

## Syllabus for Chem 314, Instrumental Analysis Spring Semester 2021

Instrumental Analysis, 3 credit hours; Prerequisite: Phys 112, Chem 212, Chem 214, and either Chem 222 or Chem 224 and 226 or permission of the instructor.

Instructor: Dr. Paul Chiarelli, Flanner Hall 102, phone 508-3106, E-mail: [mchiare@luc.edu](mailto:mchiare@luc.edu). Office hours, Monday and Tuesday 10 -11:30 AM, and Thursday 9:30 to 10:30 AM **or by appointment**. Teaching assistants are Xiomara (Xio) Martinez and Michelle Lund. Xio's ([smartinez@luc.edu](mailto:smartinez@luc.edu)) office hour is 9:30 to 10:30 AM on Thursday. Michelle's office hour ([mlund@luc.edu](mailto:mlund@luc.edu)) is from 9:30 to 10:30 AM on Wednesday. Michelle and Xio will provide recorded tutorial lectures on how to use some of the instruments.

Classes are held MWF from 12:10 to 1:00 PM via zoom. Labs are held MW from 1:30 to 5:20 PM in FH 314/315 in person.

**Course Description:** Chem 314, Instrumental Analysis, is a writing intensive, capstone course for students in BS or BA Chemistry Track. Students enrolled in this course will become familiar with modern methods of chemical analysis. Students will learn to analyze and write/present their results and analysis in a clear and concise manner. Specific learning outcomes associated with this course are listed (but not limited to) as follows:

- 1) Students will develop an understanding of the function of basic components of chemical instrumentation.
- 2) Students will be able to interpret recorded data with standard statistical methods including noise analysis.
- 3) Students will understand how different methods of chemical separation are used for the analysis of different compound classes.
- 4) Students will learn how optical spectroscopy is used to identify specific structural features of molecules.
- 5) Students will learn fundamental aspects of electrochemical analysis involving voltammetry, amperometry, and potentiometry.
- 6) Students will become familiar with how mass spectrometry is used to determine molecular weights, empirical formulas, and primary structural features of different molecules.
- 7) Students will learn how to write their experimental results in a manner consistent with chemical literature practices.
- 8) Students will learn how to use electronic resources for searching the chemical literature.

Textbook: "Fundamentals of Analytical Chemistry" (9th edition), by D.A. Skoog, D.M. West, F.J. Holler, and S.R. Crouch. ISBN-10: 0-495-55828-1. ISBN-13: 978-0-495-55828-6.

**Other Materials for lecture and lab:** You will need an inexpensive calculator having logarithmic (base 10 and base e), exponential, and trigonometric functions. Be sure you are familiar with your calculator and that it is in user-ready condition for quizzes and exams. **Calculators cannot be shared during exams and the covers must be removed while taking the exam.** You will need a laptop computer with Microsoft Excel for use in the laboratory.

## TENTATIVE CLASS SCHEDULE

<b>Date</b>	<b>Day</b>	<b>Topic</b>	<b>Chapter</b>
Jan 18	Monday	Martin Luther King Day, No class	
Jan 20	Wednesday	Introduction/Review of Fundamental Concepts	
Jan 22	Friday	Statistics, Random and Systematic Error in Measurements	5
Jan 25	Monday	Statistical Tests in Chemical Analysis	6
Jan 27	Wednesday	Statistical Tests Continued	7
Jan 29	Friday	Sampling and Calibration Methods	8
Feb 1	Monday	Calibration Methods	8
Feb 3	Wednesday	Exam 1: Chapters 5-8	
Feb 5	Friday	Introduction to Chromatography	30
Feb 8	Monday	Gas Chromatography	31
Feb 10	Wednesday	High Performance Liquid Chromatography	32
Feb 12	Friday	First Spring break , no class	
Feb 15	Monday	Ion Exchange and Ion Chromatography	32
Feb 17	Wednesday	Capillary Electrophoresis and Capillary Electrochromatography	33
Feb 19	Friday	Chromatography Detectors; FID, ECD, and Thermal Conductivity	31,32
Feb 22	Monday	Detectors continued, NPD, Photoionization, light scattering	31,32
Feb 24	Wednesday	Exam 2: Chromatography and detectors	
Feb 26	Friday	Electrochemistry: Basic Concept Review	18

