

Organic Chemistry Lab Chm310 - Fall 2021

(The Second, Advanced Part of the Organic Lab for Chemistry Majors)

IMPORTANT! YOU CANNOT TAKE CHM 310 WITHOUT ALREADY HAVING HAD ONE FULL SEMESTER OF ORGANIC LAB (CHM 309) AND HAVING COMPLETED ONE FULL YEAR OF AN ORGANIC CHEMISTRY LECTURE COURSE.

Instructor: Dr. Ralf Warmuth

Office: Wright-Rieman Labs, room 380; email: warmuth@chem.rutgers.edu

Office Hours: Tuesday 3-4:30pm (Zoom) or by e-mail appointment (use **310** in subject line of all e-mails)

Lecture: Tues 5:00 – 6:20 PM; via Zoom; links are provided via Canvas course site: login Canvas course site, select *Zoom*, then click the lecture link

Labs:	Section 1, T	7:00 – 11:00 PM	WRL 331
	Section 3, TH	1:00 – 5:00 PM	WRL 331
	Section 4, TH	7:00 – 11:00 PM	WRL 331
	Section 5, F	1:00 – 5:00 PM	WRL 331

Please, wait in the Rieman lobby until 5 min. before your lab.

Canvas course web site: Use this link <http://chem.rutgers.edu/?q=node/82> or go to the chemistry dept. web site www.rutchem.rutgers.edu, click on “Undergraduate Study”, in ‘Undergraduate Course Pages’ box (upper right corner) select “Fall 2010”, select Chm 310. Here you find previous years’ exams, syllabus & handouts.

Masks must be worn during lab meetings and while being in the Chemistry Building

In order to protect the health and well-being of all members of the University community, masks must be worn by all persons on campus when in the presence of others (within six feet) and in buildings in non-private enclosed settings (e.g., common workspaces, workstations, meeting rooms, classrooms, etc.). **Any student not wearing a mask will be asked to leave the lab.**

Masks should conform to CDC guidelines and should completely cover the nose and mouth:

<https://www.cdc.gov/coronavirus/2019-ncov/prevent-getting-sick/about-face-coverings.html>

Each day before you arrive on campus or leave your residence hall, you must complete the brief survey on the [My Campus Pass symptom checker](#) self-screening app. Also, please assure you have a mask with you.

I. LAB SCHEDULE

No labs on 9/2/21, 9/3/21 and during Thanksgiving week

BLOCK 1 (notebooks due 2nd lab of Block 2: 10/12-15).

Exp 1A: Acetylation of ferrocene; Exp 1B: TLC and column chromatography; Exp 2A: Synthesis of a propargyl ether; Exp 2B: Click Chemistry: Cu-catalyzed alkyne-azide [3+2] cycloaddition; IR; NMR; Exp 3: Identification of an Unknown (report due Dec 7-10)

Lab 1 9/7,9,10 Safety Quiz. Check-in; Exp. 1A, Acetylation of ferrocene (*Handout & Chpt 32*).

Lab 2 9/14,16,17 Exp. 1B: TLC of acetylated ferrocene mixture and isolation of products by column

chromatography (*Handout & Chpt 8&9*); IR, mp. of product (*Chpt. 11*)

Lab 3 9/21,23,24 Exp. 2A. Prepare 4-acetylphenyl propargyl ether. Confirm purity of product by TLC (*Handout*); Exp 3: Start Identification of Unknown (IR; (m.p.); EI-MS (provided); ¹H-NMR; ¹³C NMR (*Handout; Chpt11-13*)).

Lab 4 9/28,30;10/1 Exp 2B: One-pot Cu-catalyzed azide alkyne cycloaddition (*Handout*). IR and NMR & m.p. of product. Finish everything for Block 1;

Exam 1 – Sunday, October 10 from 3:00 pm – 4:20 pm (Block 1 topics)

BLOCK 2 (notebooks and Unknown folder due 1st lab of Block 3: 11/2-5).

Physical Organic Chemistry - Exp 4A: Prep. and use of benzene diazonium salts; Exp 4B: pKa values and linear free-energy relationships; Exp 5A: Synthesis of dimedone; Exp 5B: Solvent Effect on keto-enol equilibrium by NMR; Exp 6: NaBH₄ reduction of a cyclohexenone; Start essay.

