

Organic Chemistry II Lab (Chem 29B) Policies for Spring 2020

Instructor: Dr. Kawagoe

Voice Mail: 638-0300 (ext 3230)

Office: ANX-5

E-mail: Use Canvas

Text:	Required Pavia, Kriz, Lampman, Engle A Microscale Approach to Organic Laboratory Techniques, 6 th Edition ISBN: 1305968344
Software	Required Marvin Sketch (or other structure drawing program) This software is free and available for the PC and Mac
Website	Course documents can be found on Canvas
Ancillary:	Strongly Recommended (available online) Paul R. Young Practical Spectroscopy: The Rapid Interpretation of Spectral Data ISBN: 0534372309
Model Kit:	Any approved model kit (Strongly Recommended)
Calculator:	Basic scientific calculator
Class:	Friday 9 AM – 11:50
Lab Final:	The Lab will be held during the Friday <i>prior</i> to finals week, During finals week, we evaluate and organize the lab lockers and equipment.

Dr. Kawagoe's Office Hours Schedule

Office Hours PHY77

M 11:00 AM – 12:30 PM; Tu 11 AM – 12:30 PM; Friday 12 PM – 1 PM

Office hours are screencast live via Zoom (online)

The Zoom links will be posted on Canvas.

You should read your academic course catalog for policies and rules regarding **Student Conduct, Academic Dishonesty, Attendance, and Withdrawing from College** (pages 54-59) of the 2017-2018 FCC Catalog. You are responsible for knowing all that is discussed there.

Attendance Policies

I am required to drop students who do not attend the first day of class. If during the first two weeks of class you miss two consecutive labs, you will be dropped from the course. **All labs must** be completed during the course of the semester. No makeup lab work is allowed unless prior arrangements are made with the instructor. If you miss a lab for a valid, verifiable reason, you will be allowed to makeup the lab or submit other work in the place of the lab. You can be dropped for missing two consecutive weeks.

Academic Dishonesty

Cheating will not be tolerated. You will receive an "F" for the course and be reported to the VP of Students.

Academic Accommodation

"Students with disabilities who qualify for academic accommodations must provide a letter from the DSP&S and discuss specific needs with the professor, preferably during the first two weeks of class. DSP&S determines accommodations based on appropriate documentation of disabilities."

Grading

The points are distributed approximately as follows:

Formal Report	10%
Reports, questions, assignments	55%
Technique & Notebook	20%
Lab Practical	15%
Total	100%

Assignments are reduced by one grade value (10%) per day. Reports are due the week following completion of the experiment at Sunday at 11:59 PM. Assignments are submitted through Canvas (links will be posted).

All reports will be checked for plagiarism via Turn-it-in and must be original works. This includes both the report and answers to questions assigned for each experiment.

Grading Scale (subject to change by instructor)

A=90.0% and up, B=80.0-89.9%, C=70.0-79.9%, D=60.0-69.9%, F=59.9% and below

A simple grading system is employed. In general, the reports are "cleaned up" version of your lab notebook.

- Abstract & main reaction
- Results
- Analysis of spectra/data
- Discussion & Summary
- Answers to text questions
- Brevity and clarity are important aspect of report grades!

Safety & Technique: We are all responsible for safety in an organic chemistry lab. You can be badly hurt or killed if an accident occurs in an organic chemistry lab. During the first 10-15 minutes of each class, I will go over safety information and discuss any new procedures. If you perform what I consider to be an unsafe procedure because of lack of preparation, you will lose 1-10% off your **class grade** per incident. If the severity or repetition of the incident brings concern for the safety of you or other students, you will be dismissed/dropped from the class. Past the drop deadline, you will be assigned a failing grade.

Lab Rules: You are required to have read and outlined each experiment, including the safety information before you enter the lab (see notebook section below). You will receive instructions on how to safely perform the experiment or new procedures. You are very likely a safety hazard to others if you miss the pre-lab discussion on safety and safe lab procedures.

Missing or "messing up" a lab: The grade for a missed lab is a 0 (zero). If you miss or "mess up" a lab, you must make arrangements with me to make up the lab. All experiments must be completed successfully to receive a passing grade in the class.

Course Description: Continuation of Chemistry 29A and the laboratory for Chemistry 28B. Application of theories and mechanisms of organic reactions. Methods of synthesis, isolation, purification, and identification of organic compounds. Principles and hands on application of FT-NMR, FTIR and gas and solid phase chromatography. Principles and interpretation of GC-MS data.

