

HOW TO'S FOR SOLVING PROBLEMS/TASKS WITH PLATON

INDEX (Version 14 Oct 2014)

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1 – How to run PLATON/SQUEEZE together with SHELXL2014

1. Refine a non-disordered solvent model (i.e. excluding the solvent that needs to be 'modelled' by SQUEEZE) using SHELXL2014 with the files **<name>.ins** & **<name>.hkl** to convergence (Include the ACTA instruction). The result will be the files **<name>.cif** and **<name>.fcf**. Do not remove the embedded **.res** and **.hkl** files from the resulting CIF !)
2. Run PLATON/SQUEEZE in a terminal window based on the **<name>.cif** & **<name>.fcf** files produced in step 1 with the command '**platon -q <name>.cif**'. The result will be the files **<name>_sq.ins**, **<name>_sq.hkl** & **<name>_sq.fab**. The **<name>_sq.fab** file includes the solvent contribution to the calculated structure factors (details of the SQUEEZE calculation are embedded in this file as well). Inspect the listing file **<name>_sq.lis**.
3. Continue SHELXL refinement in the presence of the files **<name>_sq.ins**, **<name>_sq.hkl** & **<name>_sq.fab** from step 2 with the command '**shelxl <name>_sq**'
4. Inspect the list files and validate (i.e. run '**platon -u <name>_sq.cif**'). The result will be in the files **<name>_sq.chk** & **<name>_sq.ckf**).

Notes:

a) Changes to the above in case of disordered solvents + Twinning:

The FCF produced in step 1 should be in that case of the 'LIST 8' type. This is needed to allow PLATON/SQUEEZE to run based on twin deconvoluted reflection data (Note: the SHELXL refinement in step 3 will again be based on the twinned data). The **<name>.ins** file should include the ACTA, LIST 8, BASF and TWIN or HKLF 5 instructions

b) Generally, no recycling of steps 1 to 3 will be needed. However, to accomplish this, it is possible to start with the **<name>.cif** & **<name>.fcf** from step 1 and run PLATON/SQUEEZE with the command '**platon -qn <name>.cif**' where 'n' is the number of cycles.

c) There should be no residual unresolved (disorder) density in the discrete model part of the structure because of its impact on the quality of the difference map in the solvent region. The dataset should be reasonably complete and with sufficient resolution [i.e. $\sin(\theta)/\lambda > 0.6$]. There should be no unresolved charge balance issues that might effect the chemistry involved (e.g. the valency of a metal in the ordered part of the structure). The reported electron count in the solvent region is meaningful only with the supply of a complete and reliable reflection data set. The SQUEEZE technique can not handle properly cases of coupled disorder effecting both the model and the solvent part of the structure. The solvent region is assumed not to contain significant anomalous scatterers.

2 – How to apply a unit cell transformation to a SHELXL .ins file

What is needed is the insertion into the <name>.ins of a record with the general format:

TRMX m11 m12 m13 m21 m22 m23 m31 m32 m33 t1 t2 t3

where **m11,m12,...,m33** are the 9 components of the cell transformation matrix and **t1,t2,t3** the components of the optional origin shift after transformation.

Such a record will generally be inserted directly after the TITL record. The effect will be that the transformation will be applied on what follows, i.e. the cell parameters, the space group, the coordinates and the displacement parameters. A proper transformation matrix will be inserted on the HKLF record. In such a way, no explicit transformation of the reflection file (<name>.hkl) will be needed for the subsequent SHELXL refinement.

Implementation:

Start terminal window

Edit <name>.ins to include the TRMX record

Invoke: 'platon <name>.ins' (where <name> is substituted by the actual filename)

Click on 'Create-res' (on the Main Menu under MISC-TOOLS)

As a result, a new <name>.res file is created

Notes:

- The TRMX command may also represent a unit cell halving etc.

3 – How to RENAME atom labels with PLATON in a .res file

1. start PLATON with the command '**platon -r <name>.res**'.
- 2a. Hit return (the graphics window should be active). The program will loop over all atoms in sequence with the current label changed from white to red. Hitting return will leave the label as indicated and the new in line label will turn red. Otherwise a new label name can be entered.
- 2b. Click on the atom for the label has to be changed and enter the new name.

Notes:

- a) Atom labels that have been changed turn green
- b) In case of a conflict with a label name that is already present, the already present atom label will be changed in a new label (that can be changed later on)
- c) when finished, click on 'END'. The new RES file will be on **<name>_new.res**

4 – How to calculate the Kitaigorodskii Packing Index

A crystal structure without room for embedded solvents of crystallization will still include small volumes outside of the van der Waals radii of the atoms making up the structure. A measure for the volume taken by the atoms is the (Kitaigorodskii) Packing Index. This value will generally be in the order of 65%..

The value of the Packing Index for a given structure (as an INS or CIF file) can be calculated by clicking on the PLATON main menu function '**CALC K.P.I.**'. This calculation is part of a search for solvent accessible voids (which should be very small or absent for a meaningful P.I.)

5 – How to apply a unit cell transformation to a SHELXL.hkl file

In general, there is no need to transform the reflection file data after a unit cell transformation. That transformation will be applied automatically by SHELXL when the relevant transformation matrix is supplied in the HKLF record. In that way there is only one copy of the reflection file.

The following options in PLATON are available in PLATON to carry out a 'hard' transformation of the reflection file:

1 – Startup PLATON with the relevant <name>.ins & <name>.hkl as '**platon name.ins**' and click on the PLATON main menu option '**HKL-Transf**'. The result will be the file <name>_trans.hkl. Also, a new <name>_trans.ins will be created with the (implicit) unit matrix in the the HKLF record.

2 – Placing a 'HKLT' record after the HKLF record in the .ins has the same effect as with option 1.

