

Structure determination of alaptide, example

Unit cell: 21.14 7.22 6.14 90 90 90

Space group: $P2_12_12_1$

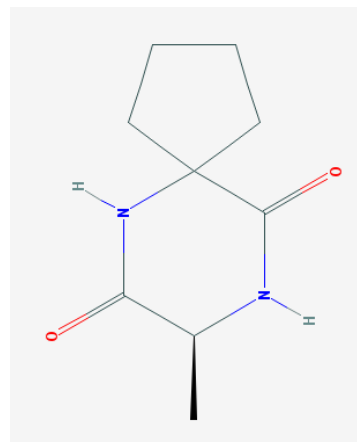
$\lambda = 0.79984 \text{ \AA}$

Programs needed

- fox.exe

Files needed

- alaptide.fhz (model file)
- synch.xy (powder pattern)



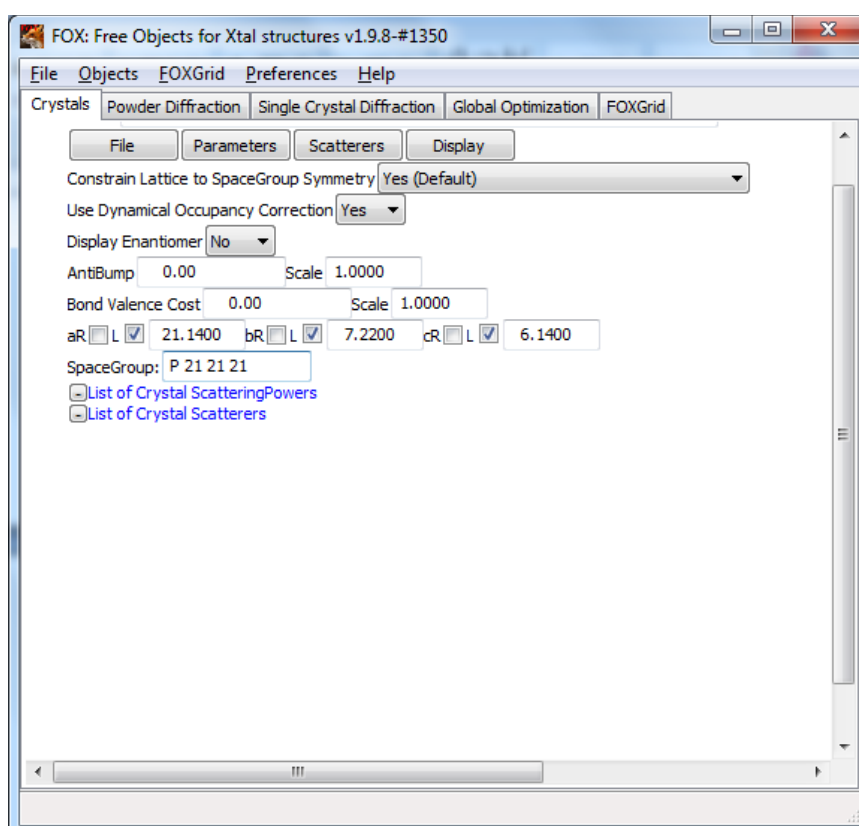
1. Define unit cell and asymmetric part of the unit cell

Start fox.exe

Define unit cell and space group

Object->New Crystal

Paste values of the unit cell and space group (21.14 7.22 6.14 90 90 90, P212121)

