

### Typical Crystal Structure Workflow

1. Grow crystal, mount crystal on a goniometer head using a microscope.
2. Intensity data are collected using APEX3.
3. If the cell is difficult to index (twinned, etc) then export the \*.p4p file, open a command prompt window, change the device and directory to the directory of the \*.p4p file, run CELL\_NOW. Import the appropriate \*.p4p into APEX3.
4. Integrate the data in APEX3. A minimum of 3 cycles of integration are recommended. In the Integration > Options menu always be sure to set “Active Image Queue Half-Width (Images)” to at least 12.
5. Apply an absorption correction using either SADABS or TWINABS.
6. Determine the space group using XPREP.
7. Solve the structure using XT or XS.
8. Label, sort, and refine the atoms using SHELXLE or OLEX2.
9. Return to the command prompt to finish refinement. Be sure to remove the “LIST 6” command before the last refinement cycle with SHELXL. When the refinement has converged, apply the checklist to look for common problems.
10. Edit the \*.pcf and \*.cif files. Run CIFTAB to update the \*.cif and create a basic report file (\*.rtf).
11. Run the CHECKCIF program to validate the structure. If the model can be changed to eliminate any alerts, do so and go to step 9. If no changes to the model can be done, add *\_vrf\_platon\_alert* records to the \*.cif for all A- and B-level alerts explaining the PROBLEM: and providing an appropriate RESPONSE:.
12. Make drawings with MERCURY. In particular, prepare a labeled drawing of all atoms (using ellipsoids), a packing diagram of the ordered part of the structure (using ball and stick), and a capped stick drawing of the ordered part of the molecule.
13. Prepare a report of the structure.
14. Archive the data set.

### Checklist for Completed Structure

1. Intensity data should have been collected to at least 0.82Å resolution with at least 98% completeness. If the intensities fall to background values at lower resolution, describe this in the \*.cif file.
2. Be sure that all bond lengths, angles, and torsion angles are *chemically* reasonable!
3. The last difference map should be relatively flat. No large peaks or valleys, i.e., map should have no peaks  $> 0.1 * Z_{\max}$ .
4. The anisotropic displacement parameters should be only slightly prolate (cigar shaped) or oblate (plate shaped). RIGU, DELU, SIMU, or ISOR restraints may be needed.
5. If the sample crystallizes in a non-centrosymmetric space group, check that the absolute structure has been correctly determined.
6. The final refinement must be converged, largest shift / esd  $< 0.05$ .
7. The weighting scheme should be adjusted so that the GoF =  $S \approx 1.0$ .
8. The difference map should be flat; no peaks  $> 0.1 * Z_{\max}$ .
9. The final  $R_1$  and  $wR_2$  should be reasonably low for the data.  $R_1 < 0.05$  for a good structure;  $R_1 < 0.10$  for an acceptable structure.

## Common Programs and I/O Files

Command-prompt programs: (I: input files, O: output files)

CELL\_NOW Index (twinned) data I: \*.p4p, O: \*.p4p  
SADABS Absorption correction I: \*.raw, O: \*.abs, \*.eps, \*.hkl  
TWINABS Twin absorption correction I: \*.mul, O: \*.abs \*.eps, \*.hkl  
XPREP Determine space group I: \*.p4p, \*.hkl or \*.raw, O: \*.prp, \*.pcf, \*.ins, \*.hkl  
XT Structure solution I: \*.ins, \*.hkl, O: \*.lxt, \*.res  
XS (Old) structure solution I: \*.ins, \*.hkl, O: \*.lst, \*.res  
XP (Old) drawing program I: \*.res or \*.ins, O: \*.ins, \*.eps (drawings)  
SHELXL Refinement program I: \*.ins, \*.hkl, O: \*.lst, \*.res, \*.cif, \*.fcf  
CIFTAB Update cif, make tables I: \*.cif, \*.pcf, O: \*.cif, \*.rtf  
SHREDCIF Read I: \*.cif, write O: \*.res and \*.hkl

GUI-based programs:

APEX3 Collect and Reduce data I: \*.p4p, O: \*.p4p, \*\_ls, \*.raw, \*.mul  
SHELXLE Molecular editor I: \*.res, \*.hkl, O: \*.res  
ENCIFER Edit cif I: \*.cif, O: \*.cif  
CHECKCIF Validate structure I: \*.cif, O: \*.pdf, \*.html  
PLATON (Squeeze) I: \*.ins, \*.hkl, \*.cif, O: \*\_sq.fab \*\_sq.hkl, Platon.out  
MERCURY Graphics I: \*.res, \*.cif, \*.pdb O: \*.png, \*.jpg

## Data files

Usually the data or project names for files will be kept the same for a set of conditions. You may want to change the project name if the cell (and hence data) must be changed. Most files are written with simple text characters and hence can be edited with any text editor.

\*.p4p Cell parameters, esds, peaks used to determine the cell  
\*\_ls Output listing file for data reduction (integration)  
\*.raw Output intensity file from data reduction; contains direction cosines and setting angles  
\*.mul Same general format as \*.raw, but for a twinned sample.  
\*.abs Printer file for absorption correction program  
\*.hkl Intensity data file contains  $hkl$ ,  $F^2$ ,  $\sigma(F^2)$ , run number  
\*.prp Printer file for XPREP program  
\*.pcf File with info to be added to \*.cif  
\*.ins Input instruction file from structure solution or refinement  
\*.lxt Printer output file from structure solution (XT)  
\*.lst Printer output file from refinement  
\*.res Results output file from structure solution or refinement  
\*.cif Crystallographic Information File, also contains last \*.res and \*.hkl  
\*.fcf Intensity data in cif format (deprecated)  
\*.eps Enhanced postscript graphics file (binary)  
\*.rtf Rich text file; a word processing file  
\*.pdf Portable Document Format developed by Adobe (binary)