

A Tutorial for Solving Crystal Structure from Powder Diffraction Data Using *EPCryst*

Requirements:

Software: [EPCryst](#), [Fullprof](#)

Diffraction Data: [La2CuO4.dat](#)

Basic Steps

1. Indexing & Space group determination. (Omitted in this tutorial)
2. Intensity extraction. (Using Fullprof or any other program that can do profile fitting)
3. Trail models generation. (Using EPCryst)
4. Solve crystal structures. (Using EPCryst)
5. Refinement. (Omitted in this tutorial)

Details

Step 1: Indexing & Space group determination.

You can finish this step in Fullprof, or any other programs that you are familiar with. Although it is not a very easy task, we will omit the details of this step here. The results we obtained for La₂CuO₄ are as following:

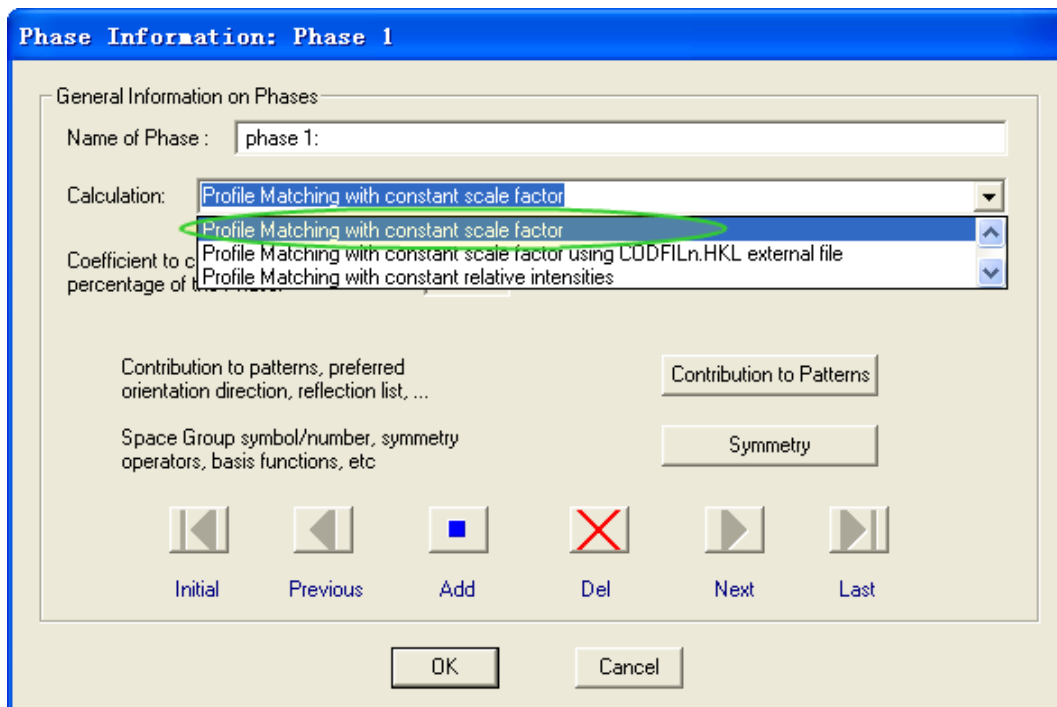
Compound	La ₂ CuO ₄
Cell Parameters:	a=5.354706 b=5.398284 c=13.151965 $\alpha=\beta=\gamma=90$
Space group:	F m m m (SG No. 69)

Step 2: Intensity extraction.

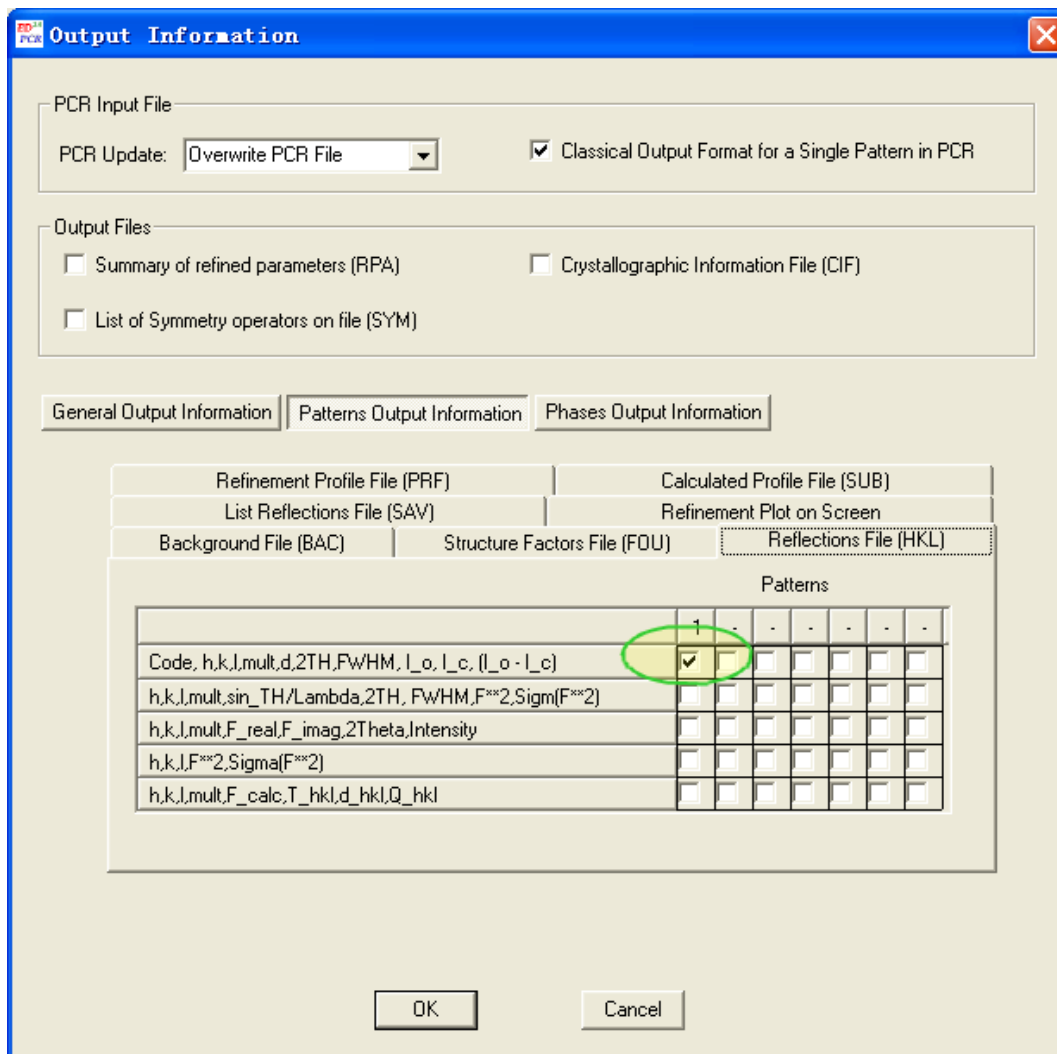
For efficiency consideration, EPCryst uses the extracted intensities. Profile fitting programs are needed to prepare the intensity file. Although the intensity file can be prepared using any profile fitting program, Fullprof is recommended. The output file of Fullprof can be read directly by EPCryst, while files from other programs must be converted to certain format first. In this tutorial we will concern only on Fullprof.

Details about using Fullprof to do full profile decomposition is out of the range of this tutorial. We will only discuss details that are related with EPCryst.

