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

***Homework Assignment 6:***

***A simple metal complex with disorder around an element of symmetry***

*Questions are 15 or 45 points, as indicated. Due date: One week after assignment.*

1) Download the compressed folder “homework 6” from the class web page. Starting from the p4p and hkl files, and with the help of the synthesis layout (below), assign as much as possible all non-H atoms and refine the structure.



Once all non-H 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.

Check for potential disorder. Use the tools of Shelxl and Shelxle for refinement of disorder such as **PART** or **negative PART** commands, **SAME**, **SADI**, **DFIX**, and **SIMU** or **ISOR** to properly refine all disordered sections of the structure. If you use a SIMU command, the recommended esd is 0.01 Å2 (i.e, use **SIMU 0.01** rather than just **SIMU**). Move atoms in meaningful positions using the **MOVE** and **COPY** commands of Shelxle (accessible via right mouse clicks on atoms). Pay attention to occupancies of disordered atoms. Make sure the final result is chemically and physically meaningful before proceeding.

Then place H atoms. Use the automatic HFIX command where 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 123). If you placed H atoms not using the “automatic HFIX” command of Shelxle, explain your choice of action used.

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?

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 that YOU think are real hydrogen bonds to the top of the res file (anywhere under the LIST or UNIT instruction). 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. 45 points

2) Fill in the missing items in the CIF. Use the instructions and items provided in the file “[CIF Entries 2018 for Purdue Quest instruments](https://www.chem.purdue.edu/xray/docs/CifFileEntries2018.docx)” on the class web page (Assume the data were collected on the copper wavelength Purdue Quest instrument). Pay attention to the Z value, the sum formula and the moiety formula.

Prepare a final checkcif report (including structure factor checks) and print out a copy. 15 points

3) Describe the disorder you refined from a **chemical** and **structural point of view** i.e., not focusing on the way it was refined, but what it means for the structure. How would you describe this in a peer reviewed publication? 15 points