

# CHEM 311: Analytical Chemistry

Trinity College

Fall 2018

## Class Meetings

MWF 10-10:50 am  
Clement 210

## Lab Meetings

Tues 1:30 pm (Kovarik)  
Clement 317

## Instructor

Prof. Michelle Kovarik  
860-297-5275  
[michelle.kovarik@trincoll.edu](mailto:michelle.kovarik@trincoll.edu)

## Office Hours

Clement 129  
Mondays 1-2 pm  
Thursdays 9-10 am  
and by appointment

## Teaching Assistants

Jessica Duong (lecture)  
[jessica.duong@trincoll.edu](mailto:jessica.duong@trincoll.edu)  
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## Inside this syllabus

Tips for Success .....	2
Resources.....	2
Important Dates .....	3
Required Materials.....	3
Grading .....	3
Course Policies.....	3-4
Laboratory Info.....	5
FAQs.....	6

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

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**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.

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**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 Lori Clapis in the Student Accessibility Resource Center at 860-297-4025 or [Lori.Clapis@trincoll.edu](mailto:Lori.Clapis@trincoll.edu).

### 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.

### 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>

### Teaching Assistant

**Jessica Duong** will hold regular review and Q&A sessions, times and location to be determined. See Moodle for announcements.

### 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 exams, including 2 in-class exams and a final. All exams will be cumulative.

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.

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 (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 .....	9/10
Withdraw Deadline .....	9/28
Exam 1 .....	10/1
Project Proposal .....	10/22
Exam 2 .....	11/5
Project Report .....	12/11
Final Exam .....	12/18

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 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 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

Although I am available to help mediate any disagreements or personal issues that arise between you and your lab partner(s), ultimately you are wholly and individually responsible for settling disputes and working together as professional adults.

Under no circumstances will excuses based on disagreements, interpersonal problems, or difficulties in communicating with your lab partner be considered valid reasons for me to change your grade.

If your partner is absent from lab you will be expected to complete the day's experiment individually and you should be prepared to do so. You may not join another laboratory group without my explicit permission.

### Lab Schedule

Week of	Lab	Assignment Due (8 am)
Sept 4	Sampling	
Sept 11	Sample Preparation	Sampling Lab Spreadsheet
Sept 18	Calibration	Sample Prep Write-Up
Sept 26	Effect of pH	Calibration Write-Up
Oct 2	Writing Workshop	pH Write-Up
Oct 9	Trinity Days – No Lab	Vanillin Introduction
Oct 16	Chromatography 1A	Vanillin Full Report
Oct 23	Chromatography 1B	Project Proposal
Oct 30	Chromatography 2A	Chromatography 1 Write-Up
Nov 6	Chromatography 2B	Revised Vanillin Report
Nov 13	Project Lab (Week 1)	Chromatography 2 Write-Up
Nov 20	Project Lab (Week 2)	Project Introduction
Nov 27	Project Lab (Week 3)	Figure & Question Outline
Dec 4	Project Lab (Week 4)	Project Methods

Lab reports are due in the Digital Dropbox on Moodle at 8 am on the due date (typically Tuesday, one week after you complete the lab). The project spreadsheet and formal report are due 12/11. If you would like feedback on your project spreadsheet before submitting your report, you may submit your spreadsheet on 12/11 and request an extension to provide time for me to review your calculations.

## 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. In general, lab absences will only be excused if due to incapacitating illness/injury, family emergency, or if arranged with your lab instructor *at least 3 days* 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.*

Date	Topic	After this class period, you should be able to...	Items Due
<i>Week 1</i>			
Lab	sampling	Use volumetric glassware correctly; explain how sample size affects sampling errors	
Sept 5	intro	Identify qualitative and quantitative analyses; describe and apply steps in the analytical method	
Sept 7	sampling and sample prep	Make practical decisions about sampling and sample preparation	
<i>Week 2</i>			
Lab	sample preparation	Compare liquid-liquid extraction and SPE	Sampling Spreadsheet
Sept 10	accuracy	Identify types of error (random vs. systemic, absolute vs. relative) and explain how they affect accuracy; calculate and interpret percent recovery values	<b>Add/drop period ends Tuesday</b>
Sept 12	precision	Express the precision of a measurement using significant figures, standard deviation, CV, RSD, and confidence intervals; apply the "real rule" for significant figures	
Sept 14	hypothesis testing	Correctly apply t-tests to experimental data; identify limitations of t-tests	
<i>Week 3</i>			
Lab	calibration	Determine the amount of vanillin in the extracted samples using UV-Vis absorbance spectroscopy	Sample Prep Write-Up
Sept 17	uncertainty	Distinguish between accuracy, precision, error, and uncertainty; perform propagation of uncertainty calculations	
Sept 19	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 21	figures of merit	Calculate and interpret calibration sensitivity, analytical sensitivity, LOD, dynamic range	

