

# Chemistry 445

Fall 2024

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Course:	Characterization Pt.B:X-ray & Electron Methods	Textbook:	(required)
Lecture:	Tuesday Thursday 6:00-7:15-P Flanner 105		Yang Leng
Instructor:	Prof. Jacob Ciszek Flanner Hall 122 E-mail: jciszek@luc.edu		Materials Characterization: Introduction to Microscopic & Spectroscopic Methods, 2nd ed. (recommended)
	Prof. Progna Banerjee Flanner Hall 402 pbanerjee@luc.edu		Bozzola & Russell Electron Microscopy 2 <sup>nd</sup> ed.
		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 (10/1, 11/14) and for crystal structure refining (11/12, 11/14).

**Office Hours:** Office hours will be held during the following time slots:

Ciszek: Monday 5:30-6:15 P, Thursday 4:15-5:30 P (no office hours 9/5)

Banerjee: Tuesday 2:30-4 P, Cuneo Hall common area (turn left exiting from elevator)

**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 3) may require you to come back and collect your experiment on a second day. You must satisfactorily image the samples, section the materials, solve the crystal's 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 two 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 3, 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 equipment 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 2 useful questions per presentation session.

Exam/Final: The majority of the grade in this course comes from examinations. The final is comprehensive covering all material, though focusing slightly more on the second half of the class. Grading is on thoroughness and detail as much as accuracy. 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%
Exam		50	B- > 80%
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 exam and final will cover a large dynamic range. Thus, grades on the 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**, 2<sup>nd</sup> ed.

**BR** Bozzola and Russel – **Electron Microscopy**, 2<sup>nd</sup> ed.

**R** Rhodes – **Crystallography Made Crystal Clear** 3<sup>rd</sup> ed.

**O** Ooi – **Principles of X-ray Crystallography**

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

**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**

**YL** Yang Leng - **Materials Characterization: Introduction to Microscopic and Spectroscopic Methods**, 2nd edition

**Due to guest speakers, this course's schedule is a bit tentative\*. Exam date will not change.**

**Tentative schedule**

8/27	Practical #1 - Electron Microscopy: SEM & TEM	Practical #1
8/29	Syllabus, EM and X-ray techniques/instruments BK1-7, 10-24; BR 3-14; R1-15; O1-7; GLR1-25	
9/3	TEM: Abbe limit, TEM construction, imaging, applications BK130-134, 180-194, 238-247, BR3-12, 150-201	Unsatisfactory Practical #1 due
9/5	TEM topics: Diffraction patterns, sample prep, science discovered BK90-99, 194-208, 230-233, 310-315	
9/10	TEM of biological samples: Sample prep, imaging, science discovered BR18-46, 74-117, 121-146	
9/12	CryoEM/Enzyme cytochemistry RefA BR 283-320	
9/17	Discussion	Discussion #1
9/19	SEM: e <sup>-</sup> interactions, SE/BSE, imaging, applications BK261-294, BR204-228	
9/24	SEM: Biological sample prep, analysis, science discovered BR49-69, 231-233	
9/26	Practical 2 –Dehydration, critical point drying/sputter coating, sectioning	Practical #2
10/1	EDX/EDS: Theory, science discovered BK271-277, 341-343	
10/3	Advanced Techniques: Liquid/thermal cells, cathode luminescence, EBSD	Unsatisfactory Practical #2 due
10/8	Fall Break	
10/10	Presentation on Practical #2	Presentation + Q&A
10/15	Review	
10/17	Exam	Exam
10/22	X-ray crystallography: Structure, lattice, unit cells, symmetry, space group BK30-65, O8-64, 44-47, R49-73, GLR73-136	
10/24	X-ray: Bragg, diffraction patterns, processing results BK55-99, GLR185-205	
10/29	Guest, TBA: Argonne National Laboratory's synchrotron utilized for crystallographic studies	
10/31	Crystal growth methods GLR33-65, B22-244	
11/5	Practical 3 – Nanocrystal Growth, ShellXL software	Practical #3
11/7	Processing tutorial (bring laptop) O66-111, M1-164	
11/12	Practical 4 -Structure Solution (bring laptop)	Practical #4 Unsatisfactory Practical #3 due
11/14	Presentation on Practical #3	Presentation + Q&A
11/19	Misc. topics: Miller indices, powder diffraction, grazing angle scattering BK36-42, 73-76, RefB, JJR185-196, 253-267	
11/21	Protein crystallography: Crystals and collection R31-89	Unsatisfactory Practical #4 due
11/26	Phases, Model, Assessing R91-209	
11/28	Thanksgiving Break	
12/3	Discussion (for upcoming tests)	Discussion #2
12/5	*Guest, TBA:	
12/10	<b>Final (cumulative) 7:00-9:00 P</b>	

