

CHEM 273/279 SPRING 2009  
TAKE-HOME FINAL EXAM, **PART 2**  
**DUE: WEDNESDAY, JUNE 10, AT NOON**

Name \_\_\_\_\_

Perm number \_\_\_\_\_

Date submitted \_\_\_\_\_

This and the following Part 3 of the final exam will be primarily built around the Sheldrick software, tutorial, and your data output. The focus is on (1) preparation and assessment of the data to be used for structure solution by Patterson or Direct Method techniques (XPREP); (2) the direct method solution procedure (SHELXS); and (3) structure refinement (XP and SHELXL).

As guides, use the shelx online tutorials: [shelx.uni-ac.gwdg.de/tutorial/english/intro.htm](http://shelx.uni-ac.gwdg.de/tutorial/english/intro.htm) and the uploaded lecture web pages.

Each individual is expected to submit his or her own, independent answers to the exam.

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		
TOTAL for Part 2		

**Questions 1–5 concern XPREP -- data preparation:**  
<http://shelx.uni-ac.gwdg.de/tutorial/english/xprep.htm>

**1.**

- (a) What is defined by Shelx as a “run”?
- (b) For your data, how many runs were made?

**2. In the Shelx tutorial, the following example is given:**

*Example one:* The reflection (326) appears three times in the list, i.e. it has been measured three times during the experiment. First, the reflection has been observed in run 1 with a intensity of 57.80 (and an error of about 6%), the second time in run 3 with a intensity of 50.34 (7% error) and the third time in run 4 with a intensity of 53.71 (6% error). For reflections measured more than once, the intensities may (and should) be averaged. In this way, the determined values become more accurate and the errors are reduced.

- (a) Define error in terms of the  $\sigma(I)$  given in column 5. Verify the values of the errors given for each of the three given measurements of the reflection (326)
- (b) In the example given in the tutorial, the mean intensity of all data divided by its error - mean ( $I/\sigma$ ) - is calculated and given as 8.86. What is the significance of this number?
- (c) How many unmerged data were measured for your crystal?
- (d) What is the value of mean ( $I/\sigma$ ) for your data?

**3. The values of mean ( $I/\sigma$ ) =  $\langle I/s \rangle$  and  $I > 3s$  are next used in the tutorial to determine the lattice type and space group by using the appropriate “absence laws”.**

- (a) Using the values of  $\langle I/s \rangle$  and  $I > 3s$ , explain how it was concluded in the tutorial example that the space group is  $P2_12_12_1$ .
- (b) Do the same analysis for your crystal structure.
- (c) Was there any change in the assignment of the unit cell at this point in your structure determination? If so, what was the change?

**4. E value statistics are introduced at this point in the tutorial.**

- (a) Define E.
- (b) What E statistic is given in the tutorial at this point? How does it confirm the space group  $P2_12_12_1$ ?

- (c) How did the E value statistic confirm the space group for your crystal?
- (d) What is meant by CFOM for the space group determination?
- (e) What is an optimum value for CFOM and what was it for your crystal?

**5. The following refers to the final analysis and merging of your data.**

- (a) What is a resolution shell?
- (b) Define completeness.
- (c) What was the range of completeness (%) for the first 11 resolution shells of your data?
- (d) Define redundancy in terms of the diffraction data that are measured.
- (e) Define  $R_{\text{int}}$  and  $R_{\text{sigma}}$ .
- (f) What was the range of  $R_{\text{sigma}}$  values for your crystal data?
- (g) Did you truncate your crystal data set? Should you have?
- (h) How many reflections are in the final data set that is used for structure determination in the Shelx tutorial example?
- (i) How many reflections were in your final data set and used for structure determination?
- (j) Give the last line data for your data set analogous to that in the tutorial, i.e.,

```
Inf - 0.80 5580 5584 99.9 6.37 64.0 20.99 0.0486 0.0344
```

What are the key parameters in this line for assessing the quality of your data?

**6. (from Professor Seshadri)**

- (a) Given only a powder diffraction dataset, what are the different pieces of other experimental information that might help solve a crystal structure ab-initio?
- (b) Give a detailed outline of the steps required to solve a structure from powder diffraction data, all the way to the final Rietveld refinement. Name typical software at every step and list all sources of information.