

## ORGANIC CHEMISTRY LAB 310

FALL 2016

(The second, advanced part of organic lab, for chemistry majors)

**Important! You cannot take chem 310 without the prerequisite credits for organic chemistry lab (chem 309) and lecture (chem 307-308, or chem 315-316).**

Lecturer and Coordinator: John Taylor, Wright-Rieman Labs, room 278. (taylor@rutchem.rutgers.edu)

Office Hours: Tue 3:30 - 4:30 pm, or by e-mail appointment (use **310** in the title of all e-mails)

Lecture for all sections: TUE 5:00 - 6:20 pm, WL-AUD

Labs: **Section 01**, THR 1:00 - 5:20 PM; **Section 02**, TUE 6:40 - 11:00 PM;  
**Section 03**, THR 6:40 - 11:00 PM; **Section 04**, WED 1:00 - 5:20 PM.

All lab sections meet in WR-331. **Wait in the Rieman Lobby until 5 min. before your lab.**

Course web site: A sakai site is set up for this course, and you should automatically be registered as a student member of this site. Important class announcements may be made through this sakai site in addition to announcements in the lecture class. The syllabus, lecture notes, material safety data sheets (MSDS), and previous years' exams for practice will also be posted on this web site in the Resources section as pdf files.

### I. SYLLABUS

**NOTE: follow the book directions for Standard Scale reactions, unless instructed otherwise.**

**BLOCK 1** (notebooks due in lab 1st week of Block 2: 10/11,12,13).

Exp. 56 – multistep synthesis of benzylic acid; IR;  
Click Chemistry.

Week 1 9/6,7,8 Safety instruction and quiz. Check-in; Exp. 56A, benzoin condensation. mix reactants only.

Week 2 9/13,14,15 Finish Exp. 56A, start Exp. 56B.

Week 3 9/20,21,22 Finish Exp. 56B. IR: KBr disks. Start Exp. 56C. Start TLC of caffeine (not in book).

Week 4 9/27,28,29 Synthesis of an Azide by SN2 Reaction (not in book). Finish Exp. 56C. IRs.  
Exp. 56 exercise questions 1-3, 5-9.

Week 5 10/4,5,6 Copper-Catalyzed Azide-Alkyne Click Chemistry (not in book). Finish everything.

**Exam 1 - October 11 in lecture** (Block 1 topics + IR, NMR identification of unknown).

**BLOCK 2** (notebooks due in lab 1st week of Block 3: 11/8,9,10)

Polymer chemistry; physical organic chemistry – prep. and use of benzene diazonium salts, pKa values and linear free-energy relationships; electrophilic aromatic substitution; TLC and column chromatography; identification of unknown by IR and NMR.

- Week 6 10/11,12,13 Polymer chemistry: Exp. 26A, and Minilab 22. Start Unknown (IR, NMR). Finish block 1 analyses, as necessary. Give lab books to TAs in lab for Block 1 grading. Exp. 26 exercise questions 2, 3ab, 5-7.
- Week 7 10/18,19,20 Start Exp. 44, electronic effect of a para-iodo substituent – synthesis of para-iodobenzoic acid (group study). Continue Unknown.
- Week 8 10/25,26,27 Finish Exp. 44 – determine pKa values and linear free-energy relationship. TLC of ferrocene. Continue Unknown. Exp. 44 exercise questions 1-3, 5-9.
- Week 9 11/1,2,3 Lecture on Suzuki coupling and regioselective NaBH<sub>4</sub> reduction. Labs: Acetylation of ferrocene and purification by column chromatography (not in book). Prepare a plan (in groups) for a study of regioselective NaBH<sub>4</sub> reduction, as directed in lecture.

**Exam 2 - November 8 in lecture** (Block 2 topics, including IR and NMR)

**BLOCK 3** (notebooks due on 12/8,9,10 for Block 3 grading, unknown folder due in lab on 11/15,16,17)

Organometallic chemistry – Suzuki reaction; Claisen-Schmidt reaction and phase-transfer catalysis; NaBH<sub>4</sub> reduction and regioselectivity.

- Week 10 11/8,9,10 Organometallic chemistry - Suzuki coupling reaction (not in book). Finish everything in block 2. Give lab books to TAs in lab for Block 2 grading. Submit typed plan for NaBH<sub>4</sub> reduction study for evaluation and feedback on paper: one plan per group.
- Week 11 11/15,16,17 Start: Exp. 43B: Claisen-Schmidt reaction using phase-transfer catalysis. NMR analysis of Suzuki coupling product (including measurement of coupling constants). Submit final corrected plan for NaBH<sub>4</sub> reduction study for grading.

Thanksgiving Week (Nov 21-25): **No lecture or labs**

- Week 12 11/29,30, 12/1 NaBH<sub>4</sub> reduction – a study on regioselectivity (not in book). IR, NMR of Claisen-Schmidt product. Exp. 43B exercise questions 5cd, 6, 7b, 8. Finish everything in block 3.

