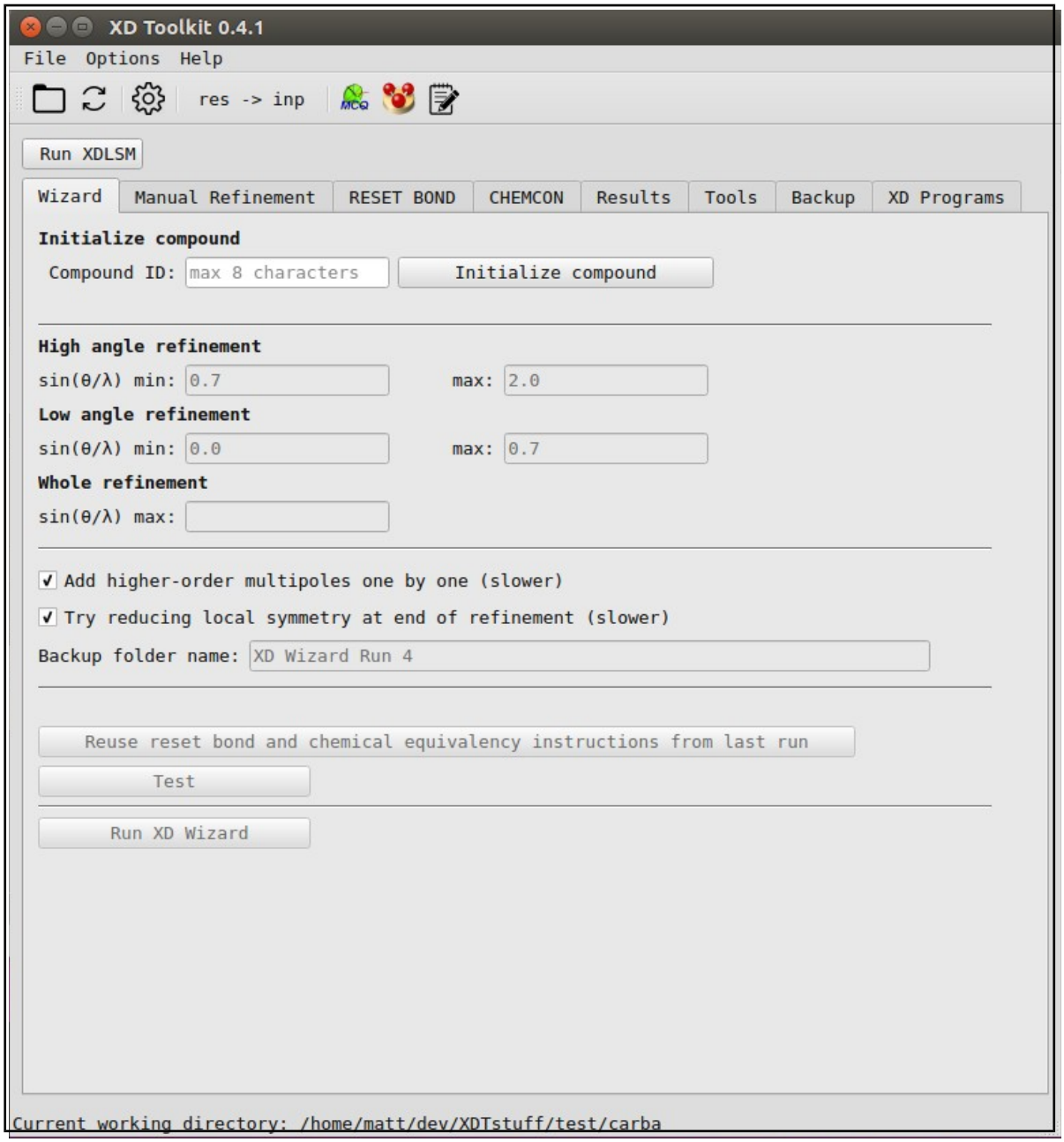
XD Toolkit Manual



General

Before you start a new refinement

1) In the folder that the SHELX structure solution was carried out, there should be a res and an hkl file.

2) Copy the res file to your project folder, and rename it “shelx.ins”.

