

Text: Introduction to Organic Laboratory Techniques, 6<sup>th</sup> edition  
Pavia, Lampman, Kriz, Engel

Calculator: A basic scientific calculator is all that is needed for this course.

Lab Notebook: Bound Comp notebook & Ballpoint Pen

Lab Supplies: Approved Safety Goggles (If you have questions ask)  
Dishwater soap

#### Dr Kawagoe's Teaching Schedule and Office Hours

Class	Days	Hours	Room
CHEM-1B	MW	9:30AM-10:45AM	MSCI-204
CHEM-1B	MW	11:00AM-1:50PM	MSCI-202
CHEM-28B	TTH	9:30AM-10:45AM	MSCI-204
CHEM-29B	TTH	11:00AM-1:50PM	MSCI-202
Office Hours	MW	2:00PM-3:00PM	MSCI-222
Office Hours	F	12:00PM-1:00PM	MSCI-202

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### Safety

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. Just about everything we use is either flammable, toxic, carcinogenic, teratogenic, and potentially deathogenic (my own word). We use most chemicals in labs because they dissolve or react with organic substances (including you). If you perform what I consider to be an unsafe procedure or cause another student to perform an unsafe procedure because of lack of preparation or irresponsible behavior, **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 dropped from the class. **Because this course is a co-requisite for Chemistry 28A, you may be dropped from both courses**. Past the drop deadline, you will be assigned a failing grade.

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### Disabled Students Programs Services

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.

### Prerequisites and Corequisites

Prerequisite: Second semester General Chemistry (Chem 1B) with a grade of C or better.

Corequisite: First semester Organic Chemistry (Chem 28A). You may have also completed Chem 28A in a prior semester.

### Course Description (Generally what is covered by the class)

Laboratory for Chemistry 28A; Isolation, purification, and identification of organic compounds. Product characterization; melting point and boiling point; index of refraction; chromatography. Principles and hands on application of FT-NMR, FTIR and gas and solid phase chromatography. Principles and interpretation of GC-MS data. Application of theories and mechanisms of organic reactions. Methods of synthesis.

## Class Policies

You should read and understand pages 47-48, RC 2022-23 Online catalog. These pages outline the college policy and rules regarding Student Conduct, Academic Dishonesty, Attendance, and Withdrawing from College. 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 lectures, you will be dropped from the course. Following the first two weeks, if you accumulate four unexcused absences, you will be dropped or your grade will be lowered by one letter grade.

## Academic Dishonesty

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

## Grading

Lab work, reports and results	75%
Notebook	15%
Lab Practical	10% (see additional provisions in <b>Safety</b> and <b>Technique</b> sections)

*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

## Proper Lab Attire

Since you are required to wear a lab coat, any clothing is allowed as long as it does not cause safety concerns. You must wear **close topped shoes**. **I strongly recommend that you wear clothing that covers your legs during lab** since a lab coat will not protect your legs. You should consider leaving a spare set of clothing in your car (for those days you forget or those days you have to change clothes after lab). Several well regarded institutions find that contact lenses are permissible in the chemistry lab (American Chemical Society and Centers for Disease Control) as long as you are wearing goggles. Eye glasses or safety glasses are not acceptable eye protection for organic chemistry. Everyone must wear goggles in the lab. Long hair must be tied back to keep it out of chemicals.

## Notebooks (See your lab text for more information: Pavia, Technique 2 pg 609)

The following summarizes how notebooks should be formatted and used. 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. Many of you are only a couple of years from the point at which you will be held to these standards, so my goal is to help prepare you.

1. Pages must be bound.
2. Write everything in unerasable ink
3. Use the first few pages for a table of contents. An acceptable format is given below. The last three columns can't be filled out until you actually do the lab or write it up.

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

4. **Number all the pages in the entire notebook by the beginning of the first experiment.** Start numbering the pages after the table of contents pages.
  - I usually only write on one side of the page, so I only number those pages.
  - Government labs and industrial labs either use computers or buy books with prenumbered pages
5. As you use the pages, put the date in the top right-hand corner of the page.
6. You are expected to summarize your procedure before starting.
  - Many laboratories require prior approval (signatures from other lab members) for new procedures.
  - In most labs, you can be fired for carrying out unauthorized procedures.
7. Record all observations, data, and calculations in your notebook.
8. Cite any reference work you use.
  - If you look up data cite the source (i.e. Melting Point 103.5°C, CRC, 75<sup>th</sup> 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.

**I may collect your notebooks and grade them at any point in the semester.** A progress grade will be placed in your table of contents. If I see white out, erasable pen, pencil, undated pages, etc., I will notify you so that you can fix these problems (i.e. I'll give you some warning before I deduct points).

