

## Syllabus – Fall 2019

### Instructor

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Guion 223

In this laboratory course, you will gain experience with synthesis and purification of organic compounds, and their characterization using NMR and IR spectroscopy.

### Meeting Time & Location

Monday 1:10-4:20 pm, Guion 216

### Required Materials

Carbon copy lab notebook

Nitrile gloves

Safety goggles, Lab coat or apron

### Expectations & Grading Policy

There will be a total of seven experiments. Most experiments will have a lab notebook component and an accompanying lab report (the report is either a typewritten summary, a data sheet, or both, depending on the experiment). For the final experiment, you will prepare an oral presentation in lieu of a lab report. A lab technique grade will also be assigned at the end of the semester.

Lab notebook	50%
Lab reports	40%
Technique grade	10%

A grade of zero will be recorded if no lab report is submitted or if a lab report is more than ten days late. Please make every effort to turn in your lab report on time.

Letter grades will be based on the following scale:

A	93-100%	C	73-76%
A-	90-92%	C-	70-72%
B+	87-89%	D+	67-69%
B	83-86%	D	63-66%
B-	80-82%	D-	60-62%
C+	77-79%	F	59% or below

**Lab notebook:** Your lab notebook must be with you at all times in the laboratory. It's where you will keep a written record of raw data collected during the experiment. You will also refer to it to help guide you while setting up the experiment (pre-lab write up). Lab notebooks must be written in ink, not pencil. Please write **legibly**; if you make a

mistake, draw a single straight line through the mistake and write the correction nearby. If your writing is messy or difficult to read, I will deduct points from your lab notebook grade for that experiment.

The basic components required for each experiment in your lab notebook are as follows:

pre-lab

1. A one-to-two sentence introduction to the day's lab including the objectives or goals of the experiment.
2. The reaction scheme with the reactant(s) and product(s) structures drawn out correctly. You don't have to write a mechanism here, just the overall reaction equation. If no reaction is being performed (e.g. an extraction), just leave this section out.
3. A list of the reactants, reagents and solvents you will be using, in tabular format, containing at minimum the following information:
  - a. Chemical name (or formula if a salt, e.g. NaOH)
  - b. Amount of substance (from pre-lab, if given)
  - c. Molecular weight
  - d. Melting point (if a solid)
  - e. Boiling point (if a liquid)
  - f. Density (if an organic liquid)
  - g. Hazards or special safety precautions
4. A description of key observations that you make during the experiment. Any data that is collected should be recorded in this section as well (e.g. melting point, TLC). Do not copy the procedure from the lab text. Include a brief summary and any modifications made by you or suggested by the instructor. Common items that should be included in this section are listed in the table below.

Item	Example
time intervals	"the solution was cooled in an ice bath for 10 minutes"
exact amts. of reagents	the procedure states to weigh out 0.150 g of NBS but you weigh out 0.148 g
color changes	"as the reaction progressed, the color darkened to a deep violet"
temperature changes	"the temperature started at 30 °C but then increased to 50 °C within 5 min"
heat or gas evolution	"after adding bicarbonate the solution bubbled vigorously"
pH	"HCl was added until the pH reached 2 using pH paper (bright red)"
solubility	"sample A was really hard to dissolve, but B dissolved instantly"
precipitate formation	you see a solution begin to turn cloudy and notice tiny yellow particles
description of products	"white, needle-like crystals"
melting points	"mp 79-82 °C"
TLC data	write down the developing solvent used, and draw a diagram of the TLC plate

Items 1-3 make up the pre-lab. These must be completed at the beginning of the lab period and will be checked by the TA or lab instructor. Incomplete pre-labs will have points deducted from the lab notebook grade.

**Lab reports:**

On a separate piece of paper, write a conclusion that summarizes the results of the experiment. This should be **typewritten** using 12-point font and **double-spaced**; handwritten summaries will not be accepted. Use the data you collected to support your conclusion(s). Provide a citation for any external sources you use for data comparison (e.g. melting points, IR or NMR data). This should be a minimum of 120 words.

Below your paragraph text, draw the chemical structure of the product or compound you are analyzing, using chemical drawing software. ChemSketch from ACD Labs is a free software program available on the Internet.<sup>1</sup> Other software programs, such as ChemDoodle and ChemDraw, are not free but do provide a free trial period.<sup>2</sup> Alternatively, the two lab computers at the back of 216 have ChemDraw loaded onto them, and can be used for chemical structure drawing.

An example of a typewritten summary is provided in the Appendix.

Attach a copy of the accompanying data sheet, if any, along with printouts of any spectral (IR, NMR) data to your typewritten summary and turn in together. **Make sure the typewritten conclusion is on the front**, with your name, date, and title of the experiment clearly visible.

Lab reports will generally be due at the beginning of the lab period one week after the completion of the experiment. See the schedule on the next page for specific due dates. Late lab reports will be deducted 10% for each day late.

*A note on academic integrity:* Although you will be working in pairs for most experiments, you are still expected to write your own lab report each and every time. Willfully copying text verbatim from a lab partner is not acceptable and constitutes an honor code violation, for which referral to the Judicial Committee Academic Chair will be made.

**Technique Grade:**

Assigned by the instructor at the end of the semester, the technique grade will be based on an assessment of 1) how well prepared you were for each lab experiment, 2) whether or not you followed instructions, including proper safety protocol, 3) your proficiency in setting up glassware, handling reagents, transferring chemicals, and using equipment and instrumentation, and 4) keeping your lab bench clean and glassware & equipment clean and put away.

*Make-up labs:* Due to the size of the labs, you should make every effort to come to your scheduled lab time. If you foresee missing a lab for an athletic event, make arrangements with the instructor ahead of time. If this is not possible because of schedule conflicts,

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<sup>1</sup> <http://www.acdlabs.com/resources/freeware/chemsketch/>

<sup>2</sup> Trial period is typically two weeks. At the time of this printing, you can purchase a six-month subscription to ChemDoodle for \$29.00

then it's up to you to fit the missed lab into next week's lab that you are performing. That means you will be doing two labs at the same time! If you don't make up the lab within one week, then a zero will be assigned for that lab. We will not be able to keep the chemicals out for extended periods of time.

*Safety:* You must read and agree to abide by **all** chemical safety orientation guidelines. Any student who willfully disregards safety protocol will be asked to leave the lab and will receive a grade of zero for that day's experiment.

Get into a habit of gearing up with personal protective equipment (PPE) before you begin each experiment. PPE includes goggles, gloves, lab apron, and proper attire: closed toe shoes (no sandals allowed), long hair tied back.

### Lab Schedule

Week	Date	Event
1	Sept 16	Safety, check-in Experiment 1 Introduction to NMR Spectroscopy
2	Sept 23	Experiment 2 Purification and NMR Analysis of Aspirin
3	Sept 30	Experiment 3 IR Spectroscopy
4	Oct 7	Experiment 4 Synthesis of Isopentyl Acetate
5	Oct 14	Exp 4 cont.
6	Oct 21	Experiment 5 Multistep Synthesis
7	Oct 28	Exp 5 cont.
8	Nov 4	Experiment 6 Extraction of Eugenol from Cloves
9	Nov 11	Experiment 7 Diels-Alder Reaction
10	Nov 18	Experiment 8 Friedel-Crafts Acylation
11	Nov 25	THANKSGIVING BREAK
12	Dec 2	Exp 8 cont.
13	Dec 9	Lab cleanup, check-out