

ORGANIC CHEMISTRY LABORATORY I
(CHEM 117) 2.0 CREDITS
Summer 2021

Instructor: Dr. Ron Davis, Jr.
voice: 202-687-3566
email: rbd34@georgetown.edu
Course Website: canvas.georgetown.edu

Text: Basic Organic Laboratory Techniques – portions provided.

Teaching Assistants

TBA

Course Objectives:

- To become familiar with basic techniques for isolating and characterizing organic compounds
- To become familiar with searching, using and citing the chemical literature
- To become familiar with aspects of technical writing as they apply to synthetic organic projects
- To become familiar with synthetic techniques by completing substitution and elimination reactions

Semester Schedule

	Day	Lab	Assigned Reading	Procedure	Other
Week 1	Tues	Introductory Lecture Review Lab Safety and Glassware	Safety Contract Online	None	
	Wed	Labs 1 & 2 Lecture	Davis Ch1,2	None	Lab equipment and safety quiz
	Thur	Melting Points and Recrystallization	Davis Ch3	CHEM117-01	Lab 1 quiz
	Fri	Boiling Points and Distillation	Davis Ch4	CHEM117-02	Lab2 quiz
Week 2	Tues	Labs 3 & 4 Lecture			Write-up 1&2 due
	Wed	TLC of Analgesics	Davis Ch 6,7	CHEM117-03	Lab 3 quiz
	Thurs	Acid-Base Extraction	Davis Ch 5	CHEM117-04	Lab 4 quiz
	Fri	Acid-Base Extraction (continued)		CHEM117-04	
Week 3	Tues	Labs 5 & 6 Lecture			Write-up 3&4 due
	Wed	Column Chromatography of Plant Pigments		CHEM117-05	Lab 5 quiz
	Thurs	Separation of enantiomers <i>Intro to Polarimetry</i>	Davis Ch 7 Stephani and Cesare <i>J Chem Ed</i> 1997	CHEM117-06	Lab 6 quiz
	Fri	Lab makeup day and mid-semester review			
Week 4	Tues	Labs 7 – 9 Lecture			Write-up 5&6 due
	Wed	Literature and Drawing	Davis Ch 8,9	CHEM117-07	Lab 7 quiz
	Thurs	Substitution Reaction: Solvolysis of <i>t</i> - butyl bromide	McMurray 11.4, 11.5	CHEM117-08	Lab 8 quiz Assignment 7 due
	Fri	Elimination Reaction: Alkenes from Alcohols	McMurray 7.1, 11.7, 17.6	CHEM117-09	Lab 9 quiz
Week 5	Tues	Review Session			Reports 8 & 9 due
	Wed	Study Day			
	Thurs	Lab Exam			Labs 1-9 covered

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Grading Scale:

10 pre-lab/safety quizzes	15%
6 Laboratory Worksheets/Write-ups:	42%
Literature and Drawing Assignment	7%
2 Laboratory Reports:	20%
<u>Lab Exam:</u>	<u>16%</u>
Total	100%

Course Policies

Course safety and administrative policies are outlined in separate documents. Please be sure read all of the following documents (available in the 'Course Documents' section of Canvas) to be sure that you fully understand all course policies:

CHEM117-00 – Safety Contract.pdf
CHEM117-00 – Online Submission Instructions.pdf
CHEM117-00 – Waste Handling.pdf
CHEM117-00 – Report Writing Guidelines.pdf
CHEM117-00 – Academic Integrity Guidelines.pdf

Laboratory Meetings

Laboratory meetings will take place on Wed/Thu/Fri. After an initial pre-lab quiz, students will participate in a live experiment conducted by the professor or a video review. Real-time attendance at these laboratory meetings is mandatory.

Procedures and Worksheets

Procedures, background reading, recitation slides and worksheets will be posted to Canvas on the weekend prior to the associated recitation. Students should review all pertinent materials before logging in to Zoom for Tuesday recitation.

Quizzes

Safety and pre-lab quizzes will post to Canvas at the beginning of the associated laboratory meeting. Quizzes and must be completed during the first 15 minutes of each lab period while logged into the course meeting. Each quiz is worth 1.5% of your final course grade.

Recitations

Recitations will be held live in Zoom web conferencing. During this time we will discuss the underlying principles, procedures and safety issues related to the week's experiments. Recitation attendance is mandatory.

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Lab Exam

The lab exam will be a common-time, 30-question, multiple choice exam covering all nine experiments as well as general lab safety and hygiene.

Worksheets and Reports

Each of the first seven experiments (1-7) will require students to complete and submit a worksheet guiding them through data analysis and prompting them to write a 500 word discussion of the goals, techniques and results.

