

Syllabus for Chem 314, Instrumental Analysis Spring Semester 2020

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 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

Date	Day	Topic	Chapter
Jan 13	Monday	Introduction/Review of Fundamental Concepts	
Jan 15	Wednesday	Statistics, Random and Systematic Error in Measurements	5
Jan 17	Friday	Statistical Tests in Chemical Analysis	6
Jan 20	Monday	Martin Luther King Day, No class	
Jan 22	Wednesday	Statistical Tests Continued	7
Jan 24	Friday	Sampling and Calibration Methods	8
Jan 27	Monday	Calibration Methods	8
Jan 29	Wednesday	Exam 1: Chapters 5-8	
Jan 31	Friday	Introduction to Chromatography	30
Feb 3	Monday	Gas Chromatography	31
Feb 5	Wednesday	High Performance Liquid Chromatography	32
Feb 7	Friday	Ion Exchange and Ion Chromatography	32
Feb 10	Monday	Capillary Electrophoresis and Capillary Electrochromatography	33
Feb 12	Wednesday	Chromatography Detectors; FID, ECD, and Thermal Conductivity	31,32
Feb 14	Friday	Detectors continued, NPD, Photoionization, light scattering	31,32
Feb 17	Monday	Exam 2: Chromatography and detectors	
Feb 19	Wednesday	Electrochemistry: Basic Concept Review	18
Feb 21	Friday	Potentiometry, Reference Electrodes	21
Feb 24	Monday	Potentiometry	21

Feb 26	Wednesday	Electrogravimetry and Coulombic methods Of Analyses.	22
Feb 28	Friday	Amperometric Analyses	22
March 2-6		Spring Break	
March 9	Monday	Exam 3: Electrochemistry Chapters 18, 21 and 22	
March 11	Wednesday	Introduction to Voltammetry Reference Electrodes	23
March 13	Friday	Stripping Voltammetry	23
March 16	Monday	Cyclic Voltammetry	23
March 18	Wednesday	Introduction to Spectroscopic Methods Electromagnetic Spectrum	24
March 20	Friday	Instrumental Components for Absorption and Emission Spectroscopy	25
March 23	Monday	Molecular basis of Fluorescence and Phosphorescence	28
March 25	Wednesday	Instrumental components and instruments Continued.	26
March 27	Friday	Raman Spectroscopy	
March 30	Monday	Exam 4: Absorption and Emission Spectroscopy	
April 1	Wednesday	Introduction to Infrared Spectroscopy	
April 3	Friday	Light Sources for IR Spectroscopy	
April 6	Monday	Detectors for IR Spectroscopy	
April 8	Wednesday	Fourier Transform IR Spectroscopy	
April 10	Friday	Good Friday, no class	
April 13	Monday	Easter Monday, no class	
April 15	Wednesday	Introduction to Mass Spectrometry	
April 17	Friday	Quadrupole Mass Analyzers	

April 20 Monday	Time-of-Flight mass analyzers
April 22 Wednesday	Ionization methods, Electron Ionization, Electrospray, DESI, and MALDI
April 24 Friday	Ionization methods continued, Detectors.

Final Exam

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

Friday, May 1, 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 three 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.

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 exam is worth 15% of your grade (total 60%). The lowest exam grade of the four taken during the regular semester will be dropped. You cannot drop the final (15% of your grade). The final is not cumulative. 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 six, five-page lab summaries and one fifteen-page report. I will correct and return the first four 5-page summaries, so you can resubmit them for a better grade. Your fifteen-page report will be concerned with the analysis of peptides and proteins using ion trap mass spectrometry. The instructor will provide you with 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 five-page summaries are worth 5% of your grade. The fifteen-page paper will be worth 12% of your grade ($6 \times 5\% + 12\% = 40\%$). 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	<u>Tentative</u> Lab Schedule		
	Group 1	Group 2	Group 3
Jan 13	Introduction, Writing Summaries and Lab Reports, Statistics, Lab Procedures		
Jan 20	No Lab, MLK day		
Jan 27	LC/MS-determination Of protein MW	Ion Chromatography	UV-Vis Spectroscopy
Feb 3	UV-Vis Spectroscopy	LC/MS-determination Of protein MW	Ion Chromatography
Feb 10	Ion Chromatography	UV-Vis Spectroscopy	LC/MS-determination Of protein MW
Feb 17	LC/MS-determination of protein MW	Ion Chromatography	UV-Vis Spectroscopy
Feb 24	GC/MS	Tandem MS of peptides	Atomic Absorption
Mar 2	No Lab, Spring Break		
Mar 9	HPLC-Fluorescence of Vitamin B homologs	GC/MS	Tandem MS of peptides
Mar 16	Tandem MS of peptides	Atomic Absorption	Capillary Electrophoresis
Mar 23	Capillary Electrophoresis	Tandem MS of peptides	Atomic Absorption
Mar 30	Atomic Absorption	HPLC-Fluorescence of Vitamin B homologs	GC/MS
Apr 6	GC/MS	Capillary Electrophoresis	HPLC-Fluorescence of Vitamin B homologs
Apr 13	No Lab, Easter Break		
Apr 20	HPLC-Fluorescence of Vitamin B homologs	Make up	Capillary Electrophoresis

Lab procedures

Students will work in groups of three in the lab at the beginning of the semester. Five-page summaries and fifteen-page papers will be submitted by each group, not by students individually. We will run three different experiments each week. There are four groups of three students in each lab section. As a result, one group will be off every three weeks. Over the course of four weeks, each group will complete three lab assignments. There are eight lab assignments per group per semester. Six of these assignments will produce six, five-page reports per group. Two of the lab assignments will culminate in a fifteen-page paper.

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.