

Quick Start Users Guide for Bruker SMART APEX II Diffractometer

APENDIX IV

INSTRUCTION FOR USING SQUEEZE

J. Tanski 2/12/06¹

Suppose your files are **f10.***

- (0) **SQUEEZE should only be used for good reason; i.e. if solvent is disordered so badly that it cannot be modeled even with restraints. *SQUEEZE in effect refines the data against the model. This is ethically questionable.*** The void space and electron count must make chemical and physical sense. You **MUST** append SQUEEZE results to the cif.
- (1) **Finish the structure completely. Perform the final refinement (XL) without any solvent residues or disordered counterions** in the atom list. Their electron density will be “squeezed” by Platon. At this point, the cell content should be in agreement with the structure without disordered solvent/counterion molecules.
- (2) Find the Platon program on the computer, double click **pwt.exe** to open the Platon Taskbar and the following window will show up.



- (3) In Platon, click **File** and select **select Data File**. Either .ins or .res file can be used. However, **.res file is recommended**. Click **Files of type** and select **SHELX file (*.res)**, then find the target file **f10.res** by browsing the computer through the **Look in** window, then click **open** to select **f10.res** file.
- (4) In Platon, click **Data** and select **SQUEEZE**, then wait until the program is done calculation. A lot of numbers will come out on the screen at this time and it usually takes a few seconds but up to a couple of minutes. It's done when you see the bottom of the screen have something like following:

Summary and Remarks : N = NOTE, W = WARNING, E = ERROR

=====

N: No S.U.'s (esd) on observed/calculated parameters.

N: SHELX-style data input.

N: No-Hydrogen atoms in this structure

N: Number of moved primary input atoms: 58

W: Low density (check!) of 0.980 gcm⁻³

N: Number of Ignored Lines on INPUT 5

of which blank in column 1 5

N: Total Potential Solvent Accessible Void Vol 1714.1 Ang³

N: Electron Count / Cell = 251 - To be included in D(calc), F000 & Mol.Wght.

=====

:: Input Data from File f10.res - Data Type RES

:: NORMAL END of PLATON : 49 Pages on:

:: f10.lis (ASCII, 132 Characters Wide)

:: f10.lps (PostScript Version of .lis)

:: SQUEEZE xyz on : f10.sqz

:: SQUEEZE CIF on : f10.sqf

:: SQUEEZE out on : f10.hkp

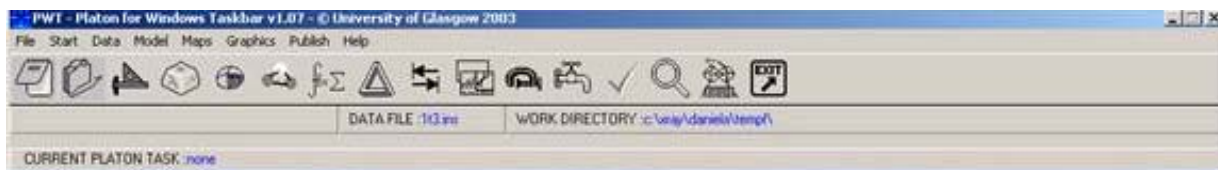
- (5) Close Platon program and any other windows that were opened by Platon.
- (6) A series of files should have been generated, including .lis, .lps, .sqf, .sqz, .hkp. Use Windows Explorer to go to the directory of your files and double click **f10.lps** to open it. Go to the **end** of this file and find lines of **Total Potential Solvent Accessible Void Vol**, for example 1714.1 Ang³s; and **Electron Count / Cell**, for example 251.
- (7) Now comes the most important part that is to calculate the correct number of solvent molecules. Refer to (a) the total potential solvent accessible void volume; (b) electron count; and (c) what kind of solvent were used during crystallization. Check the coordinates of disordered solvent molecules and see if they are at some special positions.

The most important thing is that the number of disordered solvent molecules should have some chemical sense with regarding to the symmetry and Z value of the crystal.

