

**CHEM 146A – Advanced Organic Chemistry Lab**  
**UCSC, Department of Chemistry and Biochemistry**

**Instructor:** Dr. Caitlin Binder  
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**Office Location:** Thimann Labs 313  
**Office hours:** M 11-12 PSB 240, W 2-3 PSB 145  
And by appointment (sign up on acrochem website)

**Teaching Assistants:** Grant Koch – gakoch@ucsc.edu  
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**Course Prerequisites:** CHEM 8L/M, 110L      **Course Fees:** \$55 materials fee  
**Lab Meetings:** TuTh 12:30 – 4:30 p.m.

### Required Materials

- Any Experimental Organic Chemistry textbook, such as...
  - Mohrig, J. R., *et. al* "Techniques in Organic Chemistry" Freeman (any edition)
  - Palleros, D. R. "Experimental Organic Chemistry" Wiley, 2000.
- Lab Notebook – no carbon copies needed, get a new notebook of any kind
- Goggles & Lab Coat (provided) or bring your own approved safety goggles (no glasses in Exp 4).
- Course website: [acrochem.sites.ucsc.edu/chem-146a](http://acrochem.sites.ucsc.edu/chem-146a)
  - Experiments, safety guidelines, cleaning responsibilities, lab notebook guidelines, lab report organization and writing style, rota-vap and TLC instructions, NMR & IR spectroscopy tables

### Required Assignments

There are two dry-lab and two wet-lab experiments with four written assignments to be completed using proper technical writing and specific notes given within each experiment. Structural elucidation through NMR spectroscopy is a key component of this course. Students will be given in-class time to gain a more complete understanding of this valuable tool, but this will also require time outside of class. Students are given opportunities to work on and revise reports during lab. Instructors will provide edits and feedback on drafts for students to incorporate into final reports. The minimum passing grade for each experiment is 70%.

### Laboratory Notebook

An up-to-date, current laboratory notebook should be kept while doing experiments. The notebook will be checked periodically and will be turned in with the reports. A table of contents should be kept up-to-date at all times. Students that do not have notebooks completed on experiment days cannot participate in lab and will receive zero points for that day's work. See full details in the 'keeping the lab notebook' on the 146A website and specific notes within each experiment.

### Absences & Lateness – Communication is Key!

If you absolutely need to be late for or miss a lab or two, *pre-arrange this with the instructor*. Students that are late or absent without notice will lose points on the corresponding reports. Experiments 1 - 3 **cannot** be postponed, however there is some flexibility in Experiment 4. This does not mean that you are free to miss labs on a casual basis: email the instructor the night or morning before at latest. **More than one non-prearranged absence can be grounds for a Fail (F) in this class. No make-up laboratory sessions can be arranged.**

### Illness. Casts and Crutches. Pregnancy

If you are ill, you **should not attend** lab and, as stated above, should contact the instructor as soon as possible. In the circumstance that you must wear a cast, a sling or use crutches, please contact the instructor immediately, before your next lab section, so we can arrange the best possible accommodations for you. Also contact the DRC (Disability Resource Center 831-459-2089) as soon as possible. If you are pregnant or intend to get pregnant during the course of this quarter, please contact the instructor before your next lab section to obtain important information about chemicals and pregnancy. Also, we recommend that you consult with your physician about this subject.

## Introduction to Chemistry 146A

Detailed guidelines and instructions are below and online to set you up for success in this course. Attention to detail and safety are emphasized above all else. Talk to instructors immediately if there is confusing or conflicting information. Please read the documents on the 8A website regarding safety and cleaning carefully before the first class meeting. Additional documents on lab notebooks, reports, equipment instructions, and spectroscopy tables should be consulted as needed.

We want to emphasize that a collegial and professional atmosphere is mandatory in these lab sections. The teaching assistant and instructor are here to help you. Our philosophy is that we are here to act as your research advisors and, hopefully, to convey to you some of our own passion for organic chemistry. **Your part of the deal is thinking, preparing and asking questions, no matter how small or silly you might think they are.** Collaboration and cooperation is encouraged. That means that you should feel free to consult with each other and with the TA about the chemistry that you are doing. It does not mean that the TA or another student does your lab work for you.