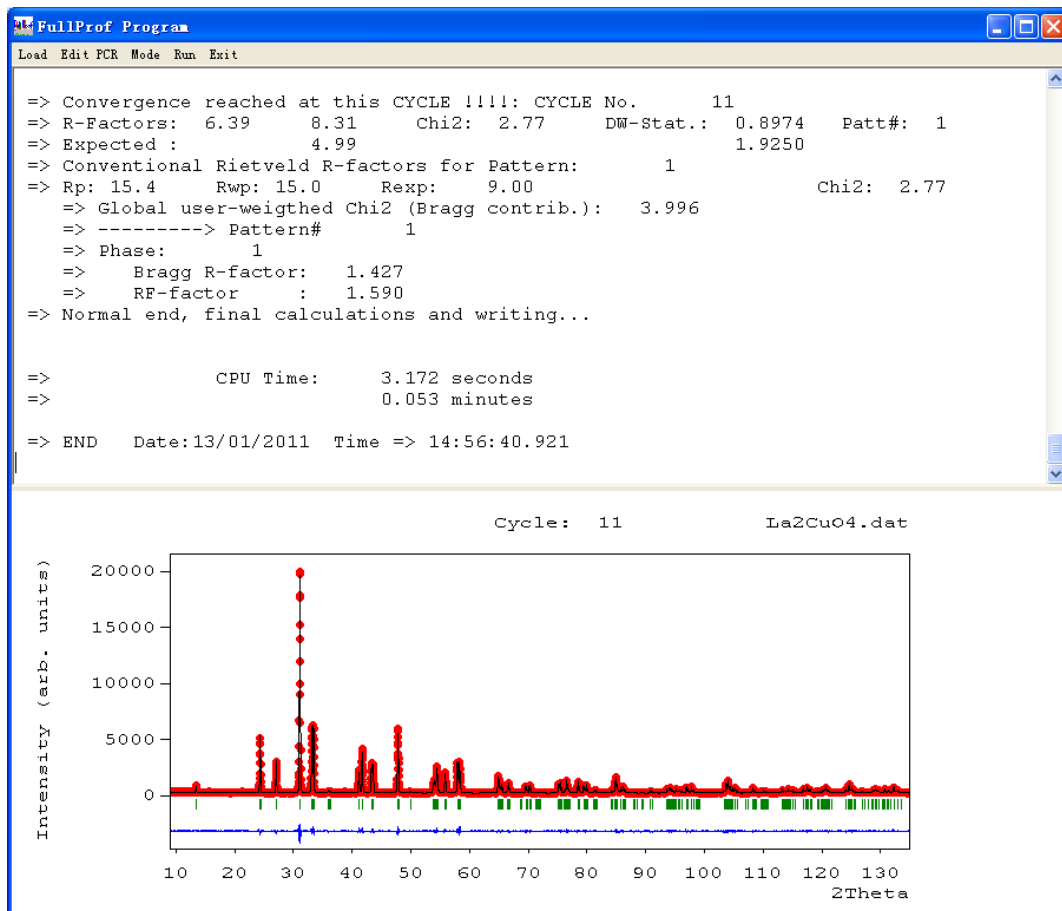
(1) Job type: Under PCR Editor->Phases



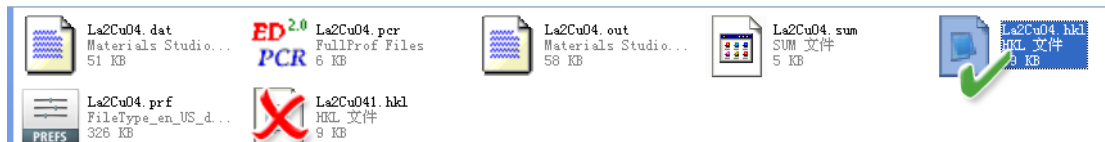
(2) Output file format: Under PCR Editor->Output



(3) When we get a reasonable Rp/Rwp, we can stop.



(4) There will be two hkl files when finished (filename.hkl & filename1.hkl). Filename.hkl is the desired file. Copy and save the file for later usage.



The .pcr file and .hkl intensity file can be download here [La2CuO4.pcr](#) [La2CuO4.hkl](#).

Step 3: Model Generation.

(1) Open EPCryst (Double click the EPCryst shortcut on your desktop). Click "New" on the toolbar to create a new file.

(2) Input the basic information:

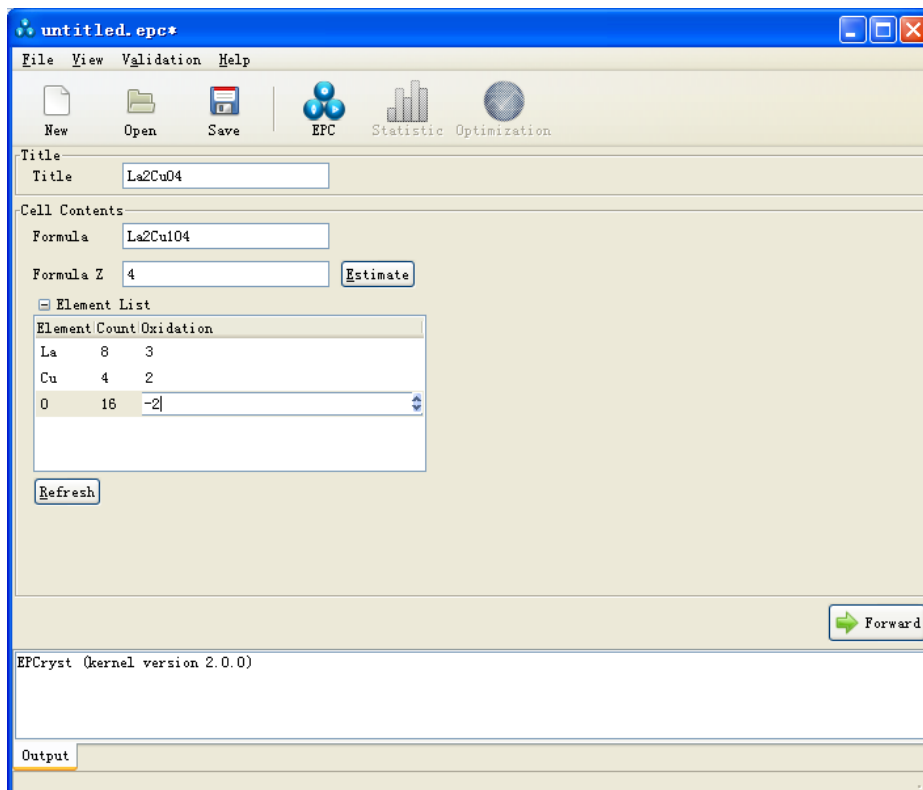
Title: A title, which also used as the output filename.

Formula: Press enter to refresh the "Element List". Do not forget the "1" subscript.

