

Chemistry 582
Advanced Chemistry Laboratory 1
Fall 2018 Version

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CHEMISTRY 582
ADVANCED CHEMISTRY LABORATORY 1
Course Syllabus, 2018

1. General Information

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Course Objectives and Methods: This course will give you practical experience with advanced techniques for organic and inorganic synthesis, and for subsequently characterizing the compounds that you make. A major goal will be to prepare you for independent research in chemistry. The course will bridge the gap between the highly structured experiments that you have thus far encountered in undergraduate laboratories, and the situation that you will find in a research laboratory, where you generally design your own experiments. You will need to use your time wisely. More than two absences over the semester is unacceptable, and can cause failure for the course.

The course is divided into two parts. The first two thirds of the semester will consist of short, limited-scope experiments, similar to those that you have performed in past laboratory courses. Each of these experiments will have features not seen in your previous lab courses, including emphasis on purity and characterization of products, more elaborate procedures and sensitive reagents. While some of these experiments could be treated as “cookbook” instructions, that would be a mistake. You are expected to understand what is happening at each step, and the purpose of each operation. During this part of the course you will also learn to use the advanced instrumentation available throughout the Chemistry and Biochemistry department for characterizing the compounds that you make. Note also that the instructions will contain fewer of the important details as the course proceeds; you are expected to apply your knowledge to decide which apparatus to use, for example. At the end of this section, a group project is carried out, where each student will have a separate role and all data must be combined to complete the experiment. In the last third you will choose a project from a selection of topics, and undertake this project more or less independently. Several projects involve adaptation of earlier experiments for new and different results.

This syllabus is the first section of a course text that will be provided. You should also consult other lab texts and the sources cited herein for procedures.

2. Course Structure

Part 1: Formal experiments (~2 months).

For each experiment you are provided with the following:

- § A detailed summary of the experimental techniques to be employed
- § Additional references to helpful textbooks and/or journal articles
- § Detailed procedures

Timeline:

§ Nominally, you will have 2 sessions to complete each experiment (see schedule below), but you will often be running more than one experiment simultaneously. This is not accidental: frequently the most efficient approach is to intersperse your work on two projects. Think of this as a challenge to your organization and scheduling abilities. You may be able to finish some experiments in less than two sessions, whereas others may take considerably longer.

§ The write up for each experiment is due at the start of class 1 week after the last scheduled date for that experiment. In some cases, you may have been unable to complete some detail of a procedure by the deadline. In that case, write up what you have, and specify exactly what you have yet to do, and how you will analyze the data. When you later get the data, you may submit a report addendum to complete your report. Such extra work may be submitted for full credit **only** if you have specified in the original report what is to be done and how: the original graded report must be submitted together with the addendum. The addendum must specify which are the new parts to be graded. Any extension over a week must be authorized by the TA. However, the absolute deadline for all write ups of this section will be one week after the completion of the final experiment of this section. If you are organized, this will allow you ample time to repeat parts of experiments that don't work the first time. All lab reports are required: only I or F grades are possible if any are missing.

§ **Important note:** You must come to lab prepared! This includes understanding the risks involved in using the substances you will handle. While we will alert you to safety hazards, you should look up the MSDS for each substance before its use. If you will prepare a substance for which no MSDS is available, you should look up the properties of several related compounds. Any substance whose hazard rating on the MSDS is 3 or above should be noted in your notebook before class. You should look up safety requirements for all substances and equipment you intend to use, and check it out with TA and/or Professor before use.

§ The nominal schedule doesn't directly take into account time needed for extra purification steps (e.g. recrystallizations), or for running characterization steps such as NMR, IR, etc. It will thus be *essential* that you make good use of any "dead time" for multi-tasking. For example: if an experiment calls for you to reflux a solution for 2 hours, you should use those two hours to filter a solution from a previous experiment, run an NMR of it, etc. Your TA will make every effort to alert you about upcoming blocks of dead time, but you will do much better if you can identify these yourself.

Making sure to be familiar with all reagents, reactions, and procedures before coming to lab will cause you lab time to be much more enjoyable and productive. Note that videos of most experimental procedures are to be found on the MIT website at the address given in the appendix on recommended references. This and other appendices should be quite helpful.

