MoPro and database transfer: a step by step tutorial

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September 23, 2011

## Contents

1 Introduction

This small tutorial explains how to do a simple refinement of a small-molecule structure with MoPro (Guillot, B., Viry, L., Guillot, R., Lecomte, C. & Jelsch, C. (2001). *J. Appl. Cryst.* **34**, 214-223; Jelsch, C., Guillot, B., Lagoutte, A. & Lecomte, C. (2005). *J. Appl. Cryst.* **38**, 38-54) using either a spherical atomic model or a multipolar atom model transferred from an experimental electron-density database. A link for downloading the program with test data can be found at the ЯÉCIPROCS site. Note that you have to fill in a registration form if you want to download MoPro for the first time in order to obtain a password.

2 Refinement using the Spherical Atom Model

The starting point are the cif-file gz1.cif, the hkl-file gz1.hkl, and the electron-density database file EL-MAN2.TXT. You can put the latter file anywhere you want, but for practical purposes we just put it in the data folder. The starting point is thus like this:
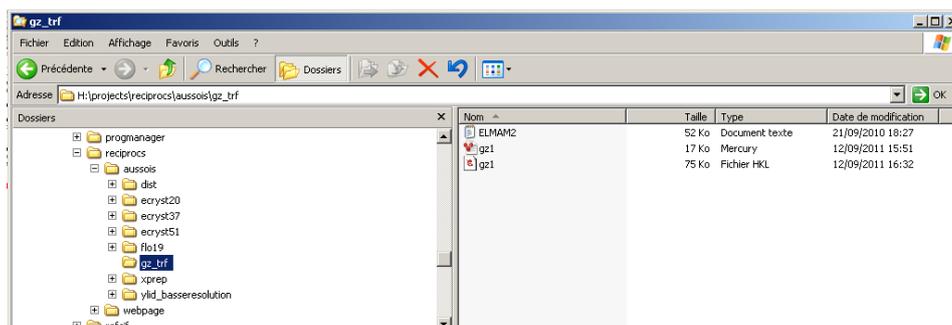

Start the MoPro interface (normally by double-clicking on the executable jar-file MoProGUI-1105.jar for windows systems), and if the program asks you for a working directory, select the directory of the gz1 files.
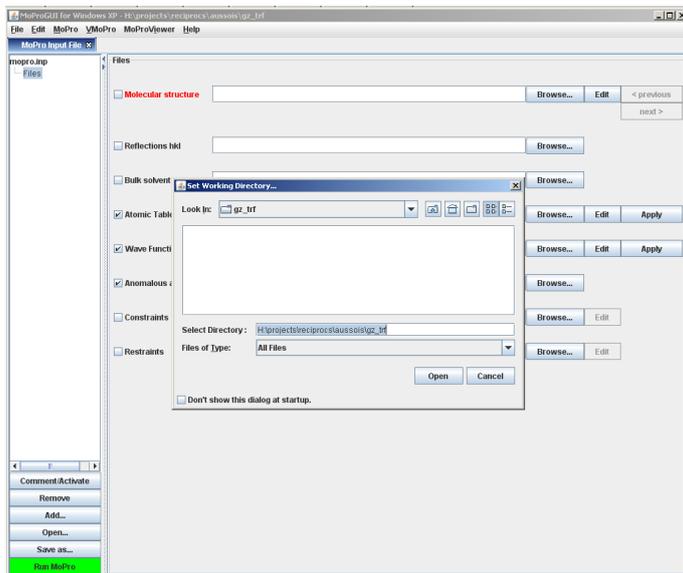
If you have not done so before, start by defining the location of a number of executable files:
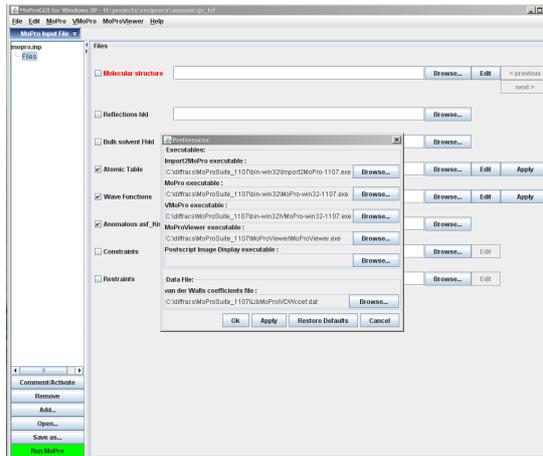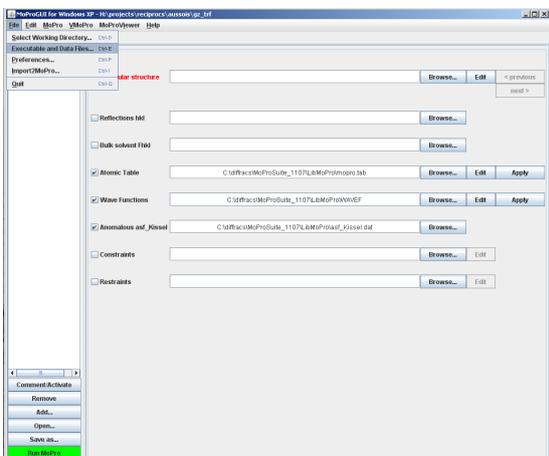

