

Chemistry 443
Advanced Analytical Chemistry
Dr. Jeremy Ramsey
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Course Description

The material presented in this course covers the basic principles of instrumental analytical chemistry, including the fundamentals of spectroscopy, separation science, and electrochemistry. Additionally, the course will cover the fundamentals of measurement science and hopefully, biological analysis.

Philosophy of Instruction

Ultimately, the purpose of this course is for you to learn the foundations upon which modern instrumental analysis is based. In my opinion, grades are secondary to your understanding of the subject, and ideally, I am willing to present each of you with an A. In reality, some students will be more/less motivated or be more/less inclined toward the material. These differences will lead to differences in performance. My goal is to see each student achieve mastery of the subject and I am dedicated to reaching this goal. The motivation, however, must begin with you. Please take advantage of the opportunities available to you and we will both achieve the goals that we have for the semester.

Prerequisites

- Chemistry 331 and 332 (or permission of instructor)
- Knowledge of chemical reaction stoichiometry and equilibrium
- Introductory statistical analysis techniques
- Basic skills in Microsoft Excel

Meeting Times

Lecture	MWF	11:30-12:20 am	215 Heim
Laboratory	T	1:00-4:50 pm	207/223 Heim

Required Course Materials

- Principles of Instrumental Analysis, 6th edition; Authors: Skoog, Holler, and Crouch
- Bound laboratory notebook
- Safety Glasses
- Calculator capable of performing logarithmic, exponential, and statistical functions

Office Hours

Since I tend to spend a lot of time in and around my office, I will not be having formal office hours for this course. As many of you know, I encourage you to just stop by when you have a question or concern.

Grading

- There will be no makeup examinations or quizzes. One legitimate exam absence (excuse must be approved by the Dean's office: for instance, an illness or a funeral) can be replaced by the average exam score for the semester.
- Makeup laboratory experiments will be nearly impossible and will only be permitted for legitimate reasons (excuse must be approved by the Dean's office). If at all possible, please contact me (not your classmates) before the laboratory period has been missed! Attendance in laboratory is mandatory since we will be working in groups.

	<u>Points</u>
Examinations (5)	450
<i>Highest midterm score</i>	105
<i>Middle midterm score</i>	100
<i>Lowest midterm score</i>	95
<i>Final exam</i>	125
<i>Final exam (ACS standardized)</i>	25
Lab Reports and Performance	100
Participation	15
Presentation	50
Safety and Cleanliness	15
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Quizzes

We will not be having regular quizzes in this course. However, an unexcused absence may be cause for an unannounced quiz in either the lecture or laboratory portions of the course. The purpose of these quizzes is to ensure attendance in class. As with the examinations, quizzes should be considered cumulative and may contain information from the laboratory or lecture portion of the course.

Department Participation

All chemists use chemical analysis techniques to some degree. This is most clearly observed by watching others discuss chemical research. For us this opportunity is chemistry colloquium and you will receive the full participation grade for attending at least FIVE colloquia (each is worth three points). If you are unable to attend colloquia due to a scheduling conflict, you can receive the participation points by summarizing research articles. You should discuss this with me before you begin.

Homework

In this course, homework assignments will not be collected, but it is strongly suggested that you attempt them. The selected homework problems provide an indication of the topics that I think are important. This makes solving them of utmost importance to your grade and your performance in the course will likely correlate with the amount of time spent solving problems. Because learning can be much more efficient through failure, I feel strongly that the problems should be attempted individually before seeking help from others. Your answers can be checked with answers in the back of the textbook. Please feel free to stop by my office to discuss any difficulties you may have with any of the suggested problems.

Lab Reports

Lab reports are due one week (7 days) following completion of the experiment, unless otherwise informed by the instructor. Grades on late lab reports will be reduced by 10% plus 5% per calendar day beyond the due date. The format of the reports is the same as that necessary for publication in the journal, *Analytical Chemistry*. A summary of the format is attached to this syllabus.

Presentation

Although it is often thought to be painful, the ability to orally present complicated concepts is one of the most important skills that you can possess. Each student will be required to present the results from the experiments that they design/perform. The grade for the presentations will be based on the evaluation by the class and the instructor. Each student will have ~20 minutes for their presentation.

Examinations

Examinations will be given during laboratory sessions and will be administered on the following dates. Because the material presented in the class builds upon concepts presented earlier (and from Chemistry 332), all exams should be considered cumulative. Changes to the exam schedule will be made only with unanimous consent of the class and must be made prior to January 14.

Examination 1	February 5
Examination 2	March 11
Examination 3	April 8
Final Examination	April 22

** The final examination time and date is established by the registrar. It cannot be changed.

Safety

Safe laboratory practices, including proper attire, will be expected at all times. Long pants are required as well as closed toe shoes (no sandals). Wearing contact lenses during laboratory session is strongly discouraged, but may be tolerated with prior approval. You will not be permitted to begin any experimental procedures until all safety concerns have been addressed. **Repeated safety violations will cause a zero to be rewarded for the experiment.**

Laboratory Topic Schedule

The class schedule presented here is tentative and can (will!) change during the semester.

