

CHEM 273/279 SPRING 2009
TAKE-HOME FINAL EXAM, **PART 1**
DUE: WEDNESDAY, JUNE 10, AT NOON

Name _____

Perm number _____

Date submitted _____

This exam is based on the experimental data that you have obtained as part of the laboratory data collection and analysis that you did. If you are not enrolled for the laboratory course, please see Guang Wu to obtain output data for a sample crystal for this exam. Each individual is expected to submit his or her own, independent answers to the exam.

Reference:

Help information of structure determination software Shelx can be found at

<http://shelx.uni-ac.gwdg.de/tutorial/english/verf.htm>

Please put your name and perm number (in case the name is difficult to read) on this front page, staple your answer sheets to the end of this package, and put your initials at the top of each page.

Problem number	Possible points	Actual points
1		
2		
3		
4		
5		
6		
7		
8		
TOTAL for Part 1		

1.
 - (a) What is the empirical chemical composition for your crystal?
 - (b) How was the composition determined?
 - (c) Summarize briefly, but quantitatively, the results of any other experimental techniques that were used to study this material (density, thermal analysis, FTIR, Raman, NMR, Vis/UV, optical microscopy, etc.)

2.
 - (a) Using what you know about the crystal composition, and assuming that you will be using MoK α radiation, calculate the linear and mass absorption coefficients for your crystal.
 - (b) Based on part (2a), what is a reasonable size crystal to use for your data collection?

3. You have selected and mounted your crystal; and, obtained sufficient data to run the Bruker *Index* Routine.
 - (a) How many frames were used in this initial data collection? How many observable diffraction peaks are used?
 - (b) Define reduced cell.
 - (c) Give the reduced cell parameters for your crystal, including standard deviations.
 - (d) Based on the histogram information given by Index, how well are the observed reflections indexed with the choice of the reduced cell?

4. What is the purpose of the Bruker program *Bravais*?

5. You've selected what appears to be the unit cell with the highest symmetry. Confirm that the transformation matrix given properly relates the reduced cell parameters to the highest symmetry cell parameters.

6. You have now run the Bruker programs *SAINT* and *XPREP*.

(a) What box size was used to measure the diffraction intensity?

(b) The output from SAINT contains the following:

●Angstrms #Obs Theory %Compl Redund Rsym Pairs %Pairs Rshell #Sigma %<2s

Explain each of these headings, and summarize the conclusion that you draw from the output.

7.

(a) What is the symmetry of your diffraction pattern?

(b) What are the possible point groups for your crystal?

(c) What is the probable Bravais lattice for your crystal?

(d) What are the systematic absences?

(e) What are the possible space groups for your crystal?

8. You first decide to see what you can learn from the heavy atom-Patterson-Fourier approach about the position of the heavy atom and the structure.

(a) Briefly explain this procedure.

(b) What is the expected symmetry of the Patterson map for your crystal?

(c) Using the layout shown on the following page, derive the Patterson vectors for your space group. If you wish you can only derive the symmetry unrelated vectors, but you must specify how many vectors you expect to observe, and how the Patterson space group symmetry relates them.

(d) Using the Patterson map intensities and Harker Lines and Planes, determine the heavy atom coordinates in your structure. Be sure to check with the UVW general position peak. How does it compare with what came out of the Bruker structure determination program that you used to determine the structure?

(e) Using the heavy atom position, calculate the Fourier map peak positions and compare a selected number of these positions with those determined by your Bruker structure program. It might help to calculate a bond distance or two to verify the reasonableness of the Fourier peak positions.

	XYZ	$\bar{X}\bar{Y}\bar{Z}$	$\bar{X}, \frac{1}{2}+Y, \frac{1}{2}-Z$	$X, \frac{1}{2}-Y, \frac{1}{2}+Z$
XYZ	000	$-2X, -2Y, -2Z$	$-2X, \frac{1}{2}, \frac{1}{2}-2Z$	$0, \frac{1}{2}-2Y, \frac{1}{2}$
$\bar{X}\bar{Y}\bar{Z}$	$2X, 2Y, 2Z$	000	$0, \frac{1}{2}+2Y, \frac{1}{2}$	$2X, \frac{1}{2}, \frac{1}{2}+2Z$
$\bar{X}, \frac{1}{2}+Y, \frac{1}{2}-Z$	$2X, \frac{1}{2}, -\frac{1}{2}+2Z$	$0, -\frac{1}{2}-2Y, \frac{1}{2}$	000	$2X, -2Y, 2Z$
$X, \frac{1}{2}-Y, \frac{1}{2}+Z$	$0, -\frac{1}{2}+2Y, \frac{1}{2}$	$-2X, -\frac{1}{2}, -\frac{1}{2}-2Z$	$-2X, 2Y, -2Z$	000

SINCE +1 OR -1 CAN BE ADDED TO ANY h, v, w (JUST LIKE x, y, z),
PEAKS SUCH AS $-\frac{1}{2}-2Y \rightarrow \frac{1}{2}-2Y$.

NOW, COLLECT REDUNDANT PEAKS:

PEAK (hvw)	RELATIVE WEIGHT		PEAK (hvw)	RELATIVE WEIGHT
0, 0, 0	4		$2X, 2Y, 2Z$	1
$0, \frac{1}{2}+2Y, \frac{1}{2}$	2	} ALONG HARKER LINE	$-2X, -2Y, -2Z$	1
$0, \frac{1}{2}-2Y, \frac{1}{2}$	2		$2X, -2Y, 2Z$	1
$2X, \frac{1}{2}, \frac{1}{2}+2Z$	2		} IN HARKER PLANE	$-2X, 2Y, -2Z$
$-2X, \frac{1}{2}, \frac{1}{2}-2Z$	2			

Example of one heavy atom in $P2_1/c$