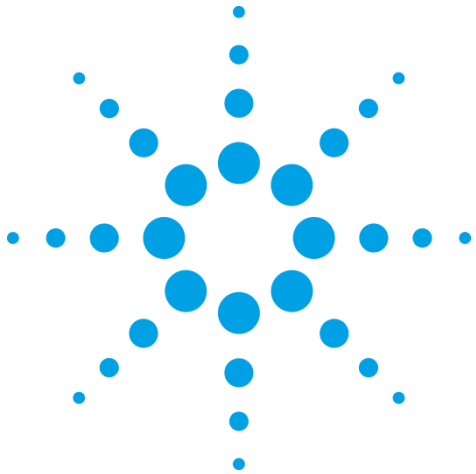

AutoChem$^{2.0}$

Automatic structure solution and refinement software for CrysAlis^{Pro}

User Manual

Version 5, July 2012

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1. Introduction

Developed exclusively for Agilent Technologies by the authors of Olex2 (OlexSys, UK), AutoChem$^{2.0}$ represents a significant advancement over its predecessor, AutoChem, and is seamlessly integrated into CrysAlis^{Pro} for automated, real-time structure solution and refinement during data collection experiments.

Since its launch in 2006, CrysAlis^{Pro} has provided automatic data collection, with concurrent data reduction making experiment workflow a very simple procedure. With AutoChem$^{2.0}$, CrysAlis^{Pro} can also complete crystallographic structure determination. AutoChem$^{2.0}$ runs continuously throughout data collection providing the user with visual feedback within the main CrysAlis^{Pro} GUI.

Should the user wish to review the structure more closely or to control aspects of structure solution, Olex2, can be launched at any time from within CrysAlis^{Pro}. Using Olex2, the user is able to review all the solution and refinement steps performed by AutoChem$^{2.0}$, step back to any stage of the process, apply changes as necessary or manually launch AutoChem$^{2.0}$ with custom settings.

An intelligent program, AutoChem$^{2.0}$ will automatically select the best solution from the various supported methods and programs available to it. Supported solution programs include ShelXS (Patterson, direct methods), Sir2008 and Sir2011 (direct methods), Superflip (charge flipping), and olex2.solve (charge flipping) formerly known as smtbx.solve, developed at Durham University in collaboration with cctbx. Refinement may be performed with either ShelXL or olex2.refine (formerly smtbx.refine).

AutoChem$^{2.0}$ is designed not only to solve and refine structures, but also to automatically generate an HTML and/or PDF report, containing a summary of useful crystallographic information. If desired, reports can be fine-tuned and manually produced within Olex2.

You should expect the majority of good quality data collections to be solved and refined using AutoChem$^{2.0}$. However, AutoChem$^{2.0}$ is currently unable to cope with significant disorder or twin decomposition. In these cases, user intervention will be required.

2. Installing AutoChem2.0

AutoChem2.0 is not included in CrysAlisPro as standard, and must be installed as a separate software plugin. If you have purchased AutoChem2.0 as part of a new system installation, an applications scientist will install AutoChem2.0 for you or provide instructions. For all other installations including trial licences (limited to 30 structures), please use the instructions below.

AutoChem2.0 is designed to be very simple to install, but an internet connection is normally required. If it is not possible to connect your PC to the internet or you encounter problems during installation, please contact xrdapplications@agilent.com, and assistance will be provided.

Please follow the below procedure for all computers covered by your licence*:

1. CrysAlisPro v171.36.xx or higher must be installed in order to interface with AutoChem2.0. If not already installed, the latest version of CrysAlisPro can be downloaded from the **Current Release** section under **Software Downloads and user manuals** on our user forum found here http://www.agilentxrdforum.com/yaf_topics48_Current-Release.aspx
2. Download and Install the AutoChem2.0 version of Olex2 (Olex2-1.2-ac2):
    - Go to http://www.agilentxrdforum.com/yaf_topics48_Current-Release.aspx and download the 32bit or 64bit installer depending on your operating system version. If you don't know which you have, you may use the 32 bit version or help can be found here: http://windows.microsoft.com/en-GB/windows7/32-bit-and-64-bit-Windows-frequently-asked-questions
    - Run the installer by double-clicking on it and following the on-screen prompts.
3. Open Olex2, by selecting the **Olex2-1.2-ac2** icon from the Start Menu.
4. Your licence code should already have been emailed to you, please paste this code into Olex2-1.2-ac2 and press return. Olex2-1.2-ac2 will now verify your licence online and download some additional files. When it has finished, follow the prompt to restart Olex2-1.2-ac2. This may take a few minutes depending on your internet connection speed.
5. Open or restart CrysAlisPro and the 'AutoChem' icon should appear on the left hand side of the main window. This indicates successful installation.

