

Chemistry 832: Solid State Structural Methods Syllabus**Dr. Allen D. Hunter****Department of Chemistry, Youngstown State University
Spring 2000**April 6th, 2000 Version of Syllabus**Course Credit:** 3 Quarter Hours of Credit (2 Hours per Week Lecture and 3 Hours per Week Lab)**Q2S Notice:** Under Semesters this course will become Chemistry 5832: Solid State Structural Methods which will have 3 Semester Hours of Credit (2 Hours per Week Lecture and 3 Hours per Week Lab) for a 50% increase in total contact time. The content will be expanded to include greater coverage of Symmetry and the Solid State, Powder Diffraction, Protein Crystallography, and High Resolution Diffraction Studies.**Lecturer:** **Dr. Allen D. Hunter** (Office 5015, NMR Lab 5031, X-Ray Lab 5024/5020, Advanced Synthesis Lab 5005)
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Dr. Hunter's Home Page: <http://www.as.yzu.edu/~adhunter/index.html>
Chemistry 832 Home Page: <http://www.as.yzu.edu/~adhunter/Teaching/Chem832/index.html>**Required Texts:**

- (1) Electronic Syllabus and Course Instruction (Required, will be updated frequently)
- (2) Glusker, J. P.; Lewis, M.; Rossi, M. "Crystal Structure Analysis for Chemists and Biologists" VCH, New York, 1994 (Required text).
- (3) X-Ray Structure Solution via SHELXTL: A Beginner's Introduction (Required Text. A master copy may be borrowed from Dr. Hunter for duplication and/or it may be obtained as a .pdf file from him for printing out and/or reading on screen).
- (4) WEB Handouts (Required)
- (5) WEB Links/References (Required, may be accessed in NT lab)
- (6) Diffraction Related Programs (Required, may be accessed in NT lab)
- (7) Hard Cover Laboratory Note Book (Required).
- (8) XSCANS Software Tutorial/Users Guide (available in lab).
- (9) XSCANS Technical Reference Manual (available in lab).

WEB Materials:**This Course will use a rich mixture of programs and WEB related materials which you will need to consult daily. You will need an email address and an account in the "NT lab" for this course, if you do not have these, see me during the first period.**

1. Chemistry 832 Home Page: <http://www.as.yzu.edu/~adhunter/Teaching/Chem832/index.html>
2. Structural Methods Textbook Reference List:
<http://www.as.yzu.edu/~adhunter/Teaching/Chem822/reflist.doc>
3. Diffraction Methods Textbook Reference List:
<http://www.as.yzu.edu/~adhunter/Teaching/Chem832/Xrayrefs.pdf>
4. X-Ray Crystallography Links
5. YSU Structure Center: <http://www.as.yzu.edu/~adhunter/YSUSC/index.html>

Office Hours: **Dr. Hunter:** Tuesday and Thursday 7:10 to 7:40 PM and 9:30 to 10:00 PM. Monday and 10:30 to 12:00, and Wednesday 2:00 to 3:30. Dr. Hunter is also available at other times by appointment. He'll be in WB 5015 (his office), 5031 (the NMR lab), or 5024/5020 (the X-ray lab)).**Lecture:** Monday and Wednesday 1:00 to 1:50 in WB 5026 and/or the Senior (NT) computer lab.**Scheduled Lab:** Wednesday 2:00 to 4:50. WB 5024/5020 (i.e. the X-ray lab) and/or the Senior (NT) computer lab.**X-Ray Access Times:** Monday - Friday, 9:00 a.m. to 5:00 p.m. and/or by scheduled appointment. The Senior (NT) computer lab is open similar hours.

