# **Chemistry 3L: Introduction to Organic Chemistry Lab**

**Description:** Application of separation techniques in the context of reactions learned in Chem

3. Use of NMR for the analysis of reactions.

**Location**: Braun Lecture Hall

**Time**: Monday and Wednesday afternoons. Lecture 1-2 p.m.; lab 2-6 p.m.

**Instructor**: Dr. Christopher R. Moylan 237 Mudd

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### **Required Materials**

1. J.R. Mohrig, C.N. Hammond, and P.F. Schatz (2010). *Techniques in Organic Chemistry* (3<sup>rd</sup> Edition). New York: W.H. Freeman and Company.

2. Laboratory Notebook with duplicate pages.

### **Schedule**

Date	Lecture	Experiment	Readings	Lab Skills
Mon. August 5 [2L Lab Reports 6 Due]	¹H NMR	7A: Synthesis of <i>N</i> -phenylmaleimide	pp. 332-352	Filtration
Wed. August 7	<sup>1</sup> H NMR	7B: Diels-Alder Reaction	pp. 352-370	NMR, TLC
Mon. August 12 Lab Reports 7 Due	<sup>13</sup> C NMR	8A: Nitration of Acetanilide	pp. 371-396	Recrystallization, NMR, TLC
Wed. August 14	Mass Spectrometry	8B: Nitration of Methyl Benzoate	pp. 405-425	Recrystallization, melting point
Mon. August 19 Lab Reports 8 Due	Review	9A: Synthesis of Polyesters	All	Drawing fibers
Wed. August 21	Quiz	9B: Synthesis of Nylon 6,6		Drawing fibers
Mon. August 26 Lab Reports 9 Due	none			

## Safety:

A safety form will be handed out at the start of each term outlining all guidelines and policies. This form **must** be signed before working in the lab.

- 1). Safety goggles must be worn in the laboratory at all times!
- 2). Required clothing and apparel include: long pants, long sleeves, full socks, and close-toed shoes. No skin should be visible except your hands and head. Even though it is warm outside, you must come prepared! You will be sent home if not dressed properly for lab!
- 3). Gloves are available for your use and must be worn for all experiments. Do not used gloved hands in common areas and in hallways (handles on doors etc.) to avoid cross contamination.
- 4). Substances that are volatile, flammable, and/or toxic should be handled in the hood. In general, all experiments will be done in the hoods.
- 5). Know the location of the eye wash, safety showers, extinguishers and exits in the lab.
- 6). If a chemical spills or an accident happens, alert the TA or instructor.
- 7). Clean up the common areas on bench tops, near the balances and instruments when you are done.
- 8). If you get a chemical on your skin, wash with water immediately and hold for 15 minutes.

#### Waste:

- -Pour nothing down the sinks but uncontaminated water!
- -All other chemicals used in the lab must be put in the appropriate waste container. There are organic, acidic aqueous, basic aqueous and solid waste available everyday and special waste containers depending on the experiment. Follow your instructions from your TA if you aren't sure.

### **Grading**

3 Lab Reports 90%

Ouiz- 8/1 in class 10%

The laboratory report component will generally consist of three parts for each experiment: a prelab assignment, in-lab data and observations, and a post-lab assignment (lab results/discussion and questions) for a total of ten points. If the lab report consists of two parts, each report will be valued at 10 points for a total of 20 points possible.

### **Pre-lab Assignment (1 pt):**

Handouts for the lab are available on coursework. Doing the pre-lab assignment is meant to prepare you for the lab. Therefore you must come to lab with it done, otherwise you will not be allowed to do the lab for safety reasons. For the pre-lab, you need to write into your notebook the following items:

- I. Experiment Title
- II. Date you are doing the lab
- III. Experiment Objective: what is the purpose of the lab?
- IV. Reaction Scheme (structural formula equation given in lab handout): Reactants with reagents over the arrow and products to the right of the arrow.
- V. Reagent Information: given to you already in table format in lab handout
  - a. Structure
  - b. Molecular Weight
  - c. Density
  - c. Melting Point/boiling point
- VI. Experimental Procedure (on divided page; on left hand side is procedure and on the right hand side is left blank for observations made during lab)

## **In-Lab** (3 pts): Data and Observations

On the right hand column of the divided notebook, write down in your notebook all data and observations during the lab so that someone could repeat your experiment and understand what you observed. This includes the precise amount of reagents and solvent that you actually used and all observations like color changes, precipitates, gas evolution etc. Data, conclusions and results for all your experimental work should be included in your notebook. A running commentary of the experimental procedure should also be included with all observations made during the experiment. Do not record what the lab handout tells you to do, but rather what you DID do. FOLLOW THE GUIDELINES AT THE END OF THIS SYLLABUS FOR GOOD NOTEBOOK KEEPING.