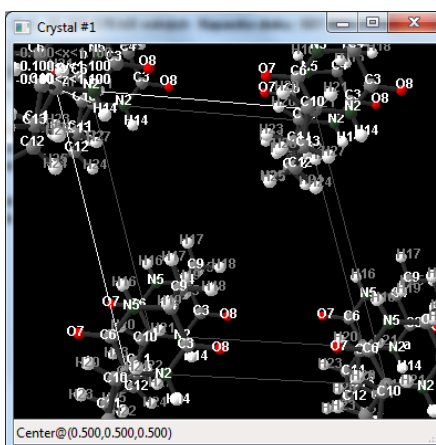
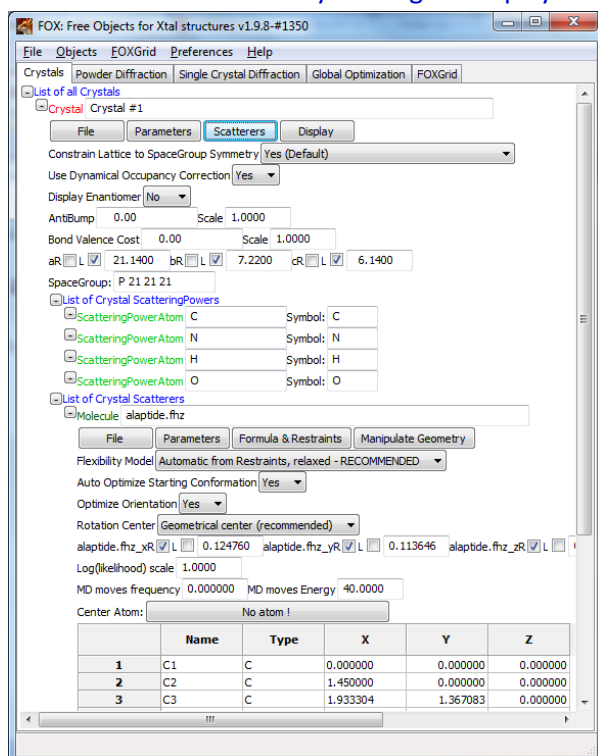


Define the asymmetric part of the unit cell

Scatterers->Import molecule from Fenske-Hall Z-matrix

- Select alaptide.fhz

Check the loaded model by clicking on Display->3D display. Please let It open.



If you set the space group to P1, the model of the molecule in 3D display will be more synoptical. After you check the model, set the space group correctly to P212121. Checking the model is important, if the model is wrong, you will never find the correct solution.

Define rigid body restraint (optional)

It helps to keep the rigid groups in the correct (known) conformation (e.g. benzene rings). This speeds up the solution process.

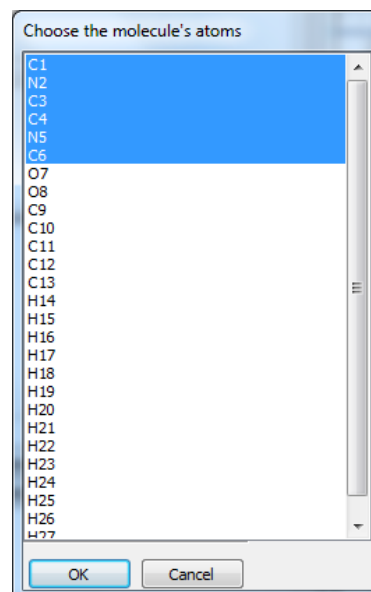
The whole molecule of Alaptide seems to be more or less rigid. This you can set by changing the "Flexibility Model" to "Rigid body".

However, it is necessary to use this option carefully. The molecule of Alaptide is not strictly rigid. There are even several possible conformations of the molecule. So, be careful!

Another possibility is to set individual rigid group(s) in **Formula and restraints->Add rigid group**

In the case of Alaptide, you can, for example, set the ring as rigid, so **Select C1, N2, C3, C4, N5, C6, O7, O8, H16 and H14 atoms.**

Sometimes, it is necessary to use a rigid body, because the model is too complex. However, in the case of Alaptide, rigid body restriction is not needed.



Save data

File->Save->FOX compressed files (*.xml.gz). Save data as "01_unitcell"

2. Define powder pattern

In this part, you have to import the powder diffraction pattern, and you also have to define the wavelength, maximal resolution (decreasing the resolution speeds up the calculations) and profile parameters.

