

Chemistry 396/445

Spring 2019

Course: Chemistry 396/445	Textbook: Microstructural
Lecture: Tuesday Thursday 4:15-5:30-P Flanner 105	Characterization of Materials by Brandon and Kaplan, 2nd edition
Instructor: Prof. Jacob Ciszek Flanner Hall 122 E-mail: jciszek@luc.edu	Website: Sakai (sakai.luc.edu)

Course Philosophy: This course is designed to familiarize the student with the techniques of X-crystallography and electron microscopy. This includes the fundamental principles which underpin the instruments, instrument and detector construction and operation, sample preparation, and sample analysis. As this course is designed to appeal to dedicated students, four hands on “Practicals” are also included in the course as are peer discussions to facilitate learning. On a side note, the small class size means distractions are easily amplified; as a consequence, laptops should not be used except for presentations (2/19, 4/9) and for crystal structure refining (3/21, 3/26).

Office Hours: Office hours will be held during the following time slots (3h total):

Monday 4:00-6:00 P

Tuesday, Thursday 5:30-6:00 P (except 4/25)

Academic Honesty & Discipline: Honesty is the foundation of the academic system and hence is of the utmost importance. All Practical reports should be exclusively your own work and no portions should be copied from any other sources. In the unfortunate event that a student is caught cheating, 50 points will be deducted from your total grade and you will be brought to the attention of the Department Chair and Dean of the College who will determine if further action should be taken.

Grading: Grading for the course can be broken down into 5 assessment categories described separately below.

Practicals: To increase the practical familiarity of the student with the instruments and techniques, 4 immersive practical experiments have been designed. These are designed to be completed in a single classroom period, though some (such as Practical 4) will require you to come back and collect your experiment on a second day. You must satisfactorily image the samples, section the materials, solve the crystals structure, and grow the crystals to receive full points. Half credit is given for performing the experiment but getting unsatisfactory results (no crystals, etc.). In the case that half credit is given, or that the practical is missed, a report can be produced which will replace the practical’s grade. These are graded on a 0-15 pt scale with 10 of 15 roughly equating to minor but regular errors and 5 of 15 equating to multiple major errors.

Discussion: On two occasions class consists of a student lead discussion of the material covered to that point. You are expected to make significant contributions to the discussion at least three times. By the end of the discussion, if you have not contributed enough, you will be asked if you wish to add anything.

Presentations: For Practical 2 and 4, it is not possible for an individual student to experience all the techniques. Thus the lecture immediately following the Practical consists of students presentation to the class. You will thus learn the other techniques from your peers. A presentation should include at a minimum 1) the scientific principle behind the technique, 2) an

explanation of how the instrumentation functions, 3) step-by-step instructions of how it was operated (including photos) 4) results obtained. All portions should have appropriate figures. You will also be graded on how you answer questions from the audience.

Presentation Q&A: You (as an audience member) must contribute at least 3 useful questions per presentation session.

Oral Exam/Final: Rather than force content into multiple choice questions, or assign open ended questions which lead to unclear answers, you will be assessed orally. The oral exams/finals will take as long as is necessary to accurately assess student comprehension. This could be as long as 20-30 minutes for the exam and 30-40 minutes for the final. Scores of 40% have only a passing understanding on instrument function and preparation techniques. Scores of 60% correspond to having a solid but rudimentary grasp of the instrument's function, and perhaps not all the details of its operation and processing. Scores of 80% would be able to expound on most of the major concepts. Scores exceeding 90% would be missing only a detail here or there.

Grading scale:

Practicals:	4 × 15 pts	60	A > 93%
Discussion	2 × 10 pts	20	A- > 90%
Presentations	2 × 15 pts	30	B+ > 87%
Presentation Q&A	6 (2 × 3 pts)		B > 83%
Oral Exam		50	B- > 80%
Oral Final		<u>85</u>	C+ > 77%
Total		251	C > 73%, C- > 70%, D > 60%

Typically, the scores on the Practicals, Discussions, and Presentations will be quite high (>95%). Scores on the oral exam and final will cover a large dynamic range. Thus grades on the oral exam/final are usually determinant as to whether a student receives an A or a C.

Resources:

BK Brandon and Kaplan – **Microstructural Characterization of Materials**, 2nd ed.

BR Bozzola and Russel – **Electron Microscopy**, 2nd ed.

R Rhodes – **Crystallography Made Crystal Clear** 3rd ed.

O Ooi – Principles of X-ray Crystallography

GLR Glusker, Lewis and Rossi – Crystal Structure Analysis for Chemists and Biologists

M Muller – Crystal Structure Refinement: A Crystallographer's Guide to SHELXL

RefA Royal Swedish Academy of Sciences – **Scientific Background on the Nobel Prize in Chemistry 2017**

RefB Feidenhans – Surface structure determination by X-ray diffraction

JJR J.-J. Rousseau – Basic Crystallography

Due to guest speakers, this course's schedule is fairly tentative. Exam date will not change.

