CHEM 312
Instrumental Methods of Chemical Analysis

Trinity College **Spring 2022**

Class Meetings
MWF 10-10:50 am
Location TBA

Lab Meetings
Mondays 1:15 pm
Clement 317 & 122

Instructor
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Office Hours
Clement 129
Mondays 11 am - noon
Thursdays 2-3 pm
and by appointment

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Inside this syllabus

Course Objectives

A lecture and laboratory course in the principles and practice of the use of instruments for quantitative and qualitative chemical measurements. Theory, optimization, and application of instrumentation for spectroscopic, electrochemical, spectrometric, and hyphenated methods of analysis are presented. Applications of computer methods of analysis as well as analog and digital manipulation of electrical signals are presented.

After completing this course, students should be able to

- Identify common sources of noise in instrumental measurements, and formulate strategies to maximize signal to noise experimentally and through data processing
- Explain the chemical basis of the signals produced by instrumental methods, including how the physical components of each instrument transduce chemical information into data
- Evaluate potential instrumental methods in the context of a specific analytical problem, identify the best method for the problem at hand, and justify your choice

Course Format

During class we will alternate between short lectures, small group work, and whole class discussion. You should do the pre-class reading in order to participate and make the most of your time in class. Sometimes I may call on you randomly to answer a question instead of asking for volunteers. I want to make sure everyone is contributing their ideas to the class, but I know that “cold calls” can be stressful. Our goal is to create a classroom environment where it’s OK to get something wrong or not know an answer. (If we all knew all the answers already, then we would just go home.) If you are anxious about this part of class, please let me know so we can discuss strategies for your participation.

During lab you will work in small groups to gain hands-on experience with the instruments we are discussing. Lab reports will mainly be informal results & discussion sections. The goal of these assignments is to give you the opportunity to discuss ideas from class in your own words while applying them to authentic data.

Tips for Success

Before class read the text pages that introduce the next topic and preview the worksheets and practice exercises that go with them.

During class participate often and ask questions. Discuss difficult concepts with your group members, and follow along in your course pack. Make a note of sections you need to review in more detail.

After class review your notes and attempt the practice exercises. As soon as you get stuck or feel uncertain, reach out to me or the TA. You will get the best results if you review the notes and start the practice exercises as soon as possible after class.

Before lab read the entire lab exercise and focus on what you need to produce in terms of data, analysis, and writing. Based on those end products, write one or more objectives in your lab notebook. Do the assigned pre-lab reading and pre-lab exercises. Prepare the rest of your lab notebook, using the rubric as a guide, while thinking actively about how you will divide tasks between yourself and your lab partner(s).

During lab stay on task. Work actively throughout the period, and avoid standing around watching your lab partner. Make sure you understand the purpose of each step, and think about your data as you collect it. Consider whether it matches your expectations and is logical based on your sample. Check in with your partner(s) before leaving to make sure you understand what the other(s) did and have all necessary data.

After lab begin the data analysis as soon as possible while the work you did is still fresh in your mind. Double check that you have all the data you need to prepare your report and contact your lab partner(s) as soon as possible if you need to get data from them.

Before exams use the learning objectives in the course schedule to focus your studying. When reviewing the course pack and homework assignments, re-work questions from scratch instead of reviewing worked solutions. For difficult concepts, practice explaining them out loud, as though you are the teacher. Keep in mind that the exam will not include questions you have seen previously, so you will need to apply your knowledge to new problems. It will not be sufficient to know the "right answer" – you need to understand why the correct answer is correct.

You should plan to spend 6-12 h per week outside of class and lab time in order to be successful.

Resources

Accessibility Requests
Please let me know if there are circumstances that might affect your participation in class or your health and safety in the lab. If you need accommodations, talk with me as soon as possible and contact Sheila Njau in the Student Accessibility Resource Center at sheila.njau@trincoll.edu.

Textbooks
If you prefer a traditional textbook, I recommend any edition of *Principles of Instrumental Analysis* by Skoog, Holler, and Crouch. A copy is on reserve in the library.

ACS Style Guide
All references must be in ACS style. You can refer to this reference for citation formatting online through the Trinity library website, and hard copies are available in the main library and the Chemistry library.

Excel for Chemists
A copy of this book is available electronically through the library, and a hard copy is available for use in my office during office hours.
http://site.ebrary.com/lib/trinity/Doc?id=10510405

Emergency & Equity Fund
Financial support to help ensure the academic success of all students. More information and an application is available at https://www.trincoll.edu/dean-of-students/campus-life-resources/student-emergency-fund/

How do you earn your grade?