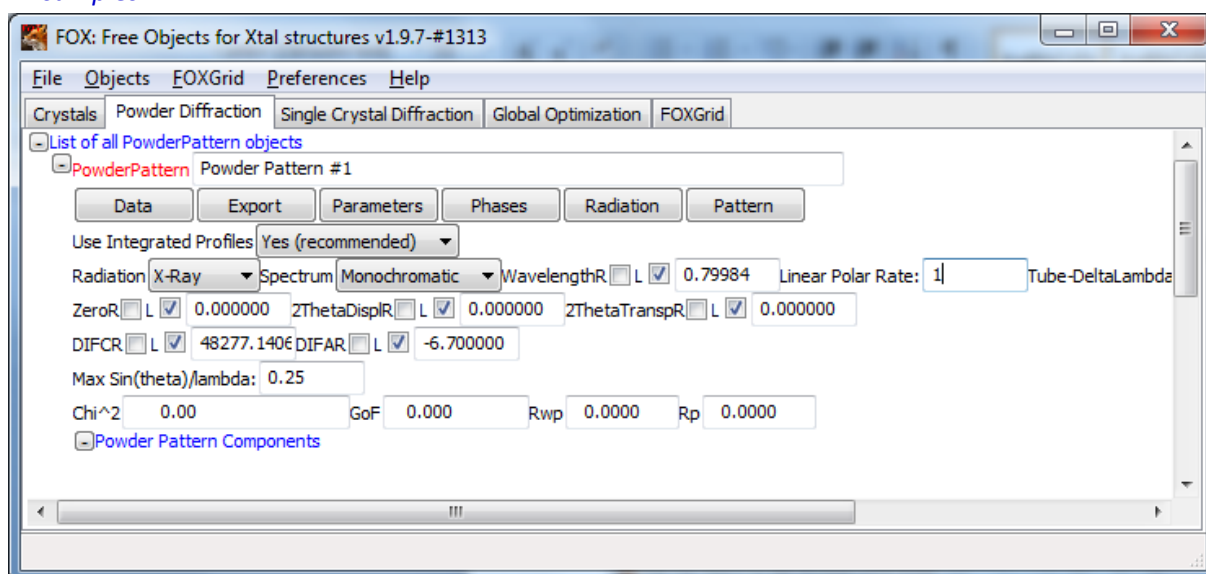
Define powder pattern object

Objects->New powder pattern

Go to the "Powder Diffraction" tab.

- Set the wavelength to 0.79984 Å
- Set linear polar rate to 1 (synchrotron data). (For laboratory data - this is not the case - set 0).
- Set Max Sin(theta)/lambda to 0.25.

$\text{Sin}(\theta)/\lambda$ is a resolution and corresponds to 2θ (try to calculate it from the Bragg's law). Resolution of 2θ is usually sufficient for structure solution by direct-space methods of organic samples.



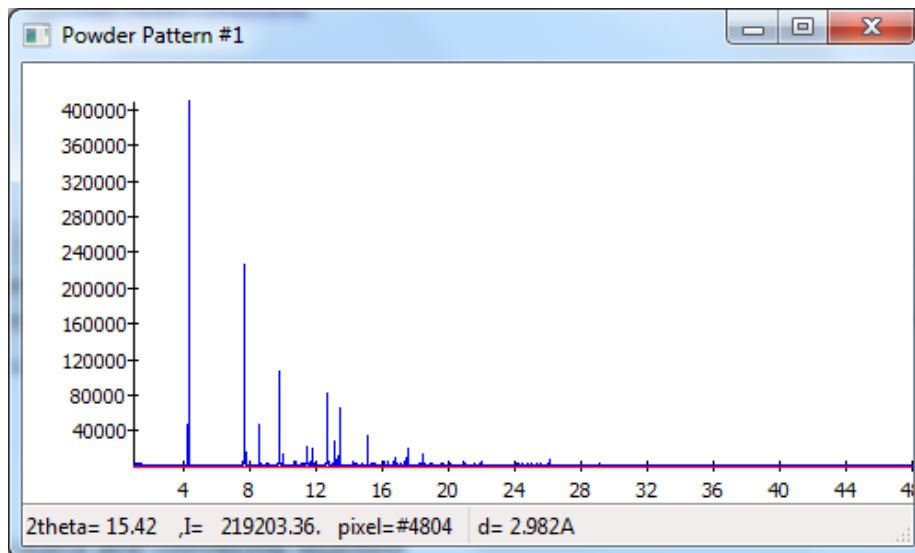
Import powder pattern

Data->Import 2Theta-Obs pattern

- Select synch.xy file

Check loaded data

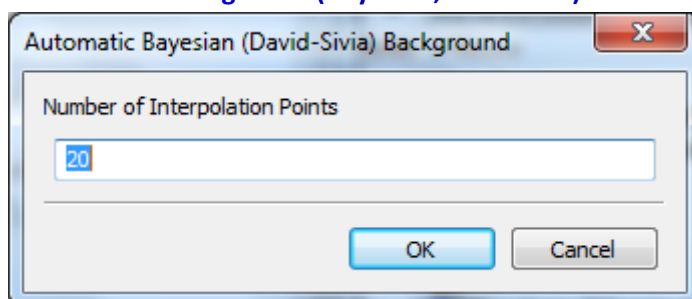
- Pattern->Show graph
- don't close the powder pattern window! Later, you will need it.



