

CHEMISTRY 425W: Electrochemistry and Separations
Fall 2017

Lecture: MWF at 10:10-11:00 AM in 217 Thomas
Lab: T (sec. 1), W (sec. 3), R (sec. 2), F (sec. 4) at 1:25-5:30 PM in 330 Whitmore
425 Instructor: Dan Sykes, dgs12@psu.edu, 330 Whitmore
Dan Koeplinger, dbk9@psu.edu, office hours WF 12:20-1:20 PM
Ryan Schonert, rks26@psu.edu, office hours M 2:30-4:30 PM
Text: Instrumental Analysis, 1st edition, Granger et al. (required)
Lab Materials: Lab notebook (Edison Notebook, Roaring Springs Paper Products, #77648)
Eye protection (UVEX Astrospec S1359)

Lecture

There are three lecture periods each week. You should make it a firm rule to attend *every* lecture regardless of their perceived entertainment value. You are responsible for all course material presented in lecture including administrative announcements, syllabus changes, and exam locations. The lectures complement the information in the assigned reading material. If you read the material carefully BEFORE you come to the lecture you will be able to stay up with the flow of the lecture and take notes of things that *add* to what you already learned or which clear up points of previous confusion. Soon after the lecture, go over the notes and work things through in detail. Note that problems on exams may be somewhat different from any problems done in class; you cannot simply memorize rote procedures. It is far better to understand the conceptual basis of the material. Examination questions for any lecture exam will cover material from any and all facets of the course, i.e., do not assume a lecture exam only covers material discussed/presented in the lecture.

The bulk of the course uses algebra to solve sets of equations. You should feel comfortable doing this and capable of doing this in a timely manner. Calculus will also be used throughout the course. You will also use Microsoft Excel for many homework and laboratory assignments. You should feel comfortable preparing spreadsheets (using absolute and relative cell referencing), preparing plots, and adding trendlines. If you do not feel confident in these areas talk with the instructor or a TA early in the semester so you can acquire some proficiency. You will not be able to focus on the chemistry behind the equations if you are focusing on the equations themselves. **Don't fall behind.** Later material in this course depends strongly on earlier material and procrastination can quickly lead to a difficult situation. You should set aside regular times during the week for focused study.

Lecture Format. For each lecture period, unless stated otherwise, the class will meet in 067 Willard for a "traditional" lecture. On days when a lecture activity is assigned, students will work in small groups to discuss a set of in-class questions. Students will record and submit their individual answers to the in-class questions prior to leaving class at 11:00 AM. **Each student should bring a calculator and any assigned reading to each class.**

There are approximately 20 take-home lecture problems that will be assigned throughout the semester. When assigned, the lecture problem will be due the following lecture period at 10:10

2. **Submitting assigned work then leaving lecture.**
3. **Submitting another student's work will earn both you and the other student a grade of zero.**

The only acceptable excuses for late work will be serious personal injury or illness (hospitalization), death in the family, and the like, and **you must let me know your reason for the absence prior to your absence (if possible) or upon your first day back in class and have written documentation.** Only 3 late submissions under such circumstances are accepted.

If you miss more than 10 lectures, you will not earn a passing grade in the course. Withdrawing from the semester would likely be a better option in case of a catastrophic illness or family crisis. Please come to see me if you are facing an extended illness or other difficulty that is causing you to miss numerous class sessions, so we can discuss your options.

You will be allowed to miss a total of 1 laboratory session for any reason (you're sick, you have an athletic absence, you're asleep--whatever). You may use your group's data for the missed laboratory exercise and receive at best 90% of the total points associated with that particular laboratory module. **There are no scheduled make-up labs.** With each unexcused absence beyond the first you will lose 100% of all points associated with the particular laboratory module your group is completing at that time.