The first thing to do is to convert the cif-file (the starting point of the refinement) to a format that is readable for MoPro. For that go to File → Import2MoPro, select the file gz1.cif and a dos-box opens:
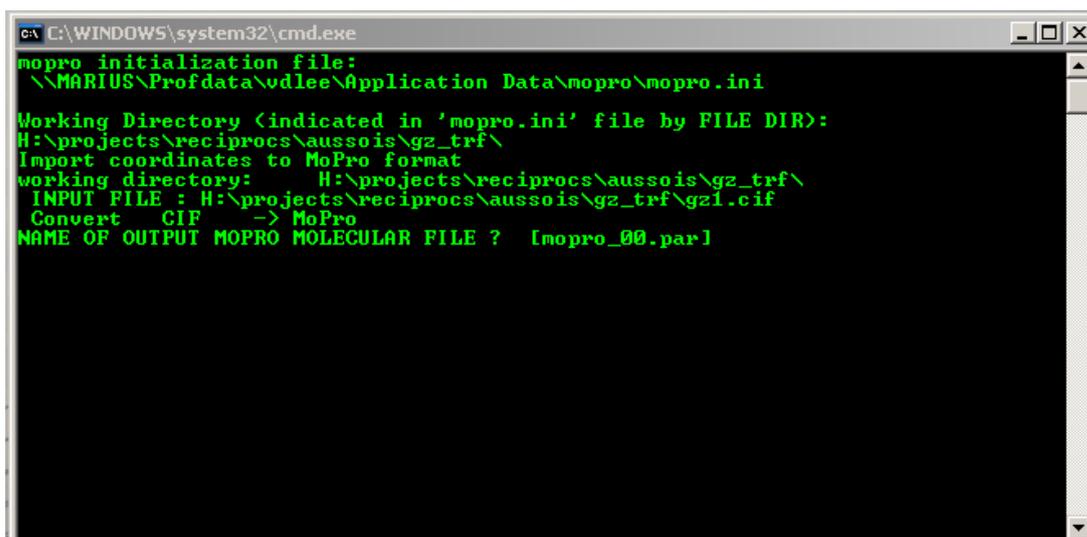
Change the default name mopro_00.par to gz1_00.par and then press <return> for all other questions. You return in the main MoPro window and now you can input your molecular structure in the upper part of the right pane. Select as well the hkl file and then the MoPro window should look like this:
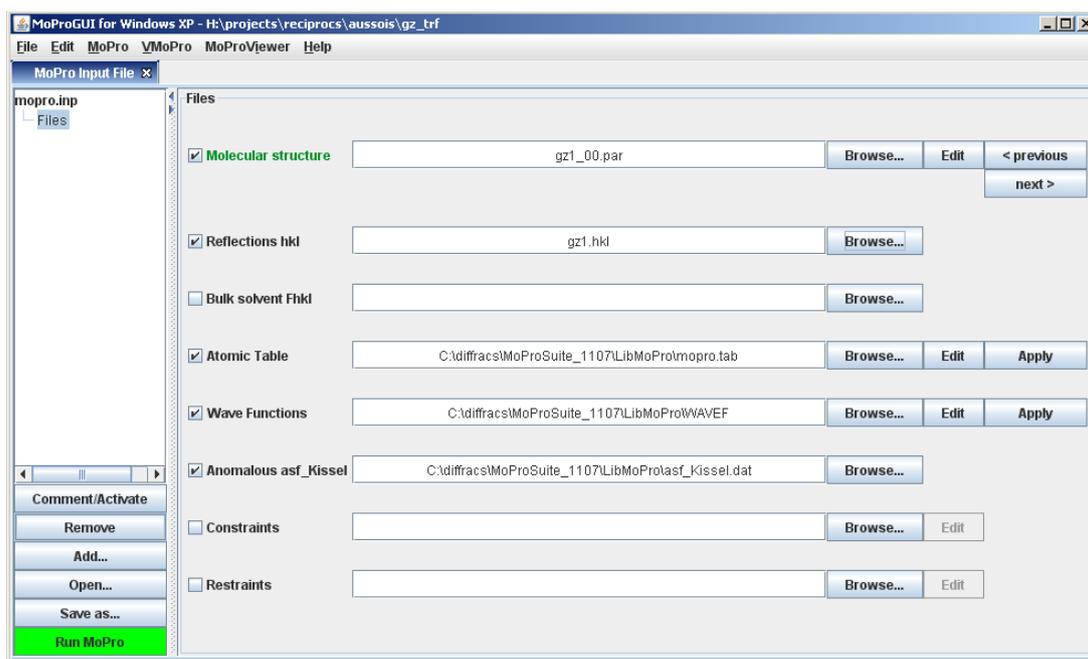
In MoPro it is important to specify the exact format of the hkl file, since not only Shelx-type HKLF4 are read. To specify the format of the file, go to Add in the left pane and and add after the selected item an 'Options tab':
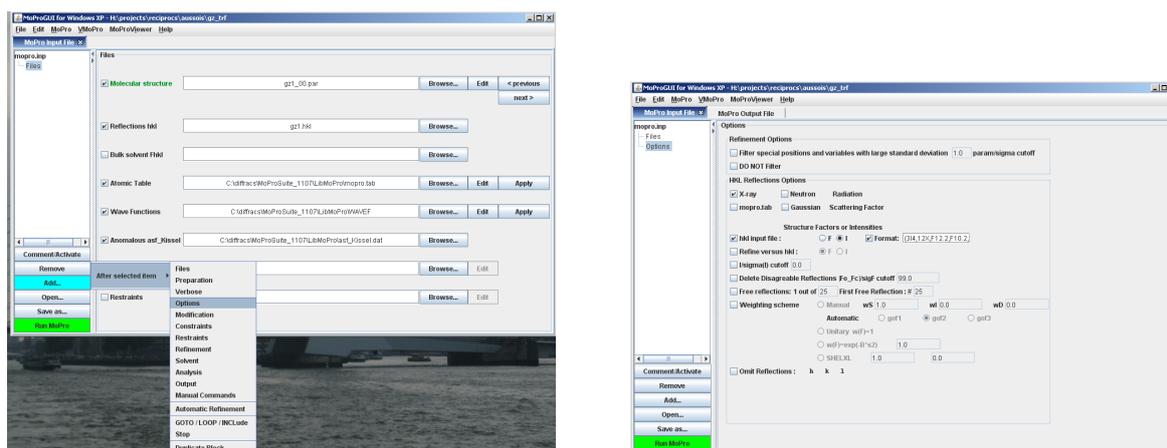