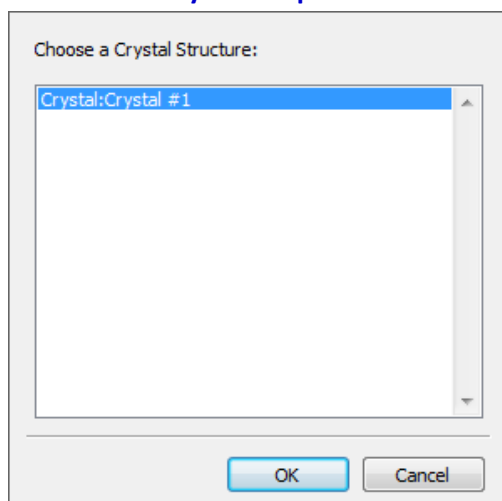
Define powder pattern parameters (background and profile)

In the Fox's main window:

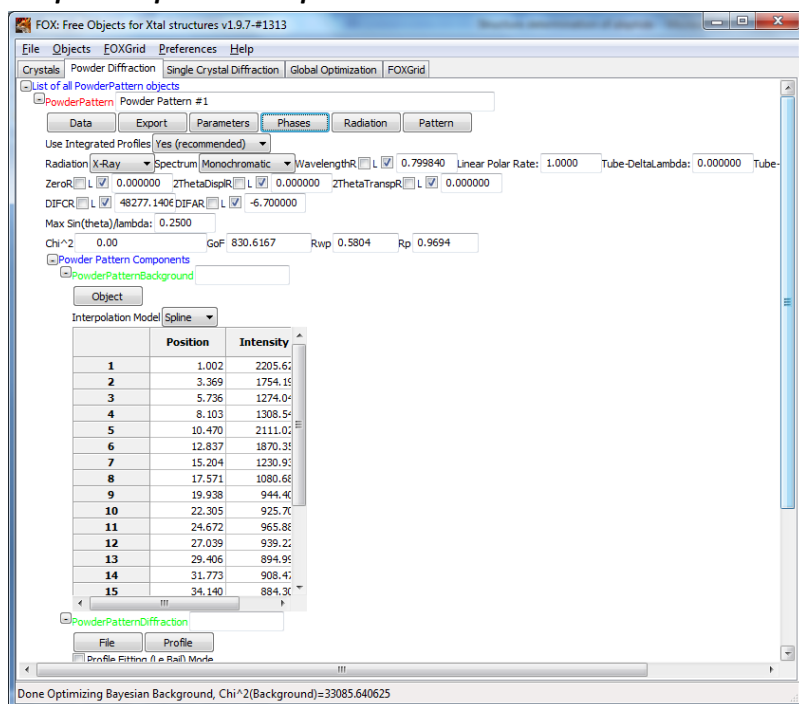
Phases->add background (Bayesian, automatic)-> 20 -> OK (Usually 20 parameters are sufficient)



Phases->add crystalline phase->OK



The powder pattern tab preview



Save data

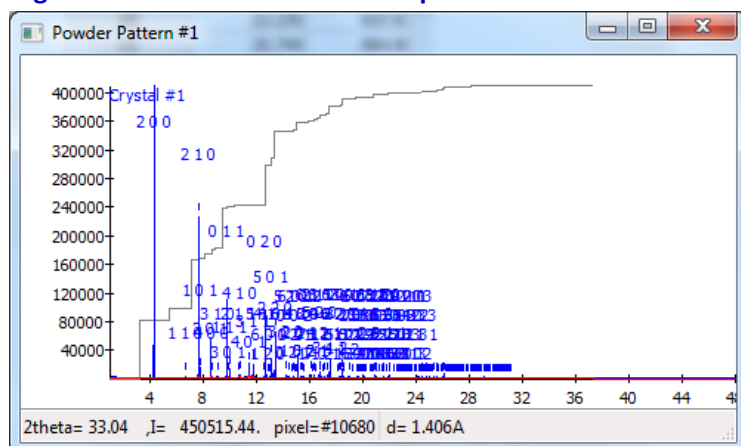
File->Save->FOX compressed files (*.xml.gz). Save data as "02_powder_pattern"

Perform Le Bail fitting to get profile parameters

At the end of this tutorial, the program will try to find a good agreement between the measured and theoretical XRPD patterns during the structure solution process. Therefore, we need to find profile parameters that guarantee that the fit of the profile will be perfect when the correct structural model is found.

