**X-ray Crystallography 12650 - CHM 69600-006**

***Homework Assignment 7:***

***Evaluation of Published Work***

Questions are 10, 20, 40 or 50 points, as indicated. *Due date: One week after assignment*.

1) Using Scifinder, the Cambridge Crystallographic Data Base, or your own information, pick a random publication with no Purdue coauthors that contains at least one single crystal structure of a molecular compound (a “small molecule” structure, not a macromolecular structure). Pick a structure of your choice for analysis.

2) Using the Cambridge Structural Data Base’s “request a structure” service at <https://www.ccdc.cam.ac.uk/structures/>?, obtain the original CIF deposited with the CCDC (for some ACS journals original CIFs are also available in the SI of the journal). Do not use the abbreviated CIF available through the Conquest software. It is truncated and incomplete.

3) Check that the CIF contains a copy of the res and hkl files before proceeding (entries \_shelx\_res\_file and \_shelx\_hkl\_file). If the CIf does not contain these entries (i.e., if refined with an outdated shelxl version or other software), pick another paper/compound.

4) Using Mercury, Ortep3, Platon, CrystalExplorer or another visualization software of your choice, prepare a plot of the molecular structure with labels for important atoms. Use the thermal ellipsoid style (“Ortep plot”) with 50% probability level. If disorder is present, try to make it “look pretty” (decipherable). The Ortep3 implementation at <http://publcif.iucr.org/services/tools/> tends to give good plots of disordered structures quite easily. If necessary for clarity, prepare a second view of the molecule. Make another plot in packing view, with unit cell boundaries but without labels. Include H-bonds if present (without dangling H-bonds). Make copies of the plots and insert them into a word file (often a screen shot works well). The cover page of the word file should include the paper’s title, the literature reference, and the author list as well as the compounds name.

20 points

5) Download and install a copy of FinalCif from <https://dkratzert.de/finalcif.html>. Download <https://github.com/dkratzert/FinalCif/raw/master/finalcif/template/template_without_text.docx>.

Open your cif in Finalcif. Click “Options” (right lower corner). Under “Report Template” (middle of page) click “Add new Template” and link the “template\_without\_text.cocx”. Place a checkmark at this selection in “Report Templates” (also make sure there is a hashed box around the selection). Click “Back to Main Table” (bottom center of page). Click “Make Report from Template”. Add the tables from the file that opens to your report.

10 points

6) On a following page, explain the physical meaning of every entry in the first table (“Table 1. Crystal data and structure refinement for …”). Give (mathematical) definitions and explanations for every entry as appropriate. One to four sentences per entry should be sufficient.

50 points

7) Extract the res and hkl file using the “shredcif” option of Shelxle (The “File” drop-down menue, select “shred a cif and load its res” (alternatively, copy out the res and hkl file form the cif and make into files readable by Shelxl/Shelxle). Run a refinement cycle to create a difference density map (change LIST to LIST 6 if required, but make no other changes to the res file).

Run Checkcif on the original cif file. Inspect the checkcif report, the res file (including difference densities), and the cif for potential problems. What would you have done differently? If changes seem necessary, try to improve the refinement to the best of your abilities (but do not invest more than 30 minutes on this).

Discuss any shortcomings (if any) of the structure, especially any level A or B checkcif alerts. What do they mean and why are they present for this structure. Also discuss avoidable or unnecessary level C and G checkcif alerts. Are the most serious alerts related to problems with the data, the refinement model, or other causes? Give an overall evaluation of the structure’s quality and of the refinement model.

50 points

8) Using the information from the above sections, prepare a 5-10 minute power point presentation on your compound discussing the most important aspects of this structure (include the plots and tables and checkcif report). Present your findings to your classmates and discuss the most important features as well as shortcomings (if any) of this structure.

50 points

10) Print out or e-mail the above MS word file as well as the powerpoint slides and hand them in as the assignment. E-mail the original cif as well as any revised res files (if any).