

ORGANIC CHEMISTRY LAB 309

SPRING 2021

For CHEMISTRY (160), BIOCHEMISTRY (115 and 694), and CHEMICAL ENGINEERING (155) MAJORS, or by special permission. (This is the first semester of the two-semester organic lab sequence 309-310, for chemistry majors)

Important! You cannot take chem 309 without already having had one full semester of an organic chemistry lecture course (Chem 307 or Chem 315).

Lecturer and course Coordinator: Leslie Jimenez, Wright-Rieman Labs, room 323.

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Office Hours: Friday 10:00 am - 12:00 pm (noon)

Lecture for all sections: Tuesday 5:00 - 6:20 pm, SEC-118

All Labs in WL331:

- Section 01, Th 1:00 - 5:20 pm
- Section 02, W 6:30 - 10:50 pm
- Section 03, M 6:40 - 11:00 pm
- Section 04, F 1:00 - 5:20 pm
- Section 05, M 1:00 - 5:20 pm
- Section 06, W 1:00 - 5:20 pm
- Section 07, T 6:40 - 11:00 pm

Course web site: located on sakai (<https://sakai.rutgers.edu/portal>).

I. SYLLABUS

1. 1/25-1/29 Exp. 13 - Investigation of a Chemical Bond by IR (Exercise 4).
Start Exp. 16 - Separation of an Alkane Clathrate. Exercises 1-3, 6.
 2. 2/1-2/5 Start Exp. 47 - Synthesis of Dimeedone and Measurement of Its Tautomeric Equilibrium Constant (mp and NMR). Exercises 1, 2, 6, 10. Complete Exp. 16.
 3. 2/8-2/12 Complete Exp. 47.
 4. 2/15-2/19 **FIRST LAB REPORT COLLECTION (bundled lab reports due in lab to grade Expts. 13, 16, 47).** Start Exp. 30 – The Synthesis of Triphenylmethanol and the Trityl Carbocation (no mp). Exercises 1, 3, 6, 7.
 5. 2/22-2/26 Finish Exp. 30. Start Exp. 18 – Structures and Properties of Stereoisomers. Exercises 1, 2, 5, 6, 7.
 6. 3/1-3/5 **SECOND LAB REPORT COLLECTION (bundled lab reports due in lab to grade Expts. 30 and 18).**
- 3/2 **FIRST EXAM** covers Exp. 13, 16, 47, IR, and NMR.

7. 3/8-3/12 Check-in. Preparation for lab-week 2: Read pp. 2-30. Read Exp. 1A, pp. 40-45 (this is a read-only expt). Answer Exercises 1, 2, 4, 5, 7, pp. 46-47 in your lab notebook. Read OPs 1, 4-7, 15-19, 25. Do your pre-lab write-up for Exp. 2 in your lab notebook or type onto paper that you will tape or staple into your lab notebook. Exp. 2 - Extraction and Evaporation. Separating the Components of "Panacetin". Exercises 1-5. **Safety quiz.**

SPRING BREAK

8. 3/22-3/26 Exp. 3 - Recrystallization and Melting Point Measurement. Identifying a Component of "Panacetin" (Exercises 1-4); Exp. 33 - Spectral Identification of Monoterpenes (IR only). Exercise 2. Read pp. 846-875.
9. 3/29-4/2 **THIRD LAB REPORT COLLECTION FOR GRADING (bundled lab reports due in lab to grade Expts. 1A, 2, 3, 33-IR).** Exp. 4 - Heating Under Reflux. Synthesis of Salicylic Acid from Wintergreen Oil. Exercises 1, 2, 4-6.
10. 4/5-4/9 Finish Exp. 4. Start Exp. 5 - **Simple** Distillation, NMR and IR analysis. Preparation of Synthetic Banana Oil. Exercises 1, 2, 3b, 4, 5. "Investigating Free Energy Relationships Using Gas Chromatography" (not in book).
11. 4/12-4/16 Finish Exp. 5 distillation and GC experiment.

4/13 SECOND EXAM (covers Exp. 30, 18, 1A, 2, 3, 33, and IR)

12. 4/19-4/23 Exp. 15 - Thin-Layer Chromatographic Analysis of Drug Components (Exercises 1-3). Start "Identification of Unknown by IR and NMR" (not in book). This will continue over several weeks, when your time allows and as directed by your TA.
13. 4/26-4/30 **FOURTH LAB REPORT COLLECTION (bundled lab reports due in lab to grade Expts. 4, GC, 5, 15).** (Not in book) – Nitration of Vanillin to form 5-Nitrovanillin. Synthesis, purification, mp, TLC analysis, and column chromatography of 5-nitrovanillin (see handout).

4/27 NO CLASS

THIRD EXAM covers Exp. 4, GC, 5, 15 during finals week.