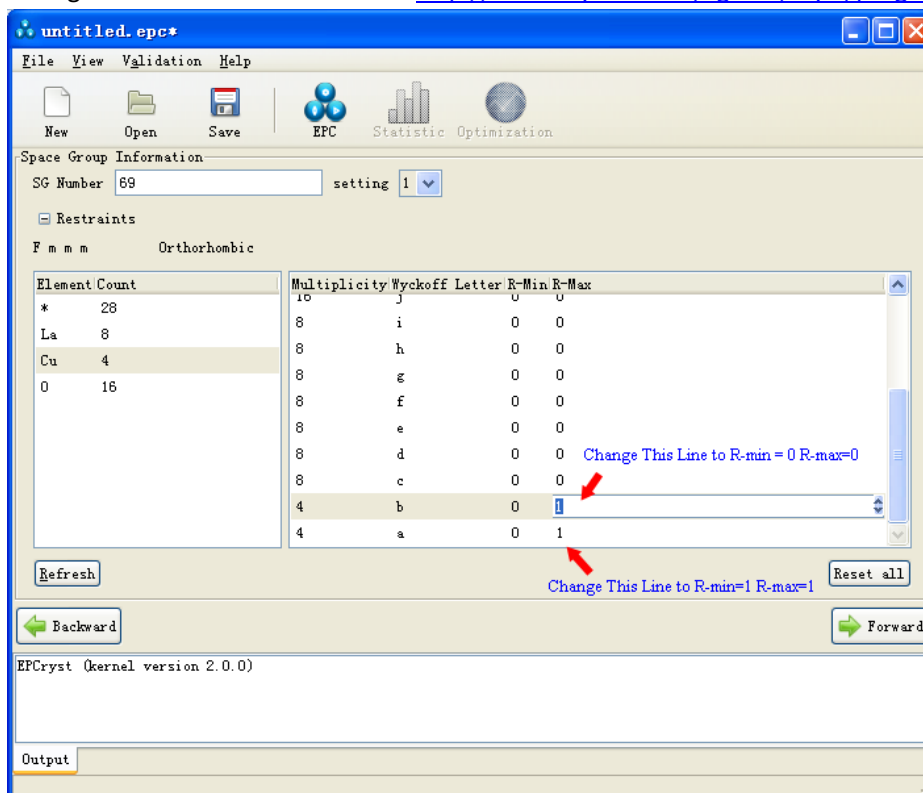
Formula Z: Formula units in the unit cell. One can estimate the value from cell parameters and the density of the compound.

Element List: Set the oxidation for each element in this list.

Click "Forward" to go to next step.



(3) Set Space group information. EPCryst use the same conventions as the *International Tables for Crystallography*. One can check here for a full list of Space group settings: <http://www.cryst.ehu.es/cgi-bin/cryst/programs/nph-wp-list>.



SG Number: Space group number. Press Enter to refresh Wyckoff position list.

Setting: Space group setting.

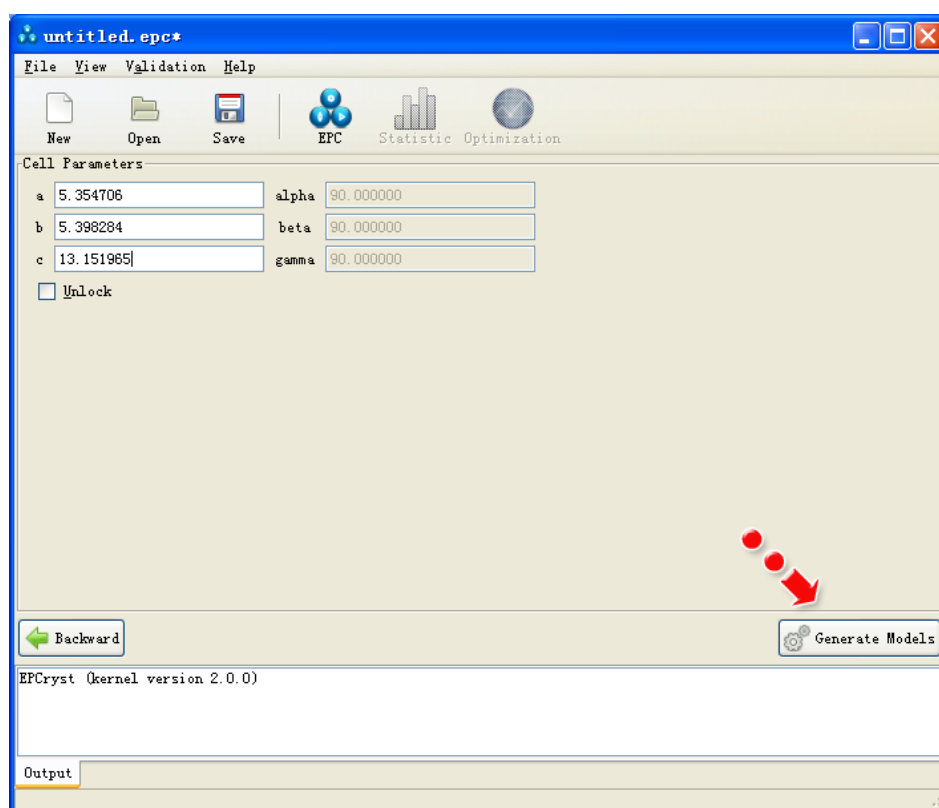
Restraints: Control the population of the generated models.

Select 'Cu' in the element list and change both the R-min and R-max value in Wyckoff position 'a' to 1 and position 'b' to 0. For this setting, only combinations with 'Cu' in position 'a' will be generated.

Element	Count	Multiplicity	Wyckoff	Letter	R-Min	R-Max
*	28	16	j	U	U	
La	8	8	i	0	0	
Cu	4	8	h	0	0	
		8	g	0	0	
		8	f	0	0	
		8	e	0	0	
		8	d	0	0	
		8	c	0	0	
		4	b	0	0	
		4	a	1	1	

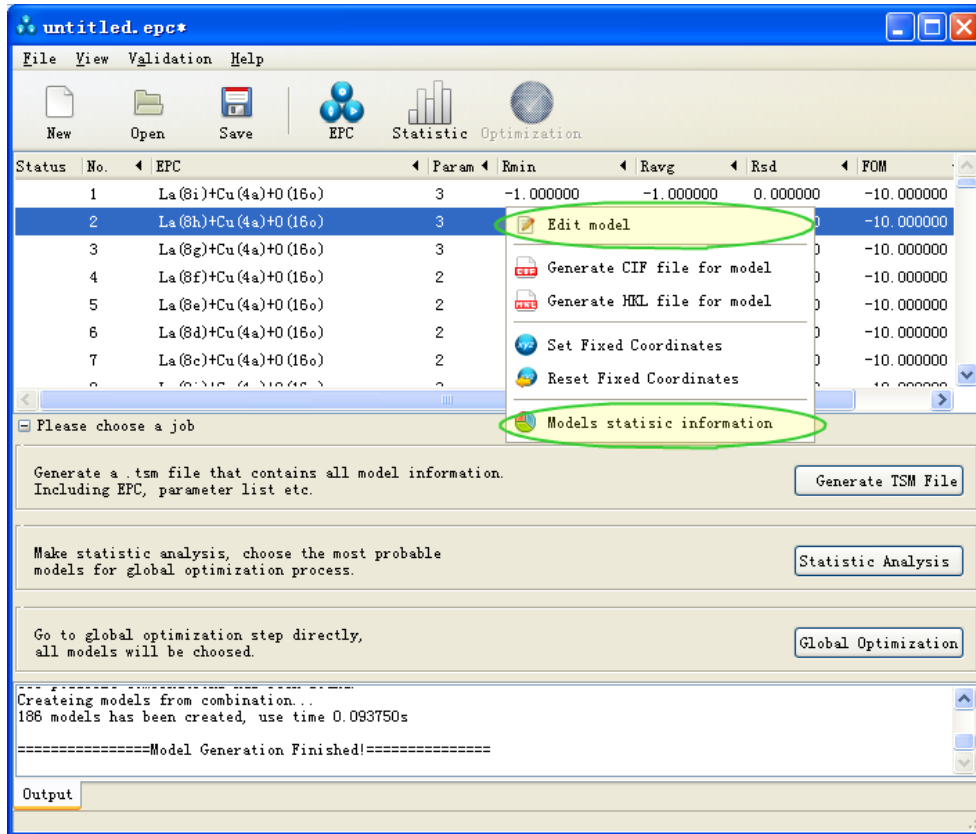
Click "Forward" to go to next step.