For systems where an older version of Olex2 or AutoChem has been in use, it may be necessary to update the AutoChem program location in CrysAlisPro to point to the correct version of Olex2, namely Olex2-1.2-ac2. This is accessible under the Programs tab in the **Options CCD** button at the bottom of the command line window.

Please note that AutoChem2.0 is only compatible with CrysAlisPro versions beyond v36.20. The older Autochem only works with CrysAlisPro v35

For installation of additional supported programs, please see the Appendix at the end of this document.

* N.B. A standard licence code covers two computers

3. Getting started with AutoChem2.0

The AutoChem2.0 functions have been developed to be remarkably simple to use. Once installation is complete, an **AutoChem** icon will be visible amongst the power tools on the left-hand side of the main CrysAlisPro window. This will be inactive (grey) until a data reduction has been completed.

Located at the bottom of the **Pre-experiment setup** window is a tick box option entitled **Attempt AutoChem**. In order to run AutoChem2.0 concurrently during a data collection, it is necessary to tick this box. It is also recommended (but not required) to input a chemical formula in the **Expected Chemical Formula** box. This does not need to be the exact formula, but should include any expected elements, particularly heavy atoms. All other aspects of the standard experiment workflow (Pre-experiment, strategy determination, crystal movie, data collection) remain the same.

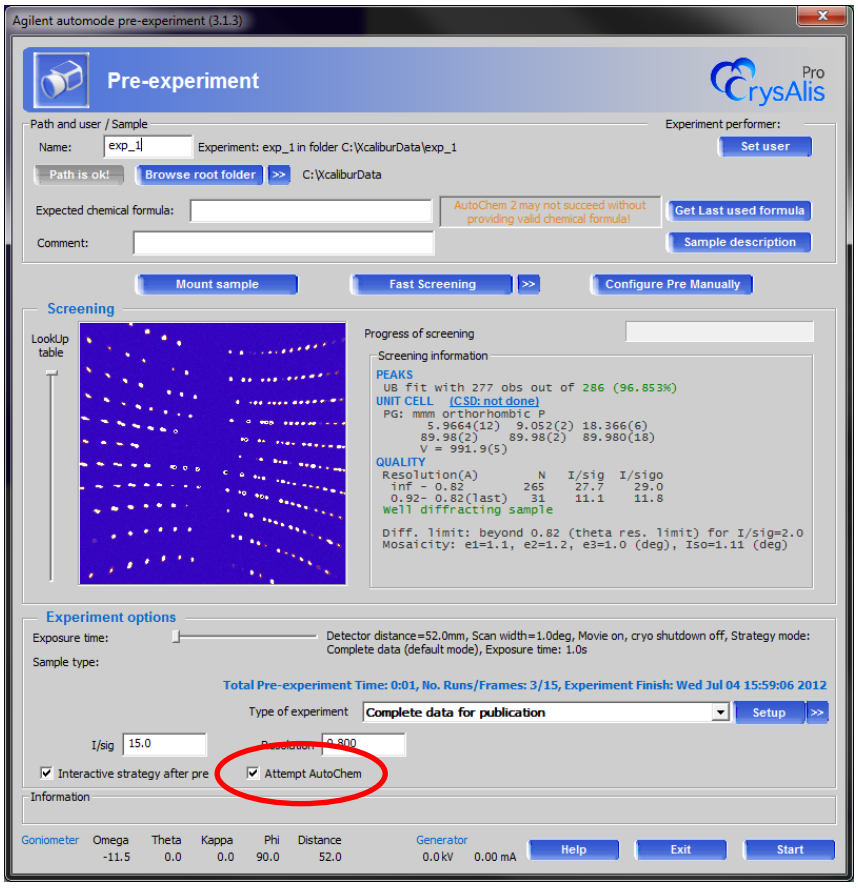
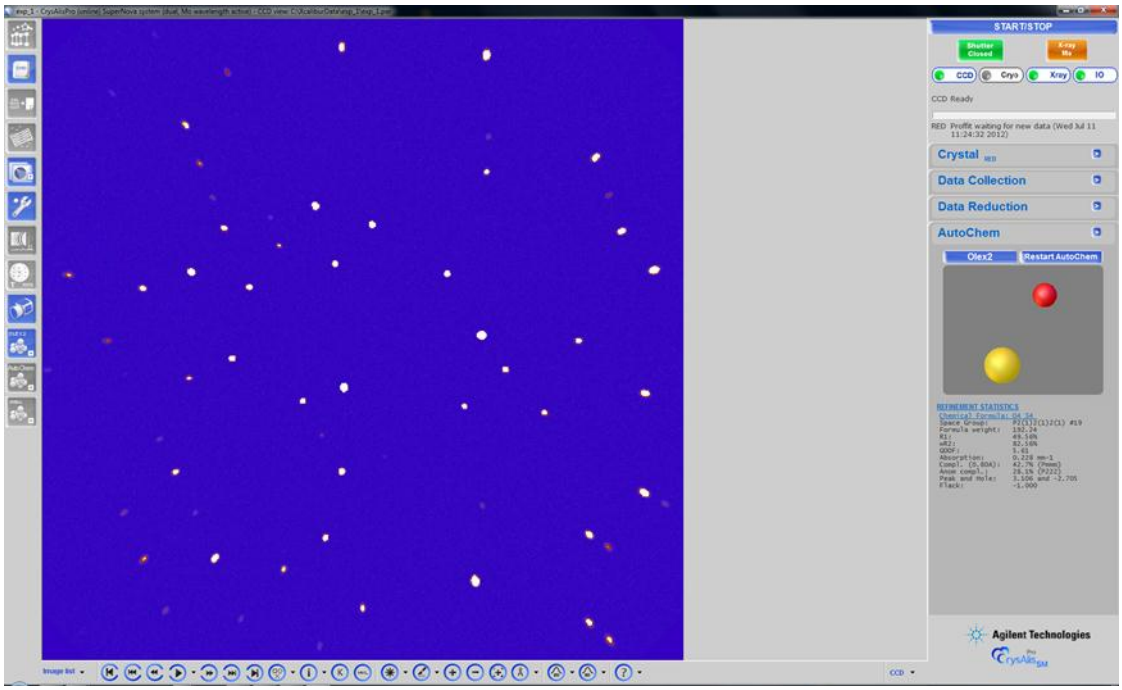