§ Nominal schedule for part 1

Experiment	Date	Comments
Safety/Check in, wash glassware.	9/5	Place glassware in oven for expt. 1.
Carbonation of an unknown aromatic halide	9/10, 9/12	
Diels-Alder	9/17, 9/19	
Fluorescein	9/24, 9/26	If practical, start 9/19
Synthesis of 6-nitrosaccharin	10/1 – 10/3	Inform TA of your choice for independent project (Part 2).
Preparation and Conductometric Analysis of $[\text{Co}(\text{NH}_3)_x\text{Cl}_y]\text{Cl}_z$	10/8, 10/10	Inform TA and stock room of requirements for your first independent experiment (part 2).
Synthesis and Metal-catalyzed Hydrolysis of <i>bis</i> <i>p</i> -nitrophenyl phosphate: Michaelis-Menten kinetics	10/15 - 10/24	
Begin individual projects		
Last day to complete Part 1 Experiments	10/29	
Final deadline for all Part 1 lab reports	11/5	
Final deadline for project reports, checkout	12/12	

Performance Evaluation for Part 1:

- § Laboratory notebook pages will be collected at the end of each day and evaluated. You will have a chance to improve on this grade if your notebooks improve over the semester.

- § You will write a lab report for each experiment, in the same format as an article submitted to an American Chemical Society (ACS) journal.

- § You will answer the questions given at the end of each experiment, and include these answers as “Supplementary Material” at the end of the lab report (as per an ACS journal article).

- § You must provide convincing evidence of the purity of any compounds you prepare. The evidence will vary depending on the experiment, but should include characterization by at least 2 physical techniques, such as melting point, NMR, extinction coefficient, IR, gas chromatography, etc. *An experiment will not be considered complete unless all synthesized compounds are properly characterized.*

Part 2: Introduction to research (~1.25 months). In this part of the course you will try to carry out original research or to reproduce a specific research project that was previously published in the primary literature, using only the original references for guidance.

What will be provided:

- § 3 possible titles for research projects
- § An outline of the desired outcome for each research project (~ half a page)
- § References to journal articles from the original literature.
- § Reports from students who worked on these projects last year, so that you may learn from their efforts, and continue the projects into new territory, rather than simply repeating what was done.

Timeline:

- § The titles, project outlines and journal article references will be handed out at the beginning of the semester. You will then have 3 weeks to choose a topic, and inform your TA of your choice.
- § You will have the whole last third of the semester to complete your project. No later than 1.5 weeks before beginning each new experiment, you *must* present a plan to your TA, *and* provide the stock room staff with a list of the chemicals and materials that you will require.
- § The final project will be due on study day before finals week, Dec. 15th. The last day of class no experimental work is to be carried out: we leave the lab in pristine condition with all glassware clean and accounted for.

Performance Evaluation for Part 2:

- § The lab report for the last third of the semester will be different from those of the first two thirds. In this case, you will attempt to create a detailed, self-contained protocol that could serve as a formal lab for subsequent generations of Chem 582 students. In other words: you will try to convert the terse, sometimes difficult-to-follow instructions of a typical journal article into a more user-friendly format. In addition, your report will put the experiment into a context that will be meaningful for the future student. The best experiments from any given year will indeed be used in future years as “introductory” experiments (i.e., experiments to be performed in the first 2 months of the semester).
- § The quality of your laboratory notebooks will be an important part of the final grade. The grade will improve if your notebooks improve in the second half of the semester.
- § You must provide convincing evidence of the purity of any compounds that you prepare, *or* plausible data-supported explanations of why this was not possible. Evidence of purity will vary depending on the experiment, but should include characterization by at least 2 physical measurements, such as NMR, melting point, extinction coefficient, IR, gas chromatography, etc.

3. Grading Policies

The grade is split into portions as follows:

Notebook	30%
Reports	30%
Product quality and characterization	30%
Technique	10%

Product quality and characterization - Throughout the semester you must provide convincing evidence of the purity of any compounds you prepare. The evidence will vary depending on the experiment, but should include characterization by at least 2 physical techniques, such as melting point, NMR, extinction coefficient, IR, gas chromatography, etc. *An experiment will not be considered complete unless all synthesized compounds are properly characterized.* In addition, you should record % yields for all compounds, *once they are purified.* In all cases high purity is considered more important than high yield. All products must be turned in to the TA in tared, properly labelled vials. “Properly labelled” means it must have the date, your name, the structure, notebook page number, and tare. Labelling will be graded.

