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. The course will require use of the fundamentals of quantitative measurement science as presented in Chemistry 332—Analytical Chemistry.

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.

Learning Objectives

Upon completion of this course, students should be able to

- Understand the principles by which chemical instrumentation operates
- Identify components of chemical instrumentation and relate it to the function of the instrument
- Understand how basic instrumentation components can be combined to produce different, working instruments
- Perform calculations involving stoichiometry and solution concentration as they relate to laboratory problems
- Use the conclusion of statistical analysis to determine sources of experimental error and to direct the path of subsequent chemical experimentation
- Analyze written scientific literature to understand chemical instrumentation
- Communicate scientific data clearly in oral presentations
- Analyze student presentations critically to determine their quality
- Use graphical analysis software to analyze data and prepare publishable quality graphs

Meeting Times

<table>
<thead>
<tr>
<th></th>
<th>MWF</th>
<th>11:30-12:20 am</th>
<th>215 Heim</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lecture</td>
<td>MWF</td>
<td>11:30-12:20 am</td>
<td>215 Heim</td>
</tr>
<tr>
<td>Laboratory</td>
<td>T</td>
<td>1:00-4:50 pm</td>
<td>207/223 Heim</td>
</tr>
</tbody>
</table>

"In the middle of difficulty lies opportunity."
-- Albert Einstein

“Success is a lousy teacher. It makes smart people think they can’t lose.”
-- Bill Gates

“Opportunity is missed by most people because it’s dressed in overalls and looks like work.”
-- Thomas Edison
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 and Igor Pro

Required Course Materials

- *Principles of Instrumental Analysis*, 6\(^{th}\) 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, so attendance in class on exam dates is mandatory. One legitimate exam absence (excuse must be approved by the Provost’s office: for instance, an illness or a funeral) can be replaced by the final, standardized exam score.
- Attendance in laboratory is mandatory since we will be working in groups. Makeup laboratory experiments will be nearly impossible and will only be permitted for legitimate reasons (excuse must be approved by the Provost’s office). You should contact the instructor (not your classmates) before any laboratory period has been missed.

<table>
<thead>
<tr>
<th>Points</th>
<th>Examinations (4)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Midterm exam 1</td>
<td>125</td>
</tr>
<tr>
<td>Midterm exam 2</td>
<td>125</td>
</tr>
<tr>
<td>Take home final exam</td>
<td>75</td>
</tr>
<tr>
<td>Final exam (ACS standardized)</td>
<td>75</td>
</tr>
</tbody>
</table>

Lab Performance

- Presentations 50
- Reports 25

Quizzes 50

Participation 15

Colloquium Attendance 15

Safety and Cleanliness 15

570

Points
Quizzes

We will have a minimum of three quizzes in this course, which may be unannounced. The purpose of these quizzes is to ensure attendance in class and to provide motivation for staying current with the course material. As with the examinations, quizzes should be considered cumulative and may contain information from the laboratory or lecture portion of the course.

Colloquium Attendance

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 will be discussed at a later date.

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 each presentation will be based on evaluation by the class and the instructor. The grades of all three group members will then be combined to produce a group grade, which will be assigned to each member of the group. It is in your interest to help your fellow group members and encourage them to perform to their best ability.

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 15.

| Examination 1 | February 16 |
| Examination 2 | April 13 |
| Final Examination* | April 29; 8:30 am |

** 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.*

The class will also be evaluated for its safety and cleanliness. If any member of the class is found to be in violation (leaving a chemical spill, not cleaning balances after using them, squirting water, etc.), the entire class will be penalized. *It is in your best interest to make sure that your classmates are acting appropriately during lab time.*