Figure 1 – Pre-experiment setup window with AutoChem activation

As in most other experiments, concurrent data reduction begins after collection of the 25th frame, and data reduction is updated in blocks of approximately 25 frames (depending on the length of the individual runs). If AutoChem2.0 has been enabled, each time a round of data reduction is completed, a new 4th tab will appear (beneath Crystal, Data Collection and Data Reduction tabs). The RED status area will detail what is happening at any one time and while AutoChem2.0 is working this will read **Waiting for Autochem report** with a counter. Generally a solution will be found quickly and will appear in the molecular viewer. If no suitable solution is found, the molecular viewer will remain blank. Timing out for every sample with a blank molecular viewer generally indicates a problem with installation.

There is a good chance that the initial solution will not be a reasonable one, but this is due to the fact that 25 frames will likely mean very low data completeness. The quality of the solution and refinement is highly dependent on the data quality and completeness value, as in any other crystallographic experiment and so things will generally improve as more data are collected.

AutoChem2.0 works by using an atom confidence figure. If the level of confidence is high enough, the refinement will proceed to the next stage (isotropic refinement ⟶ anisotropic refinement ⟶ addition of hydrogen atoms). Therefore, as the experiment progresses, the molecular model and refinement should become more complete. If necessary, AutoChem2.0 also attempts structure solution using a variety of space groups, choosing the best result as the one displayed.



Top level refinement statistics are output beneath the molecular viewer.