Introduction. Single Crystal X-ray Diffraction. X-ray crystallography is the most important method for determining the structures of materials in the solid state. When detailed information on atomic positions is required (e.g., so that one can determine bond distances and angles), X-ray crystallography is carried out on "single crystals." These are crystalline samples having distinct faces (examples from every day life include salt, sugar, and diamonds) and dimensions of 0.05 to 0.5 mm on an edge. In a single crystal, the molecules are arranged in a repeating pattern like the bricks in a wall with each repeating unit (i.e., each brick) containing an identical arrangement of atoms. The basic principle of X-ray diffraction is that when a beam of X-rays passes through a crystalline sample some of the X-rays will be bent (this process is called diffraction). The angles to which the X-rays are bent are determined only by the size and shape of the repeating units of the crystal (i.e., is it a "brick", a cube, a Lego block, a prism, etc.). The intensities (i.e., brightness) of each of the bent X-ray beams are determined by the arrangement and identity of the atoms within each repeating unit. If one knows the structure of the solid (i.e., the size and shape of each repeating unit and the arrangements of atoms within them), it is quite easy to calculate the angles and intensities of all of the bent X-ray beams. Unfortunately, working backwards from the angles and intensities of the bent X-rays to the structure of a solid is much more difficult. Fortunately, recent advances in X-ray crystallography and computers now make the successful solution of such problems by undergraduates possible.

Powder X-ray Diffraction. Powder X-ray diffraction is the method of choice for characterizing the structures of solids that are not available as suitable single crystals and for identifying compounds in solid mixtures.

X-Ray Diffractometers. An X-ray diffractometer is the instrument used to collect the raw data (i.e., measure the angles and intensities of the bent X-rays). It has several basic parts, including: an X-ray generator, a device called a goniometer that is used to orient the crystal so that the angles of the bent X-ray beams can be measured, and a detector that measures the brightness of each bent X-ray beam. We have recently purchased two such diffractometers from Siemens at a cost of over \$243,000. They were purchased with a combination of external grants from the National Science Foundation (\$71,199) and the Ohio Board of Regents (\$60,000) and internal Chemistry Department and University funds. One of these systems is optimized for determining the structures of powders and of crystals of large organic or biological materials (it has Dr. Wagner as its primary coordinator) and the other is optimized for determining the structures of inorganic and organometallic molecules, ceramics, minerals, and metals (I am its primary coordinator). These instruments are new models and, consequently, their operation is under computer control by IBM compatible computers. The software used, XSCANS, is very user friendly and Siemens provided excellent manuals suitable for use by beginners such as our undergraduates.

X-Ray Software and the YSU Diffraction Facility. To obtain structural information on new materials from this raw data, one must use sophisticated software and a powerful desktop workstation. The best software available for X-ray data analysis was supplied by Siemens with our purchase. It is a package of programs known as SHELXTL/XSHELL. These programs are exceedingly powerful and run on both high end IBM compatible personal computers and UNIX workstations. The PC/DOS version of the SHELXTL software package is reasonably user-friendly (i.e., about equivalent to many early DOS applications). However, it is not very "intuitive" for beginners and is accompanied by a manual that is most useful for someone already an expert in both X-ray crystallography and the computer system being used. To remedy this problem, I have written a users manual for the PC/DOS version of SHELXTL. The computers in the NT computer lab are equipped with the XSHELL version of this package. PCs for carrying out these calculations are available in the NMR and X-Ray labs and in the senior computer lab. YSU was a founding member of the Ohio Crystallography Consortium and is serving as one of this consortiums main nodes with the special responsibility of supporting the research and teaching needs of Ohio's predominately undergraduate institutions.

Preliminary Lecture Schedule (The order and relative weighting of these topics may change.):

- Introduction to Crystal Structure Analysis
- Crystals (overview)
- Diffraction by Crystals
- Symmetry in Crystals and their Diffraction Patterns
- Combining Waves to Obtain an Image
- Measurement of Structure Amplitudes
- Estimation of Relative Phase Angles
- Electron Density Maps
- Least Squares Refinement of the Structure
- Interpreting Atomic Coordinates, Conformation, Atomic and Molecular Displacements
- Preparative Routes for Inorganic Solid State Materials
- Characterization Techniques of Inorganic Solids
- Symmetry