(4) Set cell parameters, click 'Generate' to generate models.



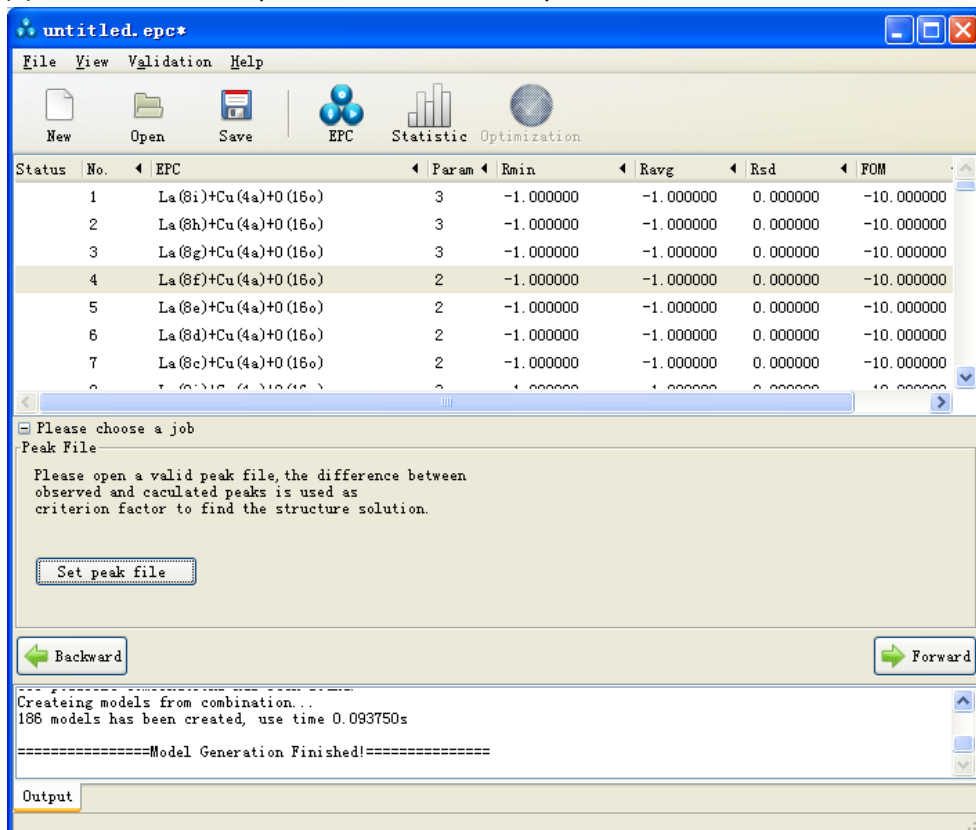
When it takes a long time to generate models or the number of generated model is very large. One should consider editing the restraints to reduce the generated models. If the problem still exists, it may mean that EPCryst is not suitable for this problem.

When the model generation process finished, we will come to the model list page. Right click on a selected model, on the popup menu, you can view and edit the detailed information of the model ("Edit Model") or view summation of all models ("Models Statistic Information"). If the "3D view" window is open (You can open it under "View"->"3d view window"), you can see the 3D plot of the selected model.

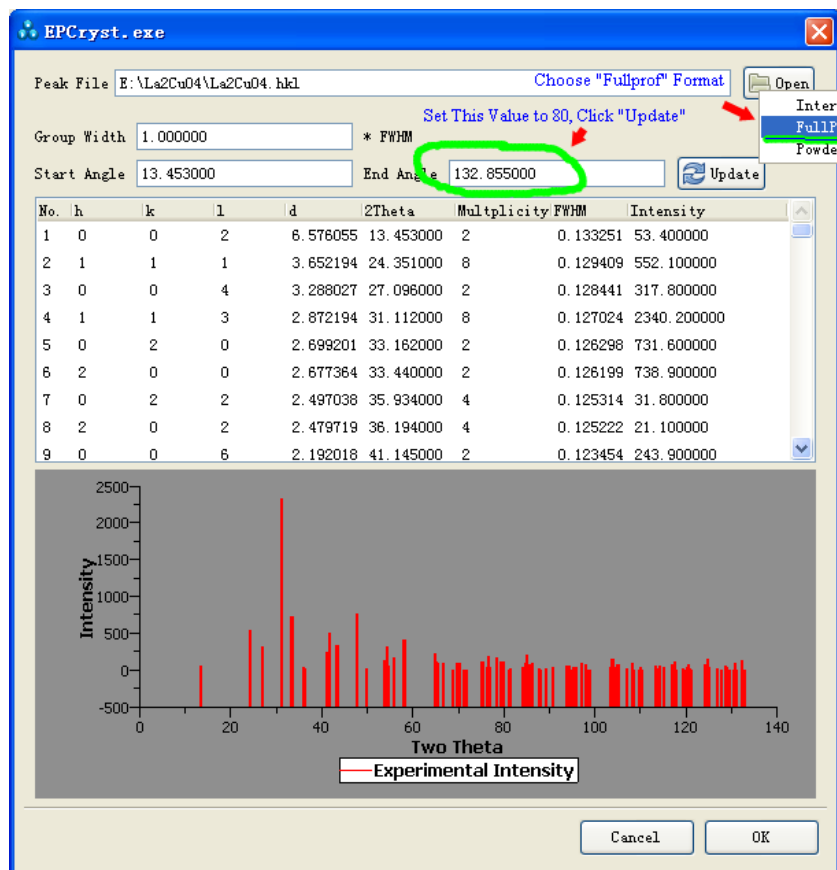


Step 4: Solve Crystal structure

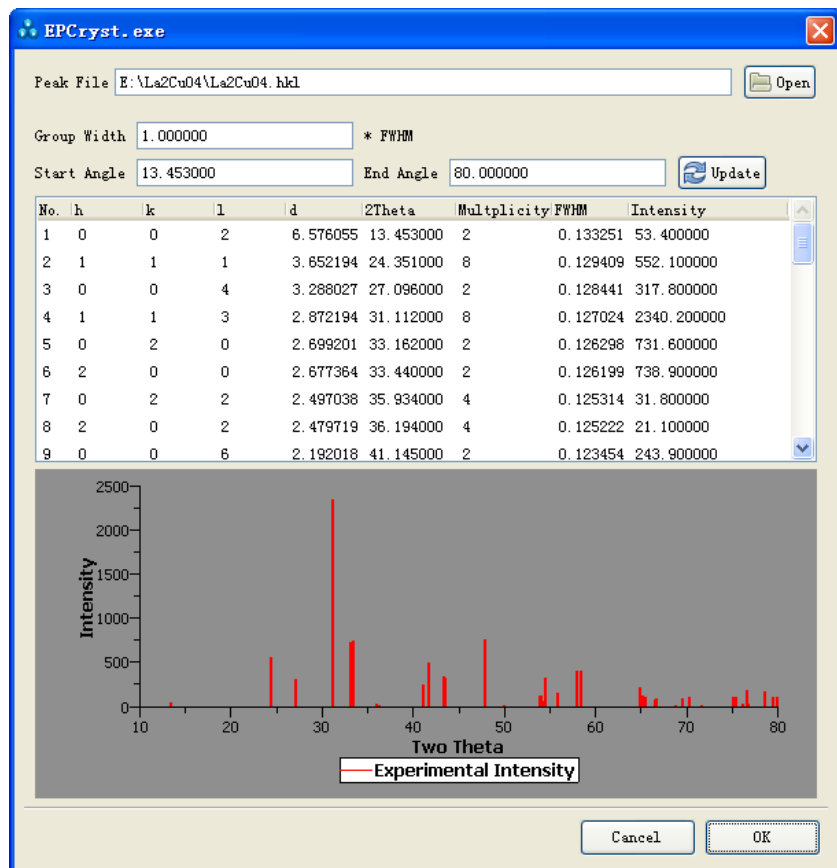
(1) Click "Statistic Analysis" to do statistical analysis.



