

TUTORIAL EXPO: SOLUTION BY DIRECT METHODS

The **Solution by Direct Methods** folder contains:

- **Example of default correct solution (Default folder)**
- **Example of correct solution obtained by exploring all the Direct Methods trials (Alltrials folder)**

• Default folder

It contains: **LaTi.exp** [the input file for the default run of *EXPO* in case of non-perovskite compound LaTi₂Al₉O₁₉, after that the cell and the space group have been determined]; **LaTi.rtv** (the file containing the experimental profile counts); **LaTi_true.cif** (the CIF file of the true model); **LaTi.pdf** (the file of the published structure).

The input file 'LaTi.exp' consists of the following lines:

```
%structure LaTi
%job LaTi
%data
cell 22.59355 10.99919 9.72968 90 98.5634 90
spacegroup C2/c
content (Al9LaO19Ti2)8
pattern LaTi.rtv
%continue
```

To run EXPO on LaTi in default way:

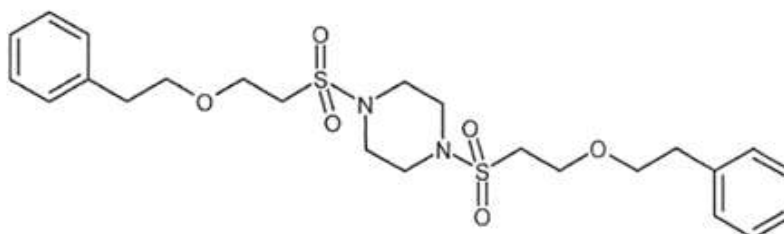
- Click on EXPO icon
- **File** in the upper Menu
- **Load & Go**
- Use 'LaTi.exp' as Input File and give the Output Filename you like (LaTi.out is the default output file name)
- **Go**
- **OK**
- Click on **Next** to go on continuously until the end of the run.

The structure model obtained at the end of the Direct Methods procedure, executed on the first set of phases (default choice), is the correct one. All the 32 atoms in the asymmetric unit are correctly located.

The obtained solution can be compared with the published fractional coordinates contained in the LaTi_true.cif file. It can be done by the graphic pathway described below (See **Comparison with an external model** paragraph).

• Alltrials folder

It contains: **piperazine.exp** [the input file for the default run of *EXPO* in case of 1,4-Bis-(2-phenethyloxy-ethanesulfonyl)-piperazine (C₂₄H₃₄N₂O₆S₂), after that the cell and the space group have been determined]; **piperazine.pow** (the file containing the experimental profile counts); **piperazine.fra** (the file of the fractional coordinates and the isotropic thermal parameters of the true model, hydrogen atoms excluded); **piperazine.pdf** (the file of the structure information published in **paper.pdf**).



The input file 'piperazine.exp' consists of the following lines:

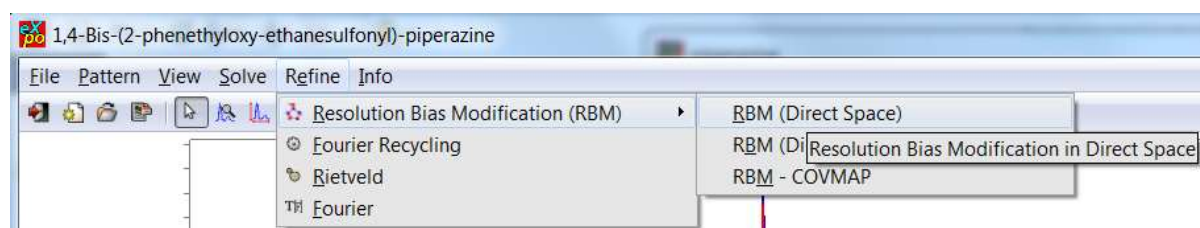
```
%Structure piperazine
%Job 1,4-Bis-(2-phenethyloxy-ethanesulfonyl)-piperazine
%Data
Cell 13.442 5.182 19.796 90 108.74 90
SpaceGroup p 21/a
Content (C24H34N2O6S2) 2
Pattern piperazine.pow
%continue
```

To run EXPO on piperazine in default way:

- Click on EXPO icon
- **File** in the upper Menu
- **Load & Go**
- Use 'piperazine.exp' as Input File and give the Output Filename you like (piperazine.out is the default output file name)
- **Go**
- **OK**
- Click on **Next** to go on continuously until the end of the run.

The structure model obtained at the end of the Direct Methods procedure, executed on the first set of phases (default choice), is not interpretable.

