Chemistry 36B Spring 2003

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Office Hours by Appointment

M/W, 36B.1 1:25 - 4:25 pm 216 Whitmore Lab
TA: Andrea Cerrone

T/Th, 36.2 1:25 - 4:25 pm 216 Whitmore Lab
TA: Mandy McElwain

Staff Assistant: Karen Heichel 863-3261
Stockroom Director: Jeff Brooks 865-7483
Required Materials


Chem 35/36 Laboratory notebook (8.5" x 11" ruled sheets with tear out pages, Hayden McNeil)

Eye protection. See options in Ch 2 of Lab Guide

Org. Lab Equipment Kit including NMR tubes, TLC plates, vials, pipet bulbs, etc. Need for 1st lab!

Combination lock, Lab apron (recommended), Gloves are supplied
Required Materials

Course CD

You will be given a course CD that contains additional information not found in the Lab Guide and any modifications to the material found in the Lab Guide.

We will follow the course syllabus found on the CD!!! We will not follow the schedule on the front cover of your Lab Guide.
Always use eye protection while inside a chemistry laboratory.

Use gloves at critical times, wash hands often.

Reactions must be run in the hoods. (TA will tell you when you can work on the bench)

Your TA must be present when you are in the lab,

NO ONE WORKS UNSUPERVISED
Lab Safety Rules

• If you wear shorts, you must have a lab apron.

• **No** open-toed shoes of any kind.

• **No** open flames in the lab unless directed otherwise.

• Report accidents immediately.
Handling of Chemical Waste

Chapter 2, Lab Guide

"The entire procedure of waste disposal starts with the laboratory worker....

Prudent Practices for Disposal of Chemicals from Laboratories
Handling of Chemical Waste

• Down the Drain (D)
• Nonhalogenated Organics (NHO)
• Halogenated Organics (HO)
• Heavy or Hazardous Metals (HM)
• Waste Bin (WB)
• Sharps
• Glass Bins
• Recycle/Reuse
Courteży w laboratorium

- Powrót wszystkich butelek z reagentami i roztworami do właściwego miejsca na półce lub naczyniu, natychmiast po użyciu!
- Utrzymuj czystość swojego obszaru.
- Wybierz i zdecyduj się na czas.
- Wyczyść rozlewy!
- Zamknij pokrywy; zlej puste butelki i odśwież nadziewanie.
- Utrzymuj czystość pomieszczenia instrumentów i pojemników.
- Unikaj awarii.
- Utrzymuj czystość wagi!
- Odprowadź chemikalia w odpowiednich pojemnikach.
Course Goals

• To build a bridge between chemical and biological processes and understand the reactions that govern them.

• To understand what organic chemists really do and bring “paper chemistry” to life.

• To enhance experimental observation and recording skills.

• To translate a set of instructions into successful action.

• To develop a keener sense of structure/property relationships (bp, mp, solubility, chromatographic behavior, spectral features).

• To develop research skills (group research project).
Each experiment will consist of three main sections:

Pre-Lab

In-Lab

Post-Lab
The Laboratory Notebook

At the top of EVERY page:

Name:___________ Desk # Course & Section #:____
Date:___________ Instructor's Name:________________
Experiment # &
Title:___________________________________________

The entire pre-lab exercise will be written in your notebook in duplicate.

You will turn in the white copy to your TA.
Pre-Lab Exercise

**Summary:** A brief summary of what will be done and why it will be done for all experimental procedures in the chapter or handout.

**Learning Objectives/Goals:** 3-4 objectives or goals for the experiment.

Examples:
- To purify a compound by recrystallization.
- To carry out a catalytic reduction.
- To learn how to run an NMR spectrum.
- To prepare an organometallic compound.
Pre-Lab Exercise

Chemical Equations/Diagrams of Apparatus: Chemical equations with conditions and/or diagrams of SPECIAL or NEW apparatus, as appropriate.

Chemical Data Table: A chemical data table giving the name, structure, melting point/boiling point, quantities required, flammability, toxicity, and disposal method for the substances (solids or solvents) with which you will be working.

Use the Aldrich Handbook of Final Chemicals and Laboratory Equipment (in your lab locker) and/or the Merck Index (on reserve).
Pre-Lab Exercise

- A Common Shelf chemical data table and blank chemical data tables are in the Lab Notebook.

- You must attach a copy of the Common Shelf chemical data table to the 2nd Pre-Lab (Distillation Experiment).

- When there are multiple entries for a chemical in Aldrich, choose the one with the lowest price/g.

**NOTE:** Make sure that you do not report information for deuterated or isotopically labeled chemicals!
Pre-Lab Exercise

Pre-Lab Questions:
• Answer all pre-lab questions.

• Show your work if you are asked to do a calculation.

• TAs in the Instrument Room can assist you with these questions, but they will not give you the answers.
Pre-Lab Exercise

The following 3 sections are **not** required for **the first three** pre-labs:

- Chromatographic Behavior Comparison of Starting Material and Product
- Spectral Features Comparison of Starting Material and Product
- Explanation of Product Isolation and Purification (“Work-Up”)
Pre-Lab Exercise

Chromatographic Behavior Comparison of Starting Material and Product: For experiments in which you will use TLC or column chromatography (CC) to separate the starting material and product, predict which compound will have the higher $R_f$ (TLC) and elute first (CC) based on molecular polarities predicted from the compounds’ structures and functional groups.
Pre-Lab Exercise

Spectral Features Comparison of Starting Material and Product: For your particular synthetic reaction, indicate how IR, NMR, and MS can be used to differentiate between the starting material and product by describing major spectral features that would be different when comparing the two.
Pre-Lab Exercise