Course objectives:

1. Compile, critically evaluate, and interpret scientific information and data.
2. Effectively communicate scientific information through written and oral means.
3. Purify organic reaction products using filtration, drying techniques, extraction, chromatographic separation, crystallization, and distillation.
4. Identify organic reaction products by measuring physical properties (melting point, solubility, boiling point, density), chromatographic retention, optical properties (index of refraction, optical rotation), optical spectroscopy (UV-VIS, IR, NMR), and mass spectroscopy.

Student Learning Outcomes:

1. Demonstrate the ability to prepare for and carry out experimental protocols using modern instrumentation and methods.
2. Utilize critical thinking skills to apply concept knowledge and adapt experimental techniques to: a) form and test hypotheses and b) solve scientific problems.
3. Compile, critically evaluate, and interpret scientific information and data.
4. Effectively communicate scientific information through written and oral means.

Lab Assignments:

Regular lab report format (see expanded descriptions below)

Abstract

Main reaction(s): not mechanism

Results section: Data *tables*, calculations, analyzed spectra

Discussion: Discuss the significance or errors of your results. May be combined with the Results section.

Post-lab questions: Assume all questions are assigned.

All data preliminary calculations, analyzed spectra, and questions and a summary should appear in your lab notebook. They should be hand written entries done during the time of the lab.

There are two **formal lab reports** this semester (see schedule). I will supply a template from the journal Tetrahedron Letters for you to follow.

Formal lab report format

Abstract

Same as regular reports

Main reaction(s)

Same as regular reports

Introduction:

Discusses the significance and characteristics of the particular reaction studied and also what was attempted in the current experiment. It may give examples of similar types of reactions and the expected outcomes. This information should be gathered from the chemical literature.

Experimental:

Gives a detailed accounting of the procedure and materials used. I prefer that quantities be done as millimoles (mmol) and concentrations as molarities or percent. Temperatures used, colors, and appearance, etc. are also given.

Results and Discussion:

A detailed narrative of the results. Describes the yields obtained from the experiment and analysis results. Generally, each results is discussed as it is presented and fit into the overall picture of what was accomplished during the experiment.

Post lab questions:

Same as regular reports. These are not part of the formal report, but they will be handed in attached to the formal report. Generally it helps to do these before you write the formal report because they will give you some insight on what to put in the formal report.

Notebooks: The following summarizes how notebooks should be treated. I am not particularly following any rules or regulations (i.e. GMP, GLP, or ISO), but drawing from my experience doing research in graduate school, various universities, industry, and government labs. There is some variation from lab to lab (but not much). I will hold your notebooks to this standard. **I will grade your lab notebooks at the end of the semester.** These are turned in at the end of the semester and I will keep these in my possession for five years. If you transfer and need your notebook, I will send it forward for you or give it to you at your request. **Here's the actual form I use for grading your notebook:**

Items Checked	Lab Evaluated	
Exp. Listed in Toc w/ correct page numbers		
Purpose		
Main reactions (don't need mechanism)		
Safety information		
Physical constants of reactants, products, reagents, etc.		
Procedure outlined		
Data tables (or spaces in procedure)		
Observations/data		
Preliminary results		
Spectra Analyzed		
Summary		
Questions (bonus point)		

Points	percent
12	55
13	59
14	64
15	68
16	73
17	77
18	82
19	86
20	91
21	95
22	100

The following may be typed:

- ❖ Purpose
- ❖ Main Reaction
- ❖ Safety Data & Physical Constants
- ❖ Procedure
- ❖ Data tables (w/o data)
- ❖ Questions

Typing other information lowers your notebook grade by 50%.

I will randomly select 2 experiments to assign your notebook grade.

Note: spectra must be inserted into your lab notebook shortly after collecting the spectra to receive credit. If you accidentally collect a bad spectrum, simply add that to your notebook on a later page. Make note of the location of the improved spectra on the original spectra.

I will be looking for your and my signature/stamp when I grade your notebook. I will note when I approve your prelab and you need to sign and date the pages of your notebook as you complete them.

Notebooks (continued)

1. Use a non-erasing pen (if you are hand writing)
2. You must use a **bound notebook (comp notebook) and pages must be numbered** (by hand is acceptable)
3. **Page numbering:** Start numbering the pages after the table of contents pages.
4. **Table of contents:** Use the first few pages for a table of contents. An acceptable format is given below.

