

## Syllabus for Chem 314, Instrumental Analysis Spring Semester 2019

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. Office hours, Monday, Tuesday, and Thursday 10 -11:30 AM **or by appointment.**

Classes are held MWF from 12:35 to 1:25 PM in FH 07 (basement of Flanner Hall) Labs are held MW from 1:40 to 5:30 PM in FH 314/315.

**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 polarography.
- 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-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 14	Monday	Introduction/Review of Fundamental Concepts	
Jan 16	Wednesday	Statistics, Random and Systematic Error in Measurements	5
Jan 18	Friday	Statistical Tests in Chemical Analysis	6
Jan 21	Monday	Martin Luther King Day, No class	
Jan 23	Wednesday	Statistical Tests Continued	7
Jan 25	Friday	Sampling	8
Jan 28	Monday	Calibration Methods	8
Jan 30	Wednesday	Exam 1: Chapters 5-8	
Feb 1	Friday	Introduction to Chromatography	30
Feb 4	Monday	Gas Chromatography	31
Feb 6	Wednesday	High Performance Liquid Chromatography	32
Feb 8	Friday	Ion Exchange and Ion Chromatography	32
Feb 11	Monday	Capillary Electrophoresis and Capillary Electrochromatography	33
Feb 13	Wednesday	Introduction to Spectroscopic Methods	24
Feb 15	Friday	Instrumental Components for Optical Spectroscopy	25
Feb 18	Monday	Molecular Absorption Spectroscopy	26
Feb 20	Wednesday	Infrared and Raman Spectroscopy	26
Feb 22	Friday	Luminescence Spectroscopy	27
Feb 25	Monday	Atomic Absorption Spectroscopy	28
Feb 27	Wednesday	Atomic Emission and Fluorescence	28

Mar 1	Friday	Exam 2: Ch 24-28, 30-33	
March 4-8		Spring Break	
March 11	Monday	Electrochemistry Review: Nernst Equation, Standard Electrode Potentials	18
March 13	Wednesday	Electrochemistry Review: Reference Electrodes	21
March 15	Friday	Potentiometry and Ion Selective Electrodes	21
March 18	Monday	Electrogravimetric and Coulombic Methods of Analysis	22
March 20	Wednesday	Introduction to Voltammetry and Polarography	23
March 22	Friday	Stripping Voltammetry	23
March 25	Monday	Cyclic Voltammetry	23
March 27	Wednesday	Exam 3: Electrochemical Methods of Analysis	
March 29	Friday	Introduction to Mass Spectrometry	32
April 1	Monday	Electron Ionization Interpretation of Mass Spectra	
April 3	Wednesday	Electrospray Ionization	
April 5	Friday	Quadrupole Mass Analyzers	
April 8	Monday	Time-of-Flight Mass Analyzers	
April 10	Wednesday	Ion Trap Mass Analyzers	
April 12	Friday	Orbitrap and FT-ICR Mass Analyzers	
April 15	Monday	Protein Mass Spectrometry	
April 17	Wednesday	Tandem Mass Spectrometry of Proteins	
April 19	Friday	Good Friday, No Class	

April 22 Monday	Easter Monday, No Class
April 24 Wednesday	Imaging MS: SIMS, DESI, and MALDI
April 26 Friday	Other Chromatography Detectors; FID, ECD, Thermal Conductivity, NPD, light scattering

### Final Exam

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

**Friday, May 3, 9-11 AM**

in Flanner Hall 07. You will have exactly 2 hours to complete the exam. 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 (lmanzan@luc.edu).

**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 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 insure that each student has "hands-on" experience with each instrument. Each group will submit one written report. The specifics of which (number, length) are described below. Groups will follow different schedules (for the most part) over the course of the semester to insure 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.

Working in groups is not easy. We expect you to make an honest effort to evaluate your own contribution and that of your partners to the group. At week three, you will be given an opportunity to choose different lab partners. 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.

**Grading:** The total grade for the course is based on four exams (including the final) and your writing assignments. Each of the four exams is worth 15% of your grade (total 60%). The final is not cumulative. Your 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 twelve page report. I will correct and return the first two summaries, so you can resubmit them for a better grade. Your twelve-page report will be concerned with identifying a protein using the ion trap mass spectrometer. The instructor will provide you with a protein (5-10K 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. You will make the corrections and return it by Wednesday April 24. Each of the three page summaries are worth 4% of your grade. The twelve 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.

Week	<b>Tentative</b> Lab Schedule		
	Group 1	Group 2	Group 3
Jan 14	Introduction, Writing Lab Summaries Reports, Statistics, Lab Procedures		
Jan 21	No Lab, MLK day		
Jan 28	GC/MS	Ion Chromatography	Cyclic Voltammetry
Feb 4	Cyclic Voltammetry	GC/MS	Ion Chromatography
Feb 11	Ion Chromatography	Cyclic Voltammetry	GC/MS
Feb 18	LC/MS of Proteins and Peptide Digests		
Feb 25	LC/MS of Proteins and Peptide Digests		
Mar 4	No Lab, Spring Break		
Mar 11	Atomic Absorption	LC-UV-Fluorescence	Make-up
Mar 18	Make-up	Atomic Absorption	LC-UV-Fluorescence
Mar 25	LC-UV-Fluorescence	Make-up	Atomic Absorption
Apr 1	GC/MS-NCI	GC-ECD	Make-up
Apr 8	GC-ECD	Make-up	GC/MS-NCI
Apr 15	Make-up	GC/MS-NCI	GC-ECD
Apr 22	Final Lab Report Due, No Lab, Easter Break		

After spring break, there will be four lab assignments and two make-up dates for those groups that wish to repeat a lab. Groups (of two) are allowed one make-up date every three weeks. The precise when and if of the make-up lab during a three week period will be negotiated with the instructor.

### **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.