Syllabus
Chemistry 5755 - X-Ray Crystallography
Spring Semester, 2017
12:20 - 1:10 pm MWF in 111 Smith Hall

Instructor - Dr. Victor G. Young, Jr.
Office – 192C Kolthoff Hall
Telephone – (612)625-6897
Email – vyoung@umn.edu
Office Hours – 3:00-4:00 pm MWF or by apt.
TA – JT Moore – moor1308@umn.edu (612)624-9309 192B Kolthoff Hall


Goals:

1) To learn the essentials of crystallography through theory and practice as it applies to modern, single-crystal X-ray diffraction methods.
2) To become familiar with crystallographic literature.
3) To become a certified user of X-ray instrumentation at the University of Minnesota.
4) To learn the methodology of acquiring research-grade data on the Bruker-AXS APEX-II / PHOTON-II diffractometers. This will require the mastery of both hardware and software.
5) To learn the essentials of the SHELXL-2014 structure solution package as it applies to data corrections, structure solution, least-squares refinement, and the preparation of publication materials. SHELXle can be downloaded to your Windows or Mac laptop.
6) To familiarize oneself with the utilities of the Cambridge Crystallographic Structural Database. The CSD may only be setup on UM computers; ~17 Gb complete install.
7) To conduct a complete structural investigation on a sample of your choice (pending approval) that you will present to the class. Dates are below. Additional details will be forthcoming. A completed sample permission slip will be required from your advisor if you are in a research group. If not, then a sample will be assigned.

Lecture and Reading Schedule:

Week 1
1-18  1: Intro., Lab. Schedule, & Rad. Safety     Ch. 1, Ch. 3, pp 13-16
1-20  2: Physics of X-Ray Scattering with Matter     Ch. 3, pp 22-26
Week 2
1-23  3: Bragg's Law into Reciprocal Space  
1-25  4: Structure Factor Derivation  
1-27  5: Symmetry: Point & Border groups

Week 3
1-30  6: Plane Groups: Oblique and Rectangular  
2-1   7: Plane Groups: Square and Hexagonal  
2-3   8: Plane Groups: Toward 3d symmetry

Week 4
2-6   9: Space Groups: P1, P2, P21, Pm, Pc, and Pn  
2-8   10: Space Groups: P2/c, P21/c, and P21/n  
2-10  11: Space Groups: Cc, C2/c, and P212121

Week 5
2-13  12: Space Groups: Pbca, Pna21, and Pca21
2-15  13: Diffraction Sym. and Reflection Conditions  
2-17  14: Data Collection and Corrections

Week 6
2-20  15: Anomalous Dispersion  
2-22  16: The Phase Problem and Structure Solution  
2-24  Examination 1

Week 7
2-27  17: Fourier and Patterson maps  
3-1   18: Patterson Analysis in P21/c  
3-3   19: Patterson Analysis in P212121 and Pbca

Week 8
3-6   20: Patterson Analysis in Pna21 and Pca21  
3-8   21: Patterson Superposition (or TBD)  
3-10  22: Crystals: Growth Strategies and Quality (JTM) 

Spring Break
3-13 through 3-17

Week 9
3-20  23: Direct Methods: Theory  
3-22  24: Direct Methods: Worked Example  
3-24  25: Direct Methods: Worked Example

Week 10
3-27  26: Completion of the Structure  
3-29  Examination 2
3-31  27: Least-Squares Background

Week 11
4-3   28: Least-Squares Theory  
4-5   29: Least-Squares Restraints and Constraints  
4-7   30: Disorder in Least-Squares Refinement
<table>
<thead>
<tr>
<th>Week 12</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>4-10 31:</td>
<td>Disorder Refinement Examples</td>
</tr>
<tr>
<td>4-12 32:</td>
<td>Derivation and Interpretation of</td>
</tr>
<tr>
<td>4-14 33:</td>
<td>Results</td>
</tr>
<tr>
<td></td>
<td>handout</td>
</tr>
<tr>
<td></td>
<td>Ch. 12, 161-169</td>
</tr>
<tr>
<td></td>
<td>ITC Vol A</td>
</tr>
<tr>
<td>Week 13</td>
<td></td>
</tr>
<tr>
<td>4-17 34:</td>
<td>Space Groups: Trig., Hex., and</td>
</tr>
<tr>
<td>4-19 35:</td>
<td>Cubic SGs</td>
</tr>
<tr>
<td>4-21 36:</td>
<td>Twinning by Merohedry &amp; Pseudo-</td>
</tr>
<tr>
<td></td>
<td>Merohedery</td>
</tr>
<tr>
<td></td>
<td>ITC Vol A</td>
</tr>
<tr>
<td>Week 14</td>
<td></td>
</tr>
<tr>
<td>4-24 37:</td>
<td>Twinning by Non-Merrohedy</td>
</tr>
<tr>
<td>4-26 38:</td>
<td>Pseudosymmetry</td>
</tr>
<tr>
<td>4-28 39:</td>
<td>Modulated structures</td>
</tr>
<tr>
<td></td>
<td>handout</td>
</tr>
<tr>
<td></td>
<td>Ch. 11, 146-155</td>
</tr>
<tr>
<td>Week 15</td>
<td></td>
</tr>
<tr>
<td>5-1 40:</td>
<td>Presentation Content Review (JTM)</td>
</tr>
<tr>
<td>5-3 41:</td>
<td>Hypersymmetry in Pna2₁ and Pca2₁</td>
</tr>
<tr>
<td>5-5</td>
<td>**Final Presentations / Take-home</td>
</tr>
<tr>
<td></td>
<td>Examination 3 (24 hrs.)</td>
</tr>
</tbody>
</table>

**Course Lecture Materials:**

All of the PowerPoint presentations (in PDF format) and other notes will be distributed via email directly to the students.

