

CHEM X36.A1 ORGANIC CHEMISTRY LABORATORY I



Organic Chemistry Laboratory I (2 units) is the first semester of a two semester series offered by the UC Berkeley Extension program taught at Cañada College from 09/01/16 through 12/15/16 with a final exam on 12/15/16.

Description:

This course will cover a variety of fundamental laboratory techniques applicable to the study, separation, purification and preparation of organic compounds. Designed to support biology majors; premedical, prehealth and returning students; and professionals interested in chemistry. This course is associated with the lecture course, Organic Chemistry I CHEM X36A. This course is transferable to the UC and CSU systems.

Prerequisite Studies:

- One year of college-level general chemistry with laboratory.
- Organic Chemistry I CHEM X36A must be taken concurrently or already completed with a C or better grade.

Materials:

- Textbook: <u>A Microscale Approach to Organic Chemistry Laboratory Techniques</u> (5th ed) by Pavia, Kriz, Lampman & Engel; Brooks Cole / Cengage Publishing ISBN: 978-1-133-10652-4
- Internet access will be required to access answer keys, lecture slides, practice problems, and other resources.

• To the second and every subsequent meeting students must bring:

- Laboratory safety goggles (can be purchased at the campus bookstore) required for all experiments. (be sure these are chemically resistant, "shop goggles" will dissolve like sugar in boiling water if there is a lab accident)
- A spiral bound notebook. It will be needed to for doing pre-lab preparations, calculations in class, taking notes, and recording experimental data.
- Pencils (two of them) with an eraser.
- A simple scientific calculator. The calculator must allow scientific notation, log and square root functions. *Anything more complicated is a not needed and will likely trip you up. Keep it simple.* Cell phones, PDAs, smart phones, laptops, and other personal electronics devices are not allowed during exams or quizzes and may be damaged by chemical fumes or spills in a chemistry lab. Do not expect to use one in place of a calculator.

CLASS STRUCTURE

Instructor:

The Fall 2016 class will be taught by Nick DeMello. Nick received his Ph.D. in synthetic organic chemistry from the University of Pittsburgh and his B.S. in chemistry from the College of Chemistry at UC Berkeley. Nick has been teaching chemistry at Cañada College since 2007. He can be reached by email. Additional class resources are available at the URL below.

Instructor eMail: <u>nick@chemlectures.com</u> Class Website: <u>http://chem.ws/36a</u>

Meetings:

Class will meet from **6:00pm to 10:00pm (4 hr) Thursdays, in room 311 building 18** at Cañada College. A complete schedule, including exam dates and holidays is included.

Attendance is required—not attending the class means not having taken it. You must sign the daily attendance sheet to have your presence recognized—not signing the sheet is the same as not attending class. Two or more excused absences may result in the student being dropped from the class.

Meetings will most often begin with a pre-lab quiz to assess students understanding of topics explored in the previous meeting and to determine students preparation for that day's experiment. The quiz will then be discussed and may be returned to the students before the end of the meeting. There are no makeup quizzes.

The instructor will provide an introduction to the days topics and lead a discussion that may involve their history, definitions, components and the laboratory techniques or strategies derived from or based on them. This discussion is also an opportunity for students to explore related topics from the companion lecture course. This discussion will conclude with the presentation of a laboratory experiment intended to illustrate and explore those topics.

ACTIVITIES

Safety:

Chemistry labs are dangerous. The chemicals we employ and study are interesting because of their tendency to change one substance into another. As a result, <u>almost every substance you will work with is either corrosive</u>, <u>toxic</u>, <u>explosive</u>, <u>carcinogenic</u>, <u>combustible</u>, <u>mutagenic</u>, <u>or otherwise dangerous</u>.

Students are required to view a safety video and sign a contract detailing the college safety policies. Students are required to achieve a passing grade on the subsequent safety quiz before conducting any experiments. Students who disregard any laboratory safety policy at any time in the semester will be asked to leave the lab and will earn no points for that days activities. At the discretion of the instructor, 10 points may be removed from the students lab safety score as a one time warning instead.

Safety policies include (but are not limited to):

- Safety goggles must be worn for all experiments, unless the instructor specifically tells you otherwise.
- Contact lenses cannot be worn in the lab. They will trap lab vapors which may cause blindness and they prevent any medical assistance if a chemical splash occurs. Jewelry is discouraged and may be damaged.
- Students must wear clothes that adequately covers legs, arms, and feet. No exposed skin below the waste or exposed shoulders. No open toed shoes, no shorts, no sleeveless shirts—no exceptions.
- No draping or baggy clothing. Long hair must be tied back. Most lab fires start in loose hair or clothing.
- No food or drink is permitted in the lab or in the halls outside the lab—ever. While there is adequate ventilation, most foods aggressively absorb lab vapors and can quickly become toxic in a chemistry lab.

Experiments:

The last three of hours of the meeting will most often involve an experiment in organic chemistry. The experiments expected for this semester (subject to change) are listed on the included schedule and detailed in the textbook. Experiments will be conducted individually or in pairs, depending on the experiment. Each experiment requires dedicated lab space, special equipment and materials. It is not possible to "make up" a missed experiment.

A pre-lab assignment will be presented the week before the experiment. Students are required to read through the experiment description in their textbook prior to the class meeting. They are required to complete this assignment and bring it to the meeting. Pre-lab assignments generally involve calculations, questions and other preparation required to effectively and safely conduct the experiment. Students are required to individually (even when working in a group) record all experimental data in their own notebooks and insure they have their individual copies of all spectra (or other instrument print outs) for their post-lab report.