The final two experiments (8 and 9) will require students instead to write a full scientific report following the guidelines and practices discussed during experiment 7 (Literature and Drawing).

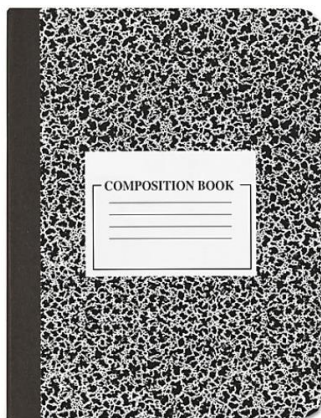
All worksheets, post-lab questions and grading rubrics will be provided via Canvas prior to the associated experiment.

Experiments

All experiments will be conducted by Professor Davis and streamed synchronously with students via Zoom conferencing. Although these experiments will be recorded, students are required to attend the live stream unless they have been granted an excused absence by the professor.

Notebooks

Documentation of experiments is a critical laboratory skill. Although students will not be performing experiments directly, students will be required to maintain a laboratory notebook as though they were conducting the experiments themselves. Copies of notebook pages must be submitted a part of each lab assignment. A new composition book with sewn pages (pictured below) is sufficient for this task.



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Summary of Experiments

Experiment 1 – Melting Points and Recrystallization

We will explore how to accurately measure the melting points of crystalline organic compounds and how to use the observed melting behavior to identify compounds and assay their purity.

We will consider how recrystallization can be used to purify crystalline organic compounds, improving the purity of a benzoic acid sample by recrystallization from water.

Experiment 2 – Distillation and Boiling Points

We will investigate the effect of pressure and purity on the boiling points of organic liquids. We will study how fundamental chemical laws like Raoult's, Dalton's and Ideal Gas Laws predict that the vapor above boiling mixtures of miscible organic liquids can change in composition. We will consider various distillation apparatus designs, their benefits, and limitations.

We will use distillation to increase the strength of an alcohol solution for use in hand sanitizer. We will test the products of both simple and fractional distillation to verify the benefits of the fractional distillation technique. We will also use the technique to identify the components of an unknown mixture of two miscible organic liquids.

Experiment 3 – Thin Layer Chromatography

We will begin by covering the fundamental concept of partitioning and how this behavior is exploited in a number of chromatographic purification strategies.

We will then analyze some over the counter analgesic products that contain non-volatile organic compounds using the technique of thin-layer chromatography.

Experiment 4 – Liquid-Liquid Extraction

We will see how partitioning can also be used to isolate and purify compounds using two immiscible liquid phases. The influence of solution pH on extraction will be investigated.

We will measure the partitioning coefficient of 2-naphthol between an organic phase and two different aqueous phases using a special apparatus called a 'separatory funnel'. We will use our results to illustrate the impact of the aqueous layer pH in this technique.

Summary of Experiments (cont.)

Experiment 5 – Column Chromatography

We will consider the application of chromatography to large-scale separations, and how scaling up a chromatographic separation requires special equipment and techniques.

We will extract the pigments (colored organic compounds) from commercially available spinach before separating them on a chromatography column. The relationship between pigment molecular structure and the order of their elution from the column will be characterized.

Experiment 6 – Polarimetry and chiral separations

The technique of refluxing, which allows the indefinite boiling of solutions, will be covered in the context of a stereospecific recrystallization of a racemic sample. We will construct a polarimeter and measure the specific rotation of aspartic acid – an essential amino acid compound.

Experiment 7 – Chemical Literature and Drawing

During this exercise we will learn to use the CambridgeSoft product ChemDraw to create depictions of structures, schemes and mechanisms using industry practices. We will also explore chemical research literature and the databases scientists use to retrieve relevant papers and reviews.

Finally, we will consider how scientific papers are organized using specific examples from the chemical literature.

Experiment 8 – Physical Organic Chemistry

We will review the science of physical organic chemistry and how fundamental physical equations and the Hammond Postulate can all be applied to probe the mechanisms of organic reactions. We will consider the solvolysis of t-butyl chloride, a fairly straightforward organic reaction whose mechanism can be elucidated by measuring the rate of the reaction under various conditions.

Experiment 9 – Using Equilibria in Synthesis

We will begin by discussing whether using an equilibrium reaction in synthesis is a proposition worth considering. We will explore how application of LeChatelier's principle and thoughtful apparatus design can overcome the yield challenges posed by equilibria.

We will prove our concept by dehydrating cyclohexanol (a disfavored equilibrium) in a fractional still and achieving a yield well in excess of that predicted by a R.I.C.E. table calculation.