<u>Week Beginning</u>	<u>Laboratory Topic</u>
January 7	Check in/Analysis of Aspirin in Alka Seltzer [*]
January 14	Analysis of Aspirin in Alka Seltzer [*]
January 21	Atomic Absorption of Copper (How Much Copper is in a Penny?)
January 28	Fabrication and Use of an Absorbance Spectrometer (Day 1) [*]
February 4	Exam 1
February 11	Fabrication and Use of an Absorbance Spectrometer (Day 2) [*]
February 18	How Hot Is That Hot Pepper? (HPLC)
February 25	Spring Break—No class
March 3	Analysis of Cocaine on Currency (GC-MS)
March 10	Exam 2
March 17	Christmas in March! Fabrication of a pH Electrode (Day 1) [*]
March 24	Christmas in March! Fabrication of a pH Electrode (Day 2) [*]
March 31	Cyclic Voltammetry [†]
April 7	Exam 3
April 14	Check Out
April 21	Final Exam

* Indicates that a presentation will be required for this experiment

† Indicates that no laboratory report will be due

Roles for Chemistry 443 Laboratory

Supervisor

The supervisor is the position of most responsibility and in turn should be the best prepared. This person will be responsible for decisions for the group and will define the roles of the others. Because supervisors are ultimately responsible for the results of the group, the supervisor will prepare for the experiment prior to the laboratory session and will prepare the report for the group.

Responsibilities

Writing a title, objective, and experimental section prior to arriving at lab

Defining the roles of the other students

Making decisions concerning the direction of the group

Writing the laboratory report that summarizes the experimental work

Summary of the work performed and roles

Chemical Technician

The technician is responsible for the majority of the wet chemistry and solution preparation.

Responsibilities

Performing solution preparation

Preparing samples for analysis

Summary of the work performed

Instrument Technician

The instrument technician is responsible for understanding the operation of the instrument and for performing the instrumental analysis. This also involves the collection, conversion, and distribution of data to the other group members.

Responsibilities

Understanding the functionality of the instrument

Performing instrumental analysis

Summary of the work performed

Example laboratory rotation of duties

Week	Experiment	Role	Assignment Due
1	Experiment 1	Supervisor: Curly Instrument: Moe Chemical: Larry	
2	Experiment 2	Supervisor: Moe Instrument: Larry Chemical: Curly	Report: Curly Work Summary: Moe Work Summary: Larry
3	Experiment 3	Supervisor: Larry Instrument: Curly Chemical: Moe	Report: Moe Work Summary: Larry Work Summary: Curly
4			Report: Larry Work Summary: Curly Work Summary: Moe

How to Write Lab Reports for Analytical Chemistry

The purpose of a lab report is to communicate the results of an experiment or set of experiments. We will be modeling our lab reports on the style used in the journal *Analytical Chemistry*. The goal of this format is to teach you to write scientific observations and procedures in a concise writing style and because of this, the written portion of your reports should be no longer than a couple of pages. When you are writing a report, you should be asking yourself: Is this readily understandable and am I using too many words to describe this procedure? Your reports will be graded not only for performance in the laboratory, but also for your ability to communicate the information in writing.

Lab reports should be typed, must include copies of all necessary graphs/figures, and should have the following sections: title, introduction, experimental, sample calculations, results and discussion, and summary of work performed.

- **Title:** This can be the title from the laboratory handout. You should also have the title in your notebook.
- **Introduction:** This section provides background information concerning the experiment that you have performed. Its purpose is to explain the reasons for performing the experiment, what prior experiments have been performed, and an explanation of the techniques chosen.
- **Experimental:** This is likely going to be the longest section of the report and also the most difficult to write. It is particularly important to clearly convey what you did when you were in the laboratory. Any techniques that you utilized should be described here. Likewise, deviations from the established procedure should be described.
- **Sample Calculations:** A sample of each calculation utilized should be represented here. There is no need to include a sample calculation for mean and standard deviation. If you choose to statistically exclude any data, you should include all calculations here and include a description of why.
- **Results and Discussion:**
 - **Results:** A tabulation of the data collected during the experiment should normally be included here. Data does not need to be limited to tables. Sometimes observations are considered data.
 - **Discussion:** You should discuss the measurements made during the experiment along with explanation of any errors (systematic) that may have occurred. In the case of errors, thought should be given to what effect the error may have had on the measured quantity. Your discussion should answer the question—Am I confident with the measurements that I have made (and explain why)?
- **Summary of work performed:** This section should describe the role that each person in the group had as well as their contribution to the experiment/laboratory report.