

Organic Chemistry Lab Chm310 - Fall 2022

(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 5:30pm-6:30pm in WL-380 or by e-mail appointment (use **310** in subject line of all e-mails)

Lecture: Tues 3:50 – 5:10 PM; SEC-202; first lecture: 9/6/22;

Teaching Assistants:

Damon Hinz; email: djh241@chem.rutgers.edu

Mark Dresel; email: mjd458@chem.rutgers.edu

Labs:

Section	Day	Time	Location	TA (Week 1-6)	TA (Week 7-13)
1	TH	1:00 – 5:00 PM	WRL 331	Damon Hinz	Damon Hinz
2	TH	7:00 – 11:00 PM	WRL 331	Mark Dresel	Mark Dresel
3	F	1:00 – 5:00 PM	WRL 331	Damon Hinz	Mark Dresel

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

Canvas course web site: Here you find previous years' exams, syllabus & handouts, MSDS of chemicals, lecture notes.

In the tab 'Modules', the Modules 'Lectures and Lab Sessions' will have a page for each lecture and lab session. The lecture pages contain the coverage of lecture, links to handouts, reading assignments and any other important information. The lab session pages will contain the lab schedule, links to handouts for the specific experiments, links to MSDS for the chemicals/solvents used in the experiment, pre-lab reading (which is often the same as the reading assignment for the corresponding lecture on Tuesday of the same week), a table with hazards of the chemicals and solvents used in the experiments.

A separate Module 'Exams/Practice Exams/Problem Sets' compiles practice exams from previous semesters, lists of topics that will be covered in each exam, and exam answer keys.

Files (pdfs) of handouts, and MSDS of chemicals can also be downloaded from the 'Files' tab.

Prerequisites: 01:160:309 and 01:160:308 or 01:160:316

Masks must be worn during lab meetings, during the lectures and while being in the Chemistry Building. Any student not wearing a mask will be asked to leave the lab/lecture room.

For more detail, please read the COVID-9 protocols <https://coronavirus.rutgers.edu/covid-19-protocols-updated-june-2022/>

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](https://coronavirus.rutgers.edu/symptom-checker/) self-screening app. <https://coronavirus.rutgers.edu/symptom-checker/>
Also, please assure you have a mask with you.

I. LAB SCHEDULE

- **Lab classes begin with check-in and experiment 56A on Thursday, 9/8/22 (sect 1&2) and 9/9/22 (sect 3). Please come prepared with your pre-lab write-up.**
- **No labs and lecture during Thanksgiving week; no lecture 12/6**

BLOCK 1 (notebooks due in lab 1st week of Block 2: 10/13,14).

Exp 56: Multistep Synthesis of Benzilic Acid; IR; Exp I: Multistep synthesis of a triazole (Exp IA: Synthesis of a propargyl ether; Exp IB: Click Chemistry: Cu-catalyzed alkyne-azide [3+2] cycloaddition); Exp. II. Identification of an Unknown (report due Nov. 17,18)

Week 1 9/8,9 Before lab: Read section “Laboratory Safety” in text (p. 13-30). Read Safety instructions; During lab: Safety quiz; Check-in; Brief lab work: Exp. 56A, benzoin condensation, mix reactants, titrate, then leave for next week.

Week 2 9/15,16 Finish Exp. 56A; start Exp. 56B

Week 3 9/22,23 Finish Exp. 56B; IR: KBr disks. Start Exp. 56C. Receive Unknown.

Week 4 9/29,30 Exp. IA: Prepare 4-acetylphenyl propargyl ether. Confirm purity of product by TLC; IR (KBr disc) (*Handout*); Finish Exp. 56C. IR of product; Exp. 56 exercise questions 1-3,6,7. Exp. II: Start Identification of Unknown (*Handout*).

Week 5 10/6,7 Exp. IB: One-pot Cu-catalyzed azide alkyne cycloaddition (*Handout*). TLC & m.p.; NMR. of product. Finish everything for Block 1;

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

BLOCK 2 (notebooks due for grading 1st lab of Block 2: 11/10,11).

Physical Organic Chemistry - Exp 44A: Prep. and use of benzene diazonium salts; Exp 44B: pKa values and linear free-energy relationships; Polymer Chemistry: Polystyrene; Nylon (NMR; Size-Exclusion Chromatography); Exp. III. Suzuki Coupling; Start essay.