**Grades:**

This is a 4-credit graduate-level course in Chemistry with **A-F grades only**. The final grade will be calculated based on exams (30%), homework (30%), final project (20%), and laboratory participation (20%). Three exams are planned. The quality and completeness of the final project will be judged on a variety of factors. The major goal of the final project is that it will be of high quality and worthy of publication in leading scientific journals. Your participation in all laboratory exercises is essential preparation for your final project. Please be on time for any scheduled laboratory!

**Final Project Presentations:**

All students will make their final project presentations during the last regularly scheduled class period Friday, May 6\(^{th}\) between 12:00 – 3:30 pm (or at another time to be arranged). The tentative location will be 193 Kolthoff Hall. **Students must provide completed reports and electronic files by 4 pm to JTM on Thursday, May 4, 2017.** PowerPoint presentations are encouraged. Judging will be made by a jury composed of 3-4 crystallographers including Victor Young and JT Moore.
Homework:

About 10 homework exercises will be assigned through the semester. This will be in addition to the projects assigned in the laboratory component.

Reading and In-Class Quizzes:

Reading and in-class quizzes will be given occasionally based on assigned reading material and the previous lecture. Grades from these will be part of the examination component. Be prepared by reading assignments before the lecture.

Academic Dishonesty:

Cheating will be dealt with in accordance to the University of Minnesota policy found at http://www1.umn.edu/regents/policies/academic/StudentConductCode.pdf. Many other policies regarding classroom expectations can be found at the same site.

Smartphone use in class:

Smartphones should not be used during class lecture periods. Usage of these distracts everyone in a small class. Please turn off all smartphones during class. This same advice extends to any other technology that distracts the class.

Laboratory Exercises:

The initial step for working in the X-ray laboratory is to pass the Radiation Safety Orientation and examination online at http://www.dehs.umn.edu/training_new_empl.htm. You will have to login with your UMN x500 computer account. Please complete both short courses prior to scheduling your first laboratory exercise. After successfully completing the test at the conclusion of the online training you will be sent an email: please forward a copy to the TA. All students must maintain a detailed laboratory notebook. A laboratory exercise schedule will be arranged during the second week of class. This will be determined by the TA once students submit their schedule of classes, TA assignments, and research group meetings.

Week 2, Jan. 23: Radiation safety instruction and exam must be completed. Email X-ray radiation safety exam confirmation to TA. X-ray Laboratory tour.

Week 3, Jan. 30: Microscope familiarization; polarization, still pictures and movies, crystal cutting, mounting, and centering.

Week 4, Feb. 6: Unit cell determination and refinement. Setting up and starting a data collection. Distribute Cambridge Structural Database self-guided tutorial.

Week 5, Feb. 13: Start to finish unaided crystal mounting, centering, and unit cell determination (peer advisement only).
Week 6, Feb. 20: Data integration (APEX-II SAINT), absorption correction (APEX-II SADABS-2014), space group determination (APEX-II XPREP), structural solution (SHELXT-2014), structural refinement (SHELXL-2014), structural manipulation (SHELXe).

Week 7, Feb. 27: Begin independent collections for final projects.

Week 8, Mar. 6: Structural refinement and report writing tutorial. Tentative all-class laboratory is scheduled for the Chemistry Microcomputer Laboratory 101D Smith Hall.

Mar. 14 – 18: Spring break – No laboratory activities.

Week 9, Mar. 20: **Practice structures 1-3 due 4 pm Friday**, March 24, 2016. Start independent data collections for final projects.†

Week 10, Mar. 27: Continue independent collections for final projects.

Week 11, Apr. 3: **Practice structures 4-6 due 4 pm Friday**, April 7, 2016. Continue independent collections for final projects.

Week 12, Apr. 10: Continue independent collections for final projects.

Week 13, Apr. 17: **Practice structures 7-9 due 4 pm Friday**, April 21, 2016.‡

Week 14, Apr. 24: Continue independent collections for final projects.

Week 15, May 1: Final project preparation week.

* Complete reports on the nine practice structures will consist of the SHELXL *.RES, the SHELXL *.LST, the completed SHELXL *.CIF, and the CheckCIF report *.PDF. Additional information will be provided in the structural refinement and report writing tutorial in week 8.

† **A laboratory practical examination** must be completed and passed prior to your independent data collection. This examination will include crystal mounting and centering, unit cell determination and refinement. All students are encouraged to acquire data as soon as possible following their successful passing of the laboratory practical examination.

‡ All 9 practice structures must be completed satisfactorily before data for your final project data will be released to you.