Chemistry C631 – Chemical Crystallography

Class – 31287, Lab – 31288

Time and location	Lecture, Tuesday 11:20 – 12:30	CH311
	Laboratory, Thursday 11:20 – 12:10	A421 and A423
	Office hours Pink, Wednesday 10 -11	A423B
	Office hours Schaugaard, *tba*	CH311

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Course description

The course "Chemical Crystallography" (3 cr.) is intended for graduate students who wish to acquire a basic understanding of crystallography, the mathematical foundations of diffraction principles, and hands-on experience in the operation of X-ray diffractometers, computer software for crystal structure determination and visualization, as well as crystallographic databases. The goal of the course is to prepare students to independently operate diffractometers and carry out X-ray structure determinations for their Ph.D. or M.S. theses. No prior knowledge of crystallography is required.

The theory portion of the course (lecture) will introduce principles of diffraction, point and space group symmetry, reciprocal space, data reduction and absorption corrections, direct and Patterson methods for structure solution, structure refinement, visualization and interpretation, as well as advanced topics such as absolute structure determination, disorder, non-crystallographic symmetry, twinning, and phase transitions.

In the laboratory portion of the course, students will learn how to design crystallographic experiments from sample preparation and the measurement of diffraction data using the instrumentation available at the IUMSC, to structure solution, refinement, and evaluation. Students will also be introduced to common crystallographic software and databases.

Students will be evaluated via class and laboratory participation, homework, quizzes, a midterm exam, and a final project. There will be no final exam. The final project will consist of a structure determination of a sample that may or may not be from the student's own research project and the preparation of a manuscript for publication in Acta Crystallographica Section C: Crystal Structure Communications or a similar journal.

Prerequisites

Calculus, physics, general chemistry, and inorganic chemistry

Textbook

Crystal Structure Determination by Werner Massa, 4th ed. (2010), ISBN 3540206442 (hardcover) or ISBN 8184898142 (soft cover). IUMSC can lend a book to you for the duration of the course. If you choose to borrow the book, we expect it to be returned at latest one week after the final project is due. The returned book must be in excellent condition, i.e. no writing, pencil marks, highlighting, or signs of damage. Otherwise you will have to purchase the book from us.

Recommended additional reading

International Tables of Crystallography, available as e-book at the IUB campus
George H. Stout & Lyle H. Jensen: *X-Ray Structure Determination: A Practical Guide*
Carmelo Giacovazzo et al.: *Fundamentals of Crystallography*
Jack D. Dunitz: *X-Ray Analysis and the Structure of Organic Molecules*

Laboratory safety

I expect that you have participated in the general laboratory safety training through IU's Environmental Health and Safety office. Additionally, the IUMSC houses potentially dangerous chemicals and

equipment. Observing best lab practices is of utmost importance. You will need to pass an X-ray safety exam and an IUMSC-specific exam before beginning class and lab work. Only after passing both exams and being registered with the IU radiation safety office will you be able to access the lab area and operate equipment. Besides ionizing radiation producing equipment we have other hazards in the laboratory, including high voltage, chemicals, cryogenic liquids, high and low pressure equipment, and sharps. If you are unsure about a lab area or specific equipment, please ask IUMSC staff for help!

Access to computer laboratory and diffractometer rooms

You will be issued a key that lets you access A421, our computer laboratory. Independent access to the diffractometers may be permitted only after successfully completing the class. Note that when entering laboratory areas, all common and X-ray equipment specific safety rules must be observed. Failure to do so will result in your access to our instrumentation being permanently revoked.

Computer access

You may use any crystallography workstation in A421 for your class work. Please use your ADS password to log onto our systems. You must store all you class work onto your "X-drive" (residing on our server and accessible to you from all IUMSC computers upon login); failure to do so may result in loss of your data! Again, no data should be stored on a local drive.
You may use the crystallography workstations also for other scientific work, e.g., searching crystallographic databases. When using our systems, please follow our guidelines about data storage and cyber security, and use our computers for academic purposes only. Please also check the IUMSC calendar at http://www.iumsc.indiana.edu/aboutIUMSC/calendar.html for room availability when planning your individual study time on our computers. Several classes utilize our lab, and our computers may be booked for them.

Software and databases

All software and databases used in the course are available on our general purpose crystallography computers in A421. We have both proprietary and public domain software. For some of the proprietary software packages we have site licenses, which allow us to install the programs on your research group's or personal computer. Please contact IUMSC staff if you have questions or need assistance with software installation.

