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

***Homework Assignment 5:***

***Refinement of a mid-sized organic molecule***

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

1) Download the compressed folder “Homework 5” from the class web page. Set up a project in Shelxtl and process the provided hkl and p4p files using XPREP. Assign the space group and explain your choice.

Using the obtained ins and hkl files, solve the structure using either direct methods or Patterson methods. With the help of the sample background (given in the powerpoint file), assign all non-H atoms (correct assigned atom types if needed) and refine the structure.

Once all atoms are assigned, continue with anisotropic refinement, rename atoms in a meaningful way, sort the atoms, and update the weighting scheme multiple times until it does not change any more. Then place H atoms. Use the automatic HFIX command were possible. For hydrogen atoms that are not automatically assigned this way, choose suitable fitting manual HFIX commands (from the table of “Common HFIX commands” powerpoint slides page 119). Explain your choice of HFIX command.

Check the list of “Most Disagreeable Reflections” in the lst file for reflections that might be obstructed by the beam stop. These would be reflections with very small observed but large calculated intensities at low resolution (i.e. with high values for the resolution in Å). Use “OMIT h k l” lines in the res file to leave these reflections out of the refinement.

Repeat updating the weighting scheme again. Investigate the structure for further changes needed. Are all atom types indeed correct? Are all hydrogen atoms there and placed correctly?

For some atoms, assignment of atom types might be ambiguous. Explain your choice of sulfur vs. chlorine atoms based on both diffraction data and chemical considerations.

When confident that your model is complete and correct, add missing items to the res file (ACTA, BOND $H, CONF). Check if the formula and Z values are correct. Update or correct them if needed.

Does the structure exhibit any hydrogen bonds? If it does, add a line HTAB and run a refinement cycle. A list of HTAB and (if needed) EQIV commands will be added after the END of the res file. Investigate the list and copy HTAB and EQIV lines for what YOU think are real hydrogen bonds to the top part of the res file (i.e. between the UNIT and WGHT lines). Explain your choice in a couple of sentences.

Run several refinement cycles again and make sure the weighting scheme is final and that the structure has settled (no large displacements any more). Once finished, run a first checkcif report to check for any oversights or mistakes. Correct as needed and repeat the last steps.

Print out a copy of the res/ins file and hand in with the assignment. Make a screenshot of the Shelxle view with atom labels, print it out and staple it to the res/ins printout.

55 points

2) Fill in the missing items in the CIF. Use the instructions and items provided with the hkl and p4p files (use the procedures given in “[CIF Entries 2018 for Purdue Quest instruments](https://www.chem.purdue.edu/xray/docs/CifFileEntries2018.docx)” on the class web page but note that the data were not collected on a Purdue instrument).

Prepare a final checkcif report (including structure factor checks) and print out a copy.

20 points