Laboratory Topic/Examination Schedule

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

<table>
<thead>
<tr>
<th>Week Beginning</th>
<th>Laboratory Topic</th>
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<tbody>
<tr>
<td>January 11</td>
<td>No lab this week (Choose absorption experiment)</td>
</tr>
<tr>
<td>January 18</td>
<td>An Experiment in Forensic Chemistry: The Breathalyzer</td>
</tr>
<tr>
<td>January 25</td>
<td>An Experiment in Forensic Chemistry: The Breathalyzer</td>
</tr>
<tr>
<td>February 1</td>
<td>Fabrication and Use of an Absorbance Spectrometer (Day 1)</td>
</tr>
<tr>
<td>February 8</td>
<td>Fabrication and Use of an Absorbance Spectrometer (Day 2)</td>
</tr>
<tr>
<td>February 15</td>
<td><strong>Exam 1 (during lab)</strong></td>
</tr>
<tr>
<td>February 22</td>
<td>How Much Copper is in a Penny? (Atomic Absorption)</td>
</tr>
<tr>
<td>March 1</td>
<td><strong>Spring Break—No class</strong></td>
</tr>
<tr>
<td>March 8</td>
<td>Christmas in March! Fabrication of a pH Electrode (Day 1)</td>
</tr>
<tr>
<td>March 15</td>
<td>Christmas in March! Fabrication of a pH Electrode (Day 2)</td>
</tr>
<tr>
<td>March 22</td>
<td>Christmas in March! Fabrication of a pH Electrode (Day 3)</td>
</tr>
<tr>
<td>March 29</td>
<td>Cyclic Voltammetry*/Unidentified GC Experiment</td>
</tr>
<tr>
<td>April 5</td>
<td>Cyclic Voltammetry*/Unidentified GC Experiment</td>
</tr>
<tr>
<td>April 12</td>
<td><strong>Exam 2 (during lab)</strong></td>
</tr>
<tr>
<td>April 19</td>
<td>Check Out</td>
</tr>
<tr>
<td>April 26</td>
<td>Final Exam (4/29; 8:30-11:30 am)</td>
</tr>
</tbody>
</table>

† 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

<table>
<thead>
<tr>
<th>Week</th>
<th>Experiment</th>
<th>Role</th>
<th>Assignment Due</th>
</tr>
</thead>
</table>
| 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 (including sample calculations), 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. These must be typed using Equation Editor (See the Moodle website).

- **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.
Towards Better Writing Skills

Guidelines for writing lab reports

Lab reports are a written version of the experiment that you performed in the lab. While industries/departments may adopt a preferred structure for lab reports, there is clearly a general format that is the norm. It includes the following sections that you will see consistently in scientific papers: Title, Abstract, Introduction, Materials and methods, Results, Discussion, Conclusion, References, and Appendices. However, your lab reports in this unit need not include all of these headings. Use the following headings in your lab reports:

- Title
- Introduction
- Materials and methods/Experimental
- Results and discussion
- References

Further guidelines are provided below.

Introduction

- Clearly and concisely tell the reader what to expect in the report.
- Use the past tense and follow the general principles of scientific writing (Handout I).
- Include the following elements:
  - Background - Briefly summarise relevant previous research and narrow the focus specifically to your experiment.
  - Problem - State the issue investigated by the experiment.
  - Aim – The last paragraph should state the aim of the experiment.

Materials and methods

- State clearly how you conducted the experiment so that it may be repeated.
- Include all chemical used and their quality (e.g. Laboratory grade, HPLC grade, Analytical grade).
- Describe the equipment used and the operating conditions.
- Describe any methods used (e.g. sample preparation steps, preparation of standard solutions).
- State how you analysed the data (e.g. statistical methods or formulas used).

Results and discussion

Results can be treated as a separate section, followed by the Discussion. An alternative approach is to combine the results and discussion sections. In the latter, it is usually best to present a result, discuss it, then present the next result and discuss it etc. In either case the following guidelines apply

- Results must be described fully in the text
- Present the data accurately, clearly and systematically.
- Use tables and figures (includes graphs, diagrams, photographs) to amplify and complement the text.
Tables and figures must have a complete caption that allows the table to be interpreted without reading the text.

- Explain and interpret the results
- State whether the results are supported by previous work. If not, provide possible reasons.
- Link your discussion back to what you said in the introduction and to relevant literature on the topic.
- Suggest further steps in the scientific process (e.g. that it may be advisable to repeat the experiment with slightly different procedures, or that future researchers could explore a different dimension to the design) where applicable.

References

- Accurately cite/reference information (i.e. ideas and words) used from other sources.
- Be consistent with in-text and end-text referencing. Suggest using the format adopted by a journal that you read.
- Include only sources you have cited in-text in the reference list.

Appendices (occasionally)

- Use appendices to include detailed information to support / substantiate something you have said in the report (e.g. raw data of your results, detailed statistical calculations).
- Number your appendices clearly and when you make reference to them within your report indicate the appendix number (as you would do for tables and figures).