- ❖ At the end of each experiment, you will submit your sample with the label indicated on page 565 of your lab text. If you do not submit a sample, you may not receive credit for the lab. Do not write directly on the sample vial! Most pen ink will not adhere to the glass surface or may become unreadable if the product is spilled on the glass (organic products tend to dissolve ink).

### Notebook checklist (see page 563 of your lab text), 1 point each.

- ❖ Record lab title and page number in index
- ❖ Write purpose of lab
- ❖ Write main reaction(s) and reaction conditions (see Pavia pg 610)
  - If a synthesis requires two steps. Write two reactions.
  - On the reaction arrow, indicate reaction conditions (i.e. H<sup>+</sup>, Δ)
  - Don't worry about the purification steps in the main reaction.
- ❖ Physical constants/data for products, reactants, and reagents (see Pavia pg 610)
  - You need this data for purposes of comparison to your results. Reagents are not usually necessary to know since you will not accidentally isolate these as your products (i.e. catalysts). However, when a reaction does not proceed according to plan, you often isolate one of your reactant or a solvent in addition to your product.
  - Examples of physical constants/data that you should find
    - Melting point

- Boiling point
- Density
- Solubility data
- Molecular weight
- Appearance (i.e. yellow-white leaf like crystals, amber liquid, etc.)
- Some sources for constants/data (All can be found in the FCC library or lab except as noted)
  - Merck Index (located in the lab)
  - CRC Handbook of Chemistry and Physics (located in the lab)
  - Sigma/Aldrich catalog (located in the lab)
- ❖ Safety Information (see pages 542-558 in your text)
  - The most common source of safety information can be found in SDSs (Safety Data Sheets). You can quickly locate SDS information for different compounds by performing a Google search with the name of the substance and SDS (i.e. isopentyl acetate +SDS). Note that MSDS is an older term that might also give you the necessary information.
  - See Technique 10 (pg 687) of your text for some useful information on solvents.
  - You may find it useful to record specific safety information in the back of your notebook for future reference.
- ❖ Summarized procedure
  - Flow charts are very useful for both the procedure and isolation steps.
    - See page 611 of your text for a good example, but lacks specific information that could be used to do a synthesis.
- ❖ Space for data and observations.
  - You should prepare data tables in advance. This helps to make sure that you record all the necessary information.
- ❖ Space for calculations and answering questions.

### Lab Reports

- Reports are to be typed.
- The report grade depends on **quality**, not quantity. For unnecessarily long reports, I will stop reading and assign a grade based on what I have read. I will also subtract 10% for being too lengthy.
- Final reports will be **typed** and turned in on Fridays by Midnight (see checklist).

### Report checklist

- ❖ Abstract (One or two short paragraphs depending on the nature of the lab.)  
It helps to answer these questions in the abstract
  - What did you do? (one or two sentences)
  - What were your results?
    - Yield and % yield
    - Results from spectroscopy
    - Boiling point
    - Melting point
- ❖ Main reaction
  - Often this is one of the lab questions. Draw the main reaction here and make reference to it in the questions section.
- ❖ Calculations and Table of Results  
(Put example calculations right before table)
  - Percent yield
  - Physical measurements (Refractive index, optical rotation, melting point, boiling point, etc.)
- ❖ Graphs and figures
  - Chromatography data (i.e. GC, TLC)
  - Spectral analysis (FTIR, NMR)
    - Draw the compound on the spectra.
    - Identify major peaks and show where that peak is derived from on the drawing of the compound.
- ❖ Answers to lab textbook questions
  - Draft responses to the questions in the “report section” of each lab will be handwritten in the lab notebook. You should do these before leaving the lab. Work with your classmates. I will clarify questions if asked.

Lab Schedule.