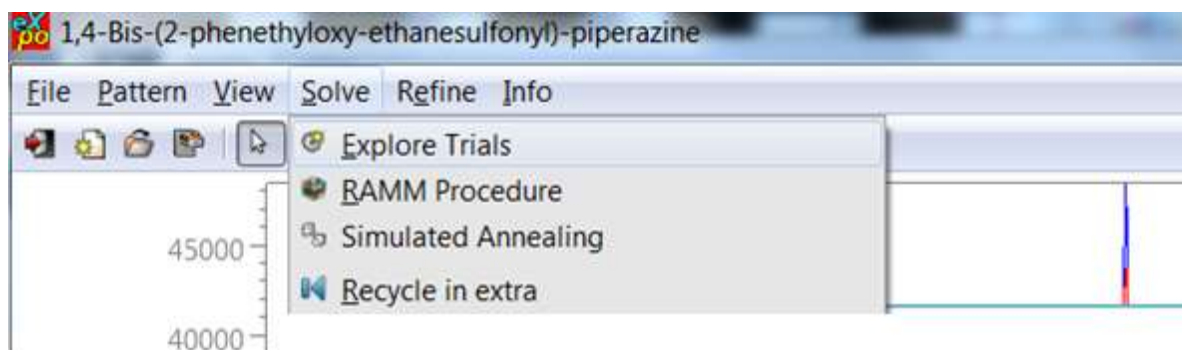
It is so rough and uninterpretable that is not advisable to try to improve it, for example, by cyclic application of RBM (RBM is advisable because the structure is organic). Indeed, by clicking on **Refine > Resolution Bias Modification (RBM) > RBM (Direct Space)** in the upper Menu,



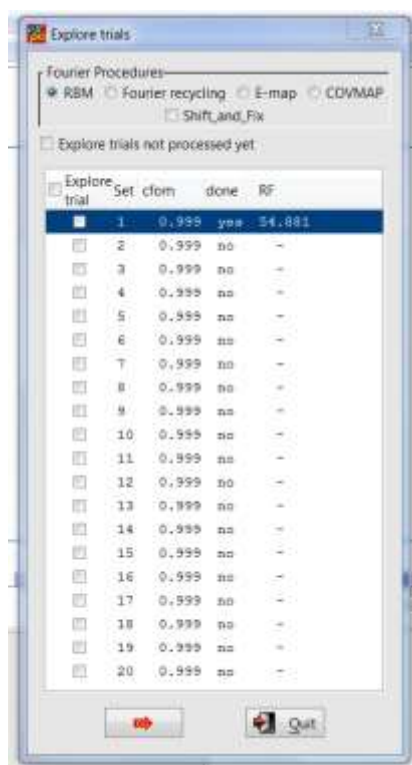
no improvement of the structure model is attained.

We can try to explore the other Direct Methods trials as follows:

Solve > Explore Trials in the upper Menu.



The following window is opened

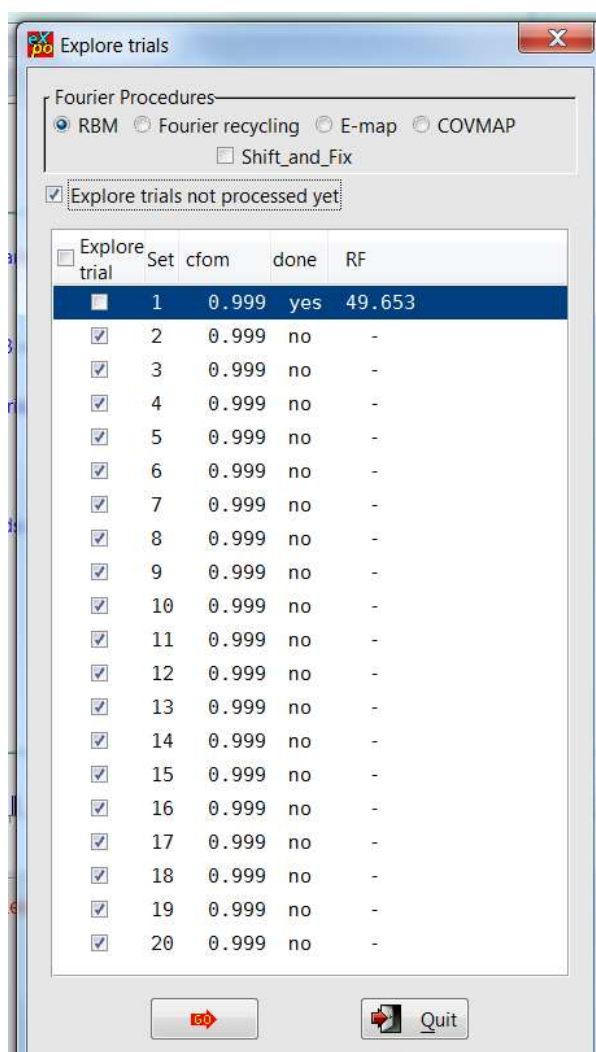


and the CFOM values, for each saved trial, can be read. The highest CFOM value could not correspond to the correct solution while subsequent different trials may be successful (in the standard run by Direct Methods only the highest CFOM figure of merit phasing trial is automatically processed).