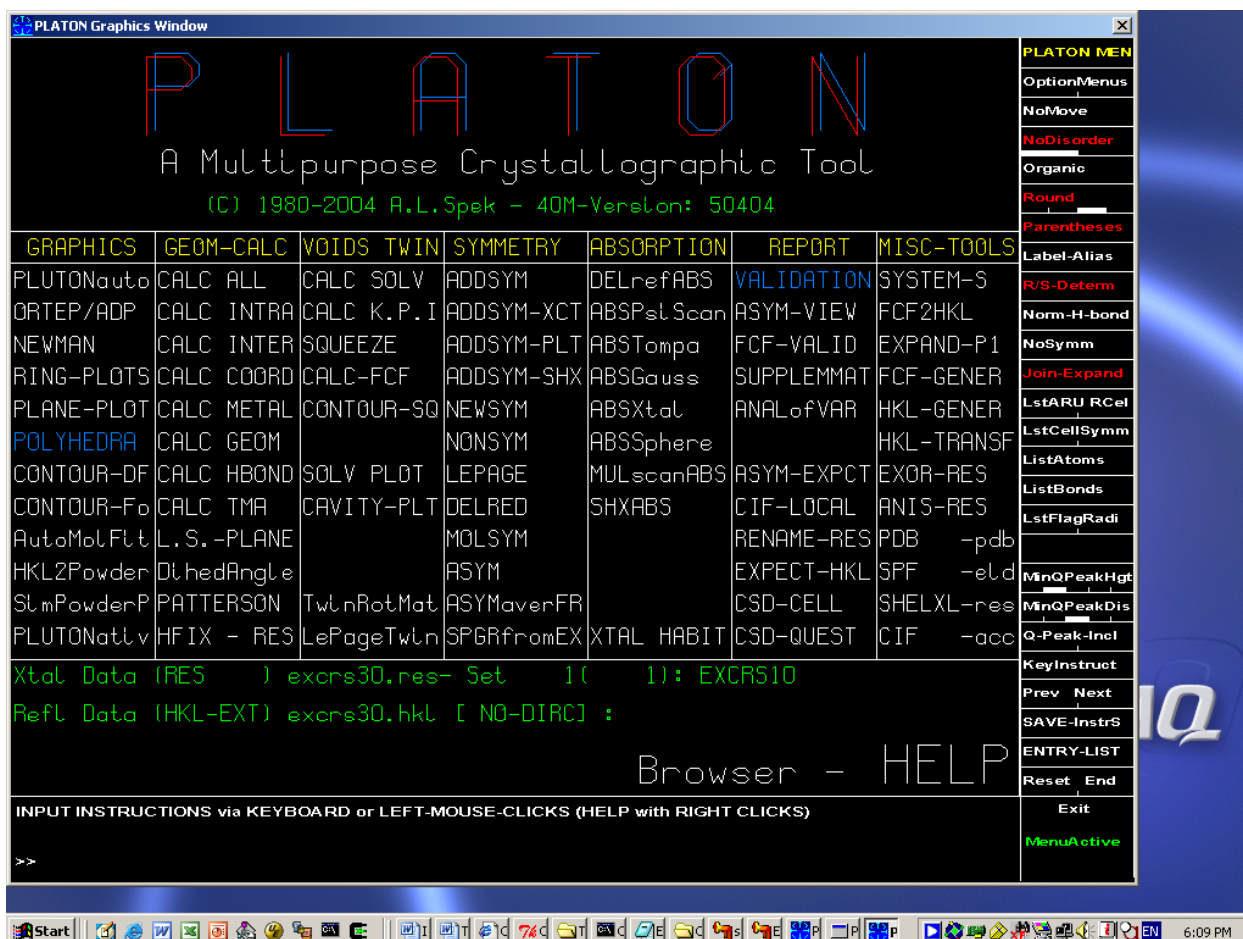
For example, expected volume for a hydrogen bonded H₂O molecule is 40 e-/Å³ while expected volume for small molecules like toluene are 100~300 e-/Å³.