Figure 2 – Initial AutoChem solution (after 25 frames)

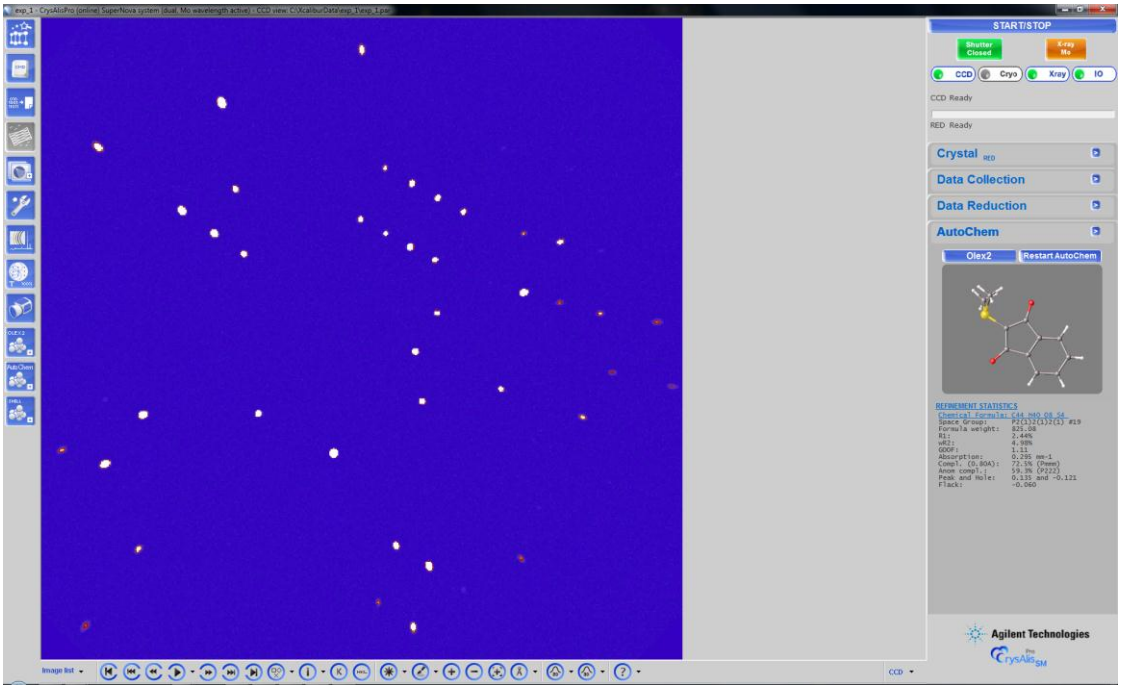


Figure 3 – Third AutoChem iteration (after 75 frames)

Certain AutoChem2.0 features can be altered at any time.

- Structure view
  - Hold left mouse button and drag for rotation
  - Hold right mouse button and drag for zoom
- Change formula
  - Click the blue, underlined formula text in the AutoChem tab

Clicking on the small blue arrow in the AutoChem tab provides access to more features;

- Draw style changes between ball and stick, ellipsoid and sphere packing views
- Labels - switches labels on and off for atoms, H atoms and Q-peaks
- Q peaks - toggles Q-peaks on and off, either with bonds or without
- Show cell toggles the unit cell on and off
- Auto rotate will keep the molecular viewer continuously rotating the molecule
- Change the background colour
- Access automatically generated reports

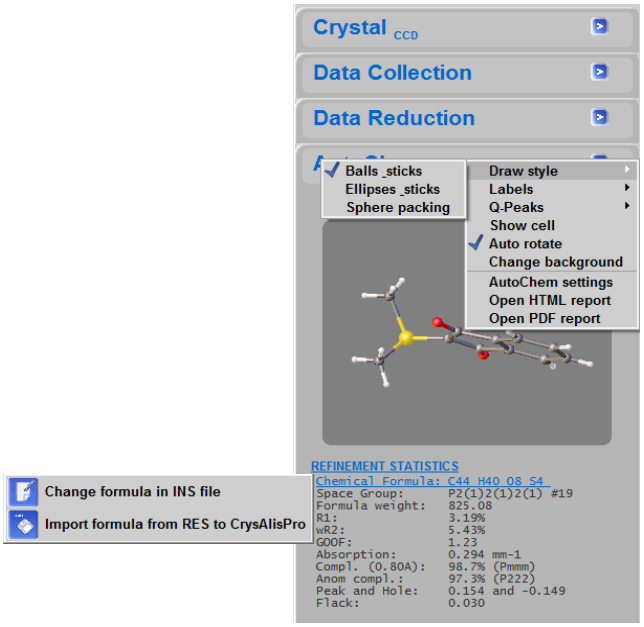


Figure 4 – Molecular viewer graphical settings

Choosing **AutoChem settings** will open a new window. The options available can be changed during an experiment, or simply left as default settings for every experiment. **Auto** mode will try all available programs and methods in order to find the best solution and refinement method for a particular structure. If desired, a particular solution and refinement method can be chosen.

