Click on Vesta icon  in the lower tool bar.

Objects->Boundary (CTRL+Shift+B); change the range of x, y, z as in the screenshot; APPLY; OK

Ranges of fractional coordinates

| | | | |
|----------|-----|----------|-----|
| x(min) = | 0.1 | x(max) = | 0.9 |
| y(min) = | 0.2 | y(max) = | 0.8 |
| z(min) = | 0 | z(max) = | 1 |

Check difference maxima. The hydrogens are nicely visible even at 3σ level.

Close Vesta, close Contour.

Double click on “New”>”New atom”. Set limits for the maxima distance from the atoms as in the case of the kinematical refinement. All the first 11 maxima belong to the hydrogen atoms. Name the hydrogens in the same way as for the kinematical refinement (H1 + name of the boron atom(s) to which it is attached).

You can see, that dynamical refinement substantially improves the visibility of the difference maxima due to missing hydrogen atoms in the structure.

☒ Skip peaks being too close to existing atoms
Minimal distance: Angs.
Show distances up to: Angs.

| List of peaks | | Equivalent coordinates of the peak | | | Shortest distance to |
|---------------|-------|------------------------------------|----------|----------|----------------------|
| Max1 | Max30 | 0.286467 | 0.153799 | 0.180884 | as typed in |
| Max2 | Max31 | 0.713533 | 0.653799 | 0.319116 | B7 1.226 |
| Max3 | Max32 | | | | |
| Max5 | Max33 | | | | |
| Max6 | Max35 | | | | |
| Max8 | Max36 | | | | |
| Max10 | Max37 | | | | |
| Max11 | Max38 | | | | |
| Max12 | Max39 | | | | |
| Max14 | Max40 | | | | |
| Max18 | | | | | |
| Max22 | | | | | |
| Max24 | | | | | |
| Max26 | | | | | |
| Max28 | | | | | |
| Max29 | | | | | |

Confirm the changes.

4. Refinement

Double click on “Refinement->Refinement commands”, set the number of cycles to 20.

OK; YES+start to start the refinement.

The refinement converges after 20 cycles to $R(\text{obs})$ 8.72% (3154 observed reflections) and $wR2(\text{all})$ 19.29% (8449 reflections). The total refinement time is about 2 hours (desktop computer, 24 physical cores). The resulting state of the Jana files is available in the tutorial folder as JANA/2-dynamic/B18H22_2-refine. You may use these files to save time, to look at the results or for comparison.

Close the output.

5. Optimization of orientation

Save a new structure "File→save as→B18H22_dyn_ori"; OK; Yes.

"Edit structure parameters→Edit parameters for electron diffraction".

Enable "Except of scale optimize also: Orientation".

Click "Run optimizations"

For each frame the orientation will be optimized.

Check the EDTheta parameter for each zone using arrows next to the zone number .

Only small number of frames have the orientation correction above 0.1° , which indicates that the frame orientation optimization done in PETS 2.0 produces precise frame orientations even for molecular crystal, which has only moderate resolution and deteriorates during the measurement.

Click on "OK" to leave the electron diffraction menu

Click on "YES" to save the changes to the M42 file

Click "Refinement->Refinement commands"

Start "Refinement->Run refinement"

The refinement converges in 5 cycles to $R(\text{obs})$ 7.28% (3166 obs reflections) and $wR2(\text{all})$ 16.40% (8466 reflections). The final state of the refinement is available in the tutorial folder:

JANA/2-dynamic/B18H22_3-refine_ori

6. Final structure

Click on "Distance calculation->Calculate distances", "Yes" to show a list of interatomic distances.

In comparison to the kinematical refinement, the standard uncertainties are now all about 0.01 Å. Bond lengths range from 1.183 to 1.451 Å.

The displacement parameters of the hydrogen atoms are now more even than with the kinematical refinement. You can see in the table below that the hydrogen atoms bonded by two electron two center bond have the ADPs about 0.055 Å² but the ADPs of the hydrogen atoms bonded by two electron three center bonds have the ADPs nearly twotimes lower (0.031 Å²). This difference stems from the higher draft of the bonding electrons by the boron backbone from these electrons, creating a more positive partial charge on these hydrogen atoms, which results in a higher scattering power of these hydrogens. On the other hand, the boron atoms have their ADPs about 0.0095 Å² with an exception of B3, which is the boron atom surrounded by three hydrogen atoms. Its ADP value is nearly doubled in comparison to the other boron atoms, so the

effect here is opposite, which is probably due to a higher electron density around this atom, and thus its lower scattering power. You can compare these results with the kinematical refinement and with the neutron data measured at room temperature published by G.S. Pawley in *Acta Crystallorg.* **20** (1966) 631. Anisotropic betas were converted to *U*iso. In the neutron data, there are no similar effects on the values of the ADPs observed in the dynamically refined electron diffraction data.

