

CHM 254

Organic Chemistry Laboratory for Majors- Fall, 2013

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Office Hours: Stop by my office any time during the day.

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The course has a Niihka site. In the resource folder of Niihka, a link to a dedicated CHM 254 website is provided. The CHM254 website contains all handouts, assignments, etc, for this lab course that will be periodically updated.

Lectures/pre-lab: Inside each Section's laboratory, a prelab introduction will be followed by a quiz.

Laboratory: Section A 1:00-5:00 W, 272 Hughes

Section B 1:00-5:00 R, last name A-L 272 Hughes, M-Z 274 Hughes

No Section Switching

Prerequisite: CHM 141.M/142.M, CHM 153/161 or CHM141/142, CHM 144/145 or equivalent

Co-requisite: CHM 251

Texts: Palleros: *Experimental Organic Chemistry* (All experiments are referred to this book unless a handout is provided). Periodic readings from CHM 251 Text are expected.

You will be encouraged to obtain information online via Tweeter. By following the class tweets, you can keep track of progress concerning specific experiments, discussions, and responses, etc. Sign up as a Follower of @gungbw. (To register for a Twitter account, go to <http://twitter.com/>.)

Additional Required Materials:

Molecular model kit.

Access to Niihka and CHM254 website for assignments, handouts, etc.

Laboratory Notebook: The Official Laboratory Research Notebook, Jones and Bartlett

ChemDraw: We have a site license for this very useful program. You will be given instructions on how to download this program to your laptop.

Course Objectives:

There are a number of important skills that you must learn to be a successful scientist. The laboratory situation is an ideal environment to teach many of these skills. The assignments and resources made available in this course are meant to advance your ability:

1. to formulate and maintain a laboratory notebook.
2. to write laboratory reports based on interpretation of experimental results.
3. to use appropriate software and websites to interpret results, enhance reports, visualize chemical structures, and search the chemical literature.

4. to communicate effectively in oral and written form, individually and as part of a group.
5. to follow written and oral directions for designing and carrying out experimental work.
6. to operate spectroscopic instrumentation and to learn how to utilize spectroscopic information to solve structural and mechanistic problems (MS, IR, NMR).
7. to operate chromatographic instrumentation for the purpose of product separation or analysis (GC, HPLC)
8. to carry out organic reactions in a safe and environmentally sound fashion.

These course objectives will not be reached in one semester. You will continue to work on these objectives in the second semester laboratory course, CHM 255, and many of these objectives will be part of additional courses and training you will receive throughout your undergraduate career.

Attendance and Preparation:

Section switching will not be allowed, and only one catch-up period is scheduled. The laboratory work schedule is demanding. You will be part of a group project this semester and you will be sharing data with lab partners or with the class in several experiments: inability to meet your obligations in these situations will be reflected in your grade. You are expected to perform pre-lab reading assignments and notebook preparation prior to coming to lab: notebooks will be checked regularly.

Notebooks:

For each experiment you are expected to maintain a notebook entry, and much of the work must be completed **before** coming to lab. For each experiment you must have the following materials prepared before coming to lab:

1. a descriptive **Title**
2. a short **Introduction** that includes the major purposes of the experiment.
3. a **Preliminary Data** section that includes a table of chemicals to be used with molecular weights and physical data (melting points, boiling points, density, etc.) for all materials for which this information is needed, and balanced chemical equations for the reactions to be carried out. Not all experiments will involve a reaction, so do not write one if no reaction takes place!
4. a **Procedure** section that outlines the procedures to be followed. Do not copy the lab manual procedure, summarize it in your own words and leave space for entries you will need to make as the lab is performed (weights, % yields, melting points, observations of color, precipitates, etc.). Take note of any procedural changes/additions given in the handouts for each experiment or that become necessary during the course of the experiment, and make sure those changes are reflected in your notebook entries.

Changes in notebook entries necessitated by procedural changes or errors are made by striking through the material to be changed with a single black line (**NO ERASURES!**) A separate **Results Section** summarizing the major experimental results of each experiment should be prepared in the notebook after the experiment is complete. Your preliminary notebook preparation will provide you necessary information for the pre-lab quizzes, and you will hand in copies of your notebook preparation at the end of each laboratory period. Failure to do so will cause a deduction in your grade for the experiment, as will improperly prepared notebook entries. You will be required to calculate the % yield or % recovery of all products as part of the notebook report for each experiment. You must provide all data necessary to carry

out this calculation and show how the calculation was performed in the notebook. Failure to do so will cost you lab points.

Writing Assignments:

There will be a formal writing report based on a library research project. The writing report will be a journal style report and will be graded using a rubric. Notebook report will also be graded based on a rubric, although different from the journal style report.

Grading:

Midterm & Final plus Literature Report	240
Quizzes	40 (8 x 5)
Lab Write ups	<u>190 (10 + 7 x 20 + 40)</u>
TOTAL	470
A	423
B	376
C	329
D	282

These cutoffs may be adjusted **downward** at the end of the semester, but not by much, if at all. Plus/minus grades are not usually used. If you want an "A", score at least 423 points!

Laboratory Schedule (Read the entire unit for each experiment)

Week	Dates	Experiment	Sample Characterization	Reading Assignments and Questions
1	08/28-29	Check-in and Modeling (10)		¹³ C NMR, Unit 33
2	09/04-05	Recrystallization (20)	MP.	See handout. For ref., see E4.1, pp 80
3	09/11-12	Extraction (20)	MP.	E5.3, pp 109
4	09/18-19	Infrared (IR) spectroscopy (20)	IR.	See handout. For ref., see Ch. 14.7 in Bruice.
5	09/25-26	TLC (20)	IR,	Unit 8, pp 172 & handout
6	10/02-03	Column Chromatography (20)	IR,	Unit 9, pp 197 & handout
7	10/09-10	Williamson Ether Synthesis (20) NMR training during reflexing	IR, NMR (¹ H)	E15.3, pp 330

8	10/16-17	Reduction of Benzophenone (20)	IR, NMR (^{13}C) E21.1 pp 451
9	10/23-24	Unknown Identification (40)	Hand out
10	10/30-31	project continued	email spectra in .png (1024x640px)
11	11/06-07	project continued	IR, NMR (^1H , ^{13}C , DEPT, COSY)
12	11/13-14	Literature project (40)	Handout
13	11/20-21	Literature project continued	
14	11/27-29	Thanksgiving week	
15	12/04-05	Finishing up and Check Out	

References: "Organic Spectroscopy", William Kemp, Third Edition, (ISBN 0-7167-2227-5); "Introduction to Organic Spectroscopy", Joseph B. Lambert, etc. (ISBN 0-02-367300-1)

Schedule for first-hour activity (e.g., quizzes, spectroscopy, and experiments)

Week	Date	Subject
1	08/28-29	Introduction, Lab Safety, hybridization and ^{13}C NMR
2	09/4-5	Recrystallization
3	09/11-12	IR, Extraction
4	09/18-19	IR spectroscopy
5	09/25-26	Mass Spectrometry, Thin-Layer Chromatography
6	10/2-3	MS, Column Chromatography
7	10/9-10	^{13}C NMR
8	10/16-17	Midterm Exam (IR, MS, ^{13}C NMR, and lab basics)
9	10/23-24	^1H NMR, Unknown identification
10	10/30-31	^1H NMR, project continued
11	11/6-7	^1H NMR, project continued
12	11/13-14	Literature project
13	11/20-21	No first-hour activity
14	11/27-28	Thanksgiving Holiday Week
15	12/04-05	Final Exam (spectroscopy, lab basics), Literature project due