

Syllabus: CHEM352L, Summer 2013

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Lab Lecture: Tuesdays and Fridays, 12-12:50, Academic IV 003 (LH4)**

Laboratory: Tuesdays and Fridays, 1-5:00**

Text: Williamson, K. L.; Masters, K. M. Macroscale and Microscale Organic Experiments, UMBC Ed., 6th Ed.; Houghton-Mifflin: New York, 2010.

Prerequisite: CHEM 351L

Pre- or co-requisite: CHEM 352 (Note - if you drop CHEM 352, you MUST also DROP 352L!)

Office Hours: Mondays 12-2 and Wednesdays 12-2 in the Chemistry Tutorial Center (MEYR145) and by appointment

CHEM352L is the laboratory complement to CHEM352. Students will gain practical experience on a variety of topics covered in CHEM352, with particular focus on chemical synthesis and spectroscopy.

Students successfully completing this course will...

- be familiar with modern spectroscopic methods used to determine the structures of organic compounds, including basic theory and instrumentation, and the interpretation of spectra.
- have acquired practical experience carrying out a variety of important synthetic transformations.
- be comfortable with common methods of separation and purification used in the organic chemistry lab, including crystallization, distillation, and extraction.
- be familiar with chemical approaches used to identify organic compounds.

<u>Date:</u>	<u>Experiment Title</u>	<u>Reading/Procedure*</u>
July 9	Williamson Ether Synthesis, Check-in	Handout (Blackboard)
July 12	IR and Mass Spec, Unknowns **	Chapters 11 and 13 (Blackboard), handout
July 16	NMR, Unknowns**	Chapter 12, handout
July 19	Spectroscopy, Unknowns	Chapters 11-13, handout
July 23	Diels-Alder Reaction	Chapter 48, procedures 1 and 2
July 26	Nitration of Methyl Benzoate	Chapter 28, procedure 2
July 30	Oxidation of Cyclohexanol	Chapter 22, procedure 4
August 2	Hydrobenzoin from Benzil	Chapter 55, procedure on p. 670
August 6	Solution Phase Amide Synthesis	Handout (Blackboard)
August 9	Solid Phase Amide Synthesis	Handout (Blackboard)
August 13	Aldol Condensation	Chapter 37
August 16	Dyes and Dyeing, Check-out	Chapter 46, procedures 4 and 5

* All procedures used will be macroscale (noted by a round bottom flask rather than a test tube in the manual). All handouts will be posted on Blackboard under "Course Documents".

** Our second and third lab lectures will be 110 minutes rather than the normal 50 minutes; report to the laboratory at 2:00 rather than 1:00. The "Unknowns" lab is due Tuesday, July 23rd.

IMPORTANT NOTE: E-mails that do not include your name and section number AND that are not sent to BOTH Dr. Smith AND your TA will not be answered.

Absences and Tardiness: Missing lecture or lab must be justified with a documented accepted excuse. Experiments cannot be made up. In the event you miss an experiment, notify your teaching assistant and Dr. Smith ASAP by e-mail; documentation for your absence must be approved by both your T.A. and Dr. Smith. After such an absence, you are required to turn in pre-lab questions, notebook copies to show that you did prepare your notebook for the experiment, and post-lab questions; these must be turned in lab period immediately following the missed experiment. *You cannot receive a grade (other than F or I) in this course if you miss more than two lab periods for ANY reason.* If you are late to lab, you will receive a zero for that day's Proficiency Points and will not be allowed to take the quiz; if you are more than 15 minutes late, you forfeit your right to carry out the experiment and will be given zero for all associated assignments.

LAB NOTEBOOKS and LAB REPORTS

A cloth-bound composition notebook or carbonless lab notebook is required for the course. The assigned readings are to be completed prior to coming to lab. You must enter the laboratory each day with a clear understanding of what you are to do and why you are doing it. Points associated with each experiment will come from:

I. Pre-lab Questions and Notebook

- a. Pre-lab questions (10%). These will be posted on Blackboard before each new experiment. Your answers to these do not go in your notebook and are handed in to your TA at the beginning of your lab period. Some of the questions cannot be directly answered from the lab text or the lab lecture and will require chemical reasoning or additional research - cite sources. Copying answers from a friend constitutes academic dishonesty.
- b. Notebook preparation prior to coming to lab (20%). This consists of items A-G on pp. 19&21 plus an outline of the procedure to be followed in column format, as shown on p. 25; the left column labeled "Procedure" is filled out prior to coming to lab, and the right column ("Observations") is used to note completion of each step as well as changes and observations during the lab period. *Note that you will not be allowed to refer to your lab manual or handouts while carrying out the experiment, so success in the lab will require good notebook preparation.* The Aldrich Catalog or Sigma-Aldrich website (sigmaaldrich.com) are good sources for physical data of reagents. This section should also include relevant reaction mechanisms using arrow-pushing formalisms for all experiments involving chemical reactions.
- c. "Record what you do as you do it" (10%). Include changes to the procedure, results of measurements taken (*e.g.* actual masses of reagents used, melting points or TLC data), and all observations (*e.g.* a white precipitate formed after 5 minutes of heating). Spectra should be taped into your notebook in this (or immediately following this) section. If you have a notebook that makes copies, you will have to photocopy your spectra to turn in.

II. Lab Summary and Post-lab Questions

- a. Results and Discussion section (10%). This is entered directly in your notebook outside of the lab after completion of the experiment. In paragraph form, completely and concisely summarize the results of the experiment. Depending on the experiment, conclusions might include the identity of an unknown based on your data (elaborate) or a percent yield (including calculations) and reasons why your yield might not be 100%. Interpretation and assignment of signals in spectra should be included in this section where relevant.
- b. Post-lab questions (10%). These will be posted on Blackboard before each new experiment (it might be useful to have a look *before* carrying out the experiment!). Your answers to these do not go in your notebook and are handed in to your TA with the copy of your lab report.