Exams	35%
Homework	25%
Laboratory	40%

We will have 3 equally-weighted exams, including 2 in-class exams and a cumulative final.

We will work on a variety of homework assignments, including quantitative problem sets and analyses of scientific articles. Unless directed otherwise, these are **due at the start of class.** For calculations, **you must show your work to obtain credit.** Please write only on one side of each page, and draw a box around your final answer.

All lab assignments will be equally weighted except the project lab presentation and the lab notebook average, which will each count as two assignments.

Course Materials

Required

- **Course pack & laboratory manual**
  Available in the bookstore, approx. $40
- **Laboratory safety glasses**
  Available in the Chemistry Office (CT 208), $5
- **Laboratory notebook**
  Spiral bound, carbonless copy, available in bookstore, ~$15.
- **Scientific calculator** (bring to class!)
- **USB drive** (bring to lab!)

Suggested

- *ACS Style Guide*
- *Excel for Chemists*
- Folder for collecting handouts and assignments
- We are using a course pack with excerpts from a freely-available textbook; however, you may wish to use a more traditional textbook as well. For suggestions, see Resources (p. 2)

Course Policies

Due Dates, Late Work, and Make-up Exams

Lab reports should be submitted on Moodle by 8 am on the due date (1 week after the lab is completed). Homework is due at the start of class. Lab notebook pages are due at the end of lab. Late assignments will be accepted with a penalty of -10% per day. Do not wait until the last moment as extensions will not be granted for technical difficulties. See p. 6 for information about how to request an extension.

Sometimes unexpected (and often unwelcome) events intrude on our plans – mental and physical illness, family needs, etc. may affect your class performance this semester. To the extent that you are comfortable sharing this information with me, I would like to know as soon as possible. In some circumstances, exams may be taken via Zoom. Please contact me in advance to arrange this. If you miss an exam due to illness, injury, or emergency, you should provide some confirmation directly to me or to the Dean of Students office. If you will miss an exam for a scheduled, College-sanctioned event (e.g., religious observance, athletics), you should discuss your absence with me in advance (preferably at least 3 days prior). In most cases, your other exams will be weighted to replace the one missed. Make-ups will be given only in special circumstances at the instructor's discretion.

Important Dates

Drop/Add Deadline 2/7

Project Proposal 2/28

Exam 1 ..... 3/2

Withdraw Deadline 3/16

Exam 2 ..... 4/13

Project Presentations 5/2

Final Exam ..... TBA

A complete schedule is available on Moodle. Check for updates often.

Moodle & Email

Moodle and email will be used extensively. All students are required to have an active email account. Students are expected to consult the course Moodle site frequently for assignments, announcements, schedule changes, lecture materials, supplementary course materials and external links.

Classroom Citizenship

This course is intended for advanced students, and I expect you to conduct yourselves as such and to be familiar with the College's policies on attendance, absences, academic honesty, and classroom behavior as outlined in the Student Handbook.

I expect you to conduct yourselves with integrity as persons. There are persistent, pervasive, and pernicious issues in academic science concerning discrimination based on race, sex/gender, sexual orientation, disabilities, religion, body type, etc. Such violations take many forms from overt harassment to seemingly smaller transgressions (unwanted comments, bullying, patronizing). While some issues may seem less serious, their cumulative effect on the recipient's career and well-being can be just as detrimental as more obvious offenses. Treating others with dignity is as integral to the proper conduct of science as keeping a good lab notebook. Discrimination and harassment persist because our scientific culture has not historically valued diverse perspectives, backgrounds, and contributions. I invite you to help me foster a community of mutual respect by reflecting on your own biases and supporting your peers to do the same. This means speaking out when you observe abuse and apologizing when your peers point out negative impacts of your behavior. I will model this openness to feedback if you notice behavior of mine that has a negative impact and bring it to my attention. Together, we can foster a climate free from bullying, harassment, and discrimination where we all can thrive and learn.

Academic Integrity

Each student should be familiar with the Trinity College Student Integrity Contract and the section on Intellectual Honesty in the Student Handbook.

> **References:** Any ideas in your writing assignments that (i) did not spring from your own mind and (ii) are not common knowledge to high school science students should be cited at the end of each assignment. Direct quotes are usually unacceptable: rewrite all ideas in your own words *and* cite them. If you have a question about whether or not your rewording is acceptable, ask before turning in the assignment. Use the *ACS Style Guide* to format your references. Plagiarism and academic dishonesty – copying from another student, copying from another source including the internet, failing to cite a reference, etc. – will be addressed through the College's jury system.
>
> **Homework:** I encourage you to discuss homework with your classmates. Your peers should be a primary resource if you are uncertain about how to proceed on a problem (although the TAs and I are happy to help, too). **You should acknowledge which classmates worked with you on an assignment by listing their names on the first page.** Additionally, I expect each of you to do your own work. Discussing homework problems with your classmates is acceptable; copying your classmate's answers or work is not. If I find evidence of copying or allowing work to be copied, we will go through the College's academic honesty proceedings. If you have any questions about whether or not your collaboration with a classmate complies with my expectations, please talk to me about it *before* turning in an assignment.