Lab 5 10/5-8 Exp. 4A/B: Electronic effect of a *para*-iodo substituent & linear free-energy relationship (*Handout & Chpt 44*). Finish up NMR & IR characterization of Click reaction product, if necessary. Continue Unknown (IR, NMR). NaBH₄ reduction procedures due to TAs

Lab 6 10/12-15 Exp 5A: Synthesis of dimedone (*Handout*); Finish up Exp 4B determine pKa values & linear free-energy relationship. Hand in lab books to TA for Block 1 grading. Feedback on NaBH₄ reduction procedures.

Lab 7 10/19-22 Exp 5B. Solvent Effect of Keto-Enol Tautomerization by NMR (*Handout*). Submit updated NaBH₄ reduction procedures, if needed. Start essay

Lab 8 10/26-29 Exp 6: NaBH₄ reduction of 3-methylcyclohex-2-enone;

Exam 3 – Sunday November 7 from 3:00 pm-4:20 pm (Block 2 topics)

BLOCK 3 (notebooks and Unknown folder due last lab of Block 3: 12/7-10).

Exp 7A/B: Organometallic catalysis: Suzuki Cross-Coupling; Exp 8: Polymer Chemistry: Synthesis and characterization of Polystyrene (NMR; Size-Exclusion Chromatography); Exp 9A/B: Host Guest Chemistry: Synthesis of a resorcinarene cavitand, self-assembly and guest encapsulation by NMR;

Lab 9 11/2-5 Exp 7A: Organometallic chemistry – Suzuki coupling: optimization of reaction conditions (*Handout*); HPLC analysis of products. Finish everything in block 2.

Lab 10 11/9-12 Exp 8: Polymer chemistry: Polystyrene; Nylon 6.10; IR of product; Endgroup analysis by NMR, GPC analysis of dispersity (*Handout and Chpt 67*).

Lab 11 11/16-19 Exp. 9A: Synthesis of a resorcinarene (handout). IR and NMR analysis of product

Week 11/22-28 **No labs; no lecture**

Lab 12 11/30-12/3 Exp 7B: Larger-scale Suzuki coupling and isolation of product (*Handout*). IR, 1D & 2DNMR analysis of product. Exp 9B: Self-assembly of resorcinarene and encapsulation studies by NMR (*Handout*)

Lab 13 12/7-10 Finish up characterization of Suzuki coupling product. Essay due end of lab. Notebooks due to TAs by end of lab. Check-out.

Exam 3 – Sunday December 12 3:00-4:20PM (Block 3 topics plus general methods)

II. REQUIRED MATERIALS

- 1. Textbooks**
Text I: *Macroscale and Microscale Organic Experiments*. 7th Ed; Kenneth L. Williamson & Katherine M. Masters. 2017, Brooks/Cole Cengage Learning; ISBN-10: 0357686853 | ISBN-13: 9780357686850. Please see eBook purchase options at this link: [eBook purchase options](#)
If you find a used copy of the 6th or 7th edition on amazon or somewhere else, both will work.
Text II: *Organic Chemistry Laboratory Techniques*. 2nd Ed. Lisa Nichols, 2016; Open Textbook Library; <https://open.umn.edu/opentextbooks/textbooks/369>
This book will supplement Text 1. It has an excellent lab techniques section with many illustrations but lacks the instrumental aspects of the organic lab.
Text III: *Operational Organic Chemistry*, 2nd, 3rd or 4th ed., John Lehman, Prentice Hall, ISBN: 9781323890240,
This book has been using so far in CHM309/310. It is unfortunately out of print. If you have a copy or find a used one on the internet, it is an excellent choice.
I will post a list of suggested readings for the Text I/II combo and for Text III.
Handouts and pdfs of journal articles will be available on the course site
- 2. Notebook** A hardbacked, bound notebook (lined or graph paper) is required and is available at the bookstore. **No spiral-bound or loose-leaf notebooks are 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 Dr. Warmuth. In light of mask wearing mandate, safety goggles instead of safety glasses are highly recommended, since the latter may fog up very quickly.
- 4. Padlock** The departmental lock will be removed from the locker after the second week. Since you are responsible for the contents of the locker, you must supply your own lock by the second week.
- 5. Markers** A permanent magic marker is highly recommended for labeling your samples
- 6. Folder** A spiral-bound or loose-leaf folder is recommended for all handouts and lecture notes. A folder is also needed for your lab reports.
- 7. Gloves** Nitrile gloves will be provided. These gloves can be reused and like face masks should be used until they break or are heavily contaminated. Depending on the size of your hands and the moisture content of your skin, it can be difficult to put them on and off. What helped me (R. W.) is sprinkling a little bit baby powder into the gloves before putting them on.