Check X-ray, hkl input file and Format. Enter in the format box '(3I4,12X,F12.2,F10.2,2X)' without the quotes. This is the Fortran-style format of the reflection lines in the reflection file. If you work with your own files, you need to change this name for sure. A typical HKLF4 file will give most probably '(3I4,F8.0,F8.0)'.

Now the constraints and/or restraints files have to be prepared. Add a Preparation tab, as was done for the 'Options' tab, and check the H-X Bond Distances and Riding Thermal Uiso check boxes. In order to be more flexible you should add as well the angles constraints. The constraints file is a text file that can be edited. Note that the constraints are equivalent to a riding motion of the protons on their parent atoms after resetting the distances to the values given in the constraint file. Note also that the parent-hydrogen distance in MoPro is set at the neutron distance (approximately 1.09 Å for a CH and 0.97 Å for an OH). This is less logical for a classical spherical atom model refinement but should absolutely be done for a refinement after transfer of the multipolar data base. The MoPro window should look now like this:
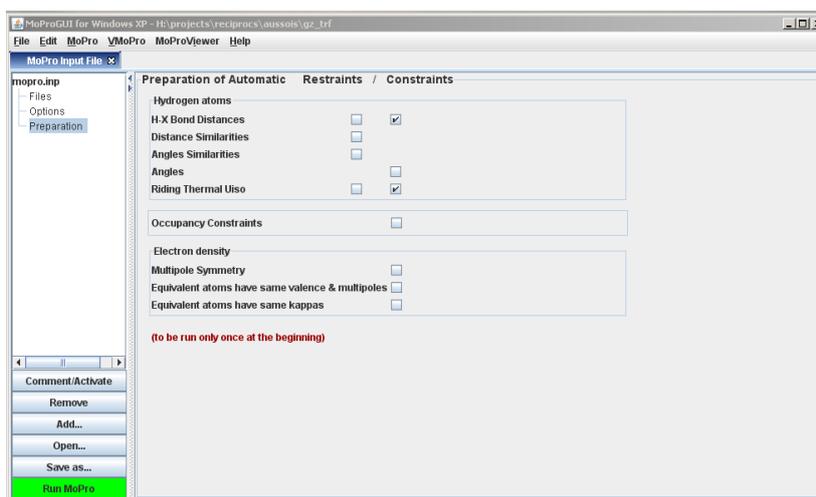
This will create a file called 'CONSTRAINT' in the working directory, when the green 'Run MoPro' in the left lower corner of the MoPro screen is pressed. To have output on your screen, answer Yes on the question whether the mopro.out file file needs to be scanned during execution. Press ok in the MoPro log window and you can check the contents in the MoPro Output File window. There is one harmless warning.
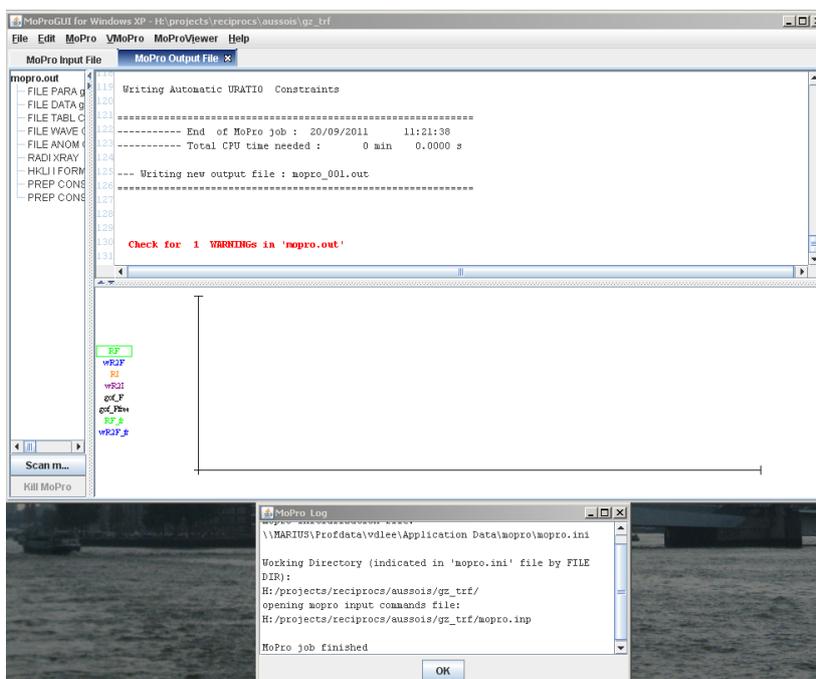

