CHEM 311: Analytical Chemistry

Trinity College **Fall 2022**

Class Meetings
MWF 10-10:50 pm
TBA

Lab Meetings
Tues 1:30 pm
Clement 317
Unless otherwise arranged

Instructor
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Office Hours
Clement 129
Mondays 11-12
Thursdays 4-5 pm
and by appointment

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Inside this syllabus

Course Objectives

After completing this course, you should be able to

- Identify types and sources of error, propose solutions to minimize these errors, and evaluate the statistical significance of results in light of uncertainties
- Design and implement suitable sampling and sample preparation protocols for real-world analyses
- Use conceptual explanations and quantitative calculations to predict the behavior of chemical equilibria
- Evaluate and optimize potentiometric measurements and chromatographic separations in the context of chemical equilibria
- Apply a working knowledge of Beer's law to spectrophotometric determinations
- Present the results of experiments in a formal report using the style and conventions of professional chemists

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 groups to analyze real-world samples. Frequently you will have the opportunity to design aspects of the experiments yourselves. You may have heard that the labs for this class tend to run long. I have been modifying the lab exercises so that we can finish on time, but you need to come to lab on time and ready to begin work in order to complete the labs in the allotted period. If you spend a significant amount of time reviewing the manual or asking questions, you may need to stay late.

Tips for Success

Before class read the text that introduce the next topic and preview the worksheets and practice exercises.

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 TAs. You will get the best results if you review the notes and start the practice problems as soon as possible after class.

Plan to spend 6-12 h per wk outside of class on this course.

Before lab read the 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 partner(s). 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.

Resources

Accessibility Requests
Please let me know if there are circumstances that might affect your full participation in class or your health and safety in the lab. If you need accommodations, talk with me as soon as possible and contact the Student Accessibility Resource Center at 860-297-4025 or SARC@trincoll.edu.

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.

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/

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

Textbooks
If you prefer a traditional textbook, I recommend any edition of *Quantitative Chemical Analysis* by Harris or *Fundamentals of Analytical Chemistry* by Skoog, West, and Holler. One copy of each is on reserve in the library.

The Writing Center
For hours or to schedule an appointment, call 297-2468 or visit http://trincoll.mywconline.com/

How do you earn your grade?

Exams	40%
Homework	20%
Laboratory	40%

We will have 3 equally-weighted, cumulative in-class exams. There will be no final exam.

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 report and the lab notebook average, which will each count as two assignments.

Course Materials

Required

- **Course pack & laboratory manual**
  Available in the bookstore, approx. $35
- **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 so that we can make a plan to minimize disruption of your academic work. If you miss an exam due to illness, injury, or a family emergency, you should provide some confirmation of the event 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 (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 9/13

Exam 1 ..... 10/3

Project Proposal 10/18

Withdraw Deadline 10/24

Exam 2 ..... 11/7

Project Report 12/13

Exam 3 ..... TBA

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

Moodle & Email

Moodle and e-mail will be used extensively. All students are required to have an active e-mail account. Please inform me during the first week of class if you prefer to use a non-trincoll address. 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.

Academic & Personal 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."

I also 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.

Laboratory Information

Please read your laboratory manual for more detail about grading, 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. Turn them in before you leave!
- You will write two formal lab reports, one on the vanillin experiments and the second on your project. For the vanillin report, you will turn in multiple drafts and revisions based on feedback.

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)
Sept 5	Sampling	
Sept 12	Vanillin 1: Sample Preparation	Sampling Lab Spreadsheet
Sept 19	Vanillin 2: Calibration	Sample Prep Write-Up
Sept 26	Vanillin 3: Effect of pH	Calibration Write-Up
Oct 3	Writing Workshop	pH Write-Up
Oct 10	No Lab (Trin Days)	Vanillin Introduction
Oct 17	Chromatography Week 1	Project Proposal
Oct 24	Chromatography Week 2	Vanillin Full Report
Oct 31	Chromatography Week 3	Chromatography R&D 1
Nov 7	Chromatography Week 4	Revised Vanillin Report
Nov 14	Project Lab Week 1	Chromatography R&D 2
Nov 21	Project Lab Week 2	Project Intro & Methods
Nov 28	Project Lab Week 3	
Dec 5	Project Lab Week 4 & Checkout	Figure & Question Outline
Dec 13	No Lab	Final Project Report

Reports are due in the Digital Dropbox on Moodle at 8 am on Tuesday one week after completion of the lab.