Le Bail fitting is the procedure that allows finding the profile parameters without the knowledge of the structure model. And this is the reason why we are going to use it right now.

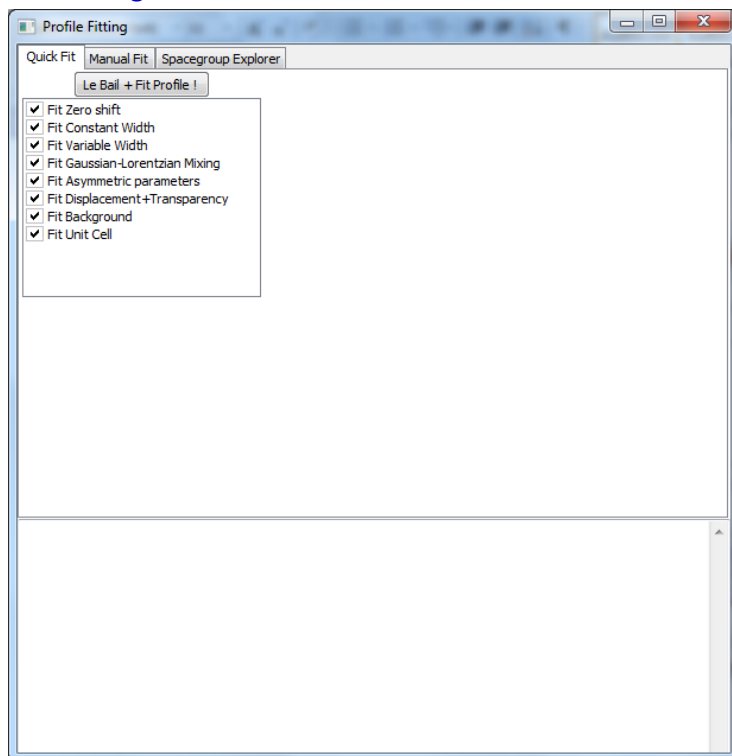
Right-mouse-click on the Powder pattern window



Select “Fit Profile + Le Bail extraction”

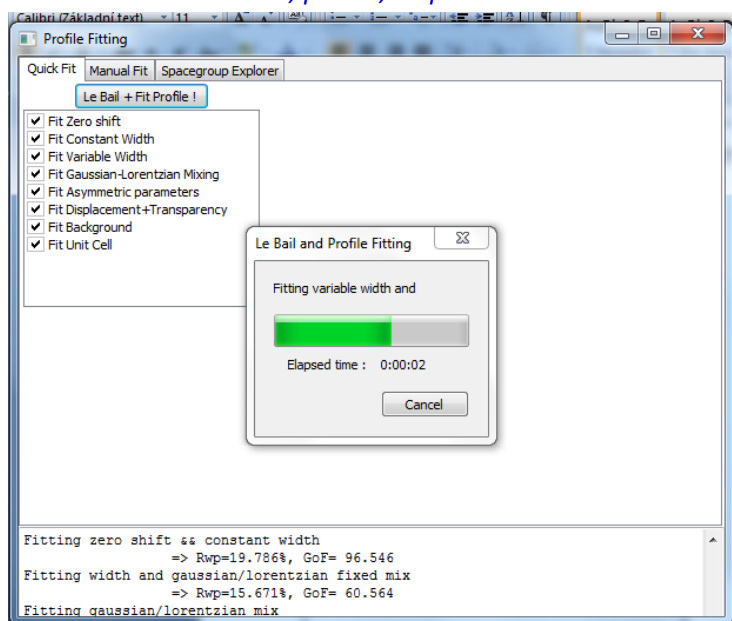
Powder Pattern
Update
Hide labels
Find peaks
Load peaks
Save peaks
Index !
Hide peaks
Add peak
Remove peak
Fit Profile + Le Bail extraction
X scale: 2theta/TOF
X scale: $Q=1/d$
X scale: $Q=2\pi/d$
Y scale: I
Y scale: $\sqrt{I}$
Y scale: $\log_{10}(I)$

Start fitting with “Le Bail + Fit Profile !” button



Do it several (three) times. After that, close the “Profile fitting” window.