<i>Week 4</i>			
Lab	effect of pH on analyses	Evaluate optimum pH for vanillin determination	Calibration Write-Up
Sept 24	internal standards	Evaluate when and why an internal standard may be needed; suggest appropriate internal standards for analytes; perform associated calculations	
Sept 26	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 28	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	<b>Last day to withdraw;</b> project lab topics due
<i>Week 5</i>			
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 1	<b>Exam 1: Selecting an Analytical Method</b>		
Oct 3	review of chemical equilibrium	Refute common misconceptions about chemical equilibria; write and use equilibrium coefficients based on balanced chemical equations	
Oct 5	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	
<i>Week 6</i>			
Lab	<b>No lab this week</b>		Vanillin Intro
Oct 8	<b>Trinity Days -- no class</b>		
Oct 10	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 12	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 $pK_a$	

<i>Week 7</i>			
Lab	chromatography rotation 1A	Determine vanillin, theobromine, and caffeine in chocolate by HPLC OR determine fatty acids in snack food by GC-MS	Vanillin Formal Report
Oct 15	solving equilibrium problems	Convert between pH, $K_a$ , $K_b$ , and concentration through acid-base equilibrium calculations	
Oct 17	evaluating assumptions	List the assumptions that may be made when solving equilibrium problems and evaluate (for specific examples) the validity of these assumptions	
Oct 19	polyprotic equilibria	Determine the predominant form and net charge of a polyprotic species at a given pH; determine the isoelectric point of a molecule from the $pK_a$ values of its ionizable groups	
<i>Week 8</i>			
Lab	chromatography rotation 1B	Data analysis for GC or LC lab	Project Proposal
Oct 22 *midterm*	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	
Oct 24	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 26	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 affect its titration curve	
<i>Week 9</i>			
Lab	chromatography rotation 2A	Determine vanillin, theobromine, and caffeine in chocolate by HPLC OR determine fatty acids in snack food by GC-MS	Chromatography Report 1
Oct 29	titrations of weak bases	Apply knowledge of weak acid titration to weak bases; calculate the pH of a solution at any point along a titration curve	

Oct 31	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 2	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	
<i>Week 10</i>			
Lab	chromatography rotation 2B	Data analysis for GC or LC lab	Revised Vanillin Full Lab Report
Nov 5	<b>Exam 2: Chemical Equilibria</b>		
Nov 7	partition coefficients	Use a partition coefficient to calculate extraction efficiency; explain why a separation step is necessary in some analyses; describe the fundamental principles of chromatography	
Nov 9	selectivity	Find $t_r$ , $t_m$ , $\alpha$ , 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	
<i>Week 11</i>			
Lab	project lab	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 Report 2
Nov 12	efficiency	Calculate plate number and plate height from a chromatogram; list the factors that contribute to band broadening in chromatography	
Nov 14	efficiency, cont'd.	Predict how changes in parameters (such as column packing, stationary phase, analyte characteristics) will affect plate height	
Nov 16	gas chromatography	Compare and contrast GC injection types, programming types (temperature vs. pressure), columns, and detectors	

<i>Week 12</i>			
Lab	project lab	Proceed with trouble-shooting and/or data collection for project lab	Project Intro Draft
Nov 19	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	
Nov 21	<b><i>Thanksgiving Break</i></b>		
Nov 23			
<i>Week 13</i>			
Lab	project lab	Proceed with trouble-shooting and/or data collection for project lab	Question and Figure Outline
Nov 26	GC/HPLC comparison	Compare GC and HPLC separations	
Nov 28	capillary electrophoresis	Explain the source of electroosmotic flow and why it might be useful in a CE separation; predict the migration order of analytes in CE; compare and contrast chromatography and CE	
Nov 30	applying chromatographic theory	Discuss the article "Plate heights below 50 nm for protein electrochromatography using silica colloidal crystals" and its relevance to this course	
<i>Week 14</i>			
Lab	project lab	Complete data collection and data analysis for project lab; draw conclusions for formal report	Project Methods Draft
Dec 3	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 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 using absorbance data; 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 an isosbestic point	

Week 15			
Lab	lab check-out		Project Spreadsheet
Dec10	course summary	Identify the main themes of this course and the important ideas that you will use in the future	
Dec 12	<b><i>Review Period / Reading Day</i></b>		
<b>Formal Project Lab Report due by 8 am on Tues Dec 11. Extensions may be requested by email if accompanied by Word and Excel files showing some progress.</b>			
<b>Cumulative Final Exam: 12 noon on Tuesday, Dec 18</b>			