CHEM 273/279 SPRING 2009
TAKE-HOME FINAL EXAM, **PART 3**
DUE: WEDNESDAY, JUNE 10, AT NOON

Name _____

Perm number_____

Date submitted _____

Parts 2 and 3 of the final exam are 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 (XSHELL and SHELXL).

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

Reminder: 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, 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.

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		
TOTAL for Part 3		

1. Shelxs Direct methods structure solution
http://shelx.uni-ac.gwdg.de/tutorial/english/shelxs.htm
The Shelxs tutorial output is reproduced on the following page.

(a) What is a TPR?

(b) How is the RE value determined?

(c) What are the criteria for good CFOM and RE values?

(d) Compare your best solution CFOM and RE values with those given for the Shelxs example.

(e) Based on this is your best solution satisfactory?

2. XShell/XP

(a) What is the purpose of the XShell (XP) program?

(b) When and why would you use it?

3. Shelxl http://shelx.uni-ac.gwdg.de/tutorial/english/verf.htm
In the refinement process the following steps are suggested in the tutorial:

1. Assignment of all atoms except for hydrogen atoms
2. Correction of wrongly assigned atoms (if necessary)
3. Refining the anisotropic displacement of the non-H atoms
4. Identification, placement and refinement of the hydrogen atoms
5. Looking at special structural features like crystal packing and hydrogen bonds, if present
6. Documentation of the structure determination with structural pictures

(a) List the output values for your refinement that are used to determine the quality of the refinement of your structure. If you did not do a structure, you may use the values given in the tutorial example (if you did do a structure, you will be given zero points for using the tutorial example!)

(b) Why would you want to use an ORTEP plot?

(c) Did you check the bond distances and angles for your structure refinement? Were there any anomalies? If so, describe.

(d) What is the purpose of HFIX? When and why is it used?

(e) What is the purpose of AFIX? When and why is it used?

(f) How would you look for hydrogen bonding?

(g) Did you check the crystal packing in your structure? How would you start this process?

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++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++
+  SHELXS-97  -  CRYSTAL STRUCTURE SOLUTION  -  UNIX VERSION  +
+  Copyright(C) George M. Sheldrick 1986-97     Release 97-2  +
+  momo-new               started at 20:26:32 on 19 Jan 2002  +
++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++

Read instructions and process reflection data
Data:    3203 unique,   2772 observed     R(int) = 0.0186  R(sigma) = 0.0280
Systematic absence violations:    3    Bad equivalents:    0

ESEL  Emin  1.200    Emax  5.000    DelU 0.005    renorm 0.700    axis 0
OMIT  s  4.00    2theta(lim)  180.0
INIT  nn   12    nf   16    s+  0.800    s-  0.200    wr  0.200
PHAN  steps   10   cool 0.900   Boltz 0.300   ns  176   mtpr   40   mnqr  10
TREF  np      256.    nE   293    kapscal  0.850    ntan   3    wn -0.375
FMAP  code  8
PLAN  npeaks   -52    del1 0.500    del2 1.500
MORE  verbosity  1
TIME  t    9999999.

    176 Reflections and   2286. unique TPR for phase annealing
    293 Phases refined using    9713. unique TPR
    412 Reflections and   15485. unique TPR for R(alpha)
   2624 Unique negative quartets found,  1376 used for phase refinement
    219 Unique NQR employed in phase annealing
    128 Parallel refinements,  highest memory =    7200 /   86987

  Try    Ralpha Nqual Sigma-1 M(abs) CFOM   Seminvariants
 990177. 0.085 -0.661  0.669  0.941  0.085* +---+ ++-++ ++-+- +---+ ---+-
Freq: 0 0 0 0 2 0 1 13 11 2 3 0 4 11 9 9 12 8 5 2 4 3 6 7 1 3 2 3 1 / 128
 491881. 0.103 -0.657  0.660  0.907  0.103  --+-- +++-+ +-+-- ---+- -+-++
Freq: 0 0 0 0 2 1 2 25 23 13 8 4 10 16 13 15 22 20 10 8 7 7 11 7 3 5 / 256

     256. Phase sets refined - best is code   990177.  with CFOM =  0.0846

Fourier and peaksearch
RE = 0.199 for  36 atoms and   857 E-values
Fourier and peaksearch
RE = 0.189 for  36 atoms and   857 E-values
Fourier and peaksearch

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+  momo-new          finished at 20:26:42   Total CPU time:       9.7 secs  +
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