On the first day of class, I will be asking you to sign the Student Integrity Statement as a way of affirming your commitment to academic integrity. You may choose not to sign, however, whether you sign or not, you are expected to behave in accordance with the statement. For your records, the statement is as follows:

> "In accordance with Article II of the Trinity College Student Integrity Contract, I hereby pledge that the papers, exams, and other academic exercises I submit for this course will represent my own work; that I will properly acknowledge and attribute any and all information and ideas that I have used from other sources; and that no collaboration unauthorized by the instructor of the course will occur in the course of its completion."

Laboratory Information

Please read your laboratory manual for more detail about lab preparation and assignments.

- Because the lab portion of the course is quite important, you should spend a substantial amount of your time and effort on lab work.
- Lab notebook pages are due at the end of lab.
- All of the written lab reports for this course are informal results & discussion sections only, unless otherwise noted.
- All lab reports are equally weighted. The average lab notebook score counts as a lab report.

Lab Partner Policy

Lab groups will be assigned and will generally rotate a few times throughout the semester so that you have the opportunity work with and get to know several of your classmates.

I am available to mediate any disagreements or personal issues that arise between you and your lab partner(s), though ultimately you are responsible for working together as professional adults. Please let me know if you would like assistance navigating any aspects of your group work in the laboratory.

Lab Schedule

Week of	Lab	Assignment Due (8 am)
Jan 31	Signal Processing	
Feb 7	UV-Vis Comparison	Signal Processing Report
Feb 14	Spectroscopy Rotation 1 (Fluorescence, IR, NMR)	UV-Vis Comparison Report
Feb 21	Proposal Workshop	Fluorescence/IR/NMR Report
Feb 28	Spectroscopy Rotation 2	Project Proposal
Mar 7	Spectroscopy Rotation 3	Fluorescence/IR/NMR Report
Mar 14	Mass Spec Rotation 1	Fluorescence/IR/NMR Report
Mar 21	No Lab: Spring Break	
Mar 28	Mass Spec Rotation 2	ICP-MS/GC-MS/LC-MS Report
Apr 4	Mass Spec Rotation 3	ICP-MS/GC-MS/LC-MS Report
Apr 11	Project Week 1	ICP-MS/GC-MS/LC-MS Report
Apr 18	Project Week 2	
Apr 25	Project Week 3	Project Outline
May 2	Project Presentations	Project Packet

Lab reports are due in the Digital Dropbox on Moodle at 8 am on the due date (typically Monday, one week after you complete the lab). The project packet is due before the start of lab on Monday, May 2. If you would like feedback on your project calculations before your presentation, you should send them to me at least 72 h (3 days) in advance.

Frequently Asked Questions (FAQs)

What should I do if I have to miss class?

Class attendance is critical to your success in the course, so please be on time and do not miss class if at all possible. If you will be absent, please do the following:

(1) Notify me as soon as possible, preferably before class and by email.
(2) Email me any assignments that are due, drop them in CT208, or send them to class with a friend.
(3) Contact a classmate to get the notes and schedule an appointment with Prof. Kovarik to address any questions you have about missed material.

What if I have to miss lab?

It is particularly important that you do not miss lab periods because the lab experience is impossible to recreate. If you must miss lab, you should inform me and your lab partner(s) as soon as possible, preferably in advance. Except in exceptional circumstances, there will be no make-up labs. All reports must still be completed by the due date using data collected by your lab partner(s). Be sure to credit your partner(s) in your report for data collection.

Can I have an extension? What is the penalty for late work?

Late assignments will be penalized at a rate of -10% per day. You may request an extension of up to 48 hours by email. All requests for extensions must be received at least 24 hours before the original deadline and be accompanied by an Excel or Word document showing that you have started working on the data analysis and/or write-up.

How can I tell what my current grade is?

Your current overall grade and your grades for individual assignments will always be available in the grade book on Moodle. If you have questions or would like to discuss the class at any time, please come by office hours or make an appointment.

Can I do extra credit?