Check the button in the first column to decide which trial will be developed and press the



button. If you check the button **Explore trials not processed yet**, all the trials not already explored will be automatically selected and developed.



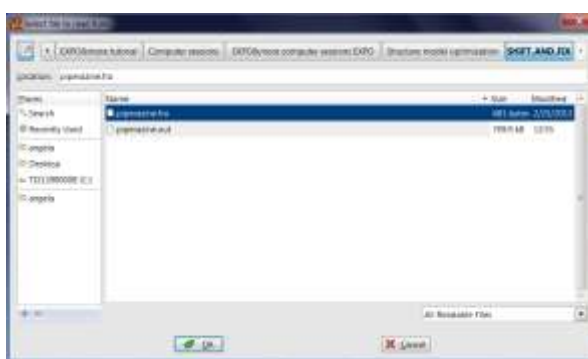
Click on **GO**.

The model first ranked by RF corresponds to the correct solution (the chemical label should be corrected: click on the Selection Mode icon  from the vertical menu of the JAV molecular viewer, left-click on the wrongly labelled atom position, right-click > Change Species and select the new label).

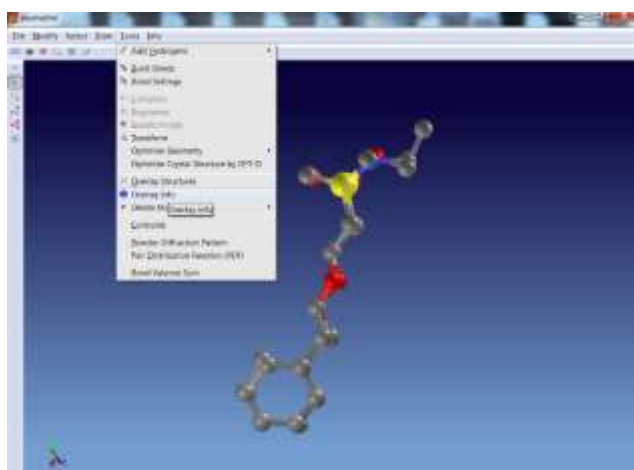
Comparison with an external model

The obtained solution can be compared with the published fractional coordinates contained in the piperazine.fra file (or LaTi_true.cif if the example of default correct solution is using). It can be done by the following graphic pathway:

Tools > Overlay structures in the upper Menu of the JAV molecular viewer and select piperazine.fra (or LaTi_true.cif if the example of default correct solution is using) and **OK**



The two models are superimposed and information on comparison can be output: **Tools > Overlay Info** in the upper Menu of the JAV molecular viewer



Overlay Into

Matches

Atom	Coordinates	Distance	Atom	xyz (pipazine.fr)	xyz (pipazine.ha)
S1	0.182 1.251 0.614	0.176	S1	0.175 1.246 0.620	
O1	0.056 1.141 0.557	0.305	N1	0.077 1.129 0.555	
O2	0.518 0.900 0.790	0.094	C5	0.518 0.892 0.789	
O4	0.437 0.839 0.717	0.452	O3	0.421 0.841 0.734	
N1	0.139 1.345 0.666	0.300	O1	0.129 1.352 0.671	
C1	0.354 1.054 0.717	0.060	C4	0.353 1.061 0.720	
C2	0.893 0.742 0.958	0.103	C10	0.893 0.738 0.952	
C3	0.262 0.998 0.647	0.273	C3	0.258 0.980 0.658	
C4	0.733 0.495 0.906	0.216	C8	0.725 0.523 0.911	
C5	0.698 0.684 0.855	0.075	C7	0.694 0.681 0.851	
C8	0.388 0.632 0.803	0.320	C6	0.386 0.651 0.796	
C7	0.247 1.411 0.567	0.499	O2	0.234 1.415 0.587	
C8	0.872 0.905 0.891	0.128	C11	0.864 0.898 0.893	
C9	0.763 0.927 0.840	0.308	C12	0.761 0.868 0.842	
C10	0.109 1.009 0.481	0.250	C2	0.104 1.024 0.482	
C11	0.013 0.934 0.577	0.308	C1	-0.005 0.873 0.572	
C12	0.819 0.543 0.968	0.168	C9	0.824 0.550 0.961	

Distance limit: 0.000 Select model: pipazine.fr

Results

Atoms in pipazine: 17
 Atoms in pipazine.fr: 17
 Matches found: 17
 Mean Phase Error: 30.260 using 463 reflections
 RMSD: 0.228
 RMSD: 0.863