(2) Set peak file: Choose "Fullprof" format and choose the "La2CuO4.hkl" file obtained at step 1.

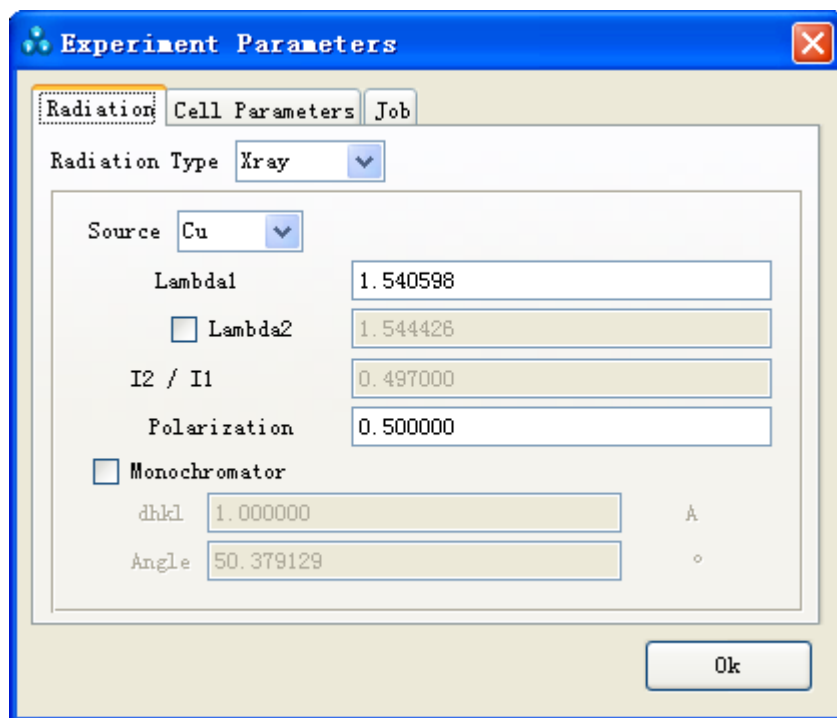


Set "End Angle" to 80 (2θ) degrees, Press "Update".



If the "Peaks" window and "Factors Plot" window are open, you will see the changes in these windows.

(3) Click "Ok" and "Forward" to go to next step. Set the experiment parameters.



Radiation: Radiation type.

Cell parameters: Cell parameters.

Job: Job control information.

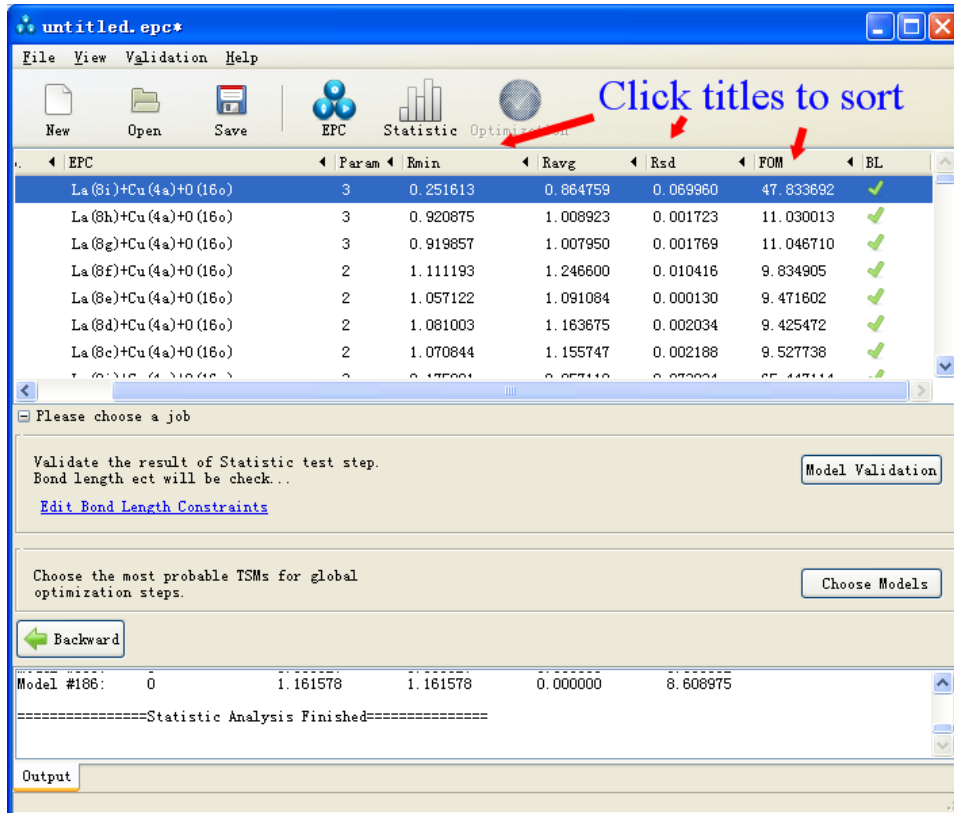
Leave all the values in these fields unchanged.

(4) Click "Ok" and "Forward" to go to next step.

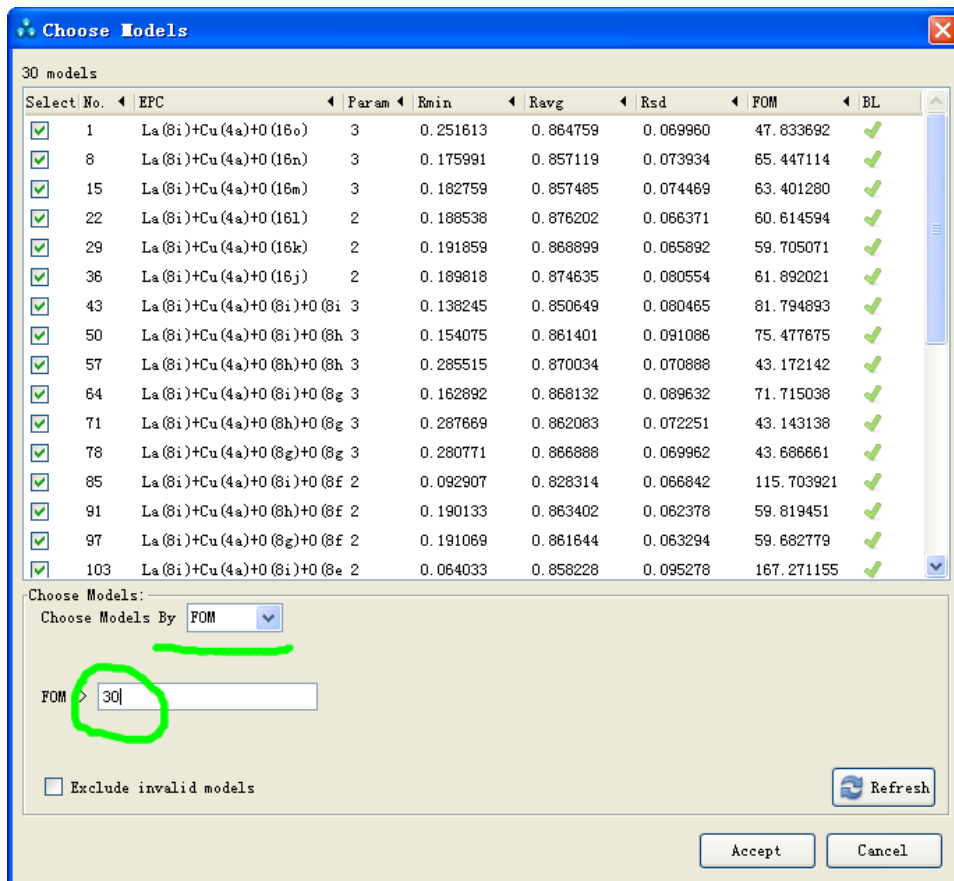
Set the number of Monte Carlo tests, check "auto" to make the program set the numbers automatically. Click Statistic to do the analysis.