You can now remove the Preparations tab by selecting it in the left pane and clicking on Remove in the left lower part of the window. Go back to the Files tab, and browse for the CONSTRAIN file that you have just created:
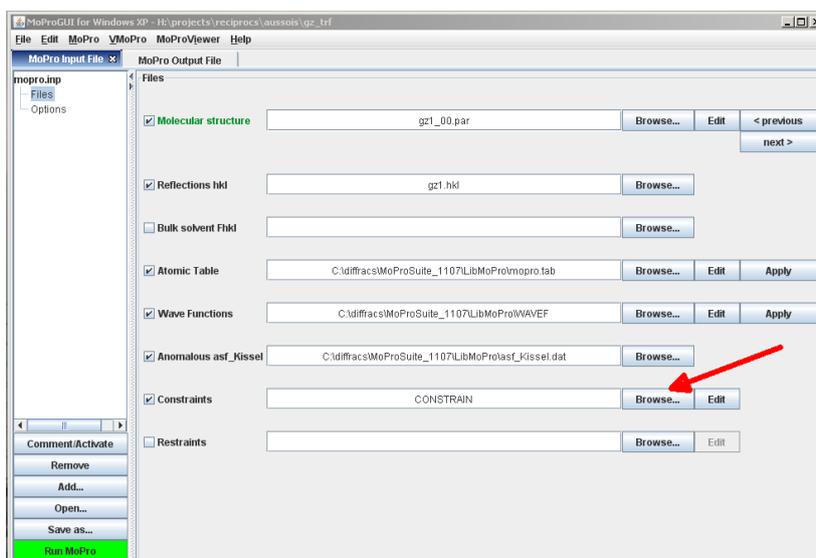
Add a Refinement tab to the list; take care to add it *after* the Options tab. Check the refinement of the scale factor and also increase the number of refinement cycles to let's say 5. The latter modification can be done in the lower part of the window:
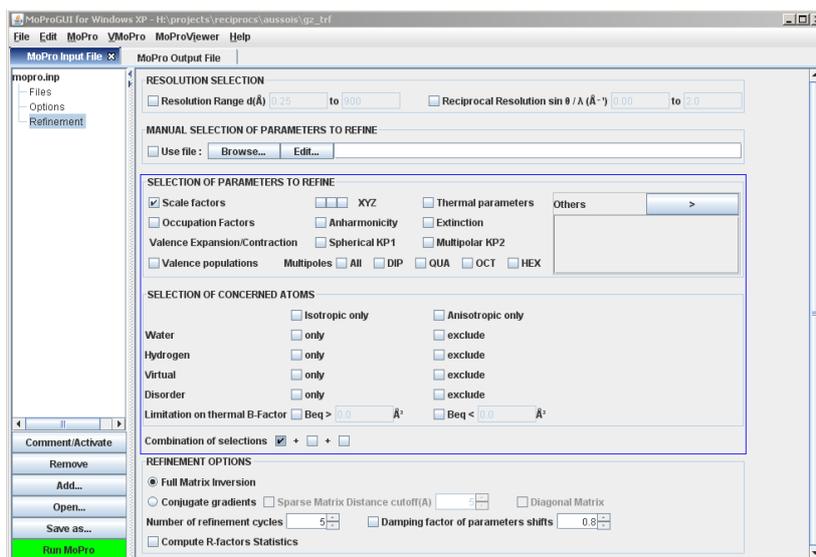

Run MoPro, and answer Yes on the question whether the existing mopro.inp file should be overwritten. The result shows that a new gz1_01.par is written and the refinement converges nicely:
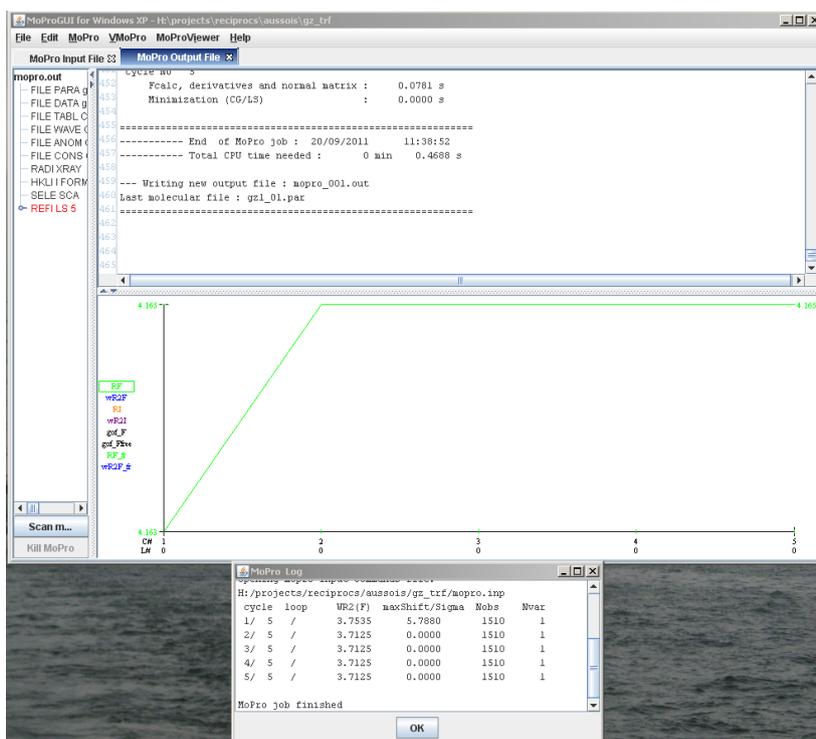
Go back to the MoPro Input File tab and therein the Files tab. For the new refinement we should select the new molecular par file. This can be simply selected by pressing next:
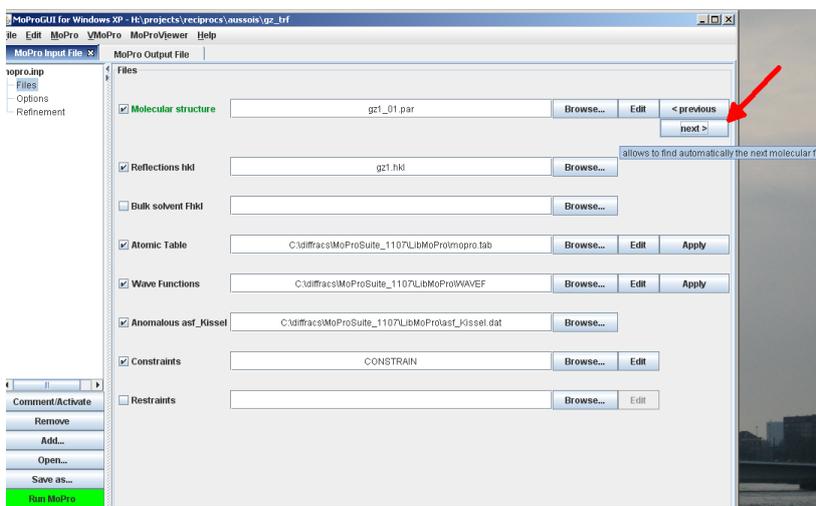