- Powder X-Ray Diffraction

Preliminary Laboratory Schedule (The order and relative weighting of these topics may change.):

- 1 Diffractometer Components, X-Ray Safety, the X-Ray Facility, and XSCANS.
- 2 Systematic Approaches to Growing Single Crystals
- 3 Selecting, Mounting, and Centering Crystals; X-Ray Diffraction Photographs.
- 4 Preparing for Data Collection: Unit Cell and Space Group Determinations (XSCANS).
- 5 Data Collection and Reduction (T/2T, Psi in XSCANS, XPREP in SHELXTL).
- 6 Trial Solutions, Refinement, and Structure (SHELXTL: XS, XP, and XL).
- 7 Tabular and Graphic Data Presentation (XP and XCIF).
- 8 Single Crystal Project

Note Books: Everything you do in the laboratory, on the bench, diffractometer, or computers, must be recorded directly into your lab book in ink!!!! These will be regularly inspected and graded.

Grading:

30	Mid-Term Exam (end of 5 th week, tentative).*
25	Laboratory Assignments and Structure Solution Problems.
30	Final Exam (<u>Friday June 9th at 10:30</u>).*
15	Project
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100	Total Points

90-100% - A; 75-89% - B; 60-74% - C; 50-59% - D; <50% - F

*Note: both exams will include questions based on the laboratory materials and there will be significant overlap in the materials covered in the two parts of the course.

Attendance: Lecture attendance is mandatory as is attendance at the scheduled laboratory demonstrations. In addition, during the days when data is being collected on the students single crystal, the students will be expected to be present for longer hours. There will be midterm and final exams that will cover the materials presented in the lectures and illustrated in the labs. **MAKE-UP EXAMS WILL NOT BE GIVEN.** Absences that have not been approved in advance will result in a grade of **ZERO** for that exam. Approved absences for sporting events, holidays, car breakdown, etc., will be given only if I am informed in advance and only if I agree. Absences for health reasons or family emergencies must be discussed with me within 24 hours of the missed quizzes for approval to be granted. The points for exams missed during approved absences will be applied to the final exam. An exam must be submitted for re-grading within 48 hours after it has been returned. The whole exam will be re-graded, not just single questions. If you miss a lab, it is your responsibility to your team to make up the work that is your part of the team's effort. **Chemistry 832 is an integrated lecture/lab class. Therefore, if you do not successfully complete all of the experiments and pass the lab, you will not pass the course.**

Reading Assignments: Mandatory reading assignments from the required texts and various WEB sites will be given each week. These will always include the chapters cited above. You will be responsible for reading this material **before** each class. Other readings from the Reading List will also be suggested.

Lecture and Laboratory Assignments: Assignments of various types will be given regularly in both the lecture and laboratory parts of this class. They are mandatory and must be handed in by the scheduled times. Late assignments will be given a reduced grade. Some assignments may be designated by me as group assignments. In these cases, single group reports should be handed in. All other assignments are *individual* assignments. You must only hand in your own work for these.

Safety Note: *The potential of risk is present in some lecture demonstrations and laboratory experiments. Accidents have been rare, but have happened. Faculty and staff members exercise great care to minimize and, where possible, eliminate all potential hazards. Additionally, minimization of risks requires that students come well prepared for each assigned exercise and are attentive in class. Safety glasses must be worn in the laboratory at all times. The X-ray diffractometers employ intense X-ray sources. If you override the safety interlocks, there could be serious injury to you and others. The posted safety rules and those in the handouts will be strictly enforced!. Failure to follow these rules will result in a reduced grade, up to and including an F, expulsion from the lab, and/or other sanctions! Unauthorized uses of the computer systems will result in similarly severe sanctions. In no case are computer disks that have EVER been used on computers outside of this lab to be used on our computers!!!!!! Computer Viruses are Endemic in the Real World and Especially on Campus!!!! You must keep your box of disks for this class in the X-ray lab, do not take them out of it!!!!*