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) Electronically submit any assignments that are due.
(3) Contact a classmate to get the notes, and schedule an appointment with me 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. Depending on the specific circumstances, we will make arrangements either for you to complete a make-up lab or to use class data. All reports must still be completed by the due date if 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.

Date	Topic	After this class period, you should be able to…	Items Due
Week 1			
Lab	sampling	Use lab equipment correctly; explain how sample size affects sampling errors	
Sept 7	intro	Identify qualitative and quantitative analyses; describe and apply steps in the analytical method	
Sept 9	sampling and sample prep	Make practical decisions about sampling and sample preparation	
Week 2			
Lab	sample preparation	Compare and contrast liquid-liquid extraction and SPE	Sampling Spreadsheet
Sept 12	accuracy	Identify types of error (random vs. systemic, absolute vs. relative) and explain how they affect accuracy; calculate and interpret percent recovery values	
Sept 14	precision	Express the precision of a measurement using significant figures, standard deviation, CV, RSD, and confidence intervals; apply the "real rule" for significant figures	**Add/drop period ends**
Sept 16	hypothesis testing	Apply t-tests to experimental data; identify limitations of t-tests	
Week 3			
Lab	calibration	Determine the amount of vanillin in extracted samples using UV-Vis absorbance spectroscopy	Sample Prep Write-Up
Sept 19	uncertainty	Distinguish between accuracy, precision, error, and uncertainty; perform propagation of uncertainty calculations	
Sept 21	linear regression	Use linear regression by the method of least squares to fit a calibration curve and determine an unknown value; calculate the uncertainty in a value calculated from a calibration curve	
Sept 23	figures of merit	Calculate and interpret calibration sensitivity, analytical sensitivity, LOD, and dynamic range	

Week 4			
Lab	effect of pH on analyses	Evaluate optimum pH for vanillin determination	Calibration Write-Up
Sept 26	internal standards	Evaluate when and why an internal standard may be needed; suggest appropriate internal standards for analytes; perform associated calculations	
Sept 28	standard addition	Evaluate when and why standard addition may be needed; perform associated calculations; compare and contrast standard addition and internal standards as means of improving the results obtained from calibration curves	
Sept 30	comparing analytical methods	Discuss the article "Comparison of HPLC and GC-MS for measurement of cocaine and metabolites in human urine" and its relevance to this course	
Week 5			
Lab	writing as a chemist	Find and place key information in a scientific paper; paraphrase and make proper use of references and citations; learn effective literature searching skills	pH Write-Up
Oct 3	**Exam 1: Selecting an Analytical Method**		
Oct 5	review of chemical equilibrium	Refute common misconceptions about chemical equilibria; write and use equilibrium coefficients based on balanced chemical equations	
Oct 7	solubility equilibria	Use concepts of solubility product, common ion effect, and complex ion formation to make qualitative and quantitative predictions about the behavior of solubility equilibria	
Week 6			
Lab	No lab this week		Vanillin Intro
Oct 10	***Trinity Days -- no class***		
Oct 12	ionic strength	Explain the concept of ionic strength and its effect on chemical equilibria; explain the difference between activity and concentration and determine when activity values should be used	
Oct 14	acid/base equilibria	Compare the strengths of acids/bases and their conjugates using K_a and K_b values; convert between pH, pOH, and [H+] and between K_a and pK_a	

Week 7			
Lab	chromatography week 1	Prepare chocolate samples for HPLC and/or GCMS; optimize chromatographic separations for the analysis	Project Proposal
Oct 17	solving equilibrium problems	Convert between pH, K_a, K_b, and concentration through acid-base equilibrium calculations	
Oct 19	evaluating assumptions	List the assumptions that may be made when solving equilibrium problems and evaluate (for specific examples) the validity of these assumptions	
Oct 21	polyprotic equilibria	Determine the predominant form and net charge of polyprotic species at a given pH	
Week 8			
Lab	chromatography week 2	Prepare chocolate samples for HPLC and/or GCMS; optimize chromatographic separations for the analysis; run prepared chocolate samples; begin data analysis	Vanillin Formal Report
Oct 24	buffers (conceptual)	Explain how a buffer is formed and why it resists changes in pH; describe factors affecting buffer capacity; select a suitable buffering agent for a desired pH	**Last day to withdraw**
Oct 26	buffers (calculations)	Use the Henderson-Hasselbach equation to determine the pH and composition of a buffer; perform calculations associated with practical buffer making; calculate the pH of a buffer solution after addition of strong acid or base	
Oct 28	titration of a weak acid	Sketch the shape of a titration curve without using a calculator; identify the pK_a of an acid from its titration curve; explain how changing the pK_a or concentration of an acid will affects its titration curve	
Week 9			
Lab	chromatography week 3	Optimize chromatographic separations for the analysis; run prepared chocolate samples; begin data analysis	Chromatography R&D 1
Oct 31	titrations of weak bases and diprotic acids	Apply knowledge of weak acid titration to weak bases and diprotic acids; calculate the pH of a weak base solution at any point along its titration curve	
Nov 2	potentiometry and ion selective electrodes	Explain how ion selective electrodes work and why they measure the activity of a given analyte; describe how a pH meter works specifically; identify common sources of error in pH measurements	

