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

***Homework Assignment 4:***

***Refinement of a mid-sized simple metal complex***

*Questions are 10, 15, 20 or 30, as indicated. Due date: One week after assignment.*

1) Download the compressed folder “Homework 4” 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. For the space group assignment step, include a screen shot (similar to that on page 88 of the class powerpoint slides). **Did you have to make any changes to the program defaults?** Discuss possible reasons.

15 points

2) Using the obtained ins and hkl files, solve the structure using either direct methods, Patterson methods or dual methods (the programs ShelXS, ShelXD or ShelXT). With the help of the synthesis layout (below), 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 139). Discuss why the automatic HFIX command failed and explain your choice of HFIX commands.

Check the list of “Most Disagreeable Reflections” might be obstructed by the beam stop (access via the SHELXL or Tools dropdown menue, depending on your version of ShelXle) in the lst file for reflections that. 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.

Do not omit outliers that seem to be not caused by a reflection being obstructed (discuss this in Question 4, below).

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?

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 line of the res file. Investigate the list and copy over HTAB and EQIV lines for what YOU think are real hydrogen bonds (explain your choice in a couple of sentences).

Run again several refinement cycles 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 it 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.

30 points

3) Fill in the missing items in the CIF. Use the instructions and items provided in the file at “[CIF items for the molybdenum radiation Bruker Quest Diffractometer](https://www.chem.purdue.edu/xray/docs/Quest%20Purdue%20Mo%202023.txt)” on the [resources web page](https://www.chem.purdue.edu/xray/instructional.html) (The data were collected on the molybdenum wavelength Purdue Quest instrument).

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

10 points

4) Discuss in a few sentences the reason/origin for any of the following checkcif alerts: PLAT910, PLAT934, PLAT213, PLAT978. Use the links provided within the checkcif report for each of the alerts for suggestions and details.

20 points (5 points for every type of alert listed).