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

***Homework Assignment 10:***

*All questions are 15 points. Due date: One week after assignment.*

1) You encounter a larger than usual unit cell and spots during unit cell measurement seem to start overlapping. Should you:

(i) Use a shorter or longer X-ray wavelength if you have a choice? What are the two most commonly used wavelengths in laboratory diffractometers and what are their values?

(ii) Move the detector closer to crystal or further away?

(iii) Use a smaller or larger crystal or a smaller or larger beam size?

2) The tabulated atomic scattering factors used in structure determination are idealized. The deviation from their idealized values allows to extract additional information from the diffracted data. Which items in the following list are among those:

Partial occupancy of an atom

Large thermal libration

Disorder of a molecule

An atom being located on a mirror plane

A wrongly assigned atom type

Presence of 13C rather than 12C.

Explain your answers.

3) A structure refinement does not settle down. Upon inspection the issue seems to be a rotating methyl group. What do you do to fix this problem?

In another case the problem seems to be associated with a rotating hydroxyl group. Another oxygen atom without hydrogen atoms is in close proximity (2.8 Å away). What do you do to fix this problem?

4) Large negative difference electron densities are found around a zinc and a bromine atom in an otherwise organic structure. What could be the cause for this? Using Shelxl as the refinement software, how do you check and resolve the problem?

5) You encounter a disordered THF molecule in s structure. Disorder is obvious, but some of the atoms are closer to each other than the resolution of the data. Which restraints to you expect you will have to use to refine the disorder?

6) You collect a data set and it appears that it will not meet the IUCr requirements for resolution and/or for data to parameter ratio. Describe what you can do to ameliorate the problem, both on the data collection as well as the data refinement side.

7) Which is correct:

Lowering the data collection temperature can help with the following:

Fluxional disorder, asymmetric peak shapes, substitutional disorder, weak high angle data, large thermal libration

Explain your choices

8) There are various choices to restrain thermal parameters. Briefly describe in a non-mathematical way the way the Shexl commands ISOR, SIMU, RIGU and EADP work. Which would you preferentially use for the following cases:

(i) Static disorder of Zn with Fe in a metal or mineral (they share the exact same position in the lattice)

(ii) A dataset from a very small weakly diffracting crystal, but with no apparent disorder.

(iii) A disordered isopropyl group with ill-defined ADPs.

Explain your choices.