

Chemistry 339: Organic Chemistry Lab Spring 2012

Instructor: Dr. Marc J. Adler (mjadler@niu.edu) **TAs:** Anila Kethe (and Jeremy Hess)
Lab Meeting Times: TuTh 2:00–4:50pm **Location:** FR 309
TA Office Hours: Th 12–2 PM, LaT 333 **TA Email:** akethe@niu.edu
Text: Harwood, L.M; Moody, C.J.; Percy, J.M. *Experimental Organic Chemistry, Standard and Microscale*, 2nd edition.

Safety: Safety is the number one priority in the organic laboratory, since all chemicals are toxic to varying degrees. **ALL STUDENTS MUST WEAR EYE PROTECTION AND CLOSED-TOE SHOES AT ALL TIMES during the class. Leg coverings are also required.** Goggles are available free of charge at the chemistry stockroom, and they must be the type approved by the Department of Chemistry. No food or drinks are permitted in the laboratory. Waste chemicals must be properly disposed. Notify TA of any glassware breakage, chemical spills, or emergencies immediately. Your lab area, lab equipment, etc. must be cleaned prior to leaving the lab. Refer to the textbook for a more comprehensive discussion of safety. The Department of Chemistry has a zero tolerance policy for safety violations, and points will be deducted for violating the safety rules (see section on grading). Pregnant students should consult with their doctors regarding the risks of being enrolled in this and other laboratory-based classes.

Class Attendance and General Advice: Regular attendance is essential for a successful and pleasant experience in organic chemistry laboratory. Lab starts promptly at 2:00 pm, so be on time. You must attend every lab session unless you have a university-sanctioned excuse, of which you have to inform your TA in advance. If for some unforeseen reason you will not be able to attend lab, please alert your TA and Dr. Adler as soon as possible. **There will be no make-up labs**, and unexcused absence during a lab will result in a zero for that experiment. Also note that some labs require multiple periods; you must attend all sessions to receive credit for the experiment.

Pre-Lab Preparation: Read all the material in the textbook pertaining to the lab before showing up to class. Make sure that you are aware of any potential safety issues regarding the chemicals and equipment be used in the lab.

Laboratory and Reports: During the laboratory, your data should be recorded into a laboratory notebook (in ink), and **the data pages must be signed by the TA prior to your departure from lab.** Laboratory notebooks must be kept neat and must have **duplicate, numbered pages.** Regular paper notebook pages or composition notebooks are not acceptable. Lab reports should be in your own writing, and copied lab reports will receive a grade of zero. Even though you may conduct your experiments in pairs, the lab reports, including all calculations and answers to questions, should be prepared individually. The reports should be written neatly and legibly in black or blue ink. Pencils, white out, or colored pens (other than black or blue) are not permitted or acceptable for your notebooks. Lab reports are due at the beginning of the following lab period after an experiment is completed (multiple-period labs will be due the next lab period after completion) as indicated in the schedule of experiments. The following information should be included:

To be prepared before lab:

- 1) Name, Date, and Experiment Title (e.g. "Extraction of Caffeine from Tea").
- 2) Purpose. A brief summary of what you are trying to accomplish and/or learn from this experiment, (not just a restatement of the title). List methods, etc.
- 3) Data. Include all information pertinent to the experiment, including any and all safety hazards; MSDS sheets can be found at <http://www.hazard.com/msds/index.php>. For example, if you are extracting caffeine, you would want to draw the chemical structure, list important physical properties (such as melting point), and note any safety hazards associated with the compound. The mechanisms of reactions (if you are running a reaction) must be shown. Include equations and quantities of materials needed.

To be recorded in lab:

- 4) Procedure. The procedure performed during the lab on that day and observations, such as color changes, formation or disappearance of a precipitate, evolution of heat or gas, etc.
- 5) Results. Weights of the products, melting point ranges, etc. Theoretical and percent yields should be calculated if it pertains to the experiment. **Obtain TA's signature at this point.**

Full reports of the experiment should be assembled outside of the lab. The laboratory reports should be typed in Times New Roman 12 point font and 1.5 spaced. If pictures or chemical equations are required, they can be drawn in by hand. Even though you may conduct your experiments in pairs, the lab reports, including all calculations, should be prepared individually. Lab reports are due the following lab period after an experiment is completed, and multiple period labs will be due the next lab period after completion, as indicated in the tentative schedule. The duplicate pages from the lab notebook and all other relevant data (spectra, graphs, etc.) should be stapled to the typed lab report.