Grading

Pre-Lab and Post-lab Questions

20

You will hand in answers to the pre-lab questions before lab each meeting. If you fail to do so, you will not be allowed to perform that day's experiment and a grade of zero for all parts of that days' experiment will be assigned. See details above in "LAB NOTEBOOKS and LAB REPORTS". Also due at this time are answers to the post-lab questions from the previous experiment.

Notebook

40

Details regarding the contents and format for lab reports is described above in "LAB NOTEBOOKS and LAB REPORTS". Your TA must initial your notebook prior to the start of each experiment to confirm that the required pre-lab information has been entered (if this is not satisfactorily prepared, you will not be allowed to perform that day's experiment and a grade of zero for all parts of that days' experiment will be assigned). Your TA must also initial your notebook at the completion of each experiment (prior to you leaving the laboratory) to confirm that the required data was recorded in your notebook during the lab period. A copy of your entire lab report (everything in your notebook, including the lab summary) is due at the beginning of the following lab meeting. Except under extraordinary circumstances or by special arrangement with your teaching assistant and Dr. Smith, lab reports will not be accepted late and a zero grade will be assigned.

Proficiency

10

Your skill in the laboratory is assessed by the quality of the results from technique labs and the yield and purity of product from synthetic experiments. Ability to work efficiently and neatness will also factor into your proficiency grade.

Quizzes

15

There will be a quiz at the beginning of each laboratory period. These will focus on the current day's experiment and will be open-notebook (no lab manual or handout). Theory, mechanisms, synthesis, and practical aspects of the experiment are all fair game for questions.

Problem Sets

15

At certain times during the course, you will be asked to complete a problem set; some will be done as group work in the laboratory and some are to be completed individually outside of class (homework, if you like).

Grades

Grades are assigned to scores calculated from the above as follows: A $\geq 90\%$; B $\geq 80\%$; C $\geq 70\%$; D $\geq 60\%$. The instructor reserves the right to lower, but not raise these cut-offs.

Academic Integrity: By enrolling in this course, each student assumes the responsibilities of an active participant in UMBC's scholarly community in which everyone's academic work and behavior are held to the highest standards of honesty. Cheating, fabrication, plagiarism, and helping others to commit these acts are all forms of academic dishonesty, and they are wrong. Academic misconduct will result in disciplinary action that may include, but is not limited to, suspension or dismissal. This instructor will pursue the severest punishment allowed under the prevailing policies against discovered fraud. To read the full Student Academic Conduct Policy, consult the UMBC Student Handbook, the Faculty Handbook or the UMBC Policies section of the UMBC Directory.

Safety

Safety is a primary concern in the chemical laboratory. Chemicals are almost all potentially dangerous. However, with the correct precautions, all the work you will be asked to do may be performed safely. It is important that you know how to handle both the chemicals and the apparatus you will be using. In addition, you must know what to do if an accident occurs. Your text has a general section on safety and special cautions associated with each experiment. Read them and learn them. Anyone who fails to follow correct safety procedures will not be permitted to complete the course and will not receive credit for the course.

IF YOU FAIL TO ADHERE TO ANY OF THE BELOW POLICIES, YOU MAY BE ASKED TO LEAVE THE LABORATORY AND RECEIVE GRADES OF ZERO FOR ALL OF THE ASSIGNMENTS ASSOCIATED WITH THAT WEEK'S EXPERIMENT.

Personal Protection

1. You must wear safety goggles in lab at all times. Even if you are not working, others may be. Safety goggles must be worn over prescription glasses. Safety goggles can be purchased in the bookstore.
2. Your feet must be **completely covered** by your footwear and **pants that completely cover your ankles** are required. Long hair must be tied back. **If you fail to comply with these rules, you WILL NOT be permitted to complete the day's experiment and will receive a grade of zero for all associated assignments.**
3. Wear old clothes to the laboratory or use a lab coat. Store coats, sweaters, book bags, and purses in the shelves provided, not at the bench where they can be ruined by chemicals or interfere with activities at the bench.
4. Do not eat, smoke, or drink in the lab. Food, cigarettes, cigars, chew, and drinks should not be brought into the lab, as these could become contaminated.
5. Treat all chemicals as toxic. Do not breathe their vapors, taste them, smell them, or touch them.

Laboratory Protocol

6. Know the emergency procedures for fires and for spills of corrosives on your person.
7. Know where the following are located: fire extinguishers, eye-wash, shower, acid-spill and base-spill wash bottles.
8. Do not dispose of organic solvents, mercury, or strong acids or bases in the sink. Waste solvents are disposed of in separate waste containers. Solid wastes, including paper towels used for organic spills, are disposed of in the dedicated bottle.
9. Broken glass must first be washed free of water-soluble residue or rinsed free of organic residue with acetone into the appropriate waste bottle, whichever is applicable. Residue-free glassware is then disposed of in the dedicated boxes. Trash is the only material discarded in the trash cans.
10. Wipe up spills immediately and keep all containers of organic solvents covered to reduce vapor concentration.
11. Do not wash chemicals from the skin with organic solvents. Use water only.
12. Do not leave reactions unattended.
13. Social visitors are prohibited from entering the instructional laboratories.
14. Never add anything TO concentrated acid, caustics or strong oxidants. Instead add acid, caustics or oxidants slowly and carefully to the other reagents.
15. Do not return reagents back into stock bottles. Take only what you need.
16. At the end of the day's work, sponge off your bench and leave it clean. Once during the semester you may be required to clean up the common areas.

NOTE – if you become pregnant during the semester, please inform your TA immediately; make your TA aware of any medical condition that prevents you from using specific chemicals.