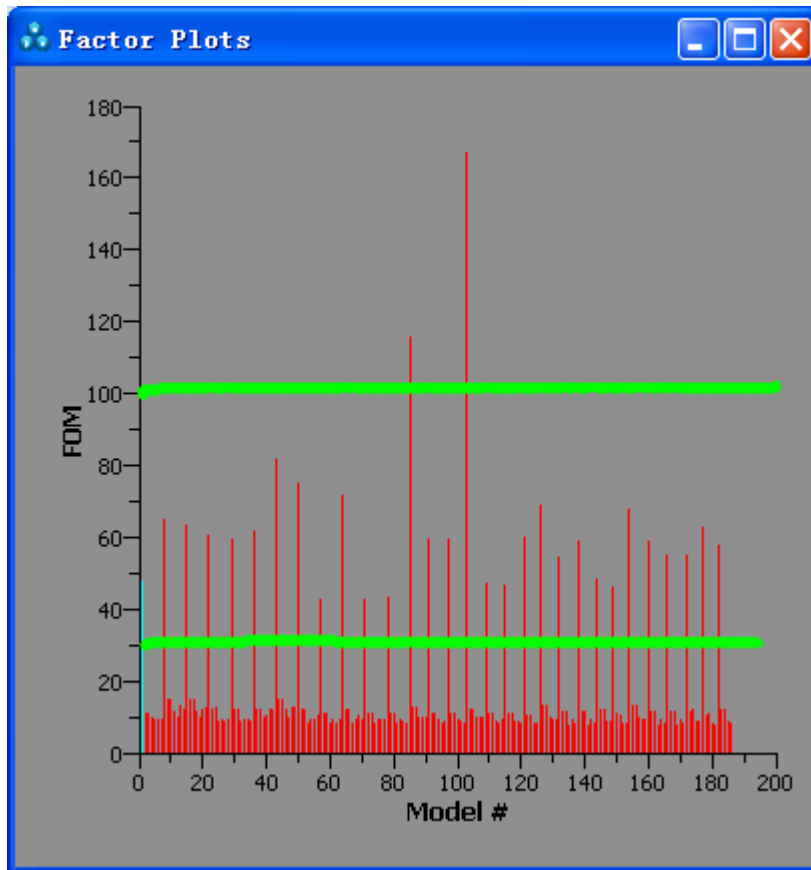
When the test finished, you can view and edit model details in the right click popup menu. You can also click the titles to sort models.



(5) Click "Choose Models" to choose models for global optimization.



We will choose model by "FOM" factor, the threshold can be set according to the FOM plots.



Here we choose 30.0. Press Enter or click "Refresh" to see the selected models. Choose "Accept" to go to next step.

(6) Global optimization.

Status	NO.	EPC	Param	Rmin	Progress	BL
	1	La (8i)+Cu (4a)+0 (16o)	3	0.251613	0 %	✓
	8	La (8i)+Cu (4a)+0 (16n)	3	0.175991	0 %	✓
	15	La (8i)+Cu (4a)+0 (16m)	3	0.182759	0 %	✓
	22	La (8i)+Cu (4a)+0 (16l)	2	0.188538	0 %	✓
	29	La (8i)+Cu (4a)+0 (16k)	2	0.191859	0 %	✓
	36	La (8i)+Cu (4a)+0 (16j)	2	0.189818	0 %	✓
	43	La (8i)+Cu (4a)+0 (8i)+0 (8i)	3	0.138245	0 %	✓
	50	La (8i)+Cu (4a)+0 (8i)+0 (8h)	3	0.154075	0 %	✓

Control Parameters
Global optimization
Set up global optimization parameters, global optimization methods, control parameters and extra options.