Technique - This is an evaluation of your technique in the laboratory, based on your instructor's observations over the semester. If your technique is poor at the beginning of the semester and then improves, your grade will be based primarily on your technique at the end of the semester. Everyone's goal should be to improve on one's technique. Good technique involves cleanliness, care of glassware and equipment, accurate following of instructions, and attention to safety issues.

Notebook -

A bound notebook with duplicating pages is required: do not use spiral bound or regular composition notebooks. All pages should be numbered; this may be done by hand if yours is not already numbered, but must be done on all pages at the start of the semester. A number stamp will be available for your use. Use either a black or blue pen only; no pencil or other colored ink will be allowed. Never erase, and tear out pages *only* to hand in the duplicates. Cross out any deletions with a single line so that the error remains readable, and add your initials and the date of correction. The initial and dating procedure is a requirement when working in industry, especially in companies that are ISO certified, so this is a good practice to follow.

Write in the notebook as you work; do not write in the notebook outside of the laboratory other than to provide data to complete the pre-lab. It records what you did, not what you intended to do. The duplicate copy of your lab notes from that day must be turned into your TA *before you leave the lab*. Your TA will grade the pages, following the criteria listed below, and return them to you the next class period with a grade and suggestions for improvement. The notebook pages will be worth a substantial amount of your final grade, 30%. Steady improvement in note-keeping over the term will compensate for errors early in the term.

Before each lab period a short pre-lab must be completed, in addition to any necessary reading material, before starting any experiments. This pre-lab must contain the title, any necessary equations, and a reagent table. The reagent table should be set up as follows:

Name	Formula / MW	State at Room Temp.	Density	Melting Point	Boiling Point	Hazards or Cautions

At the start of each class period, your lab notebook, with the pre-lab completed, must be showed to and signed off by the TA. Do not proceed with any experiments until this is completed.

Notebook Grading

Each class period a copy of your notes will be turned in before you leave. Each set of notebook pages will be worth 10 points, based on the following criteria:

Title/equation – 1 point

Date – 1 point

Reagent Table – 1 point

Procedure (what you did, not what you meant to do; time, temperature, exact quantities used) – 4 points

Observations – 2 points

Results / Conclusion – 1 point

The points for the title/equation, date, reagent table, and results/conclusion will be all or nothing, if they are present you will get the points. The points for the procedure and observations will vary depending on the context of the material. The notebook grade is for keeping a proper notebook, and does not reflect the quality of your work in other areas. Note, however, that all information in your lab report must come from your notebook, or from a properly cited source.

Sample Notebook Page

Date	Title					
Equation (s)						
$X + Y \rightarrow Z$						
Reagent Table (Include all properties relevant to your procedure)						
Name	Formula / MW	State at Room Temp.	Density	Melting Point	Boiling Point	Hazards or Cautions
Procedure						
<p>This section must be written during the lab, as it includes information about what you <i>actually</i> did, not what you meant to do. Consequently, it must NOT be started or completed before the lab begins. Include exact times, temperatures, and quantities of material used. Make this section as detailed as needed to complete an accurate and thorough lab report.</p>						
Observations						
<p>Any noticeable changes, or lack of change, observed during the experiment.</p>						
Results / Conclusion						
<p>Actual lab results and a general conclusion about the experiment.</p>						

Reports - Reports for the first part of the course will be written up in a format similar to an article submitted to an American Chemical Society (ACS) journal. More specific instructions

for writing your lab reports, as well as examples of ACS journal articles, are given in an appendix at the end of the lab manual. In addition, the ACS puts out a Handbook for Authors that is also referenced at the end of the lab manual (Ref. A2). The final report should follow the same format as the formal experiments that are found in your lab manual.

4. Laboratory Safety

- § Approved eye protection must be worn at all times in the laboratory. Wisconsin state law requires all students to wear safety goggles, which seal around the eyes and have shielded vents, while they are in the laboratory. *This will be strictly enforced.*
- § Carefully follow all safety instructions given by the Instructor, Teaching Assistant, or Laboratory Technician/Stockroom Manager.
- § You are responsible for investigating the safety of any substance you handle, and must look up MSDS data for each before coming to class.
- § Know the location of fire extinguishers, eye wash fountains, and safety showers.
- § Shoes must cover the entire foot; sandals are *not* permitted.
- § No food, gum, beverages, or tobacco products are allowed in the laboratory.
- § Dispose of all chemicals as directed by the Instructor, Teaching Assistant, or Laboratory Technician/Stockroom Manager.
- § All experiments must be approved by your TA prior to initiation. Do not attempt *any* unauthorized experiments