# Post-Lab (3 pts): Data, Calculations, Results/Discussion, & Conclusion

- VII. Data: In your notebook, list any and all data that you collected in the lab (the lab handout lists some of the important information to include).
- VIII. Calculations: Use this data to do your calculations such theoretical yield, percent yield,  $R_{\rm F}$  etc.
- IX. Results & Discussion: Discuss the results and mention any sources of error, interpretation of those results (for example, is your compound what you think it is? Is it pure?). Interpret and attach to your report any spectra or chromatogram that you refer to you in your discussion.
- X. Conclusion: Under the conclusion heading: draw any conclusion from your data and state whether you met the objective of the lab that you mentioned in your pre-lab work.

### Post-Lab Questions (3 pts):

Attached to every lab handout are three questions that can be answered in your notebook after the conclusion or attached on a separate piece of paper. If you have any questions, you can go to office hours or ask your TA for help. A sample of a pre-lab and lab report will be posted on coursework and discussed in the first day of class.

**Attendance:** There are <u>no</u> makeup labs or quizzes. If you miss a lab due to an illness or other special circumstance, you must contact the instructor immediately. If appropriate, the lab can be excused and the points normally allotted for the lab to added to quiz at the end of the quarter.

# **Handing in Lab Reports:**

Every Monday the lab report for the previous week is due. There is a cover page you need to attach to the top of your notebook pages, and any questions and spectra/chromatograms need to be attached at the end.

#### Lab Skills Online:

This semester we will be class testing a few lab techniques using a new online product called LabSkills, which is part of the online learning homework system called OWL (Online Web Learning). Please follow the instructions below to register for LabSkills in OWL. Your access will be good for 3 months.

## To register:

- 1. Go to www.cengage.com/owl.
- 2. Choose any course (such as Organic Chemistry), and then click "**Register**."
- 3. On the left menu, click "LabSkills for Organic Chemistry".
- 4. On the next pages, choose your school, and then click the blue arrow under Student Registration.
- 5. Click the blue arrow under Section 1 that matches your course (Chem 1L,2L 3L).
- 6. Enter your information in the Self-Registration Form. Check the box for the 90-day free trial, and click **Continue** to finish the registration process.
- 7. On the Successful Registration page, click "Login Page" at the top of the screen.
- 8. Bookmark the login page for future visits to OWL. To log in, enter the login name and password you used during registration. If you forget to bookmark the login page, you can always log in from www.cengage.com/owl.
- 9. Click **I forgot my login/password** on the login page if you forget your login information.

Assignments have been selected based on relevance to Chem 2L topics. You may do them if you choose.

### **Stanford Honor Code:**

You are expected to uphold the university policy on academic integrity. In the context for the laboratory, you will be working in pairs and so can help each other understand concepts and complete experiments. **However, all work completed in your notebook must be your own.** Any data that you get from another student should be clearly referenced. Once lab ends, your collaboration with your partner should end. If you have questions, go to office hours, ask the TA or the instructor.

#### **Students with Disabilities:**

Students who may need academic accommodation due to disability must initiate the request with the Student Disability Resource Center (SDRC) located within the Office of Accessible Education (OAE). SDRC staff will evaluate the request with required documentation, recommend reasonable accommodations, and prepare an accommodation letter for faculty dated in the current quarter in which the request is being made. *Students should contact the SDRC as soon as possible since timely notice is needed to coordinate accommodations*. The OAE is located at 563 Salvatierra Walk (phone: 723-1066).

#### **Notebook Guidelines**

Please review the following guidelines before making entries into your lab notebook.

- 1). The notebook should have carbon copy pages, so that they can be removed to turn in your lab report.
- 2). Your handwriting should be neat and legible. If what you wrote cannot be read, it cannot be graded (or hypothetically used by someone else to repeat what you did).
- 3). Every page in your notebook should have your name, the date of the lab, the name of your TA and the experiment number.
- 4). Use a pen to write in your notebook. If you make a mistake, draw one line through the error and continue writing.
- 5). Make sure that you use the divider to prevent writing on subsequent carbon copies. If your carbon copy is illegible, hand in the original or make a photocopy.
- 6). Graphs, spectra, charts etc. should be attached by staple to notebook pages. Each should be completely labeled.
- 7). All entries should be legible and sufficiently detailed:
- -use proper names for instruments and glassware; "10 mL Erlenmeyer flask" instead of "flask"
- -indicate the specific concentration of chemicals used: "0.1 M" rather than "dilute"
- -indicate how the reagents were measured: there is a big difference between using a graduated cylinder and a micropipette
- -record the sequence in which reagents were added, precise reaction times and how reactions were heated
- -display any calculation in full detail with units for ease of verification