This will take some time, please, be patient.



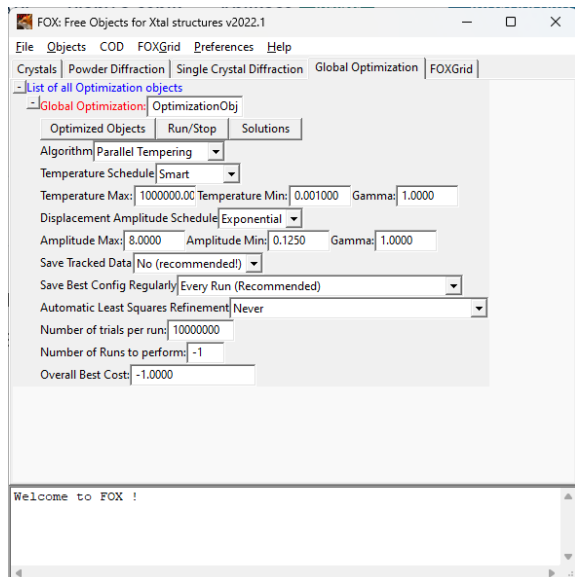
Save data

File->Save->FOX compressed files (*.xml.gz). Save data as “03_powder_pattern”

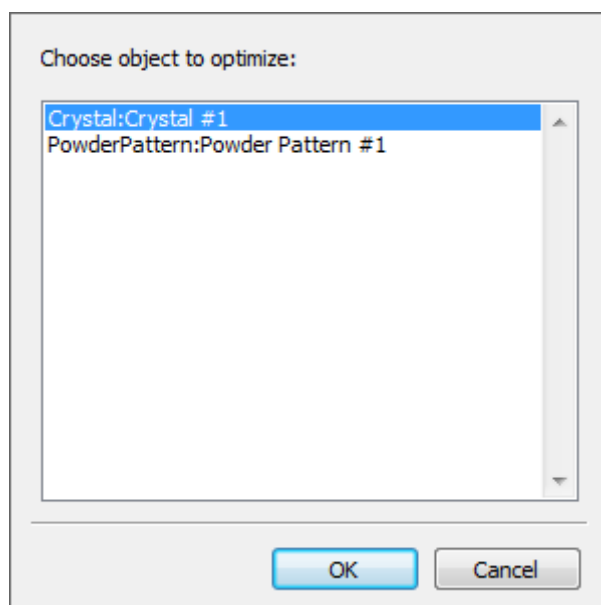
3. Define the computing algorithm and run the computation

Objects->New Monte-Carlo object

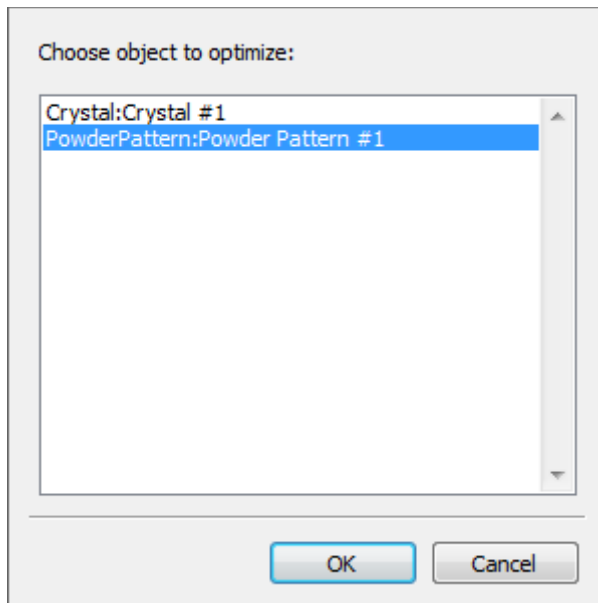
Go to the “Global optimization tab” and define objects to optimize