In this case, 6 molecules of CH₂Cl₂ in each unit cell is a good choice for an Electron Count of 251 and a Total Potential Solvent Accessible Void Vol of 1714.1 e-/Å³ since each CH₂Cl₂ molecule has 42 electrons and ~200 e-/Å³ volume. Write down 6 molecules of CH₂Cl₂.

- (8) In DOS mode, type **copy f10.hkp f10p.hkl**. Then type **copy f10.ins f10p.ins**. Then type **copy f10.pcf f10p.pcf**. A letter **p** is added to the end of file names to specifically indicate that Platon is used to squeeze disordered solvent molecules. **.pcf** file contains crystal information needed as a reference when **XCIF** runs. You will have new files of **f10p.ins** and **f10p.hkl** that will be used for later refinement.
- (9) Add calculated solvent molecules to the **SFAC** and **UNIT** list of **f10p.ins** and do final **XL**. In this case, 6 molecules of CH₂Cl₂ were found so there are 6C, 12H and 12Cl. Open **f10p.ins**, add 6C, 12H, 12Cl to current list of **SFAC** and **UNIT**. Save the file **f10p.ins** and quit. Run final **XL**. You will see the message of ****Cell content does not agree**. Ignore this because disordered solvent molecules were not located... their electron density has been “squeezed” into the rest of the molecule.
- (10) Find the Platon program on the computer and double click **pwt.exe** to open Platon again and the following window will show up.



- (11) In Platon, click **File** and select **select Data File**. Click **Files of type** and select **SHELX file (*.res)**, then find target file **f10p.res** by browsing the computer through the **Look in** window, then click **open** to select **f10p.res** file.
- (12) In Platon, click **Start** and select **Graphical Menu**. A graphical menu will show up.



(13) Under the column of **VOIDS TWIN**, click **CALC-FCF** and wait for a few seconds until it's done. Then click **Exit** on the right bottom corner to exit graphical menu. Close Platon program and any other windows that were opened by Platon.

(14) Use Windows Explorer to go to the directory of your files and double click **f10p.sqf** to open it. Something like the following will be seen in this file:

```

loop_
  _platon_squeeze_void_nr
  _platon_squeeze_void_average_x
  _platon_squeeze_void_average_y
  _platon_squeeze_void_average_z
  _platon_squeeze_void_volume
  _platon_squeeze_void_count_electrons
    1 0.000 0.000 0.000 827.0 -10.0

```

2	0.003	0.591	0.104	9.1	-0.5
3	0.000	0.500	0.500	827.0	-10.2
4	-0.003	0.091	0.396	9.8	-0.5
5	0.003	0.909	0.604	9.3	-0.4
6	-0.003	0.409	0.896	10.0	-0.4

_platon_squeeze_details

; ?

;

- (15) Open **f10p.cif** using programs like enCIFer or Notepad. **Copy all lines above from file f10p.sqf and paste them to file f10p.cif.** Find **_refine_special_details** in file **f10p.cif**, paste all above lines at the place that is a line after the line of **_refine_special_details** in file **f10p.cif**. It's very important that all lines from **loop_** to the last **;** are copied and pasted. **Don't omit any semicolon.**
- (16) Modify following sentence appropriately and use it to replace the above question mark **?** at the end of a series of lines that were just added to file **f10p.cif**.

The unit cell contains 6 methylene chloride molecules which have been treated as a diffuse contribution to the overall scattering without specific atom positions by SQUEEZE/PLATON.

- (17) Save **f10p.cif** and exit.
- (18) It's done now and you may proceed to XCIF. In the case XL needs to run again in the future, .cif file must be modified again by copying the content from .sqf file, starting from (15). You should also mention that SQUEEZE/PLATON was used in structural refinement in X-ray experimental, and consider submitting the special fcf as supplemental material.

¹Much of this was edited by Guang Zhu, past graduate student of Ged Parkin at Columbia University.