Syllabus

Instrumental analysis

Modern chemical analysis relies on instrumental methods to augment our senses. Using instruments, we can detect chemical compounds with high sensitivity, distinguish them with high selectivity, and quantify them rapidly. All of this is possible because we have devices that can control and detect physical forces; we understand how matter interacts with these forces; we have access to reference standards for laboratory measurements; and we have developed accepted protocols for comparing measurements.

You don't need to know exactly how every instrument works. In just the next decade, you will see new instruments invented and classic ones adapted for new methods. You should instead strive to understand the principles of measurement and analysis that apply to instrumental methods *generally*. You will then be able to develop, validate, and characterize new methods, and to communicate information about methods in a professional, collaborative context.

Objectives

When you finish this course, you should feel confident that you can: learn about a method using the chemical literature; select, modify, and apply a method to an analytical problem; write and implement protocols for detecting a specific analyte; evaluate the performance of an instrument empirically; and identify parameters that affect a method's performance.

If this sounds a little technical, here is another way to look at instrumental analysis. It's about constantly asking yourself:

- ❒ What property allows me to detect this compound?
- ❒ Is an analytical method working? How do I know? If not, how do I fix it?
- ❒ Where is the error in this method coming from? Can I reduce it?
- ❒ How do I make a method better? What does "better" actually mean?

What will we do in this course?

The theory of chemical analysis is surprisingly general. The principles of detection, linearity, sensitivity, selectivity, interference, statistical significance, and analytical efficiency are applicable to just about any method, and they allow us to compare analytical approaches to a problem. To master these principles, you will practice using them through a combination out-of-class study and in-class discussion.

In *practice*, however, chemists are faced with myriad analytical problems, and each must be solved in its own way. Any time you apply an instrument to a specific analyte, you have a new analytical method. (For example, separating and quantifying free amino acids in neural tissue presents different challenges and constraints than sequencing proteins isolated from the same tissue!)

Solving these problems requires an intuition for developing and implementing protocols. You will develop this intuition by practicing known, established methods and by proposing, testing, and refining your own methods.

Course information

Instructor

Simon Garcia, Department of Chemistry, Tomsich 108, voice 740-427-5077, email: garcias@kenyon.edu.

Availability

Drop-in office hours

Monday, 10:00a–11:30a; Wednesday, 10:00a–11:30a; Wednesday, 2:00p–4:00p.

Appointments

Check gmail calendar for garcias@kenyon.edu for mutually available times. Create an event in your calendar and send me an invitation. If I accept it, the appointment will show in both my calendar and yours.

Class meetings

TR 9:40a – 11:00a in Tomsich 103 and Tomsich 001

Course resources

Laboratory notebook

You need some system for recording experimental data, procedures, and calculations. There are a few possibilities. You could use a bound notebook and write procedures by hand. You could print out procedures and forms and keep them in a binder. You could even set up a spreadsheet on Google Drive, or subscribe to an electronic-notebook service. You will discuss data management with your team and decide on a system.

Calculator

You need a calculator capable of exp, ln, 10^x, log, x^y, sin, and $\sin^{-1}$ functions (devices with internet, voice, or texting functions are prohibited on exams).

Software

You will need access to software for data analysis, such as Microsoft Excel, Google Drive spreadsheets, Minitab, SPSS, Igor Pro, or Origin. Spreadsheets are better for setting up repeated calculations, while professional packages are better for making visual representations of data and results.

Textbooks

You should have regular access to a general textbook on analytical chemistry. More specialized textbooks may be helpful, depending on how much detail you need and what project your team is working on.

On analytical chemistry

Two popular titles are D.A. Skoog, *Fundamentals of Analytical Chemistry*, (Cengage), and D.C. Harris, *Quantitative Chemical Analysis*, (W.H. Freeman). Just about any edition will do, though newer editions will invariably have more current examples. Some online materials are available through Coursera and Saylor.

On chemical spectroscopy

D.L. Pavia *et al*, *Introduction to Spectroscopy*, 3rd or 4th edition, covers the theory of IR, 1-D NMR, 2-D NMR, and MS techniques, and has sample spectra for many

compounds. R.M. Silverstein *et al*, *Spectrometric Identification of Organic Compounds*, 7th edition (Wiley), is a similar text with almost the same content.

On experimental methods

D.T. Sawyer *et al*, *Chemistry Experiments for Instrumental Methods* (Wiley) describes experimental procedures for a variety of specific methods. It is organized by instrument, and is a good starting point for applying an instrumental method to a particular analyte or matrix.

On instrumentation

D.A. Skoog *et al*, *Principles of Instrumental Analysis*, (Thomson) describes the design and construction of instruments. If you want to know about the components of a spectrometer, or the countless devices for dispersing light, or the theory behind the detection of light, start here.

Course materials

Study guides

To gain the knowledge and skills necessary for method development, you will need to research several topics outside of class. I will provide study guides, which may include study questions, essays, and tutorials. Look for examples and exercises to hone your knowledge and skills.

Quizzes

I will provide quizzes for you to test your understanding of each topic. Set aside time in your study schedule to take quizzes under exam-like conditions. Compare your solutions with those in the key to see if you missed any key ideas. Consult with classmates or the instructor if you are confused about a solution.

Model experiments

I will provide model experiments for you to practice calibrating and validating methods. In each experiment, you apply an instrumental method to specific analytes in a particular matrix. The goal is to characterize the method's performance.

Each experiment is structured as a tutorial. The protocols are pretty detailed and set, so your task is to understand the logic behind each detail. Ask yourself, your classmates, and your instructor: Why do we prepare samples in this way? Why do the instruments need these settings? What kind of information do we get from this procedure? Necessary reagents will be available in the laboratory.