II. REQUIRED MATERIALS

- 1. Text** *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. The first lecture is on Tuesday, Jan. 21. Lab classes begin with check-in and experiment 2 on Monday, January 27. Please come prepared with your pre-lab write-up.
- 2. Notebook** A spiral-bound lab notebook with perforated carbonless duplicate pages is required. The Rutgers bookstore, as well as sources such as amazon.com and barnesandnoble.com sell different versions of this type of lab notebook with 50, 75 or 100 pages. During the weeks of lab notebook collection, you will hand in the original pages (staple each experiment together and clip your experiments into one bundle for your TA) and you keep your notebook with the duplicate pages for your own reference. No 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.
- 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 the second week.
- 5. Markers** A permanent magic marker is highly recommended for labeling your samples.

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 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. If you are sick or have another valid excuse, get in touch with your TA and with Prof. Jimenez 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. However, a doctor's note or a note from your Dean of Academic Affairs will be required as evidence.

V. GRADING

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

1. 500 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. **1A** (exercises only): 10; **2**: 30; **3**: 35; **33**: 15; **4**: 35; **GC**: 25; **5**: 40; **15**: 20; **5-nitrovanillin**: 40; **13**: 15; **16**: 40; **30**: 50; **47**: 50; **18**: 40. In addition, the unknown analysis and identification is worth 55 pts.
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 Operations section. Of these 11 quizzes, the lowest score will be dropped.
3. 350 pts Three Exams. Two exams given during the lecture class which will be worth 100 points each. The third exam will be worth 150 points. 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. Jimenez as soon as you are able, and certainly within one week of the exam you miss. A doctor's note or a note from your Dean of Academic Affairs will be required as evidence.
4. 100 pts TA evaluation. Your TA will assess your performance in the lab in terms of your basic understanding of the experiments, 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.

NOTE: (a) If you hand in your notebook late with no valid excuse, you will lose 3 points for every day late. This includes weekend days and Spring break!
(b) Successful completion of the labs with completed notebook write-ups should normally be sufficient for at least a C grade, unless performance in the exams is extremely poor.

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 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 use the following format.

1. Use a lab notebook with lines or graph paper, and number the pages as you go in the upper outermost corner of each page. Leave the first few pages for a Table of Contents, and keep this table up-to-date throughout the semester. Use the righthand pages (odd-numbered pages only).
2. Pre-Lab Write-up. This section of your lab report must be made IN INK, and cannot be typed. Before coming to 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. Typically one sentence.

- c. Under the heading **Equations**, balanced equations for all reactions (if any).
- d. Under the heading **Mechanism**, write complete detailed mechanisms for all reactions (if any). Use curved arrows to show electron movements.
- e. Under the heading, **Amounts and Properties**, data for all reactants and catalysts (molecular weight, grams (or milligrams) to be used, moles (or millimoles) to be used, relevant physical constants); solvents (volumes to be used); and products (molecular weight, theoretical yield in grams and moles, relevant physical properties). Please organize this information into a table. See sample headings below:

Chemical	Mol. Mass	Amt (g, mg or mL)	Actual amt (g, mg or mL)	mmol	Mp/bp	Density (g/mL)	Theoretical yield

- f. **Hazards & Safety:** Include how to safely handle & dispose hazardous chemicals.
- g. Under the heading **Procedure**, a brief description of the steps of the experimental procedure, either as a list or as a flow chart. THIS IS THE PROCEDURE YOU WILL FOLLOW WHEN YOU DO YOUR EXPERIMENT IN THE LAB. YOU WILL NOT USE THE TEXTBOOK! Do not write paragraphs, or even full sentences. Your outline should be complete and unambiguous and contain all essential details, such that you will be able to perform the entire experiment from it. It should contain no extra words to distract from the essential procedures. Your TA or any trained organic chemist should be able to repeat the experiment in exactly the same way that you did it, by following your procedure and the notes that you make during the lab (see #3, below).
- h. If not already covered above, do what is asked in the "Before You Begin" section of the experiment.

For experiment 2 only: Hand in your pre-lab (original pages) at your first lab meeting. Your TA will grade it and return it to you the following week.

For the weeks when grading occurs, hand in the original pages from your notebook. Bundle your lab reports together for your TA. You keep your lab notebook with the duplicate pages.

NOTE: If you fail to prepare your pre-lab or bring your lab notebook to your lab session, you will be told to leave and prepare the pre-lab and may be given a zero grade for that lab.

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 headings **Calculations, Discussion and 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. This portion of your lab report should be TYPED. The quality of your discussions will be the most important factor determining 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. 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." To assist the TAs in grading your notebook, **Calculations**, **Discussion** and **Conclusions** should be typed. Copies of your **Calculations**, **Discussion** and **Conclusions** pages can be stapled or taped into your notebook to give you a completed copy of your lab report.

5. After the conclusion, answer the exercises, if any (see syllabus). This section should also be typed to assist the TAs in grading. Your pre-lab, **Observations and Measurements** page(s), **Calculations**, **Discussion** and **Conclusions** pages, and answers to the assigned Exercises should be stapled together to make up your lab report.
6. 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 your discussion write-up and for your TA when grading. 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, see:
<http://academicintegrity.rutgers.edu/>