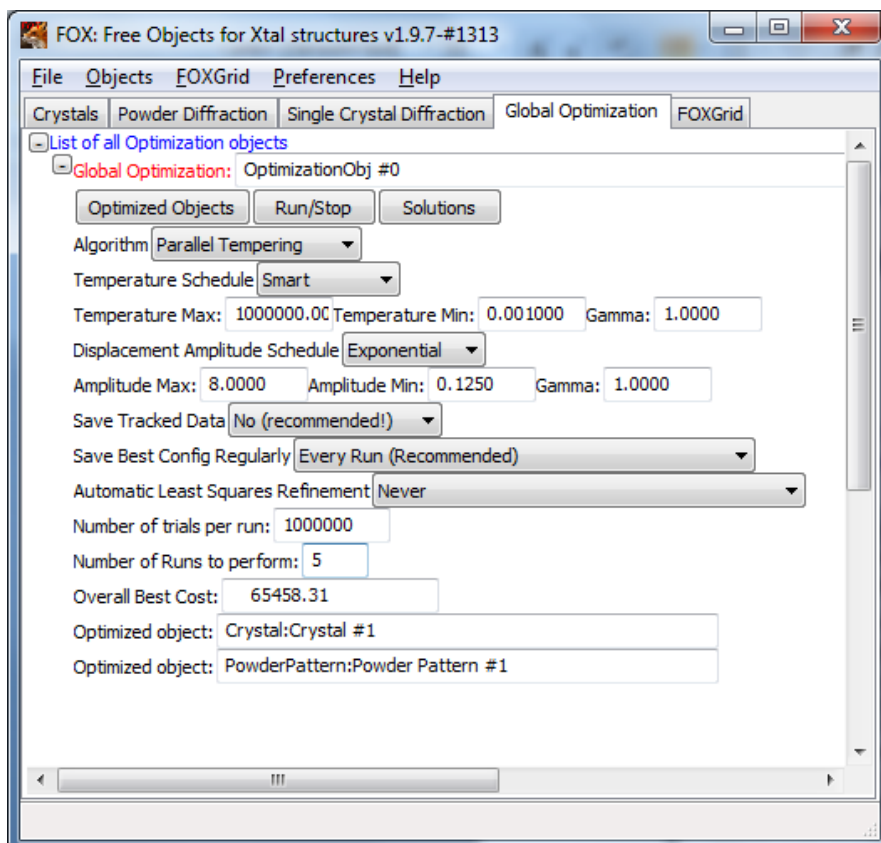
- Optimized objects->Add object to optimize->Select Crystal:Crystal #1->OK



- **Optimized objects->Add object to optimize->Select PowderPattern:Powder pattern->OK**



- **Set Number of trials per run to 500000.**
The algorithm will 500000 times change the model to find the solution. This small number is sufficient for such a simple task - DOF 6
- **Set Number of Runs to Perform 10**
The program will repeat the calculation ten times with different starting models. Note, that not every run it will reach the minimum.



Save data

File->Save->FOX compressed files (*.xml.gz). Save data as "04_final"