Now we want to refine the positional coordinates and the thermal factors, but not for the hydrogen atoms. Go to the Refinement tab and check xyz, thermal parameters, and the exclude checkbox for Hydrogens. If you want to let the hydrogen atoms ride on their parent atoms, then you should leave the exclude hydrogen check box unchecked. For the purpose of this tutorial we leave it checked.
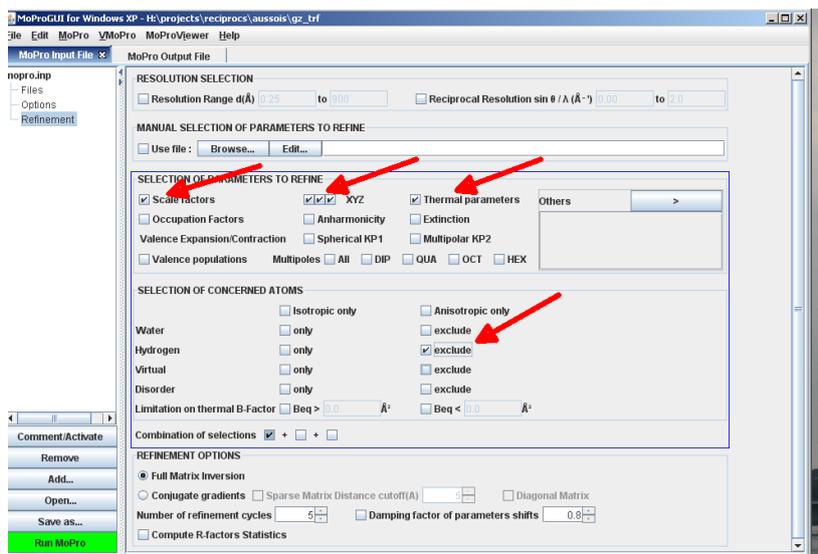
Press the green 'Run MoPro' button and let the refinement go. You should have at the end an R-factor of 3.2917.
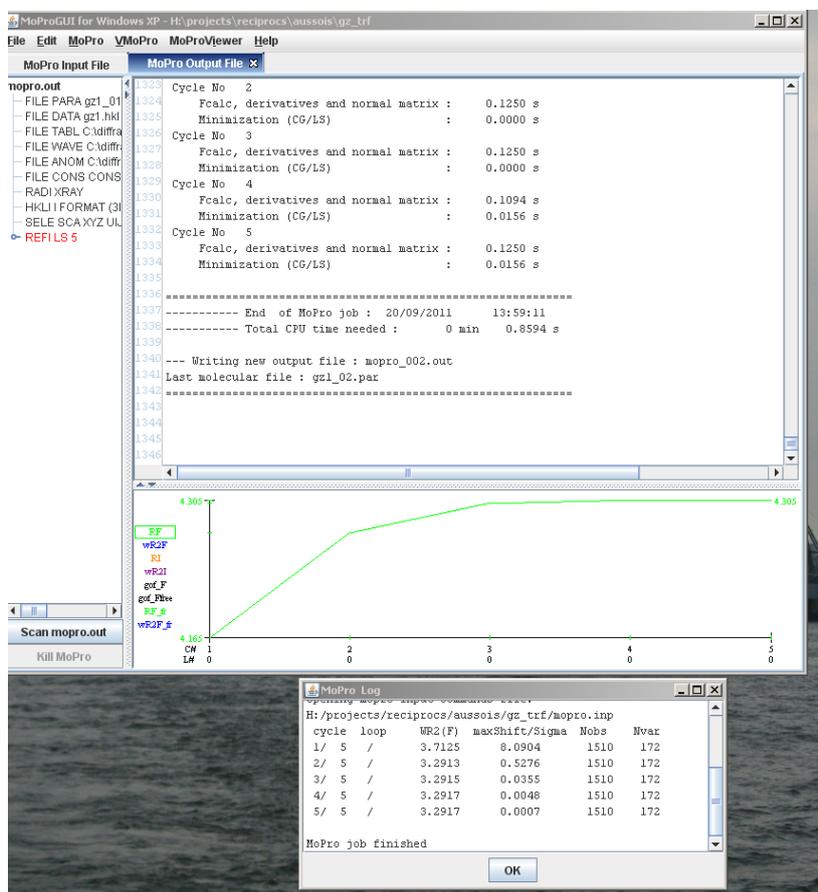
Ofcourse you can try to model better the hydrogen positions and fine-tune other options, and you should for sure inspect the difference Fourier maps with the MoProViewer. For that you need at least create the proper files by adding an 'Output' tab to the list of tabs in the upper left pane. In order to have a cif file click the cif-checkbox and if you need a Fourier file that can be visualized with MoProViewer you need to check the FOUR box.
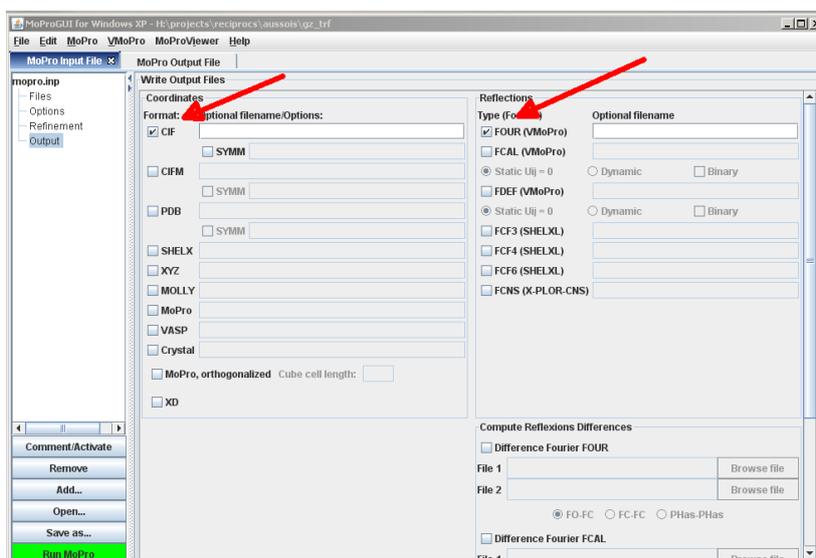
# 3 Refinement using the Multipolar Atom Model transferred from a database