**Exam 3 - December 6 in lecture** (Block 3 topics plus general methods, spectroscopy)

- Week 13 12/6,7,8 Analyses of NaBH<sub>4</sub> reduction products by IR, NMR. Check-out. Give lab books to TAs in lab for Block 3 grading.

## II. REQUIRED MATERIALS

1. **Text** *Multiscale Operational Organic Chemistry*, 2<sup>nd</sup> ed., John Lehman, 2009, Prentice Hall; ISBN-13: 978-0-13-241375-6
2. **Notebook** A hard-backed, or spiral-bound notebook (lined or graph paper) with **carbonless copying** is required and is available at the bookstore. **Loose-leaf notebooks are NOT allowed.**
3. **Goggles** Whenever you are in the lab, you must wear safety goggles that enclose the eye area completely or you will be removed from the lab immediately. Suitable goggles are available at the bookstore. Safety **glasses** may be substituted **only** with the agreement of Prof. Taylor.
4. **Padlock** The departmental lock will be removed from your locker after the second week. Since you are responsible for the contents of the locker, you must supply your own lock by week 2.
5. **Markers** A permanent magic marker is highly recommended for labeling your samples.
6. **Folders** A folder is required for the Unknown assignment in Block 2. A spiral-bound or loose-leaf folder is recommended for all handouts and lecture notes.

## III. RESPONSIBILITY FOR EQUIPMENT

Once you have signed out your laboratory equipment, you are entirely responsible for it. If you break, damage or lose anything, you will be billed later for it. Be very careful with the glassware and be sure not to leave any items out at the end of the lab. At the end of each lab period, use your own lock to secure your equipment! If you drop the course before the end of the semester, you must arrange a time to check out your equipment within two weeks of leaving the course.

## IV ABSENCES

You will be allowed to make up a lab only under very unusual circumstances, and then only if this is feasible. If you are sick or have another valid excuse, get in touch with Professor Taylor AND your lab TA AS SOON AS POSSIBLE to explain your situation. You will not lose credit for a missed lab due to isolated cases of sickness or other valid and unavoidable reasons, if you contact us promptly. However, appropriate documentation may be required as evidence in some cases. Multiple absences will require you to withdraw from the course.

## V. GRADING

Your grade will be determined as follows, from your total points score out of a maximum of 1000 points:

1. Results of Experiments, 550 points (preparation, purity and characterization of products, notebook write-up, general cleanliness and safety of operations, etc.). Your products will be graded based primarily on purity. Yield will be of secondary importance. In weeks 2-12, copies of your prelab write-ups will be collected and graded out of 10 points each. Total points per experiment will be distributed as follows (number of prelabs in parentheses): Exp. 56: 100 (2); Azide synthesis and click chemistry: 40 (2); Polymer Chemistry: 40 (1); Diazonium synthesis and pKa analysis: 50 (2); TLCs and column chromatography: 40 (1); Library assignment: 20; Suzuki coupling reaction: 60 (1); Unknown:100; Claisen-Schmidt: 40 (1); NaBH<sub>4</sub> reduction: plan 30, experiment 30 (1).

2. Lab Quizzes, 50 points. At the beginning of every lab (except the first and last), the TA will give a short 5 pt. Quiz - one or two questions dealing with the procedure to be done in that lab, possibly from the Operations section. Lowest score of 11 quizzes will be dropped.
3. Three Exams, 300 points. The three exams given during the lecture class will be worth 100 pts. each. A make-up exam will be given to anyone who missed one of these exams for a valid reason. To qualify for a make-up, you must contact Prof. Taylor as soon as you are able, and certainly within a few days of the exam you miss.
4. TA evaluation, 100 points. Your TA will assess your performance in the lab in terms of your basic understanding of the experiment, your general laboratory skills, your degree of preparedness, your care in using chemicals without waste, your attention to wearing goggles and other safety issues, and how clean you keep your bench. The average score for this evaluation is fixed at 80 points for each section.

NOTE: (a) If you hand in your notebook late with no valid excuse, you will lose 5 points for every day late, including weekends.

(b) No one who really tries will fail this course. Successful completion of the labs and notebook write-ups should normally be sufficient for at least a C grade. However, low scores on the exams (<40%), although rare, are likely to result in a grade of D regardless of lab performance.

#### IV LABORATORY NOTEBOOK (Read Appendix II, III, IV and V in the text).

Your notebook write-ups must be in your own words - cases of plagiarism will be reported to the Office of Student Conduct. Although there are many good ways to write a lab notebook, in the interest of efficient grading, we will require you to use the following format.