		Your name goes here Chemistry 29B If found, please return to FCC Chemistry department	
Table of Contents			
Experiment number (Pavia, 6 th)	Notebook Pages	Date Performed	Write up
4 Extraction	1-4	8/17, 22	5, 6

5. **Prior** to coming to the lab, the following must be in your notebook (see sections below for details):
 - a. Table of content entry (for start of lab)
 - b. Purpose & Main reaction
 - c. **Significant chemical hazards (see next page)**
 - d. Physical constants of reactants and products (any organic compound in the main reaction)
 - e. Procedure w/ room for observations and data (i.e. data tables or spaces)

Note: I will look for my signature or stickers when I grade your notebook.

You can **type** any of this information that you want, but handwritten procedures are the classroom standard. **All data, calculations, spectral analyses, summaries, and question answers must be written by hand.**

6. **Procedure:** You are expected to **summarize** your procedure before starting. You may copy textbook diagrams that you think you might need. You should be able to complete the procedure from only your notebook. Tip: Write the procedure as a numbered list. If you have any interesting observations, cross reference the observation by number.
7. **Data and observations:** Record all observations, data, and calculations in your notebook.
8. **Cite any reference work: Other than your text,** cite your sources (i.e. Melting Point 103.5°C, CRC, 75th edition, pg 105). Since our sources are generally always the same, you can reserve an inside cover or pages in the back for a bibliography (ref 1. page 105; or r1 pg105).
9. Never remove or erase anything written or attached to your notebook. Put a line through it or cross out the section you want disregarded.

Safety Information and Physical Constants

The following safety information is required:

Name of substance and CAS identifier

Hazard ratings – I like the JTBaker MSDS SAF-T-Data ratings (or “NFPA 704” are also good)

Precautions/Protective equipment (i.e. what is hazardous, how do you handle)

You can Google “JTBaker SDS bromobenzene” and see some examples. If all the hazard safety ratings are “1” you do not have to reference that material. If a majority of the ratings are “2” it should be included. Ratings of “3” or “4” must be included. Note you may also try searching MSDS instead of SDS.

Physical constants used depend on what you are doing in the experiment. Typical information includes: melting point, boiling point, density of liquid/solid, color, crystal appearance. It is often easiest to set up the safety and physical constant information in the same table: Here’s an example

Substance CAS#	Hazard	Precautions	Physical Constants
Acetone # 67-64-1	Flam = 4, React = 2	vent/hood	Clear, colorless, volatile liquid, d = 0.79 g/mL BP = 56.6°C
Methylene Chloride	Health = 3 (cancer), Contact = 3	vent/hood	Clear, colorless liquid, d = 1.318 g/mL BP = 39.8°C
AlCl ₃ , anhydrous # 7446-70-0	Ratings = 1/0	Avoid moisture (from section 10 of MSDS).	May release toxic fumes when heated to decomposition.

If the substance was already referenced earlier, you do not need to write it all down again. Reference it from the previous use (page number).

Notebook checklist (or how to get 100% on your lab notebook)

Before Lab (* can be typed)

- _ Exp. Listed in Toc w/ correct page numbers
- _ Purpose*
- _ Main reactions (don't need mechanism)*
- _ Safety information*
- _ Physical constants of reactants, products, reagents, etc.*
- _ Procedure outlined*
- _ Data tables (or spaces in procedure) (without the data)*
- _ Dr. k's signature

After lab (The following must be hand written)

- _ Observations/data
- _ Preliminary results
- _ Questions
- _ Spectra Analyzed
- _ Summary
- _ Your signature and date (on each page and at the end of the experiment).

* To put your typed information into your notebook

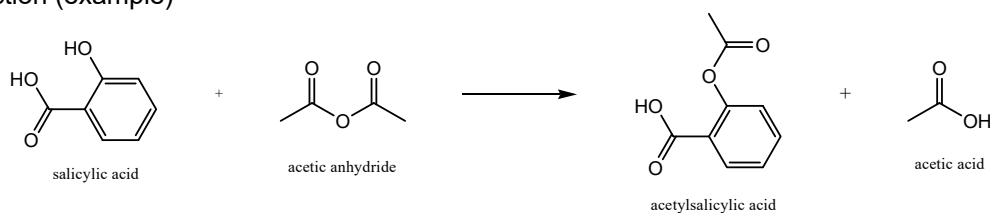
1. Cross out the section under the pasted insert.
2. Use tape to neatly tape the information onto the page.
3. Sign (initials are usually okay) and date across the tape so that the tape cannot be removed without tearing the signature.