Global optimization

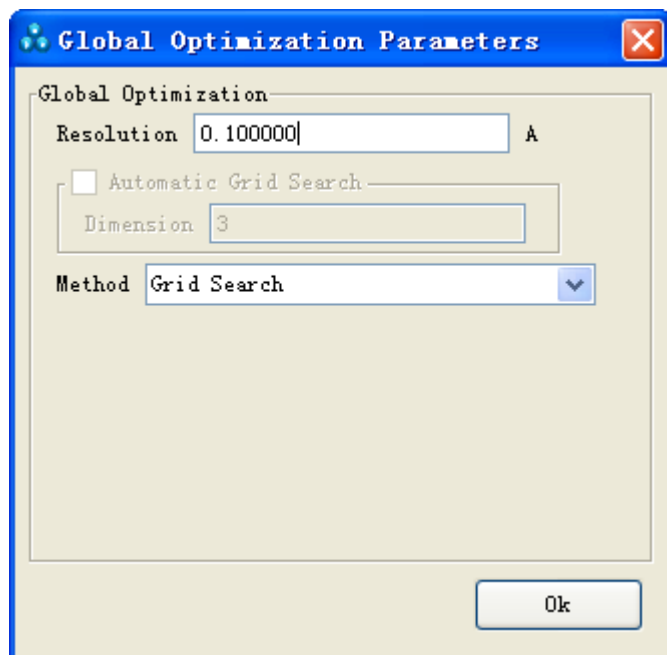
Backward Forward

Model #188: 0 1.161578 1.161578 0.000000 8.608975

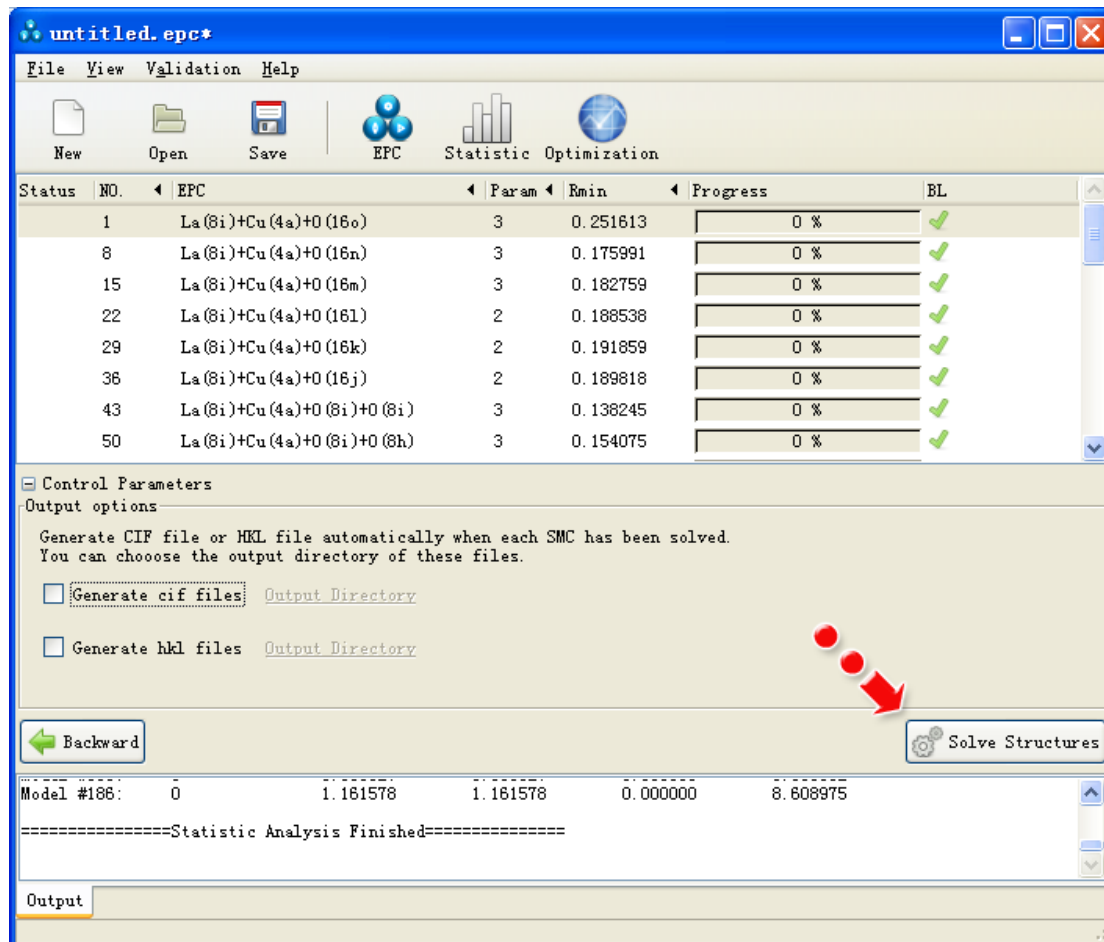
====-Statistic Analysis Finished-====

Output

The parameter counts of the selected model are in the range of 0~3, we will use the "Grid search" method to find the final solution. The searching resolution is set to "0.1" angstrom first to do a fast search. We will select the most possible solution models according the fast search result, and do a finer search for the reselected models.



(7) Click "Ok" and "Forward" to go to next step.



Click "Solve Structures" to do fast search for the selected models.

The screenshot shows the EPC software interface with the following components:

- Model List:** A table with columns for Status, NO., EPC, Param, Rmin, Progress, and BL. Model 1 is highlighted in blue and shows 100% progress.
- Control Parameters:** A section with checkboxes for "Generate cif files" and "Generate hkl files", both currently unchecked.
- Factor Plots:** Two sub-plots showing "Bragg Factor" vs "Step" (0-2800) and "Best Bragg Factor" vs "Step" (0-2000).
- 3D View:** A 3D ball-and-stick model of a crystal structure.
- Peaks:** A plot of Intensity vs Two Theta, comparing observed (blue) and calculated (yellow) peaks.

It may take some time to finish this step. When this process finished (You can also use the right click popup menu to generate *.cif files), we will choose models for finer search.

The screenshot shows the EPC software interface after solving several models. The model list is updated with the following data:

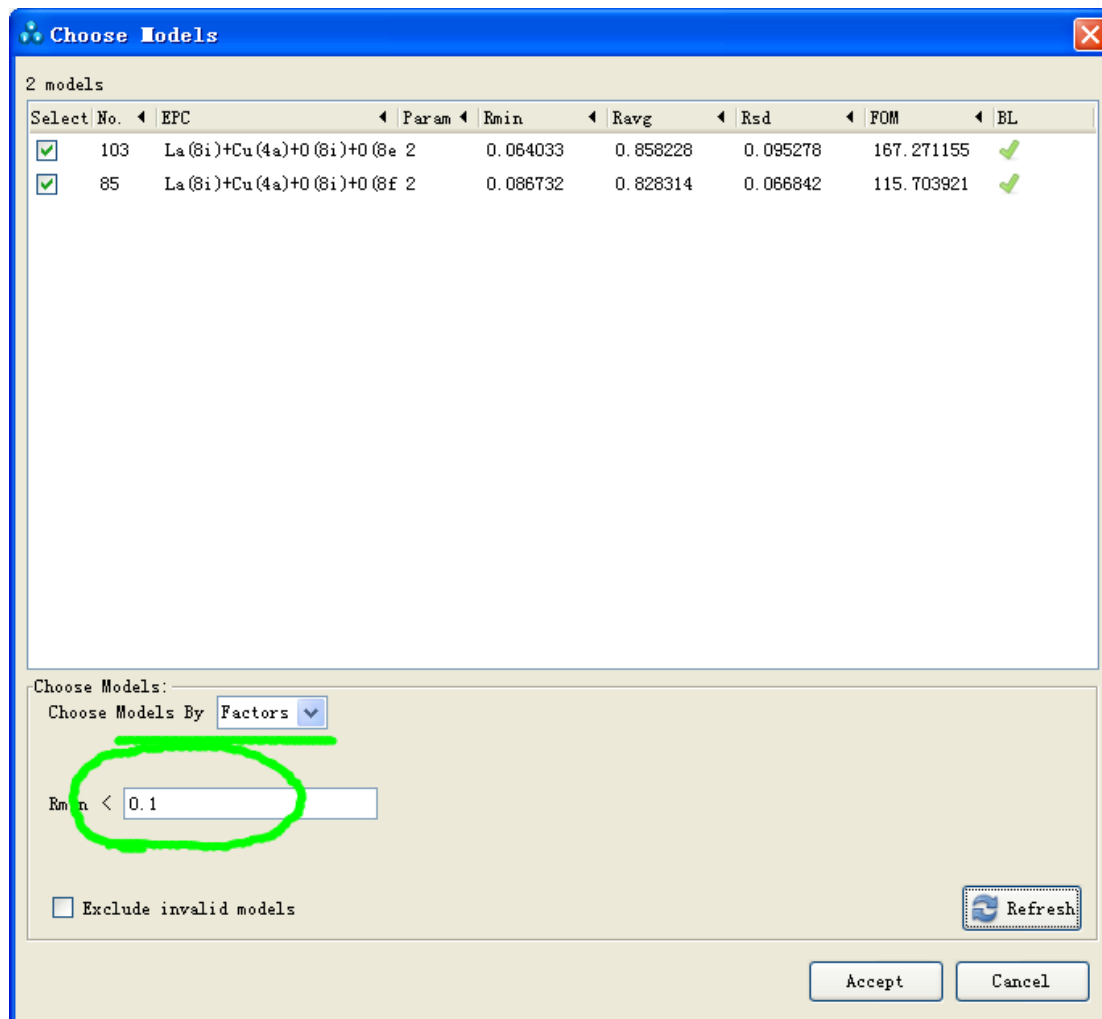
Status	NO.	EPC	Param	Rmin	Progress	BL
✓	144	La(8i)+Cu(4a)+0(8f)+0(8d)	1	0.206568	100 %	✓
✓	149	La(8i)+Cu(4a)+0(8e)+0(8d)	1	0.195495	100 %	✓
✓	154	La(8i)+Cu(4a)+0(8i)+0(8c)	2	0.167893	100 %	✓
✓	160	La(8i)+Cu(4a)+0(8h)+0(8c)	2	0.194400	100 %	✓
✓	166	La(8i)+Cu(4a)+0(8g)+0(8c)	2	0.209308	100 %	✓
✓	172	La(8i)+Cu(4a)+0(8f)+0(8c)	1	0.200440	100 %	✓
✓	177	La(8i)+Cu(4a)+0(8e)+0(8c)	1	0.185850	100 %	✓
✓	182	La(8i)+Cu(4a)+0(8d)+0(8c)	1	0.206265	100 %	✓