1. Use a hard-backed or spiral-bound notebook with lines or graph paper and carbonless copying. The pages must be numbered and should preferably open like a book, **not** a notepad. Leave the first few pages for a Table of Contents, and keep this table up-to-date throughout the semester. All entries must be made IN INK, and cannot be typed. Exceptions are the prelabs written in the two weeks of grading for blocks 1 and 2, and any graphs or spectra (see part 2h below).
2. Pre-Lab Write-up. Before coming to lab, you MUST prepare a "pre-lab" section for each experiment, **AND HAND IN A PHOTOCOPY TO YOUR TA AT THE BEGINNING OF EACH LAB FOR GRADING**. The pre-lab write-up consists of the following components:
  - a. The experiment number (if applicable), a title, and the date, at the top of the page.
  - b. The main **purpose** of the experiment. If you are working as part of a group, this section should indicate the names of all members of your group, and their assigned roles. If the assigned roles change, these changes should be noted during the performance of the experiment (section 3, below)
  - c. Under the heading **Safety and Risk Assessment**, a 3-column **table** listing (i) all the chemicals to be used, (ii) their **most important** hazardous properties (if none, say so), and (iii) actions to be taken in case of an emergency arising from their use. The manufacturer's Material Safety and Data Sheet (MSDS) for each chemical required for each experiment is provided as a pdf file on the course sakai site for your use to prepare this section. This section must be concise and written in a way that is immediately **useful** to you in the lab.
  - d. Under the heading **Equations**, balanced equations for all reactions (if any).
  - e. Under the heading **Mechanisms**, write complete detailed mechanisms for all reactions you will perform.
  - f. Under the heading, **Amounts and Properties**, data for all reactants and catalysts (molecular weight, grams to be used, moles to be used, relevant physical constants); solvents (volumes to be used); and products (molecular weight, theoretical yield in grams and moles, relevant physical properties).

- g. Under the heading **Procedure**, a brief description of the steps of the experimental procedure, either as a list or as a flow chart. Do not write paragraphs, or even full sentences. Your outline should be complete and unambiguous and contain all essential details, such that you or your TA (or any trained organic chemist) could perform the entire experiment from it in exactly the same way. It should contain no extra words to distract from the essential procedures. Diagrams of essential equipment set-up may be included, as necessary and/or useful to you.
- h. If not already covered above, do what is asked in the "Before You Begin" section of the experiment. **For the two weeks when you have handed in your notebook for grading of blocks 1 and 2 (and only for these weeks), write (or type) your pre-lab on paper that you will tape into your book when you get it back. As always, you must provide a copy of these pre-labs for your TA.**
3. During the lab, under the heading **Observations and Measurements**, write all observations and measurements. It is nice if your entries are neat, but it is far more important that you write your observations **as you make them**. **It is prohibited to make notes on scrap paper to write up later.** One approach to organizing your lab book is to split the pages into two vertical columns, and write your prelab **Procedure** in the column on the left side of the page, and your ongoing **Observations and Measurements** in the column on the right side of the page. Space your entries out to allow flexibility, including possible changes in procedures during the experiment.
4. After the lab, using some or all of the appropriate headings **Calculations**, **Discussion** and/or **Conclusions**, write all new calculations resulting from the experimental data (e.g. yield), and the discussion of your experimental work and data, and the conclusions. **The quality (precision, not length) of your discussions and conclusions will be the most important factor determining your score for each experiment**, so be sure to think about this section carefully and thoroughly. Mention any changes or improvements you would consider, if you were to repeat the experiment. If you made a mistake that could not be corrected during the experiment, discussing it precisely will be helpful. Finish with a conclusion that addresses the main purpose of the experiment and relates the outcome. This might be a very simple statement, such as "Cholesterol was prepared in 78% yield by weight, and in high purity as assessed by melting point;" or "Unknown #3 was identified unambiguously as benzoic acid by IR and NMR spectroscopy."
5. After the conclusion, answer the exercises assigned, if any (see pages 1 and 2, and lecture notes).

**Use plenty of space** in your notebook, and try not to crowd things together. This will make reading and analyzing your methods and data easier for you during your discussion write-up and for your TA when grading. Leave blank pages between experiments, in case you need to add more information later.

Note that, when writing a lab book in the chemical industry, you are often required to do the exact opposite: leave no spaces. This helps support patent applications by authenticating the dates of accomplishments and claims. Thus, the different goals of academia and industry can produce different approaches to the same tasks.

## VII ACADEMIC INTEGRITY

For a description of the Rutgers policy on academic integrity, read the following web site:  
<http://academicintegrity.rutgers.edu/academic-integrity-at-rutgers>

**Shared Work**: Note that you will be asked to collaborate or work in groups on certain lab assignments. **In all cases where collaborative work is involved, your lab write-ups should indicate the names of all participants in shared experimental work and/or the contributions of others to the data analysis. You are never allowed to take credit for another person's work: shared work requires sharing the credit.**