For this we need to define the database by adding a 'manual commands' tab. Enter the commands displayed in the below figure:
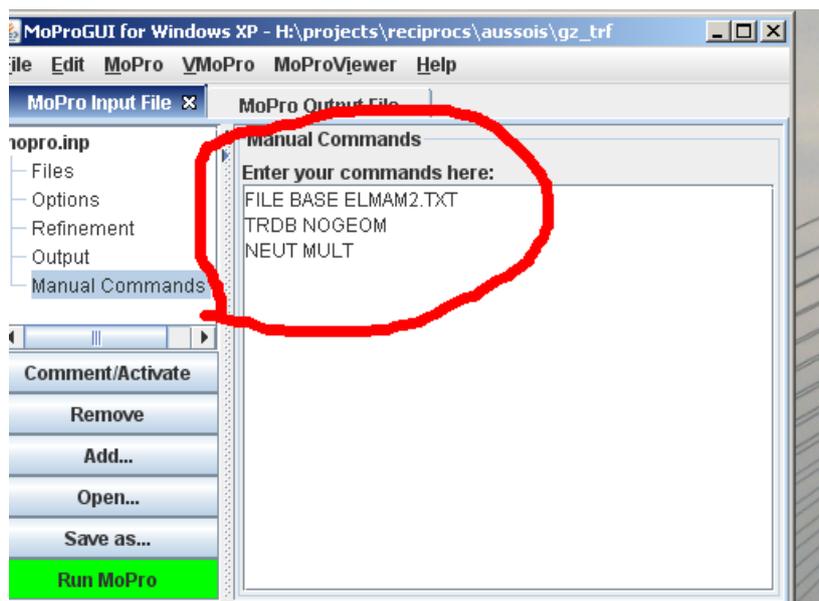
If the file ELMAM2.TXT is not in the working directory you need to specify the complete path. Note that in a futur version of MoPro there may be a GUI checkbox to click in order to transfer the database to the actual model. The transfer of the data base is for the moment not compatible with any refinement, so remove the Refinement tab, and go the last molecular model by pressing 'next' at the right of the molecular structure file name. You do need to remove the Refinement tab, if you have a very recent version of the executable of MoPro; for the Windows platform mopro-win32-1109.exe or later will do the job.
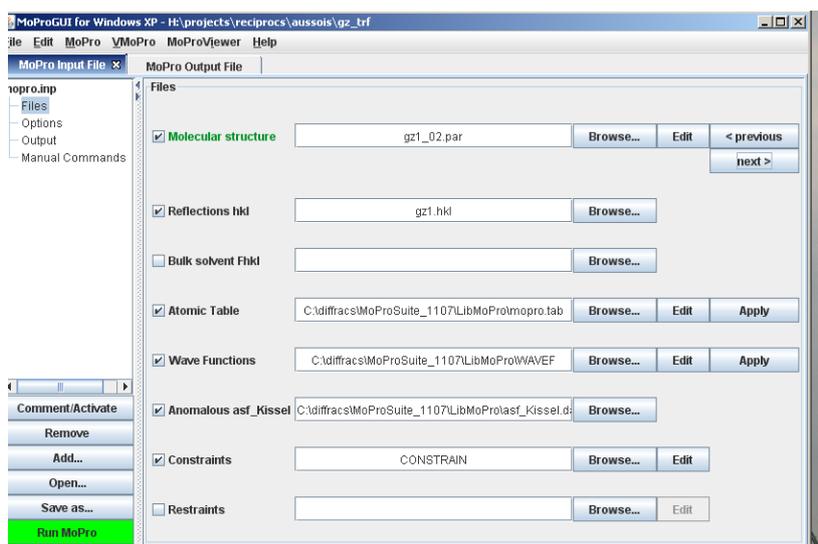
Run MoPro and check whether the transfer has taken place (see below figure). There are some warnings because we have asked to output a cif-file and MoPro cannot find any variance-covariance matrix to calculate standard deviations.