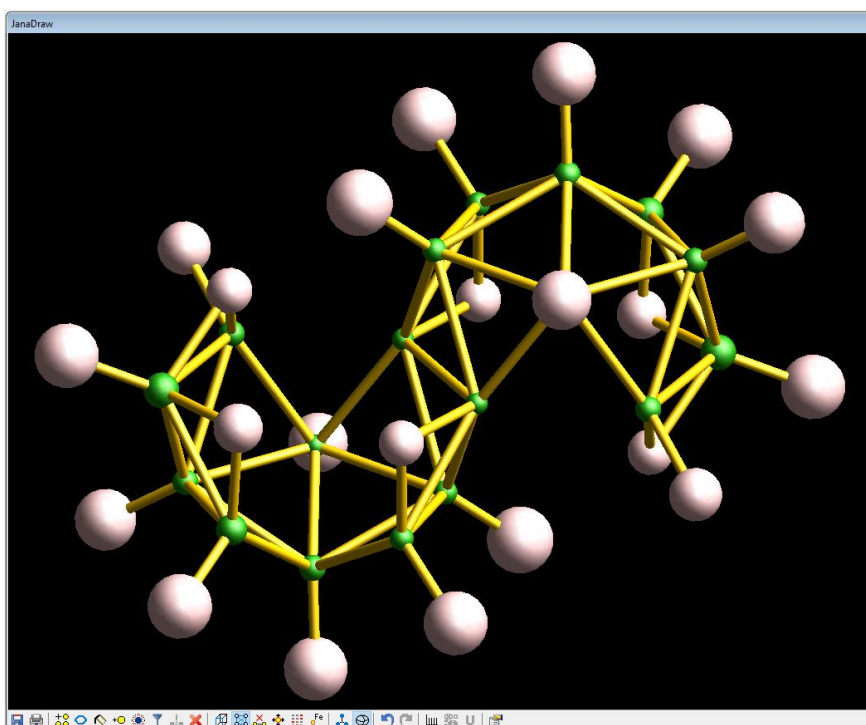
When we look at the B-H distances of B3, we can see that for kinematical refinement the distances to H1b3b8 (1.21(4) Å) and H1b3 (1.16(4) Å) are within 1 σ the same. The distance to H1b3b7 is 1.33(4) Å. But for the dynamical refinement, the distances are 1.450(10), 1.280(13), and 1.419(11) Å, respectively, and thus it is easy to determine, by which type of bond is the hydrogen atom to the boron attached.

Dynamical refinement

| Atom | Ox. | Wyck. | Site | S.O.F. | x/a | y/b | z/c | U [Å ²] |
|--------|-----|-------|------|--------|---------|---------|---------|---------------------|
| B1 | 8e | 1 | | | 0.43212 | 0.35366 | 0.39516 | 0.0082 |
| B2 | 8e | 1 | | | 0.68052 | 0.44819 | 0.41197 | 0.0068 |
| B3 | 8e | 1 | | | 0.54485 | 0.53529 | 0.20017 | 0.0179 |
| B4 | 8e | 1 | | | 0.58894 | 0.33778 | 0.34732 | 0.0093 |
| B5 | 8e | 1 | | | 0.65497 | 0.44078 | 0.24862 | 0.0092 |
| B6 | 8e | 1 | | | 0.55762 | 0.38351 | 0.50110 | 0.0084 |
| B7 | 8e | 1 | | | 0.64288 | 0.57084 | 0.32828 | 0.0092 |
| B8 | 8e | 1 | | | 0.49517 | 0.39300 | 0.22952 | 0.0133 |
| B9 | 8e | 1 | | | 0.57525 | 0.53391 | 0.49383 | 0.0065 |
| H1b7 | 8e | 1 | | | 0.71869 | 0.65645 | 0.32324 | 0.0412 |
| H1b3b7 | 8e | 1 | | | 0.53500 | 0.61710 | 0.29490 | 0.0306 |
| H1b1b9 | 8e | 1 | | | 0.36134 | 0.44482 | 0.38948 | 0.0304 |
| H1b1 | 8e | 1 | | | 0.36304 | 0.26575 | 0.41089 | 0.0552 |
| H1b5 | 8e | 1 | | | 0.73941 | 0.41790 | 0.18312 | 0.0530 |
| H1b6 | 8e | 1 | | | 0.58921 | 0.32185 | 0.58637 | 0.0635 |
| H1b8 | 8e | 1 | | | 0.45452 | 0.33407 | 0.13840 | 0.0565 |
| H1b4 | 8e | 1 | | | 0.61383 | 0.24142 | 0.33650 | 0.0561 |
| H1b2 | 8e | 1 | | | 0.78679 | 0.44754 | 0.45536 | 0.0511 |
| H1b3b8 | 8e | 1 | | | 0.42448 | 0.48890 | 0.23075 | 0.0313 |
| H1b3 | 8e | 1 | | | 0.53647 | 0.58761 | 0.09678 | 0.0599 |

