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

***Homework Assignment 9:***

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

*1) Assuming both are available, would you use Cu or Mo radiation to determine the following structures:*

*a) C6H4Br2 (crystals available are > 0.3 mm in every direction)*

*b) C6Cl4Br2 (crystals available are < 0.05 mm in every direction)*

*c) C36H12O18Ru6*

*d) Absolute configuration of C24H42N2O8*

*e) Absolute configuration of C24H40Br2N2O8*

*Would you change your mind for any of a) to e) if crystals available would be*

*a) > 0.3 mm in every direction*

*b) < 0.05 mm in every direction*

*2) A synthetic organic material yields a good triclinic dataset. The structure solves easily in P1, but not in P*$\overline{1}$*. What should you do next? Give your sequence of steps and explain each step.*

*3) Why do we bother fiddling with a) hydrogen atoms, b) disorder, c) solvate molecules? Comment briefly on different techniques available solving these problems adequately but as concise as possible.*

*4) What is the “riding model” in parameter refinement?*

*5) Discuss the placement of H atoms on:*

*(i) terminal hydroxyl groups*

*(ii) ligating water molecules*

*(iii) uncoordinated molecules of water of crystallization*

*For each case, give the Shexl commands you would use. Would you use different commands or numbers of commands depending on the quality of the structure (e.g. high and low resolution data)?*

*6) Which of the following space groups could be twinned by exact merohedry (other than inversion twinning)? P*$\overline{1}$*, P21/c, P212121, Cmca, I*$\overline{4}$*, P3121, R3m, P63/mmc, Pa*$\overline{3}$*. Hint: look up the definitions of the three primary directions for each Laue class.*

*7) You collected a dataset on a large crystal (> 0.4 mm in each direction) of an organic compound, but no data were observed beyond a resolution of 1.5 Å, despite long exposure times. What could be the cause? Discuss several possibilities. Do you expect to be able to refine the dataset to IUCr requirements? Discuss your options.*

8) Which of these symmetry elements make a four membered MXMX ring strictly planar? In each case, how many bond lengths are independent. Draw a sketch for each case and indicate symmetry elements and equivalent bond lengths and angles.

(i) an inversion center

(ii) a two-fold axis normal to the mean plane of the ring

(iii) a two-fold axis through the two M atoms

(iv) a mirror plane through the M-atoms but not through the L atoms

(v) a mirror plane through all four atoms