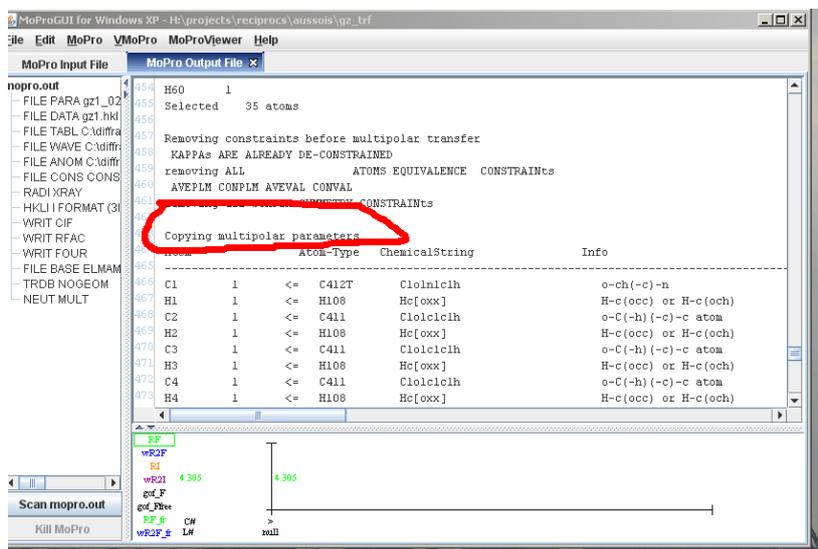
Now go in the MoPro Input File to the last molecular file (gz1_03.par) and add again a refinement tab. We remain prudent and refine first only the scale factor for 5 cycles:
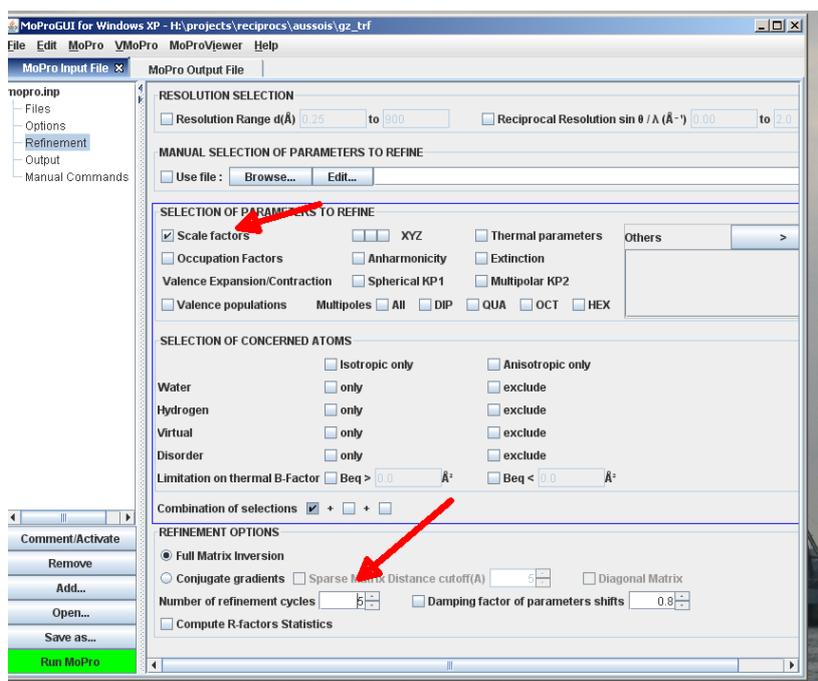
Run the refinement which should converge rapidly. Check then in the refinement tab the xyz coordinates, the thermal parameters and exclude the hydrogens. Select the latest molecular file (gz1_04.par) in the Files tab and run MoPro.
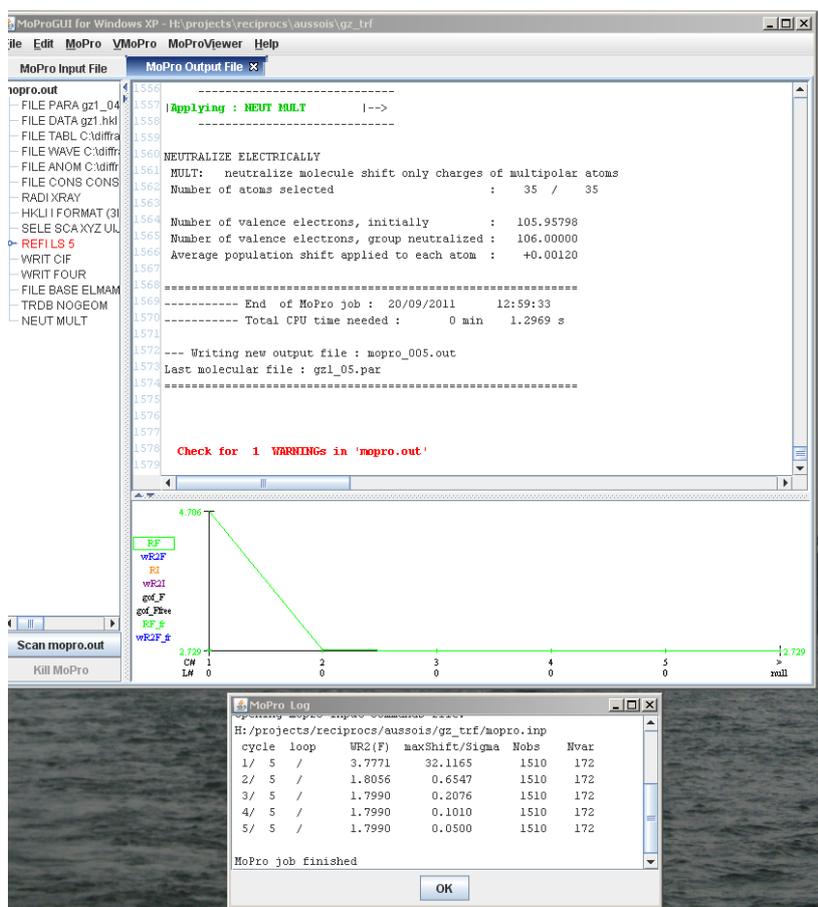
The refinement should give a large improvement with a final R-factor of 1.799. At this point you should check the difference fourier maps and compare them with those after the Spherical Atom Model refinements. Note that there are no supplementary refinement parameters compared to the earlier refinement. Try as well to include the hydrogens in riding motion or using restraints or let them completely free. There are basically