There will be no extra credit in this class. Please don't ask! My philosophy is that you should spend your valuable time succeeding at the primary objectives for the course. If you have not completed them, you should not be spending time on additional work. If you have completed them, your grade should not be in need of a boost.

Why are we doing so much group work?

A large body of educational research shows that students learn more and perform better on exams when they are actively engaged, rather than passively listening, in class. This is especially true when students work in groups because of the opportunity to learn from each other, rather than just from the instructor. Working with other people is also a key skill for almost every professional occupation, so a complete education should include practice at this skill. (For recommendations to graduate programs and references for job openings, I am almost always asked to comment on a student's ability to work on a team.) Finally, when you work in groups, it makes your thinking clear to me. If I talk and you listen, there aren't many opportunities for me to learn how things are going before the exam. When I hear you discussing ideas in class each day, it gives me important feedback about which topics we have mastered and which need to be revisited.

Tentative Schedule

Check Moodle regularly for updated versions! Note homework due dates as they are set.

Although not mentioned separately below, you should be able to explain the chemical basis of the signal obtained by each instrument, evaluate common limitations or sources of noise, and sketch a block diagram of common instrument layouts.

Date	Topic	After this class period, you should be able to…	Items Due
Week 1			
Lab	signal processing	Explain common signal processing methods; identify their strengths and limitations	
Jan 31	signals and noise	Summarize the basic operating principle of all chemical instrumentation; calculate S/N ratios	
Feb 2	sources of noise	Identify types and sources of noise in measurements	
Feb 4	noise reduction	Explain the hardware and software methods that can be used to improve S/N; select an appropriate method to reduce noise in a given application	
Week 2			
Lab	comparing instruments	Compare the figures of merit for various UV-Vis instruments; solve for the concentration of multiple unknowns using Beer's law	Signal Processing Report
Feb 7	interactions of light and matter	Describe the ways in which matter can interact with light; list and arrange the basic components of all spectroscopic instruments	**Add/drop ends**
Feb 9	light sources	Compare and contrast the main types of light sources for spectroscopic methods; summarize their characteristics, explain how they work, and perform relevant calculations	
Feb 11	wavelength selectors	Compare and contrast the main types of wavelength selectors for spectroscopic methods; summarize their characteristics, explain how they work, and perform relevant calculations	
Week 3			
Lab	spectroscopy rotation 1	Apply the principles of fluorescence or IR or NMR spectroscopy to experimental data	Comparing UV-Vis Instruments Report
Feb 14	detectors	Compare and contrast the main types of photodetectors/transducers; summarize their characteristics, explain how they work, and perform relevant calculations	

Feb 16	atomic absorption (AA) spectroscopy	Compare and contrast sample atomization methods for AA; identify potential sources of interference	
Feb 18	atomic emission spectroscopy	Compare and contrast atomic emission sources, identify advantages and disadvantages of AES relative to AA	
Week 4			
Lab	proposal workshop	Prepare for your project lab	Fluorescence, IR, or NMR Report
Feb 21	molecular absorbance spectroscopy (UV-Vis) and Beer's Law	Explain why spectra obtained from molecular spectroscopy differ from those of atomic spectroscopy; obtain quantitative information from an absorbance spectrum; identify causes of deviation from Beer's Law and suggest means to minimize or eliminate them	
Feb 23	molecular luminescence spectroscopies	Use block diagrams to demonstrate why chemiluminescence and fluorescence are more sensitive than absorbance methods; explain why phosphorescence is more long-lived than fluorescence; describe the information which can be obtained from fluorescence lifetimes	
Feb 25	molecular luminescence spectroscopies, cont'd.	Cont'd from previous.	
Week 5			
Lab	spectroscopy rotation 2	Apply the principles of fluorescence or IR or NMR spectroscopy to experimental data	Project Proposal
Feb 28	applications of optical spectroscopy	Discuss the article "Measuring atomic emission from beacons for long-distance chemical signaling" and its relevance to this course	
Mar 2	***Exam 1: Signals and Noise, Spectroscopy***		
Mar 4	IR theory and spectra	Identify major interferents in IR spectroscopy; qualitatively and quantitatively interpret IR spectra; identify sources of error in quantitative IR spectroscopy	
Week 6			
Lab	spectroscopy rotation 3	Apply the principles of fluorescence or IR or NMR spectroscopy to experimental data	Fluorescence, IR, or NMR Report