The Report Abstract:

- ❖ Abstract (One short paragraph. See examples below for what I consider “short”) In writing the abstract, it helps answering these questions:
 - What did you do?
 - What did you make and how did you make it?
 - What type of reaction and what conditions?
 - What separation technique was performed?
 - What measurement techniques were used?
- ❖ Abstract example for a synthesis/reaction lab – Note that most synthetic labs have very similar abstract formats.

Acetylsalicylic acid (**What was made**) was prepared by acylation (**Type of reaction**) of salicylic acid with acetic anhydride. (**Give the main reaction at the end or beginning of the abstract**). Starting with 0.0513 g of salicylic acid, 0.0421 g of acetylsalicylic acid were produced for a 62.92% yield (**Percent yield – use stoichiometry to determine theoretical yield**). Tests for phenol were slightly positive (**Test Results**) indicating the presence of starting material. Crystals were white and needle like, but rather sticky. (**Physical appearance**) The melting point range was 131.2-134.4°C also indicating impurities (**physical constant comparison**).

Main Reaction (example)



- ❖ Abstract example for a technique lab
An acid compound was separated from a mixture by extraction, purified, and identified by melting point. (**What was done**) Starting with 0.1097 g of mixture, 0.0735 g of crude acid sample was collected (crude recovery: 67.00%). (**Note that the term “recovery” is used rather than percent yield since you are only purifying a substance**) Following recrystallization, the percent recovery was 46.82%. The melting point was 134-135°C indicating that it was possibly acetylsalicylic acid. This was verified by the mixture-melting point method. IR spectroscopy are also consistent with phenol.

The Report Data & Results:

- ❖ Data, Results, and Discussion (**Results and Discussion Section**)
For clarification, **data** is actually what you measured. A **result** is anything you can calculate or deduce from your data. Data should be presented in table format (sometimes alongside the results). Here are some examples
 - Mass/volume of product (Data).
 - % yield (Results) (show calculations)
 - Summarize your results and compare to the literature values for the same substance. (Discussion)
 - Melting Point (Data)
 - Results of Chemical Tests (Data)
 - Spectra (data)
 - Identification of peaks in spectra (results)
 - Meaning of peaks in spectra (Discussion. Doesn't belong here.)
 - GC-MS Chromatograms Peak Areas (data)
 - GC-MS Percent Composition (results)

Lab Schedule for Spring 2020 Chemistry 29B

date	Friday
Week 1 Jan 13-17	NMR – hands on with the instrument NMR spectra of some common functional groups.
Week 2 Jan 20-24	Exp 35B Benzoic Acid (Grignard Reaction)
Week 3 Jan 27-31	Exp 43B Preparation of 1,4-diphenyl-1,3-butadiene using EtO^-Na^+
Week 4 Feb 3-7	NMR – Structure determination of unknowns using NMR.
Week 5 Feb 10-14	Friday: Lincoln Holiday – No Lab, but I will be around all week for any makeup work.
Week 6 Feb 17-21	Exp 69 – Esterification Reactions of Vanillin: The Use of NMR to Solve a Structure Proof Problem.
Week 7 Feb 24-Feb 28	Exp 39 – The Aldol Condensation Reaction: Preparation of Benzalacetophenones (Chalcones). Your product for this reaction will be your reactant for exp 41.
Week 8 Mar 2-6	Exp 41 – Preparation of an α,β -Unsaturated Ketone via Michael and Aldol Condensation Reactions.
Week 9 Mar 9-13	Exp 50 - N,N-Diethyl-m-toluamide: The Insect Repellent “OFF”
Week 10 Mar 16-20	Exp 44 - Relative Reactivities of Several Aromatic Compounds (new this year)
Week 11 Mar 23-27	Oxidation of Cyclohexanol with Bleach (handout). Your product for this reaction will be your reactant for week 12.
Week 12 Mar 30-Apr 3	Acetyl Cyclohexanone (Handout)
Apr 6-10	Spring Break
Week 13 Apr 13-17	Acetyl Cyclohexanone: isolation and analysis. We may also do Exp 29
Week 14 Apr 20-24	Exp 60 – Friedel-Crafts Acylation
Week 15 Apr 27-May 1	Exp 60-Continued
Week 16 May 4-8	Exp 56 – Luminol or Tie Dye (preparation of azo dyes, handout) Possibly we will try Exp 47 for the tie dye (synthesis of indigo)
Week 17 May 11-15	Lab Practical
Week 18	Lab Cleanup