Students who do not come properly dressed for the lab will not be allowed to participate in lab. This includes the first day of class or check-in, which may result in you being dropped from the class. Students who do not follow the safety rules during the course of the lab will be asked to leave. This may result in failing the class.

Please remember that the amount of time available for laboratory work is limited. The laboratory is the place to make observations; use time outside the lab for planning and consulting the literature. It is essential that you have a plan of action when you arrive at the laboratory. This means that you must plan ahead for the experiments. You cannot afford to look information up as needed, unless the procedures specifically instruct you to do so.

The accurate and concise communication of research results and the ideas generated by these results is paramount in every branch of science. Maintenance of a neat, professional, and **permanent laboratory notebook** is an important objective of this course. Anyone should be able to read your writing. In addition, please remember that "bad results" are at least as important as "good results." All entries in your notebook should be made in ballpoint pen (so it will not "run" if wet). Your lab book must have bound, consecutively numbered pages.

Take good care of your bench space. Keep it clean and organized at all times. Keep the reagent counters, fume hoods, and the balances clean. If you spill a small amount of a solid when transferring or weighing it, brush it onto tare paper then dispose in the solid-waste container; wear gloves during this process. Keep reagent bottles and Erlenmeyer flasks well capped. Do not move reagents around the lab.

There are two main adjoining rooms for the bulk of student work with a third adjoining room with the reagents, waste, and IR. Be considerate of the fact that one TA is primarily supervising 16 students. The instructor (Caitlin) will either be across the hall in her office or in the lab. **Students should stay in the room with their assigned drawer, going into the third room only to get necessary materials then returning to the designated workspace.** *In other words, know what you need before you move to a different room!*

All products made or purified in the lab should be stored in a screw-cap vial or a plastic bag that is properly labeled with the experiment number, compound's name & structure, mass, your name and the date. The vials or bags are to be turned in immediately after completion of the experiment to the stockroom or your TA unless requested to do otherwise. Any compounds kept for the next lab period must be in a labeled vial in the desiccator in your assigned drawer.

Have fun, and learn as much as you can! Again, your TA and instructor are committed and approachable!

## Assignments

### NMR Activity – dry lab

Students work together to describe the different features of NMR spectroscopy. Structures of several organic compounds are given for the purpose of predicting the  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra for each using chemical shift tables and background knowledge of chemical equivalency and splitting patterns. The goal of this activity is to refamiliarize students with NMR spectroscopy in a forward sense (given structure, predict spectra) in preparation for structural elucidation in a backward sense (given spectra, draw structure). This will be turned in by the end of the second lab meeting (10/3).

### Experiment 1: Identification of a Single Unknown – dry lab

The structure of an unknown compound with no more than nine carbon atoms will be determined by IR spectroscopy, given chemicals test results, and provided  $^1\text{H}$ -NMR. Students work individually on this assignment.

### Experiment 2: Distillation & Column Chromatography – wet lab

The experimental purpose is to refamiliarize students with lab techniques & equipment while the learning objective is isolation and separate of an essential oil. Students carry out a steam distillation of spearmint, lavender, rosemary, or citrus oils. The oil is analyzed by GC then separated on a silica gel microcolumn. IR and TLC will be used to analyze the separated extract.

### Experiment 3: Separation & Identification of Two Unknowns – dry lab

Students will be assigned a theoretical mixture of two compounds (acid and base). Students will devise a detailed procedure for separation, after which spectra (IR,  $^1\text{H}$  and  $^{13}\text{C}$  NMR) will be given to determine the structure of the unknown. The lab report will consist of a record of the procedure (with a CHEM 8M student as the audience) as well as the fully assigned spectra with discussion of the structural analysis.

### Experiment 4: Synthesis of Ibuprofen – wet lab

Students will carry out the multi-step synthesis of ibuprofen using a literature procedure. This will be performed twice. Students will reflect on the first round of synthesis and implement concrete suggestions for improvements in the second round. IR and  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectroscopic analysis will be performed at several steps. Details on the lab report will be made clear in class.