Homework

There will be individual and group homework. For the latter, please list on your homework sheet with whom you worked. Please turn your homework assignments in on the due date, typically one week after the assignment has been given. If you are one week late you will only receive 50% of the credit; after this time no partial credit will be given, resulting in 0% credit.

Office hours and getting help

Office hours will be determined in the first week of class via web poll. Additionally, IUMSC staff will help with technical problems on computers and instrumentation any time during normal lab operation hours.

Quizzes and Exams

Lab quizzes. There will be a 5 min online, oral, or written quiz preceding every lab that will assure that you understand the topic covered in the following laboratory exercise. A passing grade is necessary to proceed with the lab exercise.
Lecture quizzes: There will be 4 quizzes throughout the course, ca. 15 minutes each during lecture time, and they will not be announced. They will be scheduled at the beginning of class time and there will be no make-up times for latecomers. It is your responsibility to be on time for each quiz. Quizzes will cover topics covered in the preceding lectures.

Midterm: One Midterm exam will be scheduled during laboratory time. This exam will test your knowledge of information covered in all prior lectures and lab sessions.

Final Project: There is no final exam, but instead a final project. Data collection for final projects will be scheduled during the last month of classes (**12-Nov to 07-Dec-2015**). The final project consists of an individual X-ray structure determination from selection of the crystal including measurement, structure solution and refinement, the production of a publication quality representation of the structure including a Crystallographic Information File (CIF) ready for submission to *Acta Crystallographica C*, or *E* or the

Journal of Chemical Crystallography. The compound can be chosen from your own research project. Alternatively, we can supply one. Please make arrangements for instrument use ahead of time. The deadline for measurement time sign-up is **12-Nov-2015 at 5 p.m.** Please also consider that your first choice compound may not work and have a back-up plan. If problems arise during your final project you may only consult with the instructor. While group work in the laboratory is encouraged during exercises, consultation with any other person than the instructor for your final project is strictly forbidden. You will give a formal oral presentation on your project to the class during finals week. You will also score and critique your peers. The written report for your final project is due on **16-Dec-2015 at 5 p.m.** No late submissions will be permitted.

Group Projects

There will be several laboratory group projects (2 people per group) to conserve instrument time. Please be considerate and work in such a way that both you and your partner get a fair share of practice.

Attendance and participation

Attendance is important! You are expected to attend class and laboratory, to arrive on time and to stay for the duration of the class and lab period. You are expected to participate in class discussions and laboratory exercises. The instructor will keep track of your participation. If you need to miss a class or lab session, please inform the instructor well in advance. You are responsible for all announcements and material covered in class, whether or not the topic is in the textbook. It is your responsibility to obtain missed lecture and lab notes, copies of handouts, and announcements regarding changes in the syllabus. Further, it is your responsibility to make arrangements for missed laboratories.
No cell phone use during class and lab exercises please, neither for talking nor texting!! Put your cell phone on silent for the duration of the class or laboratory. If you need to use your cell phone during your individual practice time in our laboratory, please be considerate and either step out of the laboratory or make sure that you do not disturb other activity in the lab.

Grading

The full range of letter grades and +/- will be given.

50% Class participation, homework, lab work, and quizzes
20% Midterm
30% Final project and presentation

Credits and workload expectations

The definition of one credit hour at IU is defined as one hour of classroom or direct faculty instruction and a minimum of two hours of out-of-class student work each week for approximately 15 weeks for one semester of credit. Please expect to spend a substantial amount of time (at least 6 hours per week) individually in the lab and on computers to achieve proficiency in the method taught.

Students with Disabilities

Students with disabilities that affect their ability to participate fully in class or to meet all course requirements can arrange reasonable accommodations through the Disability Services for Students at IUB, http://studentaffairs.iub.edu/dss/, 812-855-7578. Students who have concerns about disabilities should contact this office within the first week of class.

Course Drop Policy

It is the student's responsibility to know about the deadlines for dropping C631; please see the official academic calendar on the IU Registrar's webpage for deadlines. Dropping the class after the official deadline will result in a grade of "W" (withdrawal) unless the student qualifies for an incomplete ("I"). Please note that according to the official Academic Guide, "The grade of Incomplete may be given only when the completed portion of a student's work in the course is of passing quality."