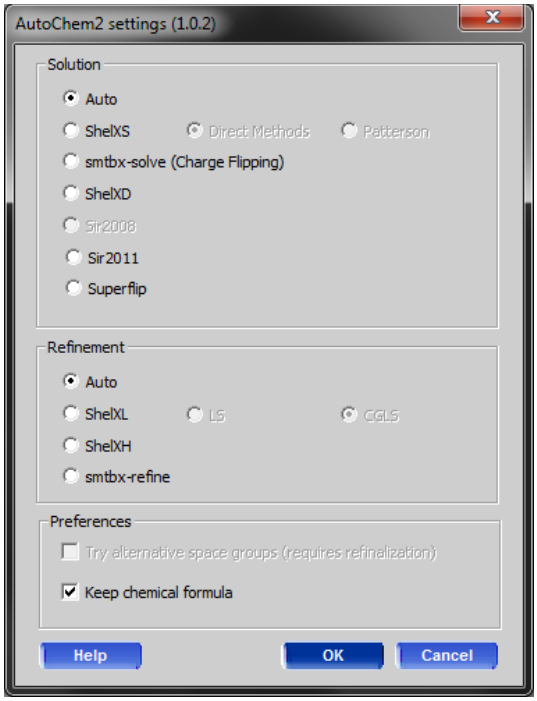


Figure 5 – AutoChem settings window

In order to manually manipulate structures, the blue **Olex2** button located above the molecular viewer is provided. This will open the structure in Olex2. The adjacent **Restart AutoChem** button is provided to reactivate AutoChem2.0, if desired, once you have finished. For more information about the use of Olex2-1.2-ac2, visit www.olex2.org.

If the model is changed in any way and a refinement is carried out, the change will also be updated in the molecular viewer in CrysAlisPro.

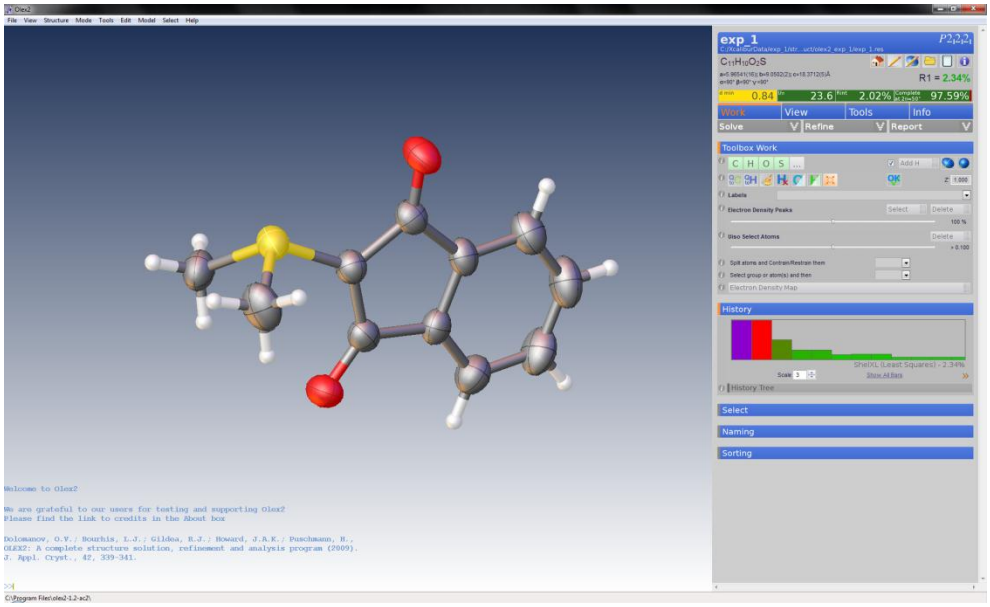


Figure 6 – OLEX2 window with current structure displayed

All generated files including automatic reports are located in the folder;

\experiment_name\struct\Olex2_experiment_name

e.g. \test\struct\Olex2_test

The final files simply retain the name of the experiment (.ins, .res, .hkl, .fcf, .cif, .cif_od). The cif_od file contains cif formatted information pertaining to data collection. A screenshot.png file is present, which is an image of the molecule as it was left in Olex2.