Kinematical refinement

| Atom | Ox. | Wyck. | Site | S.O.F. | x/a | y/b | z/c | U [Å ²] |
|--------|-----|-------|------|--------|---------|---------|---------|---------------------|
| B1 | 8e | 1 | | | 0.43445 | 0.35487 | 0.39565 | 0.0033 |
| B2 | 8e | 1 | | | 0.68030 | 0.44880 | 0.41293 | 0.0014 |
| B3 | 8e | 1 | | | 0.54349 | 0.53238 | 0.20201 | 0.0057 |
| B4 | 8e | 1 | | | 0.58898 | 0.33734 | 0.34794 | 0.0075 |
| B5 | 8e | 1 | | | 0.65511 | 0.43798 | 0.24796 | 0.0084 |
| B6 | 8e | 1 | | | 0.55899 | 0.38277 | 0.50211 | 0.0048 |
| B7 | 8e | 1 | | | 0.64344 | 0.57126 | 0.32784 | 0.0093 |
| B8 | 8e | 1 | | | 0.49569 | 0.39647 | 0.23019 | 0.0109 |
| B9 | 8e | 1 | | | 0.57658 | 0.53342 | 0.49288 | 0.0059 |
| H1b3b8 | 8e | 1 | | | 0.44094 | 0.49575 | 0.22316 | 0.0251 |
| H1b3 | 8e | 1 | | | 0.54157 | 0.58136 | 0.10883 | 0.0281 |
| H1b7 | 8e | 1 | | | 0.71616 | 0.64759 | 0.31492 | 0.0482 |
| H1b4 | 8e | 1 | | | 0.62022 | 0.23213 | 0.31854 | 0.0220 |
| H1b2 | 8e | 1 | | | 0.79404 | 0.45559 | 0.45447 | 0.0632 |
| H1b3b7 | 8e | 1 | | | 0.53624 | 0.60695 | 0.29368 | 0.0221 |
| H1b8 | 8e | 1 | | | 0.46070 | 0.32564 | 0.12515 | 0.0701 |
| H1b5 | 8e | 1 | | | 0.73743 | 0.39968 | 0.18340 | 0.0283 |
| H1b1b9 | 8e | 1 | | | 0.36127 | 0.44474 | 0.39803 | 0.0187 |
| H1b6 | 8e | 1 | | | 0.58648 | 0.32039 | 0.59949 | 0.0336 |
| H1b1 | 8e | 1 | | | 0.36606 | 0.26634 | 0.41287 | 0.0480 |



Pawley 1966, 293 K, neutron single crystal

| Atom | Ox. | Wyck. | Site | S.O.F. | x/a | y/b | z/c | U [Å ²] |
|--------|-----|-------|------|--------|----------|----------|----------|---------------------|
| B1 | | 8e | 1 | | -0.06590 | 0.14480 | 0.10390 | 0.0204 |
| B2 | | 8e | 1 | | 0.17870 | 0.05320 | 0.08700 | 0.0191 |
| B3 | | 8e | 1 | | 0.04580 | -0.03310 | 0.29800 | 0.0244 |
| B4 | | 8e | 1 | | 0.08820 | 0.16230 | 0.15130 | 0.0208 |
| B5 | | 8e | 1 | | 0.15410 | 0.06170 | 0.25020 | 0.0225 |
| B6 | | 8e | 1 | | 0.05800 | 0.11630 | -0.00050 | 0.0182 |
| B7 | | 8e | 1 | | -0.07590 | 0.03180 | -0.00590 | 0.0173 |
| B8 | | 8e | 1 | | 0.14410 | -0.06750 | 0.17180 | 0.0216 |
| B9 | | 8e | 1 | | -0.00300 | 0.10590 | 0.26990 | 0.0230 |
| H1b3b8 | | 8e | 1 | | -0.06290 | 0.01170 | 0.26380 | 0.0227 |
| H1b3 | | 8e | 1 | | 0.03940 | -0.07620 | 0.39030 | 0.0307 |
| H1b7 | | 8e | 1 | | 0.21250 | -0.13550 | 0.17680 | 0.0264 |
| H1b4 | | 8e | 1 | | 0.12100 | 0.24920 | 0.16380 | 0.0247 |
| H1b2 | | 8e | 1 | | 0.26930 | 0.05830 | 0.05160 | 0.0217 |
| H1b3b7 | | 8e | 1 | | 0.04520 | -0.10380 | 0.20920 | 0.0226 |
| H1b8 | | 8e | 1 | | -0.04080 | 0.15940 | 0.35190 | 0.0287 |
| H1b5 | | 8e | 1 | | 0.23150 | 0.09110 | 0.31750 | 0.0271 |
| H1b1b9 | | 8e | 1 | | -0.13020 | 0.05040 | 0.09810 | 0.0190 |
| H1b6 | | 8e | 1 | | 0.08720 | 0.16410 | -0.07880 | 0.0208 |
| H1b1 | | 8e | 1 | | -0.13060 | 0.21380 | 0.08800 | 0.0252 |