The following information should be included:

- 1) Name and Date.
- 2) Experiment Title (e.g. "Extraction of Caffeine from Tea").
- 3) Purpose/Objective: A brief summary of what was trying to be accomplished with and/or learned from this experiment (not just a restatement of the title). List methods, goals, etc.
- 4) Discussion: A brief discussion (~3 paragraphs) of the methods and concepts used in the experiment. For example, for the Diels-Alder reaction lab, the discussion should include its discovery, use, and mechanism.
- 5) Procedure: The general procedure that was performed during the lab on that day. Observations (such as color changes, formation or disappearance of a precipitate, evolution of heat or gas, etc.) should also be noted.
- 6) Results: Theoretical and percent yields should be calculated if it pertains to the experiment. Boiling and/or melting point ranges should be included here.
- 7) Conclusions: A brief, but informative conclusion to the lab stating the results obtained and discussing the possible reasons for those results. Include possible errors, and how they could be avoided in the future. (This may improve your techniques for later experiments.)
- 8) References: Outside resources used in writing your lab report should be *properly cited*.

NOTE: Certain practices are appropriate for college-level assignments. For example, it is expected that reports will be stapled and that correct syntax, spelling, punctuation, and grammar will be used.

Research Project: One of the most exciting aspects of this lab is the opportunity to work on a novel research project under Dr. Adler's direction, making molecules that in some cases have never been made before. It is important to maximize yield and ensure purity of these compounds. More details will be given as the time approaches.

Grading: Your grade in organic laboratory is largely based on the work done performing the assigned experiments and your understanding of the techniques/procedures. The lab reports will be graded based on the quality of your data and your presentation of the results. You must prepare your report individually, and violation of this rule will result in zero points for both laboratory partners. Following safety rules is an important part of any laboratory work, thus the points indicated may be deducted for the following safety violations:

Goggles/shoes not being worn (at all times except pre-lab discussion)	20 points
Chemical spill not cleaned (at the balance, your work area, in fume hoods)	10 points
Food/drink in lab	5 points
Improper waste disposal	10 points
Disposing of glass in waste basket (use the "Glass Waste" container)	5 points

The course grade will be assigned based on your point totals from the lab experiments and reports, quizzes, and the final. The points total is as follows:

Lab Reports (17 at 50 points each)	850 points
<u>Research Project</u>	<u>150 points</u>
Total	1000 points

Approximate Course Grading Scale:

A	900–1000 points
B	800–899 points
C	700–799 points
D	600–699 points

Schedule of Experiments

Lab	Date	Experiment	Due Date
	Tu 01.17	Check In	
01	Th 01.19	Experiment 1: Preparation of Isoamyl Acetate	Tu 01.24
02	Tu 01.24	Experiment 82: Measurement of Solvent Polarity	Tu 01.31
	Th 01.26		
03	Tu 01.31	Experiment 57: Friedel–Crafts Reaction	Tu 02.07
	Th 02.02		
04	Tu 02.07	Experiment 11: Peracid Epoxidation of Cholesterol	Tu 02.14
	Th 02.09		
05	Tu 02.14	Experiment 17: Reduction of Benzophenone	Th 02.16
06	Th 02.16	Experiment 5: Preparation of <i>E</i> -Benzaldoxime	Tu 02.21
07	Tu 02.21	Experiment 50: Preparation of Stilbene with Ylide Generation	Tu 02.28
	Th 02.23		
08	Tu 02.28	Experiment 19: Stereospecific Reduction of Benzoin	Tu 03.07
	Th 03.01		
09	Tu 03.07	Experiment 41, Part 2: Preparation of Dibenzylidene Acetone	Th 03.08
10	Th 03.08	Experiment 40: Preparation of Cinnamic Acid	Tu 03.20
	Tu 03.13	Spring Break – No Classes	
	Th 03.15		
11	Tu 03.20	Experiment 4: Protection of Ketones	Tu 03.27
	Th 03.22		
12	Tu 03.27	Experiment 7: Hydration of Alkenes	Th 03.29
13	Th 03.29	Experiment 70, Part 1: Dyes: Preparation and Use of Indigo	Tu 04.03
14	Tu 04.03	Experiment 75: Preparation of Copper Phthalocyanine	Tu 04.10
	Th 04.05		
15	Tu 04.10	Experiment 60: Diels–Alder Reaction Experiment	Th 04.12
16	Th 04.12	Experiment 66: Dipolar Cycloaddition	Tu 04.17
17	Tu 04.17	Experiment 76: Preparation of Copper Complex of Tetraporphyrin	Tu 04.24
	Th 04.19		
	Tu 04.24	Research Project (TBD)	
	Th 04.26		
	Tu 05.01		
	Th 05.03		