Reports are automatically generated in PDF and html formats by AutoChem$^{2.0}$ after the final cycle of refinement. These can be found in the above folder and will have a name such as experiment_name_tables.pdf (or html). If manual changes have been made these reports will need to be regenerated to update them, the procedure for which is covered in the next section.

4. Using AutoChem$^{2.0}$ offline

If CrysAlis^{Pro} is open in an offline thread (RED), either on the diffractometer PC, or on a different computer, AutoChem$^{2.0}$ can still be used to automatically solve and refine structures. This is achieved by loading the appropriate experiment and clicking on the **AutoChem** icon in the power tools on the left-hand side on the window.

If the data reduction for the experiment concerned has been run more than once, and different files have been output (or if the data has been refinalized with different output file names) a window will appear asking which *.ins file AutoChem$^{2.0}$ should be run with. Once initialised, AutoChem$^{2.0}$ will run in the normal way, and all the same functions are available.

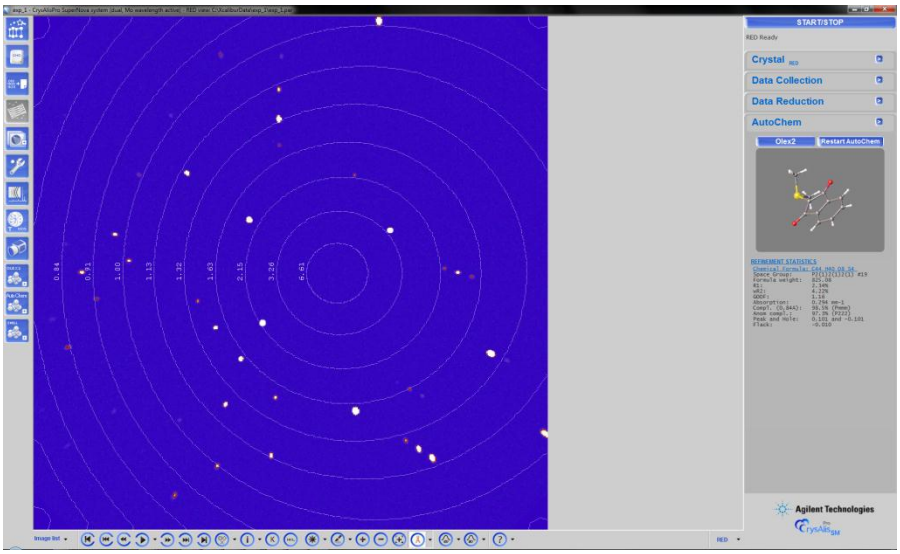


Figure 7 – AutoChem viewer in an offline thread (RED)

AutoChem$^{2.0}$ can also be run independently of CrysAlis^{Pro}, by opening Olex2-1.2-ac2 and selecting the **Run AutoChem** button which is found in the **AutoChem** menu under the **Tools** tab. Parameters for the run may be altered here also and manual triggering of report generation based on the most recent refinement is possible using the **Report** link. Alternatively you may issue the command **ac2** on the olex2 command line to start an AutoChem run.

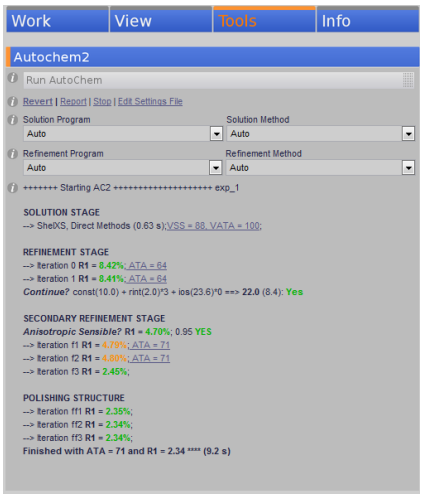


Figure 8 – AutoChem options accessible in OLEX2

Apendix: Optional solution and refinement programs

AutoChem2.0 supports several optional solution and refinement programs, ShelXS, ShelXD, ShelXL, Sir2008, Sir2011, and Superflip. These are not required however, to get the most out of AutoChem we recommend installing some or all of these programs. Due to licencing issues the user must obtain these directly. If any of these programs are desired, it is the user's legal responsibility to ensure that any necessary and appropriate licences are obtained before use.