**Explanation of Product Isolation and Purification ("Work-Up")**: Explain why each product isolation step (distillation, extraction, recrystallization, CC, etc.) was performed and what was removed or accomplished in the process.
Observations and Data: After the Pre-Lab section, your notes should contain a concise, accurate record of what you do and what you observe in the laboratory as you do it.
Post-Lab Notebook Work

**Results and Discussion**: Conclude each experiment with an analysis of your results and your comments and conclusions regarding the experiment. Answer assigned questions. See Chapter 3 in the Lab Guide for sample Results and Discussion sections. Also, regard the Grading Sheet for each experiment to make sure that you include all required details.
Before You Leave Lab

At the end of each lab period, you must have me sign and date your Lab Notebook before you leave.
Grading Sheets

Grading Sheets: At the end of the Lab Guide. These sheets contain the points distribution for each section of the lab. They also contain information concerning the content of each section of the lab report. Regard the Grading Sheet for each experiment to make sure that you include all required details. You will loose points for work that is missing from the lab report!

You will be not be using the Grading Sheets in your Lab Guide for the following experiments:

• Distillation
• TLC
• Column Chromatography
Course Grade

We will follow all the grading policies listed in Chapter 1 (Section 3) in the Lab Guide. The breakdown of points will not be the same as Chem 36.

Break Down of Points for Chem 36B

**Technique Portion**

- Technique/Synthetic Experiment Prelabs (100 pts each) 600 pts
- Technique/Synthetic Experiment Postlabs (100 pts each) 600 pts
- Quizzes (2) (100 pts each) 200 pts
- Unknown Homework 100 pts
- TA Evaluation 100 pts
Course Grade

Projects

- Literature Reviews 50 pts
- Proposal Critique 100 pts
- Group proposal (First Submission) 100 pts
- Group proposal (Second Submission) 100 pts
- Thought Questions (5) (10 pts each) 50 pts
- Mid Project Progress Report 100 pts
- Group Poster 300 pts
- Formal Report 300 pts
- Peer Evaluation 100 pts

Total Points 2800 pts
Grading

Each technique and synthetic experiment will be graded on the basis of 100 points for the Pre-Lab and 100 points for the Final Report (see Grading Sheets for points distributions).

100 points of your overall grade will be based on my evaluation of your performance and behavior in lab. (Hours put in, promptness of clean-up, dependence upon me, organization of time and desk space, cleanliness, safety consciousness, etc.)
Spectral Unknown Homework
Chapter 10 Lab Guide

Start Right Away!

- No Pre-Lab
- Do not waste your solid unknown
- Take a mp (Chapter 4).
- Write structures of possible matches from unknown list on Chem 36 Web page:

http://courses.chem.psu.edu/chem36

- Obtain NMR spectrum (Chapter 11, Section 6)

  !!! Sign up to use the NMR soon !!!
  !!! Prepare your sample during lab !!!
Spectral Unknown Homework
Chapter 10 Lab Guide

• Obtain IR spectrum (Chapter 11, Section 4)
  !!! Sign up to use the IR soon !!!
  !!! Prepare your sample during lab!!!

• Progress check with IR/NMR and unknown possibilities due in 12th Lab.

• Bring your IR, NMR and "best guesses" to me and I will initial your MS Analysis Request Form → MS spectrum (Chapter 11, Section 5) should allow you to determine the exact identification of your unknown.

• Final Report: Use the grading sheet in back of Lab Guide. Due March 26th (the 18th lab period).
Synthetic Experiments

• You will be assigned one synthetic experiment. Every student in the class will be assigned a different experiment and will be required to characterize his/her own product.

• This experiment will expose you to reactions that you probably will not encounter during the completion of your research project.

• Details concerning this assignment will be presented in the 3rd lab lecture.
Additional Requirements for Synthetic Experiments

• In addition to the reaction equation, you must:
  • Write down the name of the reaction, if applicable (example: Diels-Alder, Friedel-Crafts Acylation).

  • Write out the mechanism for every step of the reaction.

  • Include a written description of the mechanism.
Additional Requirements for Synthetic Experiments

After you list 3-4 learning objectives/goals, please write a few sentences indicating the relevance of the chemistry involved in the experiment- i.e. identify the functional group(s) involved in the chemistry, typical uses for this chemistry, any caveats associated with this chemistry, etc. This will help you recognize specific functional groups and understand their reactivities.
Group Research Projects

You will be divided into 4 research groups. Each group will be given a research project that will be carried out during the second half of the semester. The project will be bioorganic in nature and will require each group to do the following:

1. Carry Out a Literature Review
2. Write a Proposal
3. Carry Out the Proposed Experiments
4. Create a Poster Presentation of the Project
5. Write a Final Report on the Project
Group Research Projects

• The project assignments will be given out at the beginning of the 4th lab period.

• A lab lecture will be given during the 7th lab period to introduce the literature review.

• The first project assignment will be due at the beginning of the 8th lab period.
Thought Questions

Thought questions are designed to aid you in your problem solving skills. The questions will be based on literature assignments that pertain directly to the group research projects. You will be asked to read a literature article before coming to lab and then your research group will be asked answer the thought question at the beginning of the lab period. You will do one thought question for each project that is being carried out this semester.
Familiarize Yourself with the Library Web Resources

A tutorial was written by the library staff to familiarize students with the library searching for research projects. The tutorial is located at:

:www.libraries.psu.edu/crsweb/infolit/andyou/infoyo.htm

Another important website to check out is the Chem 36 website that contains Chem 36B course info and organic lab info.
What to Bring to the Next Lab

• All required course materials.

• A completed Pre-Lab for the entire Recrystallization/Melting Point Experiment (See Modifications for Technique Experiments on CHEM 36 B CD)

**NOTE:** You will need to take your Aldrich Catalog with you in order to complete the Pre-Lab!