Week start date	Experiment	
Week 1 1/9/23	Lab Check in, work on spectroscopy	
Week 2 1/16/23	Experiment 33: An Oxidation-Reduction Scheme: Borneol, Camphor, Isoborneol.	Clean glassware and dry glassware for Exp 35B
Week 3 1/23/23	Experiment 35B: Benzoic Acid	
Week 4 1/30/23	Experiment 36: Aqueous-based Organozinc Reactions (tentatively scheduled).	
Week 5 2/6/23	Experiment 60: Friedel-Crafts Acylation.	
Week 6 2/13/23	Experiment 43B: Preparation of 1,4-diphenyl-1,3-butadiene Using Sodium Ethoxide To generate the Ylide.	
Week 7 2/20/23	Experiment 69: Esterification Reactions of Vanillin: The Use of NMR to Solve a Structure Proof Problem.	
Week 8 2/27/23	Experiment 49: Benzocaine.	
Week 9 3/6/23	Experiment 39: The Aldol Condensation Reaction: Preparation of Benzalacetophenones (Chalcones).	Double reactant p-anisaldehyde. Keep product.
Week 10 3/13/23	Experiment 41: Preparation of an $\alpha,\beta$ -Unsaturated Ketone via Michael and Aldol Condensation Reactions.	Use product from 39 as reactant. Type report on combined exp 39 and 41.
Week 11 3/20/23	Exp 26: Preparation of Soap Exp 61: Analysis of Antihistamine Drugs by GC MS	Two labs this week.
Week 12 3/27/23	Enamine Reactions: 2-Acetylcyclohexanone.	
Spring Recess 4/3/23	Spring Break	
4/10/23 Week 13	Experiment 50: N,N-Diethyl-m-toluamide: The Insect Repellent "OFF.", Experiment 48: Sulfa Drugs: Preparation of Sulfanilamide.	
Week 14 4/17/23	Experiment 58: Preparation of a C-4 or C-5 Acetate Ester.	If we get the resources for testing the sulfa drugs, we will skip experiment 58
Week 15 4/24/23	Experiment 64: The Aldehyde Enigma.	
Week 16 5/1/23	Tie-dye	Bring some fabric if you want something to dye.
Week 17 5/8/23		
Week 18 5/15/23	Finals	



The following information is provided to you in the case you need to show a transfer institution the content covered in your first semester organic chemistry laboratory course (Chem-29A). It contains portions of the official course outline of record (COR).

#### Course Description:

Chemistry 29B is the second of two laboratory courses in organic chemistry, and as such it is primarily concerned with introducing intermediate level techniques used in organic chemistry. Although many of the familiar, introductory techniques from 29A will be used in 29B also, additional methods of analysis such as NMR spectroscopy, mass spectrometry, and computational methods will be utilized. In CHEM 29B students will carry out multi-step syntheses, and additional emphasis will be placed on problem solving, application of theory, and structural identification. PREREQUISITE: Chemistry 29A. COREQUISITES: Chemistry 28B. ADVISORIES: English 1A or 1AH. (A, CSU-GE, UC, I)

#### Learning Outcomes:

- Synthesize organic compounds and subsequently purify/isolate these compounds using appropriate techniques such as recrystallization, liquid-liquid extraction, distillation, and chromatography
- Identify and safely use organic chemistry glassware (including that containing ground glass joints)
- Analyze organic compounds and measure their physical properties using a variety of techniques including melting point, solubility, boiling point, density, chromatography (solid and gas phase), optical properties (index of refraction, optical rotation), op
- Interpret data and draw meaningful conclusions about experimental work; clearly communicate findings both orally and in writing

#### Course Outline:

A. Safety: Safety policies, waste disposal, MSDS, glassware

B. Synthesis (including multi-step synthesis):

Reaction methods and mechanisms

C. Isolation, Purification and Characterization:

Extractions, crystallization methods, distillation (simple, fractional, steam), thin-layer chromatography, column chromatography, melting point, boiling point, gas chromatography, polarimetry, refractometry, infrared spectroscopy, nuclear magnetic resonance spectroscopy (hydrogen-1 & carbon-13)

D. Spectral Analysis:

Infrared Spectroscopy (IR), Nuclear Magnetic Resonance (NMR) – Carbon-13, Hydrogen-1, Mass spectrometry (MS)

E. Example Experiments (\*multistep syntheses):

1. The Diels-Alder Reaction of Cyclopentadiene with Maleic Anhydride
2. Friedel-Crafts Acylation
3. Preparation of 1,4-diphenyl-1,3-butadiene Using Sodium Ethoxide to generate the Ylide
4. Methyl Salicylate (Oil of Wintergreen)
5. Esterification Reactions of Vanillin: The Use of NMR to Solve a Structure Proof Problem
6. Preparation of Benzocaine
7. \*Preparation of an  $\alpha,\beta$ -Unsaturated Ketone via Michael and Aldol Condensation Reactions
8. Preparation of a Soap
9. Enamine Reactions: 2-Acetylcyclohexanone
10. \*The Aldol Condensation Reaction: Preparation of Benzalacetophenones (Chalcones)
11. \*N,N-Diethyl-m-toluamide synthesis
12. \*Sulfa Drugs: Preparation of Sulfanilamide
13. Preparation of a C-4 or C-5 Acetate Ester
14. The Aldehyde Enigma (reaction and identification of an unknown aldehyde and its products)
15. \*An Oxidation-Reduction Scheme: Borneol, Camphor, Isoborneol