ShelXS, ShelXD, and ShelXL

Where to get them: Follow instructions at http://shelx.uni-ac.gwdg.de/SHELX/

Superflip

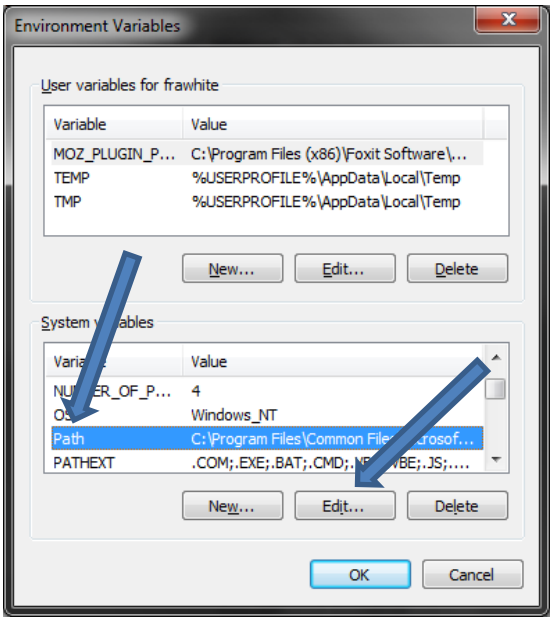
Where to get it: Download superflip and edma from http://superflip.fzu.cz/
In most cases choose the 'zipped executable for windows' option

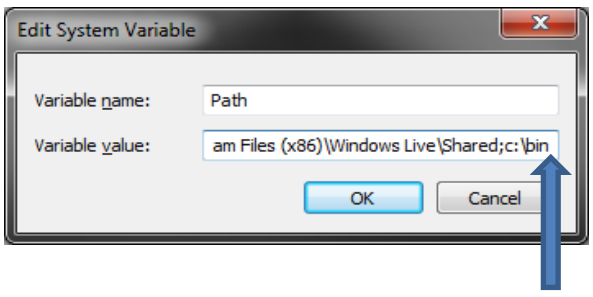
Sir2008 and Sir2011

Where to get them: Follow instructions at http://www.ic.cnr.it/icnew/

AutoChem2.0 needs to know where to find these programs on your system in order to use them. For Shelx and superflip, you can simply copy them into the Olex2-1.2-ac2 installation folder; however this method will not work for the Sir programs. To map Sir programs to AutoChem2.0 follow the instructions below:

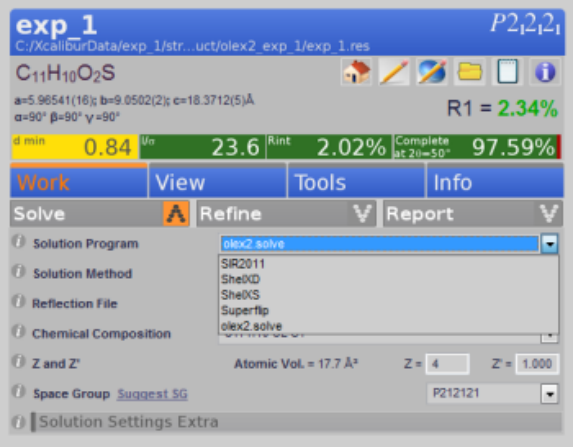
1. Run the installation programs for sir2011 and sir2008 accepting all default options
2. Go to the Start Menu > Control Panel > System
3. In the System Properties window click on the Advanced tab then click Environment Variables
4. Locate the Path environment variable under system variables select it and click the "Edit..." button
5. WITHOUT DELETING THE EXISTING TEXT (press cancel if you do by mistake) add the following (including the leading semi-colon) to the end of the line with no spaces or line breaks
   ;c:\program files\sir2011\bin;c:\program files\sir2008\bin
6. Press OK on all three windows when finished
7. Now restart Olex2-1.2-ac2





8. Click on the down arrow next to Solve under the Work Tab.

9. Look in the drop down box next to solution program and you should see the newly added programs.



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