## **Grading**

Punctuality, Preparedness, & Cleanliness: **100 points, 10%** - a little less than 6 points per day

NMR Activity: **150 points, 15%**

Each lab report includes a grade for safety, technical compliance (following instructions), and notebooks...

Experiment 1: **150 points, 15%**

Experiment 2: **150 points, 15%**

Experiment 3: **200 points, 20%**

Experiment 4: **250 points, 25%**

Note: **\*\*All drafts & reports must be printed and in-hand at the beginning of lab\*\*** All experiments and written assignments must be completed on time to pass the course. Please communicate with us if you're having a hard time completing an assignment on time. A minimum grade of 70% will be needed to pass each experiment.

A typical grade distribution is as follows:

A = 90-100%; B = 80-89%; C = 70-79%; D = 60-69%; F = < 50%.

## CHEM 146A – Advanced Organic Chemistry Lab Schedule

Arrive on time and dress appropriately for every lab meeting, even if no experiment will be performed that day. Bring your notebook to every lab. Reflect on the day's activity with a notebook / journal entry before leaving: challenges vs. success, suggestions for improvements, etc.

	Tuesday	Thursday
		9/26 – No student meeting
<b>Chemdraw &amp; SciFinder tutorials in McHenry Library Digital Scholarship Commons.</b> Register for passwords & find the DSC by 10/8 (instructions on Canvas)		
1	10/1 – <b>Orientation &amp; Safety</b> <b>** Students must be present this day to secure their space **</b>  <b>NMR Activity, Part 1</b> – bring laptop or tablet if you have one	10/3 – <b>NMR Activity, Part 2</b>
2	10/8 – <b>Experiment 1</b> (dry lab) <b>Group A</b> <b>Identification of an Unknown</b> *Pre-lab Q's due for both groups  <b>Group B – Meet at DSC</b>	10/10 – <b>Power Outage – no lab</b>
3	10/15 – <b>Experiments 1 &amp; 2</b> <b>Group B</b> <b>Identification of an Unknown</b> (dry lab) <b>Distillation &amp; Analysis of Essential Oil</b> (wet lab) <b>**BYO lavender, rosemary, or mint</b>  <b>Group A – Meet at DSC, come to lab after</b> <div style="text-align: right;">Exp 1 draft due</div>	10/17 – <b>Experiment 2</b> <b>Distillation &amp; Analysis of Essential Oil</b> <b>Group A</b> <b>**BYO lavender, rosemary, or mint</b>  All students report to lab  <div style="text-align: right;">Exp 1 Report Due</div>
4	10/22 – <b>Experiment 2</b> Draft Due Writing workshop – peer review	10/24 – <b>Experiment 3</b> (dry lab) <b>Separation &amp; Identification of Two Unknowns</b>
5	10/29 - <b>Experiment 3</b> (dry lab) <b>Separation &amp; Identification of Two Unknowns</b> <div style="text-align: right;">Exp 2 report due</div>	10/31 – <b>Exp 3</b> Draft Due, Writing Workshop <b>Experiment 4</b> Intro / Prep
6	11/5 – <b>Experiment 4 – Group A pairs</b> <b>Synthesis of Ibuprofen</b>	11/7 – <b>Experiment 4 - Group A pairs</b> <b>Synthesis of Ibuprofen</b>
7	11/12 – <b>Experiment 4 - Group B pairs</b> <b>Synthesis of Ibuprofen</b>	11/14 – <b>Experiment 4 - Group B pairs</b> <b>Synthesis of Ibuprofen</b>
8	11/19 – <b>Experiment 4</b>	11/21 – <b>Experiment 4</b>
9	11/26 – <b>Experiment 4</b> <b>Experiment 4</b> Draft Due	11/29 – Turkey Day – No Lab
10	12/3 – Lab Clean-up Writing workshop – peer review	12/5 – Writing workshop

\* Monday 12/9 – Lab reports due to Grant Koch's mailbox – 2<sup>nd</sup> floor of PSB between 230 & 240