Electronic Devices. Ah, the electronic age! Electronic devices like pagers, cell-phones, iPods, watches with alarms, etc., are **NOT** welcome in either lab or lecture. Please show some consideration and respect for your classmates and your professors. It is quite distracting to have a phone ringing, for instance, during class. **If a device in your possession sounds more than once during the semester, you will lower your final grade for the course by a letter.** Other than calculators, all electronic devices are to be kept stowed. **If a student is caught with an electronic device during, or while in, the lab (e.g., checking/sending messages, device laying on a benchtop or other surfaces) then that student will forfeit all points associated with the lab activity.**

Course Objectives:

1. To explore the practical instrumentation skills necessary to obtain high quality results.
2. To identify the intermolecular forces driving chromatographic separations.
3. To introduce the basic principles of operation and theory of ion sources and mass filters.
4. To introduce common electroanalytical techniques for analyte characterization

Learning Objectives: After completing this course, you will be able to ...

1. Identify the factors (and associated equations) which influence the efficiency and resolution of a chromatographic separation and be able to quantify these contributions.
2. Identify which factors influence column performance.
3. Identify the chemical interactions (intermolecular forces) which give rise to analyte separation and use this knowledge to develop isocratic- or gradient-based elution methods.
4. Evaluate the instrumental contributions (extra-column) to the resolution and efficiency of chromatographic separations.
5. Independently operate, troubleshoot, fix and maintain HPLC instrumentation.

6. Explain the thermodynamic underpinnings which give rise to analyte separation and use this knowledge to develop isothermal- or gradient-based elution methods.
7. Independently operate, troubleshoot, fix and maintain GC instrumentation
8. Develop an optimized LC-MS method for Q1, EPI and MRM analysis of analyte mixtures.
9. Interpret EI mass spectra and identify molecular structures based on their EI mass spectra.
10. Perform SPE and derivatization methods of sample pretreatment.

Grading

In fairness to the instructor and TAs who must grade the required course work, and to the other students, **late work will not be accepted.** Think about this. A substantial number of late or missing work will affect your grade. Stay caught up.

<u>Element</u>	<u>Points</u>
Lecture Exams 3@ 200 points each	600
Lecture Problems 20@ 10 points each	200
Lecture In-class Activities 10@ 20 points each	200
Total Lecture-based Points	1000 points
Laboratory Notebook – 1 st module	200
Laboratory Notebook – 2 nd module	200
Laboratory Notebook – 3 rd module	200
Laboratory Notebook – 4 th module	200
Poster Symposium	200
Total Laboratory-based Points	1000 points
Total Course Points	2000 points
Points Earned	Letter grade
1860-2000	A (93%)
1800-1859	A- (90%)
1740-1799	B+ (87%)
1640-1739	B (82%)
1560-1639	B- (78%)
1500-1559	C+ (75%)
1400-1499	C (70%)
1200-1399	D (60%)
<1200	F (<60%)

There will be no grading curve for this course. The number of points earned determines your grade in this course.

Safety Precautions.

1. **Chemicals** - Many chemicals and much of the equipment you will be using are quite hazardous if misused. Handle such materials with caution.
2. **Safety Glasses** - Safety glasses must be worn during the time you are in the laboratory. Inexpensive but adequate glasses can be obtained at the bookstore. Don't remove your glasses because you aren't doing anything -- your neighbor may be doing something hazardous.
3. **Laboratory Clothing** - Don't wear good clothes or clothes with ties, loose cuffs, *etc.* that could get into a flame or solution. Wear shoes that adequately protect your feet. **NO SANDALS OR BARE FEET ARE ALLOWED. NO SHORTS ALLOWED.**
4. **Fire Extinguishers** - Be sure you know the location of the nearest fire blankets, CO₂ extinguishers, sand, *etc.*
5. **Spills** - If you accidentally spill some chemical reagent on yourself, immediately wash the affected area with cold water. A special fountain for washing eyes and a shower are located in the northeast corner of the room.
6. Report any accident, no matter how trivial, immediately to the instructor or teaching assistant.
7. Dispose of all laboratory waste properly.
8. **Label all containers** - this is not only a good safety practice, but it also avoids lost samples.
9. Perhaps the most prevalent potential hazard you will encounter in this course is electricity. Don't underestimate it. Handle any electrical circuits or components with caution.