Scholastic dishonesty

I am expecting that you have read and understood IU's ***Code of Student Rights, Responsibilities and Conduct*** and that you will uphold and maintain academic and professional honesty and integrity. For more information please see, http://www.iu.edu/~code/code/responsibilities/academic/index.shtml.

Lecture and lab calendar*

Week	Lecture topic	Laboratory exercise
1	*Tuesday, 25-Aug-2015* Crystallization (nucleation and growth), crystallization techniques. X-ray generation. Scattering and diffraction.	*Thursday, 27-Aug-2015* X-ray safety and site orientation. ***Radiation and IUMSC safety exam.***
2	*Tuesday, 01-Sep-2015* Crystals and symmetry. Symmetry elements and point groups. Crystallographic point groups. Crystal systems.	*Thursday, 03-Sep-2015* Sample selection and preparation, mounting techniques
3	*Tuesday, 08-Sep-2015* Tessellation: Plane groups and pentagon tiling. The crystallographic unit cell. Centering. Bravais lattices. Fractional coordinates. Miller indices.	*Thursday, 10-Sep-2015* Unit cell determination.
4	*Tuesday, 15-Sep-2015* Space groups. Derivation of space groups, equivalent positions, special positions. Chiral molecules and space groups.	*Thursday, 17-Sep-2015* Unit cell determination followed by face indexing.
5	*Tuesday, 22-Sep-2015* Diffraction in crystals. The Bragg and Laue equations. Reciprocal space and reciprocal lattice. The Ewald sphere.	*Thursday, 24-Sep-2015* Data collection
6	*Tuesday, 29-Sep-2015* Reciprocal Space, cont. Atom form factors, structure factors, and X-ray intensities.	*Thursday, 01-Oct-2015* Diffractometer and experiment geometries, detectors. Data space & data reduction
7	*Tuesday, 06-Oct-2015* Structure factor, cont. Systematic absences and space group determination. Friedel's law. Intensity statistics.	*Thursday, 08-Oct-2015* Data evaluation and reduction, i.e., integration. Statistics and space group determination.
8	*Tuesday, 13-Oct-2015* Data reduction (absorption, Lorentz, polarization corrections). Structure factor and electron density. Fourier transforms and Patterson Method.	*Thursday, 15-Oct -2015* ***MIDTERM***
9	*Tuesday, 20-Oct-2015* Structure solution: Fourier transforms. The Phase problem. The Patterson Method.	*Thursday, 22-Oct-2015* Structure solution
10	*Tuesday, 27-Oct-2015* Structure solution: Harker-Kasper inequalities. Direct methods and newer variants. Charge flipping.	*Thursday, 29-Oct-2015* Structure solution (Patterson, direct methods, dual space methods, intrinsic methods, charge flipping).

11	*Tuesday, 03-Nov-2015* Structure refinement. Least-squares refinement. Model building and the difference electron density map. Constraints and restraints. Thermal displacement. Hydrogen atoms. Disorder.	*Thursday, 05-Nov-2015* Structure refinement.
12	*Tuesday, 10-Nov-2015* Fourier maps. Model evaluation. Disordered structures.	*Thursday, 12-Nov-2015* Disorder refinement.
	Thursday, 12-Nov-2015 5 p.m. Sign up for final project measurement time.	
12 - 15	***FINAL PROJECT DATA COLLECTIONS (please reserve the instrument) AND REFINEMENT (on your own using lab computers in A421).***	
13	*Tuesday, 17-Nov-2015* The crystallographic information file (CIF). Data bases in crystallography	*Thursday, 19-Nov-2015* Exercises using CSD and ICSD searches.
14	*Tuesday, 01-Dec-2015* Final Refinement. Interpretation and presentation of results. CIF generation.	*Thursday, 03-Dec-2015* CIF evaluation tools
15	*Tuesday, 08-Dec-2015* Special topics: Anomalous scattering and absolute configuration. Twinning. Pseudo symmetry. Phase transitions.	*Thursday, 10-Dec-2015* Refinement of Flack parameter. Twin indexing. Data reduction for twins. Twin refinement.
16 – Finals week	***10 MIN FORMAL ORAL PRESENTATION AND 5 MIN QUESTION/ANSWER/REVIEW PERIOD ON YOUR FINAL PROJECT, CA. 2.5 HRS TOTAL.***	
	16-Dec-2015 5 p.m. - deadline for submission of final project	

* The outline is tentative and subject to change.