## **Loyola Formal Statements:**

### **Course Learning Outcomes**

The course has the following Learning Outcomes many of which are already detailed above:

- Have a working level understanding of instrument design/function, sample preparation, instrument operation, image-generation/structure-processing, and associated theoretical underpinnings.
- Have a working level understanding of data analysis and scientific conclusions that can be generated via EM and X-ray techniques
- Familiarity (theoretical, experimental, and analyses) with advanced techniques and troubleshooting of the more common impediments to good image/structure acquisition.

### **Pass/Fail Conversion Deadlines and Audit Policy**

A student may request to convert a course into or out of the "Pass/No-Pass" or "Audit" status only within the first two weeks of the semester. For the Fall 2024 semester, students are able to convert a class to "Pass/No-Pass" or "Audit" through Monday, September 9th. Students must submit a request for Pass/No-Pass or Audit to their Academic Advisor.

### **Final Exam**

The University sets the schedule for all final exams. The final will be held on:

**12/10 7:00-9:00**

You will have exactly 2 hours to complete the exam. Additional time will not be granted, even if you start late. There will be no make-up final exams given under any circumstance, and the exam will not be given early, either.

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 Adam Patricoski, Assistant Dean for Student Academic Affairs, CAS Dean's Office ([apatricoski@luc.edu](mailto:apatricoski@luc.edu)).

### **Course Repeat Rule**

Effective with the Fall 2017 semester, students are allowed only THREE attempts to pass Chemistry courses with a C- or better grade. The three attempts include withdrawals (W). The Department advises that it is preferable to complete a course with a grade of C or C-, and to demonstrate growth in future coursework, than to withdraw from a course.

After the second attempt, the student must secure Department approval for a third attempt. Students must fill out the [Permission to Register Form](#), and arrange a meeting with the Undergraduate Program Director, Assistant Chairperson, or Chairperson in Chemistry. If approved, a signed copy of this form is then sent to the student's Advising office to secure final permission for the attempt.

### **Student Support: Requests for Accommodation**

Loyola University Chicago provides reasonable accommodations for students with disabilities. Any student requesting accommodations related to a disability or other condition is required to register with the Student Accessibility Center (SAC). Professors will receive an accommodation notification from SAC, preferably within the first two weeks of class.

Students are encouraged to meet with their professor individually in order to discuss their accommodations. All information will remain confidential.

Please note that in this class, software may be used to audio record class lectures in order to provide equal access to students with disabilities. Students approved for this accommodation use recordings for their personal study only and recordings may not be shared with other people or used in any way against the faculty member, other lecturers, or students whose classroom comments are recorded as part of the class activity. Recordings are deleted at the end of the semester.

For more information about registering with SAC or questions about accommodations, please contact [SAC](#) at 773-508-3700 or [SAC@luc.edu](mailto:SAC@luc.edu).