March 1	Monday	Potentiometry, Reference Electrodes	21
March 3	Wednesday	Potentiometry	21
March 5	Friday	Electrogravimetry and Coulombic methods of Analyses.	22
March 8	Monday	Second Spring break	
March 10	Wednesday	Second Spring break	
March 12	Friday	Amperometric Analyses	22
March 15	Monday	Exam 3: Electrochemistry Chapters 18, 21 and 22	
March 17	Wednesday	Introduction to Voltammetry Reference Electrodes	23
March 19	Friday	Stripping Voltammetry	23
March 22	Monday	Cyclic Voltammetry	23
March 24	Wednesday	Introduction to Spectroscopic Methods Electromagnetic Spectrum	24
March 26	Friday	Instrumental Components for Absorption and Emission Spectroscopy	25
March 29	Monday	Molecular basis of Fluorescence and Phosphorescence	28
March 31	Wednesday	Instrumental components and instruments Continued.	26
April 2	Friday	Easter break	
April 5	Monday	Easter break	
April 7	Wednesday	Raman Spectroscopy	
April 9	Friday	Exam 4: Absorption and Emission Spectroscopy	
April 12	Monday	Introduction to Infrared Spectroscopy	
April 14	Wednesday	Light Sources for IR Spectroscopy	
April 16	Friday	Detectors for IR Spectroscopy	

April 19	Monday	Fourier Transform IR Spectroscopy
April 21	Wednesday	Introduction to Mass Spectrometry
April 23	Friday	Quadrupole Mass Analyzers
April 26	Monday	Time-of-Flight mass analyzers
April 28	Wednesday	Ionization methods, Electron Ionization, Electrospray, DESI, and MALDI
April 30	Friday	Ionization methods continued, Detectors.

### **Final Exam**

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

**Tuesday, May 4, 1:00-3:00 PM**

You will have two hours to complete the exam once you receive it in your E mail. Additional time will not be granted, even if you arrive 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 Lester Manzano, Assistant Dean for Student Academic Affairs, CAS Dean's Office ([Imanzan@luc.edu](mailto:Imanzan@luc.edu)).

### **Recording of Zoom class meetings**

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 course has concluded. Students will be required to turn on their cameras at the start of class. Students who have a need to participate via audio only must reach out to me to request audio participation only without the video camera enabled. The use of all video recordings will be in keeping with the University Privacy Statement shown below.

### **Privacy Statement**

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.

**Grading:** The total grade for the course is based on four exams, the final, and your writing assignments. Each exam is worth 14% of your grade. The final is worth 18% of your total grade. The lowest exam grade of the four taken during the regular semester will be dropped. You cannot drop the final. The final is not cumulative. The lecture portion is 60% of your total grade. Your lab writing assignments will constitute the other 40% of your grade. In order to do well on the exams, you need to take good notes in class, read the material in the textbook, and do the assigned problems at the end of each chapter. I do not give multiple-choice exams.

There are eight laboratory assignments outlined in the table below. Your writing assignments will consist of seven, three-page lab summaries and one nine-page report. I will correct and return the first four 3-page summaries, so you can resubmit them for a better grade. Your lab reports are due one week after you complete the lab. Your nine-page paper will be concerned with the molecular weight and amino acid sequence analysis of using ion trap mass spectrometry. The instructor will provide you will a protein (5-10,000 amu molecular weight) to identify. The first draft of the report will be due the last week of March. I will correct these reports and return them to you within a week's time. Each of the three-page summaries are worth 4% of your grade. The fifteen-page paper will be worth 12% of your grade ( $7 \times 4\% + 12\% = 40\%$ ). The grading scale is as follows:

**A** 100-93; **A-** 92-89; **B+** 88-85; **B** 84-81; **B-** 80-77; **C+** 76-73; **C** 72-69; **C-** 68-65; **D** 64-57; **F** <56.