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 if you are sick or have another valid excuse, and then only if this is feasible. Also, if you are told to quarantine, or are experiencing symptoms of any transmittable disease, please remain at home and don't attend any lab sessions. In all instances where you have to miss a lab/lecture, submit a [Self-Reporting Absence Note](#). Also inform Dr. Warmuth and your TA, that you will be missing a lab, so that we can arrange a way for you to make-up the missed work. You will not need documentation unless you will be absent for an extended period of time (over one week), in which case you should contact the Dean of Students at this link: <http://deanofstudents.rutgers.edu/>.

V. EXAMS

	DAY	TIME	LOCATION	COVERAGE	POINTS
EXAM 1:	Sunday 10/10/2021	3:00-4:20pm	CCB1303	Block 1 topics	100 pts
EXAM 2:	Sunday 11/7/2021	3:00-4:20pm	CCB1303	Block 2 topics	100 pts
EXAM 3:	Sunday 12/12/2021	3:00-4:20pm	CCB1303	Block 3 topics	100 pts

Exam Conflicts: Please see below VI.3. and the [Common Hour Exam Policy](#) for what constitutes an exam conflict.

VI. GRADING

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

1. 510 pts Results of Experiments. (preparation, identification of unknowns, 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. Points per experiment will be distributed as follows: Exp. 1A/B: 50; Exp 2A/B Click Chemistry: 60; Exp 3 Unknown: 100; Exp 4A/B: 40; Exp5A/B Dimedone & Keto-Enol Tautomerization: 50; Exp 6 NaBH₄ reductions: 40; Exp 7A/B Suzuki coupling reaction: 80; Exp 8 Polymer Chemistry: 40; Exp 9A/B Host-Guest chemistry & molecular recognition: 50.
2. 50 pts Lab Quizzes. At the beginning of every lab (except the first and last), the TA will give a short 5 pt. Quiz – either one or two questions dealing with the procedure to be done in that lab, possibly from the Questions section. The lowest score of 11 quizzes will be dropped.
3. 300 pts Three Exams. The three exams given in-person on the above dates (See V. Exams) and will be worth 100 pts. each. A make-up exam will be given to anyone who has an exam conflict or missed one of these exams for a valid reason (health/medical related; an accident or a death in the family). If you have an exam conflict, please contact Dr. Warmuth within the first two weeks of the semester, so that a make-up option can be arranged. If you miss or missed an exam for a valid reason, you must fill a student absence report and contact Dr. Warmuth as soon as you are able, and certainly within one week of the exam you miss, if that is possible.
4. 100 pts TA evaluation. 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.

5. 30 pts Research Essay. Your essay should be 3-4 pages long and must be submitted via Canvas. 5 points per day will be deducted for each day that you submit late.
6. 10 pts Library Homework Assignment. Handout on Canvas

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

VI. LABORATORY NOTEBOOK (Read Text 1 pp. 18-25; & 308-310).

Your notebook write-ups, lab reports and essay must be in your own words – cases of plagiarism will be sent to your Dean. Although there are many good ways to write a lab notebook, in the interest of efficient grading, we will require you to follow the following format:

1. Use a hardbacked, bound notebook with lines or graph paper, and number the pages as you go in the upper outside corner of each page. 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.
2. Pre-Lab Write-up. Before coming to the lab, you must prepare a ‘pre-lab’ section for each experiment, consisting of the following:
 - a. The experiment number, title, and date, at the top of the page
 - b. The main **purpose** of the experiment.
 - c. Under the heading **Equations**, balanced equations for all reactions (if any).
 - d. Under the heading **Mechanism**, write complete detailed mechanisms for all reactions.
 - e. Under the heading **Amounts & Properties**, list data for all reactants and catalysts (molecular weight, grams to be used, moles to be used, relevant physical constants); solvents (volumes to be used) other reagents (grams to be used) and products (molecular weight, theoretical yield in grams and moles, relevant physical constants).
 - f. Under the heading **Procedure**, a brief description of the steps of the experimental procedure, as a list and 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.
 - g. If not already covered above, do what is asked in the “Before You Begin” section of the experiment. **For the labs when you have handed in your notebook for grading (and only for these weeks), write your pre-lab on a piece of paper that you will tape into your book when you get it back.**
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 and write them up later.
4. After the lab, under the appropriate heading **Calculations** write all new calculations resulting from the experimental data (e.g. yield).
5. **Discussion, Conclusions and Answers to Exercises** should be typed as a separate report. On top of the first page of your report type your name, Chm310, your section number and date. Underneath type the experiment number and title. Under the headings **Discussion** and **Conclusions** type the discussion of your experimental work and data, and the conclusions. If your discussion includes structural drawings of compounds, diagrams or figures, leave some space and hand-draw them. For drawing structures, you may also use the program ‘Chem Draw’, for which Rutgers University has a site license. The quality of

your discussions will be the most important factor determined your score for each experiment, so be sure to think about it 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 fully will be helpful. Finish with a conclusion that addresses the main purpose of the experiment and relates the outcome. It might be a very simple statement such as “Compound A was prepared in X% yield by weight and high purity, as assessed by melting point.” or “Unknown #3 was identified unambiguously as benzoic acid by IR and NMR spectroscopy and melting point.”

6. After the conclusion, answer the exercises assigned, if any. Again, you may hand-draw structures or mechanisms or use Chem Draw. Staple your report together and place it into your ‘Lab Reports’ folder.
7. Use plenty of space on the pages of your notebook and try not to crowd things together. This will make reading and analyzing your methods and data easier for you during discussion write-up and for your TA when grading. Note that, when writing a lab book in 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 task.

VII. ACADEMIC INTEGRITY

For a description of the Rutgers policy on academic integrity, read the following web site:

<http://academicintegrity.rutgers.edu/integrity.shtml>

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.**

VIII. DISABILITY POLICY

Students with disabilities requesting accommodations must follow the procedures outlines at <https://ods.rutgers.edu/students/getting-registered>. Please see also below **X. Disability Services**

IX. STUDENT-WELLNESS SERVICES

Counseling, ADAP & Psychiatric Services (CAPS)

(848) 932-7884 / 17 Senior Street, New Brunswick, NJ 08901 / <http://health.rutgers.edu/medical-counseling-services/counseling/>

CAPS is a University mental health support service that includes counseling, alcohol and other drug assistance, and psychiatric services staffed by a team of professionals within Rutgers Health services to support students’ efforts to succeed at Rutgers University. CAPS offers a variety of services that include: individual therapy, group therapy and workshops, crisis intervention, referral to specialists in the community, and consultation and collaboration with campus partners.

Crisis Intervention : <http://health.rutgers.edu/medical-counseling-services/counseling/crisis-intervention/>

Report a Concern: <http://health.rutgers.edu/do-something-to-help/>

Violence Prevention & Victim Assistance (VPVA)

(848) 932-1181 / 3 Bartlett Street, New Brunswick, NJ 08901 / www.vpva.rutgers.edu/

The Office for Violence Prevention and Victim Assistance provides confidential crisis intervention, counseling and advocacy for victims of sexual and relationship violence and stalking to students, staff

and faculty. To reach staff during office hours when the university is open or to reach an advocate after hours, call 848-932-1181.

X. DISABILITY SERVICES

(848) 445-6800 / Lucy Stone Hall, Suite A145, Livingston Campus, 54 Joyce Kilmer Avenue, Piscataway, NJ 08854 / <https://ods.rutgers.edu/>

Rutgers University welcomes students with disabilities into all of the University's educational programs. In order to receive consideration for reasonable accommodations, a student with a disability must contact the appropriate disability services office at the campus where you are officially enrolled, participate in an intake interview, and provide documentation: <https://ods.rutgers.edu/students/documentation-guidelines>.

If the documentation supports your request for reasonable accommodations, your campus's disability services office will provide you with a Letter of Accommodations. Please share this letter with your instructors and discuss the accommodations with them as early in your courses as possible. To begin this process, please complete the Registration form on the ODS web site at: <https://ods.rutgers.edu/students/registration-form>.