Tentative schedule

1/15	Practical #1 Electron Microscopy	Practical #1
1/17	Syllabus, EM and X-ray techniques/instruments BK1-7, 10-24; BR 3-14; R1-15; O1-7; GLR1-25	
1/22	TEM: Abbe limit, TEM construction, imaging, applications BK130-134, 180-194, 238-247, BR3-12, 150-201	Unsatisfactory Practical P#1 due
1/24	TEM topics: Diffraction patterns, sample prep, science discovered BK90-99, 194-208, 230-233, 310-315	
1/29	TEM of biological samples: Sample prep, imaging, science discovered BR18-46, 74-117, 121-146	
1/31	CryoEM/Enzyme cytochemistry RefA BR 283-320	
2/5	Discussion	Discussion #1
2/7	SEM: e ⁻ interactions, SE/BSE, imaging, applications BK261-294, BR204-228	
2/12	SEM: Biological sample prep, analysis, science discovered BR49-69, 231-233	
2/14	Practical 2 –Dehydration, critical point drying/sputter coating, sectioning	Practical #2
2/19	Presentation on Practical #2	Presentation + Q&A
2/21	EDX/EDS: Theory, science discovered BK271-277, 341-343	Unsatisfactory Practical #2 due
2/26	Review	
2/28	Oral Exam	Oral Exam
3/5&7	Spring break	
3/12	X-ray crystallography: Structure, lattice, unit cells, symmetry, space group BK30-65, O8-64, 44-47, R49-73, GLR73-136	
3/14	X-ray: Bragg, diffraction patterns, processing results BK55-99, GLR185-205	
3/19	Guest, Prof. Lee: Chemical principles observable by X-ray crystallography	
3/21	Processing tutorial (bring laptop) O66-111, M1-164	
3/26	Practical 3 (bring laptop)	Practical #3
3/28	Misc. topics: Miller indices, powder diffraction, grazing angle scattering BK36-42, 73-76, RefB, JJR185-196, 253-267	
4/2	Crystal growth methods GLR33-65, B22-244	Unsatisfactory Practical #3 due
4/4	Practical 4	Practical #4
4/9	Presentation on Practical #4	Presentation + Q&A
4/11	Protein crystallography: Crystals and collection R31-89	Unsatisfactory Practical #4 due
4/16	Phases, Model, Assessing R91-209	
4/18	Easter break	
4/23	Discussion	Discussion #2
4/25	Guest, Prof. Liu: Biochemical principles observable by crystallography	
4/30	Final (cumulative, oral) 4:15-7:00 P	

Loyola Formal Statements:

Final Exam

The University sets the schedule for all final exams. The final will be held on: 4/30 4:15-7:00p in FH-105. There will be no make-up final exams given under any circumstance.

Instructors may not reschedule final exams for a class for another day and/or time during the final exam period. There can be no divergence from the posted schedule of dates for final exams. Individual students who have four (4) final examinations scheduled for the same date may request to have one of those exams rescheduled. If a student reports having four final examinations scheduled for the same date, students should be directed to e-mail a petition to Lester Manzano, Assistant Dean for Student Academic Affairs, CAS Dean's Office (lmanzan@luc.edu).

Student Accommodations

If you have any special needs, please let me know in the first week of classes. The university provides services for students with disabilities. Any student who would like to use any of these university services should contact the Student Accessibility Center (SAC), Sullivan Center, (773) 508-3700. Further information is available at <http://www.luc.edu/sac/>.

Academic Integrity

All students in this course are expected to have read and to abide by the demanding standard of personal honesty, drafted by the College of Arts & Sciences, which can be viewed at: <http://www.luc.edu/cas/advising/academicintegritystatement/>

A basic mission of a university is to search for and to communicate the truth as it is honestly perceived. A genuine learning community cannot exist unless this demanding standard is a fundamental tenet of the intellectual life of the community. Students of Loyola University Chicago are expected to know, to respect, and to practice this standard of personal honesty.

Academic dishonesty can take several forms, including, but not limited to cheating, plagiarism, copying another student's work, and submitting false documents.

Any instance of dishonesty (including those detailed on the website provided above or in this syllabus) will be reported to The Chair of The Department of Chemistry & Biochemistry who will decide what the next steps may be.

Loyola University Absence Policy for Students in Co-Curricular Activities (including ROTC):

Students missing classes while representing Loyola University Chicago in an official capacity (e.g. intercollegiate athletics, debate team, model government organization) shall be allowed by the faculty member of record to make up any assignments and to receive notes or other written information distributed in the missed classes.

Students should discuss with faculty the potential consequences of missing lectures and the ways in which they can be remedied. Students must provide their instructors with proper documentation (develop standard form on web) describing the reason for and date of the absence.

This documentation must be signed by an appropriate faculty or staff member, and it must be provided as far in advance of the absence as possible. It is the responsibility of the student to make up any assignments. If the student misses an examination, the instructor is required to give the student the opportunity to take the examination at another time.

(<https://www.luc.edu/athleteadvising/attendance.shtml>)

Accommodations for Religious Reasons

If you have observances of religious holidays that will cause you to miss class or otherwise effect your performance in the class you must alert the instructor ***within 10 calendar days of the first class meeting of the semester*** to request special accommodations, which will be handled on a case by case basis.