Academic Integrity is an essential component of your education. The following is quoted from the "PSU Faculty Senate Policies for Students". "It is the pursuit of scholarly activity free from fraud and deception and is an educational objective of this institution. Academic dishonesty includes, but is not limited to, cheating, plagiarizing, fabricating of information or citations, facilitating acts of academic dishonesty by others, having unauthorized possession of examinations, submitting work of another person or work previously used without informing the instructor, or tampering with the academic work of other students." All University and Eberly College of Science policies regarding academic integrity/academic dishonesty apply to this course and the students enrolled in this course. Refer to the following URL for further details on the academic integrity policies of the Eberly College of Science : <http://www.science.psu.edu/academic/Integrity/index.html>. Matters of academic dishonesty will be turned over to the University disciplinary system and may result in the failing of the course.

Code of Mutual Respect and Cooperation for Faculty, Staff and Students in The Eberly College of Science

The Eberly College of Science is a community dedicated to personal and academic excellence. The Code of Mutual Respect and Cooperation was developed to embody the values that we hope our faculty, staff, and students possess, consistent with the aspirational goals expressed in the Penn State Principles. The University is strongly committed to freedom of expression, and consequently, the Code does not constitute University or College policy, and is not intended to interfere in any way with an individual's academic or personal freedoms. We hope, however, that individuals will voluntarily endorse the 12 principles set forth in the Code, thereby helping us make The Eberly

College of Science a place where every individual feels respected and valued, as well as challenged and rewarded.

The 12 Principles of the Code are:

1. Treat everyone equally and with respect
2. Be courteous
3. Be ready to communicate
4. Encourage others and share your expertise with them
5. Give and accept constructive criticism
6. Be receptive to change
7. Be a team player
8. Get involved
9. Have a positive attitude
10. Be honest and accept responsibility
11. Recognize other people's priorities
12. Strive to do your best

Services for Students with Disabilities

Penn State welcomes students with disabilities into the University's educational programs. Every Penn State campus has an office for students with disabilities. The Office for Disability Services (ODS) Web site provides contact information for every Penn State campus: <http://equity.psu.edu/ods/dcl>. For further information, please visit the Office for Disability Services Web site: <http://equity.psu.edu/ods>.

In order to receive consideration for reasonable accommodations, you must contact the appropriate disability services office at the campus where you are officially enrolled, participate in an intake interview, and provide documentation: <http://equity.psu.edu/ods/doc-guidelines>. If the documentation supports your request for reasonable accommodations, your campus's disability services office will provide you with an accommodation letter. Please share this letter with your instructors and discuss the accommodations with them as early in your courses as possible. You must follow this process for every semester that you request accommodations.

CHEM 425W

**Lecture Schedule
Fall 2017**

The “Assigned Reading” is material students are required to read prior to the date of the lecture period it is listed beside. For example, students are required to read Chapter 22 prior to lecture on August 29.

Date		Topic
Aug	21	Course Introduction
	23	Analytical Separations - review
	25	Solid-phase extraction
	28	Solid-phase extraction
	30	Chromatographic methods
Sept	1	Basics and terminology
	4	Labor Day/No class
	6	Basics and terminology
	8	Basics and terminology
	11	HPLC column basics
	13	HPLC column basics
	15	HPLC particles
	18	HPLC bonding and packing
	20	HPLC phase chemistry
	22	HPLC phase chemistry
	25	HPLC phase chemistry
	27	HPLC phase chemistry
	29	HPLC method development
Oct	2	HPLC method development
	4	HPLC method development
	6	HPLC method development
	9	Ion-Exchange Chromatography
	11	Molecular Exclusion Chromatography
	13	Other HPLC applications
	16	GC basics
	18	GC injection
	20	GC injection
	23	GC phase chemistry
	25	GC phase chemistry
	27	GC phase chemistry
	30	GC Detectors

Nov

1 GC Detectors
3 GC method development
6 GC method development
8 Mass Spectrometry
10 Mass Spectrometry
13 Mass Spectrometry
15 Mass Spectrometry
17 Mass Spectrometry
20 **Thanksgiving Break**
22 **Thanksgiving Break**
24 **Thanksgiving Break**
27 Capillary/Gel Electrophoresis
29 Capillary/Gel Electrophoresis

Dec

1 Capillary/Gel Electrophoresis
4 Capillary/Gel Electrophoresis
6 Special topic
8 Special topic