Control Parameters section includes:

- Buttons for "Model Validation" and "Refine Models". A red arrow points to the "Refine Models" button.
- Text: "Validate the result of the solved TSMs. Bond length ect will be check...".
- Text: "Continue structure solving, choose the TSMs for solving use finer parameters."
- Buttons for "Backward" and "Output".

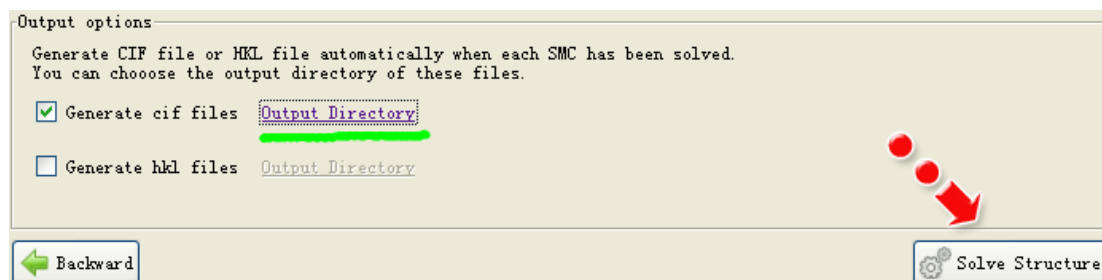
Output window shows: "Model 182 has been solved. Best Bragg factor 0.206265, use time 0.031250s. Total time used: 213.984s. Structure solving finished!"

Select models by "Factors" (Rmin), the threshold can be set according to the search results (Click titles to sort). We set the value to "0.1" here. Click "Refresh" to see the selected models.

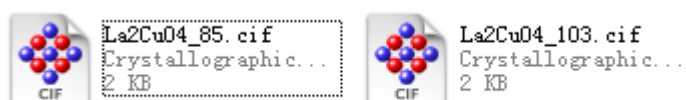


Click "Accept" to go to next step. This time we will set the searching resolution to "0.01" angstrom.

Click "Forward" and check the "Generate cif files" option. Set the output file directory.



Click "Solve Structures". The generated cifs files are in the "Output Directory" folder.



When finished, we can see that the two models almost have the same "Rmin" factors.

Status	NO.	EPC	Param	Rmin	Progress	BL
✓	103	La(8i)+Cu(4a)+O(8i)+O(8e)	2	0.059971	100 %	✓
✓	85	La(8i)+Cu(4a)+O(8i)+O(8f)	2	0.075350	100 %	✓

Most of the time these models are equivalent models, and for this example, we can identify the correct solution by bond length validation.

Click "Model validation" button, we can see that the contents of "BL" columns change.

Status	NO.	EPC	Param	Rmin	Progress	BL
✓	103	La(8i)+Cu(4a)+O(8i)+O(8e)	2	0.059971	100 %	✓
✓	85	La(8i)+Cu(4a)+O(8i)+O(8f)	2	0.075350	100 %	✗

Choose Model "No. 85" and right click on the item, in the popup menu choose "Edit Model". Expand the "Details" and check the "Hide valid Bond Lengths", we can see that 'O' and 'O' atoms are too close to each other.

Model Information
✕

Model #

EPC

Parameter Count

Atom	Oxidation	Wyckoff	Position	Fix	X	Fix	Y	Fix	Z
La	3	8i		<input type="checkbox"/>	0.000000	<input type="checkbox"/>	0.000000	<input type="checkbox"/>	0.362683
Cu	2	4a		<input type="checkbox"/>	0.000000	<input type="checkbox"/>	0.000000	<input type="checkbox"/>	0.000000
O	-2	8i		<input type="checkbox"/>	0.000000	<input type="checkbox"/>	0.000000	<input type="checkbox"/>	0.184003
O	-2	8f		<input type="checkbox"/>	0.250000	<input type="checkbox"/>	0.250000	<input type="checkbox"/>	0.250000

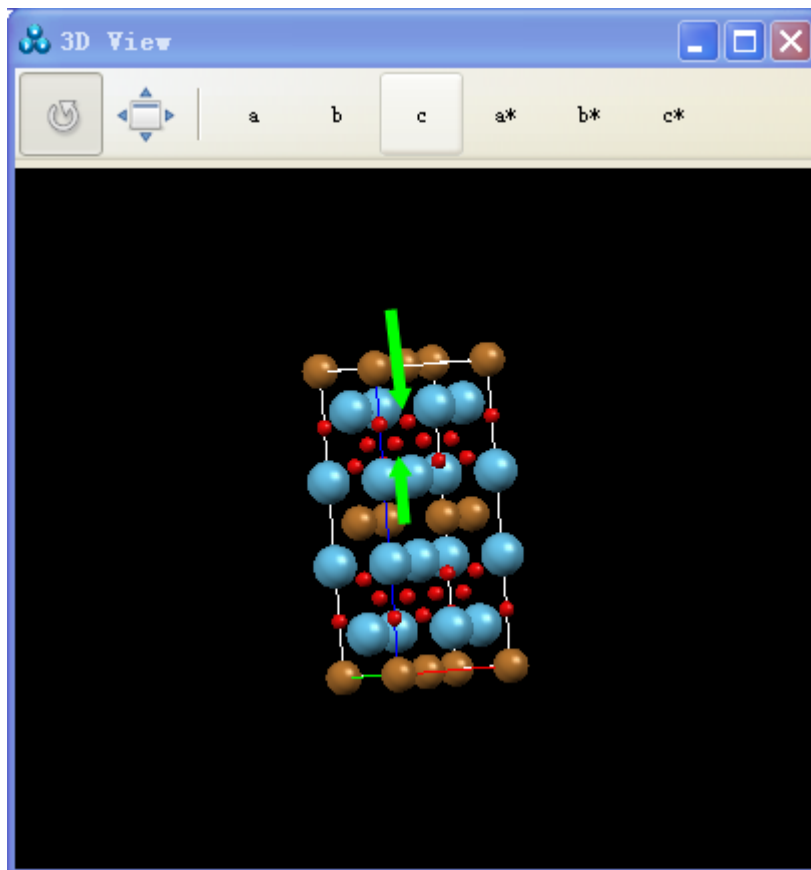
Model validation

Bond Length ✗

Validity	Element1	Oxidation1	Element2	Oxidation2	Bond Length
✗	O	-2	O	-2	2.089690

Hide Valid Bond Lengths

This is also can be seen directly in the 3D view window:



The result structure solution should be model No. 103, the generated cif file can be found in the cif output directory.

Step 5: Refinement.

Do a structure refinement with the obtained cif file. Details are not discussed here.