Grading

The total grade is based on participation (25%), completion of the method-development project (25%), and the final exam (50%).

Method-development project

Your task is to solve an analytical problem, given a restricted set of resources (instruments, reagents, and equipment), by developing a method. In most cases, you can find a variety of protocols already developed, so you need to adapt them for the specific instrument or sample you are working with. You may request alternative reagents and equipment (within the stated budget), if the chemical literature suggests they would work.

Your ultimate goal is to create a public document reporting on the development, implementation, and validation of the instrumental method. This document will serve as evidence of your ability to carry out method development and to communicate your findings. It must meet the following criteria:

- ❒ Is accessible online
- ❒ Provides sufficient experimental detail for other experimenters to replicate the results
- ❒ Assesses the performance of the method

Before beginning each experiment, you must submit a proposal to the instructor and schedule an appointment between the instructor and all team members. The proposal must list reagents, equipment, and instrumentation requested; provide a detailed procedure; specify how waste will be disposed; and verify that at least one team member knows how to use the instrumentation requested.

Your instructor will provide you with a list of possible analytical problems, but you are welcome to suggest your own.

Participation

Method development is a complex process. It requires constant communication among team members and documentation of discussion, decisions, and actions. We will use class meetings to model this process as you plan and discuss your method-development project. We will observe basic principles of project management.

Part of the grade for this course certifies your experience in project-oriented, collaborative work. Missing class, refusing to contribute to discussions, or failing to respect team members deprives your fellow students of this experience, and will thus incur a penalty of 10% from your participation grade.

Waiver. Because there are various reasons to miss class, such as illness, athletic competitions, and religious obligations, I will waive the penalty for the first three absences, *with no questions asked*. Additional absences will not be waived.

Examination

The final examination will be 2 hours long. Because of limited time, it is impossible to include every concept or skill encountered in class. For this reason, the exam will *sample* topics, so you should expect that *any* topic may appear. 10–20% of each exam will include novel problems that test not just your knowledge, but your mental agility as well.

You should bring a calculator and a sheet of personal notes for reference. You may write on both sides of this note sheet.

The Registrar will set the date and time of the final exam. It is your responsibility to check the Registrar's schedule before you make travel plans. *The College explicitly requires professors to give the final exam at the time scheduled by the Registrar.*

Optional exam. For your convenience, I will administer an optional examination on October 08. If your score on this exam is higher than on the final exam, then it will replace half of the final exam grade.

Policies

Attendance. Attendance at all class meetings is expected. Excessive, unexplained absences will result in dismissal from the course. Only the Dean of Students can excuse an absence. If you must miss a class meeting, or will be late, please notify the instructor by email, or ask a friend to tell the instructor.

Etiquette. Upon arriving in class, please greet the instructor and the students sitting near you. Silence any devices you have. Maintain a respectful presence in class, and avoid disrupting or distracting fellow classmates once class begins. If you bring a computer or tablet to class, please close it when the instructor is addressing the class. If you require frequent trips to the restroom, please seat yourself near the door so that you can exit unobtrusively. Finally, keep in mind at all times how your actions affect the people around you.

Classroom Space. We will move quite a bit during class, so please keep unnecessary items off tables. You only need a notebook, pencil/pen, and calculator at the desk. Place bags and food at the edges of the classroom or on window sills.

Academic honesty. Please read the College's statement on Academic Honesty in the *Course of Study*, pages 26–29. Pay special attention to the definition of plagiarism and to the examples of activities that violate the standards of academic honesty. I expect you to avoid plagiarism and cheating and to avoid even the mere appearance of possible plagiarism or cheating in all of your work.

Excused absence. There are no excused absences.

Unexcused absence. There are no unexcused absences.

Discretionary absence. All absences are considered discretionary. This means that you are responsible for judging the necessity to miss class. Your first three absences will carry no penalty.

Extenuating circumstances. In special cases, such as an extended or serious illness that confines you to your bed (the “Dorm List”), long-term emotional distress due to bereavement, MagiKarp evolution, or other emergency situation, please inform the Dean of Students, who will notify me of your absence (while keeping the reason confidential). Afterward, consult with me and the Dean of Academic Advising to make special arrangements for completing your course work.

Late work. A late assignment is the first step in a vicious cycle of late work. In addition, g*rading late work is disruptive to the schedule and is unfair to everyone in the class*. For these reasons, late work is unacceptable and credit will be reduced if your absence is unexcused. If your absence is excused, consult with me at least two weeks in advance to determine when you will turn in the assignment. In most cases, it will be earlier than the deadline.

Disability services and accommodations. If you have a learning disability or other disability that impacts your ability to learn, or think that you might have one, please schedule an appointment with Disability Services *as soon as possible*. Only the Coordinator of Disability Services is authorized to review your documentation and to recommend an accommodation. All discussions with the Coordinator are confidential. The Coordinator will work with you to design an accommodation tailored to your exact situation. If your accommodation requires special testing conditions (*e.g.*, extra time, separate venue, extra breaks, assistive devices, assistive services), then you must inform me *at least two weeks in advance*. It is your responsibility arrange these conditions with the Office of Disability Services.

Changes to syllabus. The policies articulated in this Syllabus are subject to change in response to unusual situations. You will be notified of any changes at least 48 hours before they go into effect.

Exam dates. The optional exam is October 08, 2013. The final exam for Period B is posted on the Registrar's site. Check your calendar to ensure these dates are free.