*If you use the Testing Center, please schedule all of the tests for this class at the beginning of the semester. If a scheduled test date changes, you will still be accommodated if you had scheduled your test in advance.*

*If you have any questions or concerns regarding the implementation of your accommodations in this course, please contact the SAC for assistance.*

### **Academic Integrity**

Academic integrity is the pursuit of scholarly activity in an open, honest, and responsible manner. Academic integrity is a guiding principle for all academic activity at Loyola University Chicago, and all members of the University community are expected to act in accordance with this principle. Please open and read the foldout for the third item, "Academic Integrity" in the [Graduate School Academic Policies](#)

Academic dishonesty can take several forms, including, but not limited to cheating, plagiarism, copying another student's work, submitting false documents, and deliberately disrupting the performance of other class members. Standards apply to both individual and group assignments.

Regarding the use of Artificial Intelligence: our Provost has expressed to "Let us all make sure we are learning and sharing best practices and not allowing AI to do the learning for us." In this course, any work you submit for credit must represent your own ideas and understanding of the assigned material. If you are uncertain about any case where your use of AI may be in conflict with University or course standards, please see me to discuss your concerns.

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

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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 i.e., "[Athletic Competition & Travel Letter](#)" 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 to the professor in the first week of a semester. It is the responsibility of the student to make up any assignments. If the student misses an examination, the instructor is required to allow the student to take the examination at another time.

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

Students who will miss class for an academic competition or conference must provide proper documentation to their instructor as early in the semester as possible.

Advance notice must be sent to the instructor through Loyola email.

### **Accommodations for Religious Reasons**

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If you have observances of religious holidays that will cause you to miss class or otherwise effect your academic work in the course you must alert the instructor ***no later than Friday of Week 2 in the semester*** to request accommodations. Advance notice must be sent to the instructor through Loyola email by this deadline.

### **Universal Absence Accommodation Policy and Late/Missed Assignment Policy**

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The purpose of a universal absence accommodation policy is to account for emergency circumstances (e.g., serious illness, caring for a family member, car accident) that require you to be absent from class, while maintaining fairness in grading for students who attend and complete all in-class graded assignments. We believe that class attendance and participation are essential for your success in this class, and that your health is important to us and our shared community. Please use good judgement and stay home if necessary/prudent for your circumstances.

This is the universal accommodation policy for in-class graded assignments:

- Practicals are made up via the same mechanism used for "unsatisfactory practicals"
- Discussions should be participated in remotely, otherwise are made up via an oral quiz on a subsequent day.
- Presentations are made up via an oral quiz on a subsequent day.
- Exams will be made up via an oral exam on a subsequent day.

Accommodating a quiz or exam requires documentation justifying the absence. These accommodations are automatically available to all students.

### **Recording via Panopto**

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In this class, software will be used to record live class discussions. As a student in this class, your participation in live class discussions will be recorded. These recordings will be made available only to students enrolled in the class, to assist those who cannot attend the live session or to serve as a resource for those who would like to review content that was presented. All recordings will become unavailable to students in the class when the Sakai course is unpublished (i.e., shortly after the course ends, per the [Sakai administrative schedule](#)).

### **Privacy Statement**

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Assuring privacy among faculty and students engaged in online and face-to-face instructional activities helps promote open and robust conversations and mitigates concerns that comments made within the context of the class will be shared beyond the classroom. As such, recordings of instructional activities occurring in online or face-to-face classes may be used solely for internal class purposes by the faculty member and students registered for the course, and only during the period in which the course is offered.

Students will be informed of such recordings by a statement in the syllabus for the course in which they will be recorded. Instructors who wish to make subsequent use of recordings that include student activity may do so **only** with informed written consent of the students involved or if all student activity is removed from the recording. Recordings including student activity that have been initiated by the instructor may be retained by the instructor only for individual use.

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#### **Copyright/Intellectual Property reminder**

Course materials provided by your instructors at Loyola, including my materials, may not be shared outside any course without the instructor’s written permission. Content posted without permission will be in violation of Copyright/Intellectual Property laws. Class meetings may not be recorded without the instructor’s written permission.