6 – How to generate an ab-initio HKLF4 reflection file for test purposes from a .res or .cif

Sometimes, an ab-initio HKLF4 style reflection file with 'observed' data generated from the coordinates and displacement parameters present in a supplied **.ins**, **.res** or **.cif** can be useful for program(ming) test purposes. This can be accomplished with the **HKLF-Gener** tool on the PLATON main menu. The generated dataset will comprise the asymmetric part of the reflection sphere with systematic absences removed. Artificial sigma(I)'s are calculated as **Sig(I) = MAX(0.01, SQRT(I) + 0.02 * I)**. Data are generated within the 'Copper Sphere' (i.e. $\sin(\theta)/\lambda = 1/1.5418 = 0.65 \text{ \AA}^{-1}$). To set the resolution to 0.6 \AA^{-1} : **SET PAR 540 0.6**. Reflection data (SHELXL HKLF 4 format) are written to a file with extension **_gener.hkl**. I and sig(I) are written as integers but are on absolute scale when read as (3I4,2F8.2).

Note: The alternative keyboard instruction is (The menu default is **ASYM GENERATE**):

ASYM (AVF) GENERATE

The **AVF** keyword determines whether or not for non-centrosymmetric space groups the Friedel related reflections will be averaged (i.e. for the monoclinic space group P21 half a sphere of data or one quarter will be generated).

7 – How to calculate Least-Squares planes and plane-plane, plane-line or line-line angles interactively

Various geometrical calculations can be performed interactively by invoking tools available from the PLATON main menu. Clicking on either **L.S.-plane**, **DihedAngle**, **AngLsplLin** or **AngleLines** will bring up an ORTEP view on which the relevant atoms can be clicked. The displayed molecule can be rotated to a suitable orientation when needed to get an optimal unobscured view.. Associated derived parameter su's are included when su's are supplied with the coordinate data (e.g. as a CIF). Atoms are treated with unit-weight in the least-squares plane calculation by default. Alternatives are weighting based on atomic weights and standard deviations (esd, su). The weighting scheme may be changed using the (UAE)WLSPL button. The result of the calculation is shown both on the graphics window, the terminal window and on the listing file.

*Interactive calculation of least squares planes with the **L.S.-Plane** tool.*

The red menu item **LsplDistEnd** in the options menu (with three sub-boxes) on the right indicates that the program is ready to receive the names of the atoms that will determine the least-squares plane of interest by clicking on the corresponding atom centers. This sequence should be terminated (and the calculation initiated) by clicking in the **End** field in the red menu item. Atoms defining the least-squares plane may be separated from additional atom labels for which only the distance to the plane has to be calculated by clicking in the **Dist** field between the clicks on atoms. Alternatively, an instruction similar to **LSPL c3 c4 c5 DIST c1** could be issued from the keyboard.

*Interactive calculation of dihedral angles between least squares planes with the **DihedAngle** tool.*

The red menu item **LsplWithEnd** in the options menu on the right indicates that the program is ready to receive the atom names that will determine the first least-squares plane by clicking on the corresponding atom names. This sequence is ended and the sequence for the second plane started by clicking in the **With** side-menu box. The second sequence is ended (and the calculation initiated) by clicking in the **End** field. Alternatively, an instruction similar to **LSPL c3 c4 c5 WITH c1 c2 c6** could be issued from the keyboard.

*Interactive calculation of the angles between a least squares plane and a bond with the **AngLsplLin** Tool.*

The red menu item **LsplWithEnd** in the options menu on the right indicates that the program is ready to receive the atom names that will determine the least-squares plane by clicking on the corresponding atom names. This sequence is ended and the sequence for the two bond atoms started by clicking in the **With** side-menu box. The second sequence is ended (and the calculation initiated) by clicking in the **End** field. Alternatively, an instruction similar to **LSPL c1 x2 c3 c4 with c5 c6** could be issued from the keyboard.

Interactive calculation of angle between two lines with the AngleLines tool.

The red menu item **Angle2Lines** in the options menu on the right indicates that the program is ready to receive the atom names that will determine the two lines respectively by clicking on the four atom names of the two lines/bonds. Alternatively, an instruction similar to **ANGLE c1 c2 c3 c4** could be issued from the keyboard.

8 – How to get the Hall symbol for a given symmetry and other symmetry related issues

PLATON includes a build-in space group symmetry handler with multiple functions. Several of those functions can also be used interactively without the need to supply coordinate or reflection data.

a) The terminal command **platon spgr 'P212121'** will report the Hall symbol and an explicit list of symmetry operations for space group P212121.

b) The terminal command **platon Hall '-P 2yab'** will report the H-M symbol (when in a known setting) and an explicit set of symmetry operations.

c) The terminal instruction **platon spgr** gives access to the other options: e.g. the option to build up the set of symmetry operators with **LATT** and **SYMM** input records followed by a **LIST** instruction.

Note: only the subset of space group generators will be needed.

9 – How to recover a .ins and .hkl from a shelxl.cif (+ shelxl.fcf)

A **.ins** and **.hkl** might be needed for additional calculations on a published structure. With SHELXL2014 those files are embedded in the CIF and can be extracted with the **shredcif** utility that is provided with the SHELX distribution. Alternatively, recovering those files can be accomplished with the terminal window instruction **platon -H <name>.cif**. The result will be two files: **<name>_sx.ins** and **<name>_sx.hkl**. In most other cases (e.g. with a **shelxl97** based CIF) also a **<name>.fcf** will be needed. A **<name>_sx.ins** file will in that case be created from the data in the CIF and a **<name>_sx.hkl** file from the observed data in the **<name>.fcf**. The latter file includes necessarily only the merged reflection set and does not include the systematically absent intensities.