3) Copy the hkl file to your project folder, and rename it “shelx.hkl”.

4) Alternatively, 2 and 3 can be done in XD Toolkit by clicking “File – Load Starting Files from Struture Solution Folder”. This will prompt you to first select your structure solution folder, and thenyour new project folder.

Wizard

The Wizard tab takes some user input, and then goes through an entire standard refinement strategy from start to finish.

Guide

1) Type a name for your compound (max. 8 characters) in the "*Compound ID*" box, and click initialize compound.

NOTE: If you have a large structure this may take a short while, but shouldn't take longer than a minute.

2) Enter your desired sin(θ/λ) cutoffs.

3) If you have H atoms in your structure, go to the "*RESET BOND*" tab. Click "*Automatically add reset bond instructions*".

If the program says it cannot add reset bond instructions for any H atoms, go to the "*Manually add reset bond instructions*" section, and input an appropriate bond length for these atoms.

NOTE: Clicking the "*Get other bond lengths*" button in the "*Manually add reset bond instructions*" section will take you to a paper with tables of standard bond lengths from neutron diffraction.

4) Go to the "*CHEMCON*" tab. Leave "*Automatically add chemical equivalency*" checked and click "*Add CHEMCON*".

5) Choose if you want to introduce higher-order multipoles one by one, and if you want to try lowering local symmetry at the end of the refinement, with the relevant check boxes in the wizard tab.

Both of these options are recommended, but doing them both makes the refinement strategy twice as long, so you may not wish to use one/both of them for larger structures.

6) Click "*Test*". If everything is fine then you will get the message "*Ready to run XD Wizard*" folllowed by an estimated running time. Otherwise you will get instructions telling you what else you need to do.

7) Click "R*un XD Wizard*".

Manual Refinement

This tab provides the ability to do everything that the wizard does, but with more button clicks.

Guide

1) Type a name for your compound (max. 8 characters) in the "*Compound ID*" box, and click initialize compound.

NOTE: If you have a large structure this may take a short while, but shouldn't take longer than a minute.

2) Enter your desired sin(θ/λ) cutoffs or leave the boxes blank.

3) If there are instructions below the drop down menu of refinement types, follow them.

4) Click "*Setup xd.mas*". If the program cannot setup anything, this will be displayed.

5) Make any desired additional changes to xd.mas.

6) Click "*Run XDLSM"* near the top left of the window.

7) Once XDLSM has finished running click the "*Show results"* button to view the output of the refinement.

8) Use the default backup name or choose your own and click "*Save backup*".

9) Click the "*res→inp"* button in the toolbar.

10) Repeat steps 2-9 for every refinement type in the drop down menu.

RESET BOND

Automatically add reset bond instructions:

This will automatically detect H atoms in the listed environments and add reset bond instructions with the bond lengths displayed to xd.mas.

If any H atoms are found for which the program cannot recognise the environment, their names will be displayed below the "*Automatically add reset bond instructions*" button.

Manually add reset bond instructions:

All H atom labels entered will be set to the bond length entered.

If the "*All"* box is checked, all H atoms will be set to the bond length entered.

Tables of standard bond lengths can be found by clicking "*Get other bond lengths*".

Formatting:

- Space or comma separation of atom labels are both fine, i.e. "*h1, h2, h3*" or "*h1 h2 h3*".
- Any of these atom formats are fine: *H(1) = h(1) = H1 = h1 = 1*

Disable/enable reset bond instructions:

This leaves the reset bond instructions in xd.mas, but comments them out, or uncomments them.

Delete all reset bond instructions:

Removes all reset bond instructions from xd.mas.

CHEMCON

Automatically add chemical equivalency:

This will automatically find chemically equivalent atoms and make them chemically equivalent in the CHEMCON column of xd.mas.

It works by finding all the possible paths through the structure, starting from every atom. If two atoms have exactly the same paths through the structure they are defined as chemically equivalent.

Other options:

The other two options are there to aid in defining custom chemical equivalency, however unless you have your own reasons for adding custom chemical equivalency, the automatic option is recommended.

Results

Tools

Backup

XD Programs