Week 6 10/13,14 Polymer chemistry: Exp. 26; Polystyrene; exercises 1,2,3a-b,5-7; Minilab 20: Nylon 6.10; IR of products; Endgroup analysis by ¹H NMR, GPC analysis of polydispersity (*Handout*); Hand in lab books to TA for Block 1 grading.

Week 7 10/20,21 Exp. 44A: Electronic effect of a *para*-iodo substituent & linear free-energy relationship (group study). Continue Unknown

Week 8 10/27,28 Exp 44B: Determine pKa values & linear free-energy relationship. Exp. 44 exercise questions 1-3, 6-9. Continue Unknown

Week 9 11/3,4 Exp. III: Organometallic chemistry – Suzuki coupling (*Handout*); TLC, m.p., ¹H NMR, and H,H COSY of product; Finish everything in block 2.

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

BLOCK 3 (notebooks due last lab of Block 3: 12/8,9).

Exp IV: Organocatalysis – asymmetric aldol reaction; Column Chromatography; TLC; Chiral HPLC; Exp. V: Regiochemistry of enone reduction

Week 10 11/10,11 Exp. IVA: Start: Proline-catalyzed aldol reaction (*Handout*).
Submit (Canvas) typed plan for NaBH₄ reduction study for evaluation and feedback: one plan per group. Hand in lab books to TA for Block 2 grading.

Week 11 11/17,18 Exp. IVB: Column chromatographic purification of aldol product (*Handout*). ¹H, ¹³C, COSY of aldol. Chiral HPLC. Mosher Ester Analysis.
Submit final corrected plan for NaBH₄ reduction study for grading.
Unknown report due this week (Canvas)

Week 12 12/1,2 Exp V: NaBH₄ reduction of *tert.*-butylcyclohexanone – a study of the effect of CeCl₃ on regioselectivity (*Handout*).

Week 13 12/8,9 Analysis of NaBH₄ reduction products by IR and NMR. Finish everything in Block 3; Check-out. Essay due this week (Canvas).
Notebooks and lab reports (Canvas) due to TAs by end of lab.

Exam 3 – December 22 at 3:50 pm in SEC202 (Block 3 topics plus general methods, spectroscopy)

II. REQUIRED MATERIALS

- 1. Textbooks** *Operational Organic Chemistry*, 4th edition, John Lehman, Pearson/Prentice Hall, ISBN-13: 978-0-13-600092-1 or *Multiscale Operational Organic Chemistry*, 2nd edition, John Lehman, Pearson/Prentice Hall, ISBN-13: 978-0-13-241375-6. This book contains both microscale and standard scale versions of the experiments. We will be using the standard scale (SS) procedure for all experiments.
Handouts for experiments **IA/B, II, III, IVA/B and V.**
- 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.
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](https://sims.rutgers.edu/ssra/). <https://sims.rutgers.edu/ssra/> 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:	10/11/2022	3:50-5:10pm	SEC202	Block 1 topics & IR, NMR	100 pts
EXAM 2:	11/08/2022	3:50-5:10pm	SEC202	Block 2 topics & IR, NMR	100 pts
EXAM 3:	12/22/2022	3:50-5:10pm	SEC202	Block 3 topics & IR, NMR	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. 530 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. 56A-C: 100; Propargyl ether & Click reaction: 60; Polymer chemistry: 50; Exp. 44A/B: 60; Suzuki coupling: 45; Asymmetric aldol reaction: 60; NaBH₄ reduction: Procedure 20, Experiment 35; Unknown 100.

2. 50 pts Lab Quizzes. At the beginning of every lab (except during the first and last week), 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. **Please note**, that the safety quiz during Week 1 will not count as lab quiz.
3. 300 pts Three Exams. The three exams given at 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, military obligations). 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 submit 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 producing excess waste, your attention to wearing goggles and other safety issues, and how clean you keep your bench & hood and leave the common lab equipment/instrumentation after use.
5. 20 pts Library Homework Assignment. Submission via Canvas. 5 points per day will be deducted for each day that you submit late. The guest lecture by RU Librarian Ms. Laura Palumbo on 10/25/22 will provide the background information for assignment.

NOTE: (a) If you hand in your notebook/submit your lab report 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 Appendix II, III, IV and V in the text).

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 ‘ChemDraw’, for which Rutgers University has a site license (Handout with instruction how to obtain the program is on Canvas). 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 ChemDraw. Submit your report via Canvas.
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 outlined 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>.