Nov 4	titration of particles and cells	Discuss the article "Cell surface groups of two picocyanobacteria strains studied by zeta potential investigations, potentiometric titration, and infrared spectroscopy" and its relevance to this course	
Week 10			
Lab	chromatography week 4	Complete data analysis for chocolate samples	Revised Vanillin Report
Nov 7	**Exam 2: Chemical Equilibria & Cumulative from Exam 1**		
Nov 9	partition coefficients	Calculate extraction efficiency or the parameters needed to achieve a certain efficiency; discuss the role of pH and ionization in specific extractions; explain why a separation step is necessary in some analyses; describe the fundamental principles of chromatography	
Nov 11	selectivity	Find t_r, t_m, α, and k from a chromatogram; explain the relationship between k and the partition coefficient; suggest way to improve the selectivity of a separation by modulating retention; calculate resolution from a chromatogram	
Week 11			
Lab	project lab week 1	Check that analyte(s) can be detected and/or separated using standards; start any prolonged sample preparation steps; identify experimental steps which require trouble-shooting	Chromatography R&D 2
Nov 14	efficiency	Calculate plate number and plate height from a chromatogram; list the factors that contribute to band broadening in chromatography	
Nov 16	efficiency, cont'd.	Predict how changes in parameters (such as column packing, stationary phase, analyte characteristics) affect plate height	
Nov 18	gas chromatography	Compare and contrast GC injection types, evaluate temperature programs, describe common GC columns and detectors	
Week 12			
Lab	project lab week 2	Proceed with trouble-shooting and/or data collection for project lab	Project Intro and Methods
Nov 21	HPLC	Explain the relationship between particle size, separation efficiency, and separation pressure; select an appropriate separation mode for an HPLC sample; compare and contrast HPLC detectors; predict HPLC elution order and suggest mobile phase changes to optimize resolution	

Nov 23 Nov 25	***Thanksgiving Break***		
Week 13			
Lab	project lab week 3	Proceed with trouble-shooting and/or data collection for project lab	
Nov 28	GC/HPLC comparison	Compare and contrast GC and HPLC separations	
Nov 30	other forms of chromatography	Explain the separation mechanisms of size exclusion, ion exchange, and affinity chromatographies; select an appropriate separation method for a given sample and justify your choice	
Dec 2	capillary electrophoresis	Explain the source of electroosmotic flow and its utility in CE; predict the migration order of analytes in CE; compare and contrast chromatography and CE	
Week 14			
Lab	project lab week 4	Complete data collection and data analysis for project lab; draw conclusions for formal report	Question and Figure Outline
Dec 5	absorbance and Beer's Law	Convert between transmittance and absorbance and justify the use of absorbance in calibration curves; use Beer's law to determine the concentration of an unknown; identify potential sources of deviation from Beer's Law	
Dec 7	absorbance and molecular structure	Explain how molecular structure affects absorbance spectra; predict how pH or a reaction could affect an absorbance measurement; identify isosbestic points	
Dec 9	applying chromatographic theory	Discuss the article "Plate heights below 50 nm for protein electrochromatography using silica colloidal crystals" and its relevance to this course	
Week 15			
Lab	lab check-out	*If you did not complete lab checkout last week, make an appointment to finish checkout during Reading Days*	Project Spreadsheet & Final Report
Dec 12	course summary	Identify themes and important ideas that you may use in the future	
TBA	**Exam 3: Separations, UV-Vis & Cumulative from Exams 1 and 2**		