Mar 7	IR instrumentation	Compare the operation of FT instruments to dispersive instruments, and explain the advantage of FT; compare and contrast light sources and transducer/detectors for IR; explain the principles behind ATR-IR measurements	
Mar 9	Raman spectroscopy	Suggest a suitable source for a Raman experiment and predict the resulting Stokes and anti-Stokes lines; compare and contrast IR and Raman spectroscopies to explain how they can be complementary techniques	
Mar 11	NMR theory and spectra	Explain how and why NMR sensitivity depends on magnetic field strength; describe processes of relaxation in NMR and their effect on line broadening; explain how the signal is converted to a spectrum showing chemical shift	
Week 7			
Lab	MS rotation 1	Apply the principles of various MS techniques to experimental data	ICP-MS or GC-MS or LC-MS Report
Lab	GCMS or LCMS	Use GCMS or LCMS to investigate the identity and/or concentration of analytes	
Mar 14	NMR instrumentation	Explain the function of each NMR component in a simplified block diagram; describe methods used to account for magnetic field fluctuations and inhomogeneities	
Mar 16	intro to mass spec	List and diagram the basic components of all mass spectrometers; perform mass resolution calculations; calculate mean free paths; explain the energetics of fragmentation in MS	**Midterm (Last Day to Withdraw)**
Mar 18	ionization methods	Compare and contrast common ion sources in mass spectrometry, describe their characteristics, and explain how they work; explain how ESI and MALDI have influenced modern applications of mass spec	
Week 8			
Spring Break: No Class or Lab			
Week 9			
Lab	MS rotation 2	Apply the principles of various MS techniques to experimental data	ICP-MS or GC-MS or LC-MS Report
Mar 28	ionization cont'd. intro to mass analyzers	Identify suitable mass analyzers for given experimental requirements; explain the operation of magnetic sector mass analyzers	

Mar 30	mass analyzers	Explain the operation of time-of-flight mass analyzers and perform related calculations; explain the operation of quadrupole mass analyzers	
Apr 1	mass analyzers, cont'd.	Explain the operation of ion trap, and FT-ICR mass analyzers	
Week 10			
Lab	MS rotation 3	Apply the principles of various MS techniques to experimental data	ICP-MS or GC-MS or LC-MS Report
Apr 4	tandem MS	List the ways in which molecules can be fragmented for MS-MS experiments; describe the type of information that can be obtained by each fragmentation method	
Apr 6	interpreting mass spectra	Interpret mass spectra to obtain structural information about a sample	
Apr 8	proteomics	Differentiate between top-down and bottom-up proteomics experiments; apply de novo sequencing methods to MS-MS data	
Week 11			
Lab	Project Week 1	Ensure that you can detect a signal for your analyte in a standard; prepare calibrators and blanks	ICP-MS or GC-MS or LC-MS Report
Apr 11	proteomics applications	Discuss the article "Examining the proteome of Drosophila across lifespan" and its relevance to this course	
Apr 13	***Exam 2: IR, NMR, and MS***		
Apr 15	intro to electrochemistry	Identify redox reactions and calculate values for the currents and voltages associated with these reactions; convert between diagrams and line notation of electrochemical cells; explain the relationship between E° and the equilibrium constant and describe what happens as a galvanic cell reaches equilibrium	
Week 12			
Lab	Project Week 2	Trouble-shoot method; prepare samples	
Apr 18	ion and electron transport	Explain the difference between Faradaic and non-Faradaic (charging current) and how each contributes to electrochemical measurements; list the three ways ions are transported to the electrode surface; compare and contrast common reference electrodes	
Apr 20	potentiostats	Apply the rules of op amp performance; recognize and interpret the circuits used in a potentiostat	

Apr 22	cyclic voltammetry	Obtain qualitative and quantitative information from a cyclic voltammogram; explain the origin of the shape of a CV; describe how CV data depends on scan rate	
Week 13			
Lab	Project Week 3	Complete data collection and data analysis for project lab	Project Outline
Apr 25	amperometry chemically modified electrodes	Describe major applications of amperometry; compare amperometric detection with cyclic voltammetry and fluorescence; list common methods and reasons for electrode modification	
Apr 27	electrochemical detection *in vivo*	Discuss the article "Subsecond dopamine release promotes cocaine seeking" and its relevance to this course	
Apr 29	student choice topic	*As a class, you will select a topic of interest for this class period.*	
Week 14			
Lab	project presentations and lab check-out	Present (as a group) the significance, methodology, results, and interpretation of your project lab experiments	Project Packet (Title, Abstract, ToC Figure, and Spreadsheet)
May 2	student choice topic	*As a class, you will select a topic of interest for this class period.*	
May 4	student choice topic	*As a class, you will select a topic of interest for this class period.*	
May 6	course summary	Identify the main themes of this course and the important ideas that you will use in the future	
Cumulative Final Exam: TBA			