I may adjust this scale (in your favor) over the course of the semester.

### Lab Procedures

**Laboratory Assignments:** Students are expected to arrive with a working knowledge of the content of the assigned lab and be ready to begin promptly in order to complete the various tasks. Students are expected to read the material made available via Email on Sakai prior to the lab and are encouraged to read web-based materials on various subjects as well. Students who do not prepare adequately for lab will lose points on their lab reports. Students will work together in groups of two to ensure that each student has "hands-on" experience with each instrument. Groups will follow different schedules (for the most part) over the course of the semester to ensure that students obtain experience with a wide variety of instrumentation. Because several instrumental techniques are being covered simultaneously in the laboratory, the lecture and the lab will not run in synch. **Time will be carved out of the lecture schedule to discuss basic concepts and procedures associated with each laboratory. We will also discuss how lab assignments should be written.** The first scheduled lab period will be used for these purposes as well.

Students will work in groups of two in the lab at the beginning of the semester. Three-page summaries and nine-page papers will be submitted by each group, not by students individually. We will run two different experiments each week. There are three groups of two students in each lab section. As a result, one group will be off every three weeks. Over the course of three weeks, each group will complete two lab assignments. There are eight lab assignments per group per semester. Seven of these assignments will produce one three-page reports per group. The Tandem mass spectrometry analysis of peptides will be used to generate the nine-page paper.

Working in groups is not easy. We expect you to make an honest effort to evaluate your own contribution and that of your partner to the group. At week three, you will be given an opportunity to choose a different lab partner. If an individual performs so poorly within a group that they are not “desirable” they will be expected to complete the entire lab on their own.

Week	<b><u>Tentative</u></b> Lab Schedule	
	Group 1	Group 2
Jan 18	No Lab, MLK day	
Jan 25	Introduction, Writing Summaries and Lab Reports, Statistics, Lab Procedures	
Feb 1	Ion Chromatography	UV-Vis Spectroscopy
Feb 8	UV-Vis Spectroscopy	Ion Chromatography
Feb 15	Ion Chromatography	UV-Vis Spectroscopy
Feb 22	HPLC-Fluorescence analysis of Vitamin B homologs	LC/MS-determination of protein MW
March 1	LC/MS-determination of protein MW	HPLC-Fluorescence analysis of Vitamin B homologs
March 8	HPLC-Fluorescence analysis of Vitamin B homologs	LC/MS determination of protein MW
March 15	Tandem MS of peptides	Capillary Electrophoresis of Drugs
March 22	Capillary Electrophoresis of Drugs	Tandem MS of peptides
March 29	Tandem MS of peptides	Capillary Electrophoresis of Drugs
March 29	Atomic Absorption	GC/MS of halomethanes
Apr 5	No Lab, Easter Break	Make up
Apr 12	Atomic absorption	GC/MS of halomethanes
Apr 19	Atomic absorption	GC/MS of halomethanes
Apr 26	Make up	Make up

### **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).

After the second attempt, the student must secure approval for a third attempt. Students must come to the Chemistry Department, fill out a permission to register form or print it from the Department of Chemistry & Biochemistry website:

<http://www.luc.edu/chemistry/forms/> and personally meet and obtain a signature from either the Undergraduate Program Director, Assistant Chairperson, or Chairperson in Chemistry. A copy of this form is then taken to your Academic Advisor in Sullivan to secure final permission for the attempt.

### **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 Chemistry and Biochemistry who will decide what the next steps may be. Cheating on an exam or plagiarizing a lab report will result in a grade of zero for that assignment.

### **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. An appropriate faculty or staff member, and it must sign this documentation 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.