three ways to treat the hydrogens. You can use the CONSTRAIN file in the file tab, but then you can not refine the positions since the protons are reset to the geometry specified in the CONSTRAIN file; take care to exclude the refinement of the protons in the Refinement tab. Aternatively you can use the RESTRAIN file - deselect the CONSTRAIN file - and then you should include the protons for refinement in the Refinement tab. Or you use a partial or complete free refinement of the protons. For a complete free refinement you remove the RESTRAIN and CONSTRAIN file from the File tab. It may be better to leave the isotropic atomic displacement parameters for the protons restrained to the equivalent atomic displacement parameters of the parent atoms. To do this, if you have already created a RESTRAIN file, you can edit it by pressing 'Edit' on the File tab. It will open in a new tab and you delete all lines not related to PREP REST URAT (see below figure).
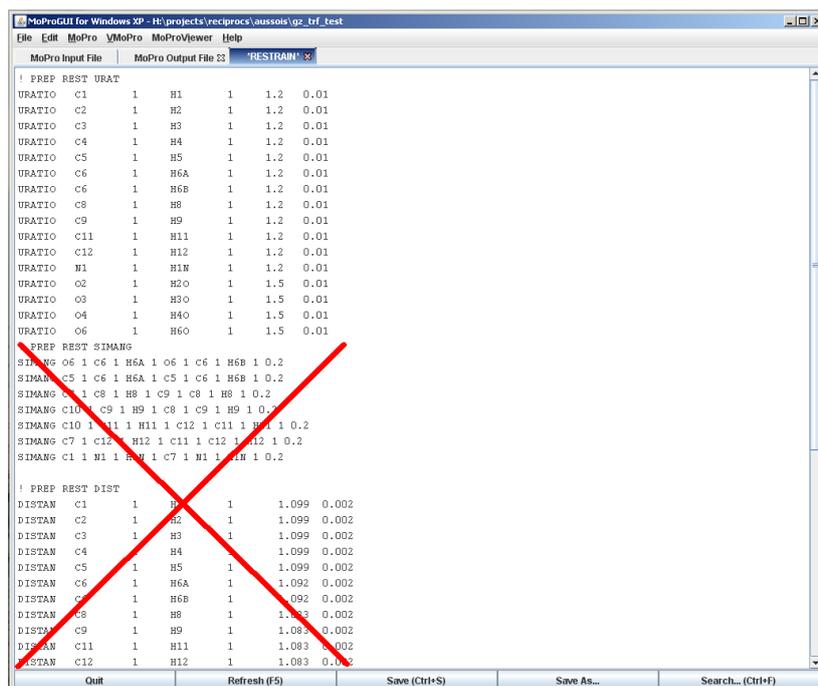
4 References and links

- The ЯÉCIPROCS website

- The MoPro website

- The main publications concerning MoPro:

  - Guillot, B., Viry, L., Guillot, R., Lecomte, C. & Jelsch, C. (2001). *J. Appl. Cryst.* **34**, 214-223
  - Jelsch, C., Guillot, B., Lagoutte, A. & Lecomte, C. (2005). *J. Appl. Cryst.* **38**, 38-54

- Experimental database transfer applications with MoPro:

  - Domagała, S, Munshi, P, Ahmed, M., Guillot, B. & Jelsch, C. (2010). *Acta. Cryst.* B**67**, 63-78
  - Ejsmont, K., Boeglin, J., Didierjean, C, Guichard, G. & Jelsch, C. (2010). *Acta Cryst.* C**66**, o292-o294