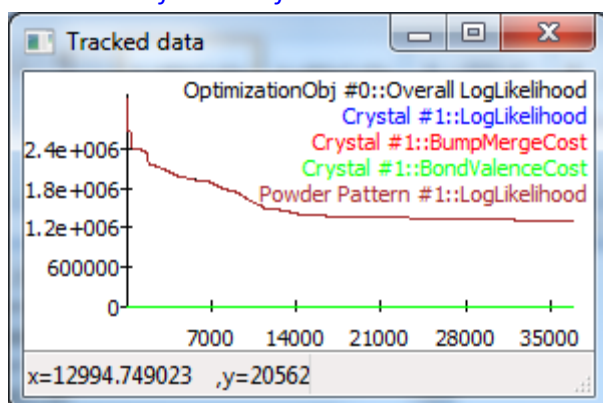
Start computation

Everything is set, and the only thing we need to do is to start the calculation

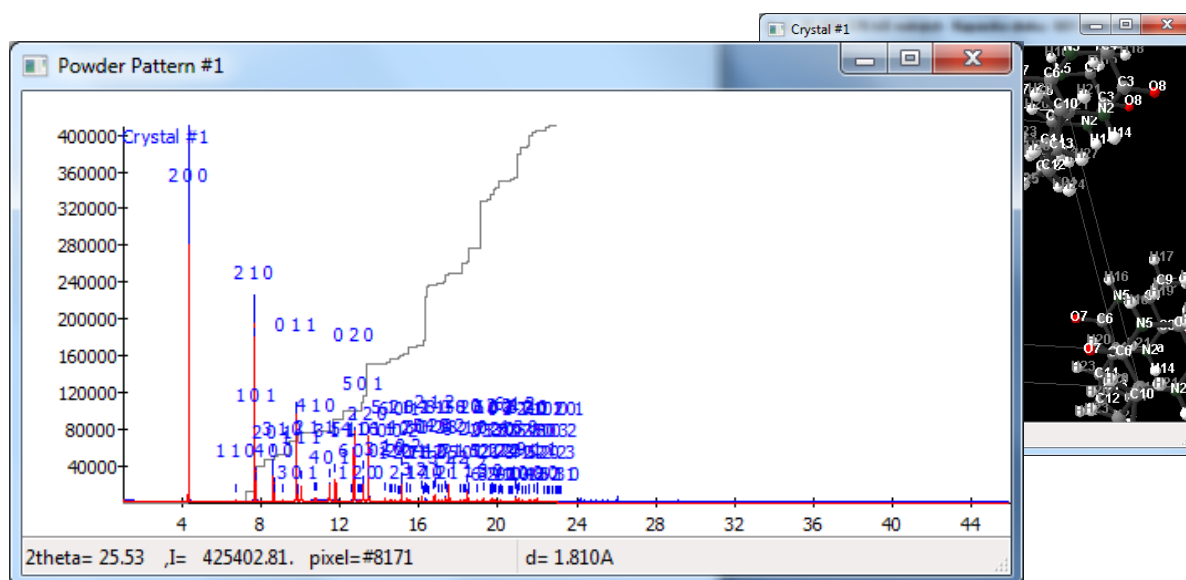
Run/stop-> Multiple runs

Fox will show you a new window (Tracked data).

This shows you the decrease of the cost function in time. The x-axis is the number of trials, and y-axis is the value of the cost function



You can also watch the structure determination process on the Crystal #1 and powder pattern windows.

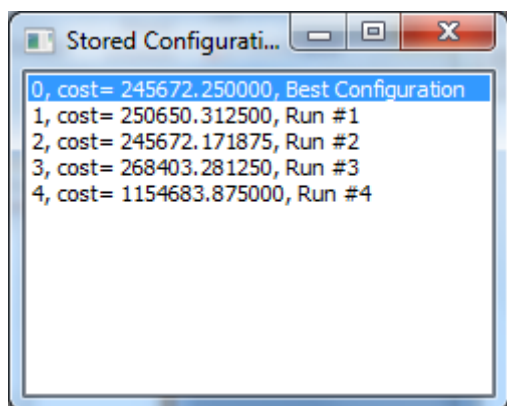


4. Saving the result

When the FOX finishes the computation, the folder contains several (10) *.xml files. File names contain information about the Run number and also the final value of the cost function. So, if you close the FOX program, your results will be still there. But don't close the FOX at this moment. We will use its solution browser...

In the „Global optimization tab“

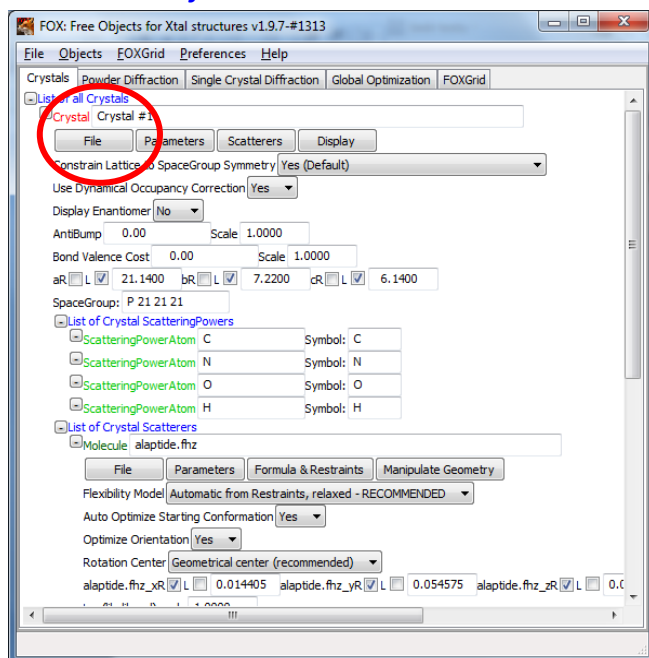
Solutions->Browse solutions



If you click on some row, the FOX will show you immediately the result in the Crystal #1 and powder pattern windows. You can select the result which you find as correct (usually the best one, which is on the top).

In the “Crystals” tab

File->Save as Cif



You can continue, and you can save another solution from the solution Browser as well.