Students who do not demonstrate sufficient preparation on the pre-lab quiz or who fail to complete the pre-lab portion of their report before coming to lab may not be allowed to participate in that days lab activity.

Reports:

Students are required to individually produce a post-experiment report for each experiment. The report is due at the beginning of the period, one week after an experiment is concluded. All lab reports require a cover sheet (a generic cover sheet is available on the class website). Your pre-lab assignment must be attached as the first section of your report. Post-lab analysis (calculations) and conclusions will also be required. More information about specific reports will be provided in the pre-lab discussion.

Students may submit reports only for activities in which they participated. If you did not do the experiment, you cannot report on the experiment.

Exams & Quizzes:

Exams dates are included in the attached class schedule. Write these dates into your calendar now. Make up exams are not possible. Exams cannot be taken early. Students are required to bring a scientific calculator, pencils (2), and an eraser to each period, these will be needed for exams and quizzes. No scantron forms or blue books are required. A periodic table will be provided. No other resources (books, notes, etc) are permitted. The final exam is comprehensive and is required. Not taking the final exam will result in a failing grade for the course.

GRADES

Assessment:

There is no curve. There is no extra credit. Your letter grade in this class is determined from a percentage of total points achieved to total points possible. Points are achieved through performance on exams, quizzes, experiment reports and lab safety. The total points expected to be offered this semester is approximately 400.

possible		percent		
60	Midterm Exam (60 pts)	15%	Exame 40%	
100	100 Final Exam (comprehensive; 100 pts)		LXa1115 40 /0	
100	Quizzes (best 10 scores; 10 pts each)	25%		
120	Reports (12 experiments; 10 pts each)	30%	Other 60%	
20	Lab Safety (20 pt safety quiz less any safety penalties)	5%		
400	total	100%		

Students who achieve 90% or more of the available points will receive an A grade. Students achieving less than 90% but 80% or more of available points will receive a B grade. Less than 80% but 70% or more of available points is a C grade. Producing between 70% and 60% will receive a D grade.

100-90%	89-85%	84-80%	79-75%	74-70%	69-60%	< 60%
А	B+	В	C+	С	D	F

Where allowed by campus policy, a "+" prefix will be attached to B and C grades when a student earns points in the top half of each respective range. Students who achieve less than 60% of the total possible points or who fail to take the final exam will receive a failing grade for the class.

Grade Options (Petition for Grade Change):

The default grading option is CLG (letter grade). Other grading options may include P/NP (pass not pass), NC (not for credit) and W (withdrawal). The deadline for changing grading options is the last class meeting. Withdrawal (W grade) from the class must be initiated by student.

Petition for Grade Option Change Form:http://extension.berkeley.edu/upload/grade_option_change.pdfPetition to Withdraw Form:http://extension.berkeley.edu/upload/petition_to_withdraw.pdf

If you opt to change your grade option, you must inform your instructor as follows. The default for all student's is to receive a letter grade. If you opt to change your grade option to a pass/no pass basis (P/NP) or a noncredit basis (NC), you must complete and submit the grade option change form above to your instructor before the last class meeting. Extension will not accept any late grade option change form and cannot change a P/NP grade or NC grade option to a letter grade after recording it.

- Passed and Not Passed (P/NP): Passed/Not Passed can only be assigned to students who complete the requirements for credit. The student must have earned at least a "C-" to receive a Passed (P) grade.
- Not for Credit (NC): Not for Credit is assigned to students whose attendance is satisfactory and may not be assigned to students who stop attending class.

You may also submit a request to withdraw by logging into your student account and submitting the request on your enrollment history page before the last course meeting. Due to the hands on nature of laboratory coursework, "Incomplete" grades will not be granted to students in this laboratory course under any circumstances.

Other Grade Policies:

http://extension.berkeley.edu/info/grades.html

GENERAL POLICIES

Classroom Visitors:

Auditing is not permitted in UC Berkeley Extension courses. You must formally enroll in the course and pay all fees before the second classroom meeting.

Student Disability Services:

All students who have special needs can receive appropriate accommodations. The EXDSS office must determine or verify these accommodations before they can be offered. Students who are requesting academic accommodations are responsible for contacting the EXDSS Coordinator before your course begins or immediately upon the start of the course. Students may submit their request by email: extension-dss@berkeley.edu or phone: (510) 643-5732.

Important Deadlines:

Course Transfer: three business days after the first class meeting Drop Course: three business days after the first class meeting Withdraw or Grade Option Change: before the last class meeting

Academic Integrity and Student Conduct:

Academic misconduct is any action or attempted action that may result in creating an unfair academic advantage for you or any other members of the academic community. This misconduct includes a wide variety of behaviors such as cheating, plagiarism, altering academic documents or transcripts, gaining access to materials before they are intended to be available, and helping another student to gain an unfair academic advantage.

As a student of UC Berkeley Extension, you are encouraged to reach out to your fellow students in your class to avoid isolation, to discuss materials, and to ask each other questions, but there are limits to this collaboration. Please review the following document on academic integrity (http://extension.berkeley.edu/upload/ academic_integrity.pdf), which clearly defines what constitutes cheating, as well as plagiarism and other forms of academic misconduct. Students are also responsible for informing themselves about UC Berkeley Extension's Code of Student Conduct and its grounds for discipline (http://extension.berkeley.edu/static/studentservices/ policies/#conduct).

UC Berkeley Extension takes academic misconduct very seriously. Depending upon the nature of the incident, the academic disciplinary sanction may vary but can result in consequences such as a failing grade for the course or even suspension and dismissal.

Reasonable Accommodation for Religious Beliefs, Observations and Practices:

In compliance with Education code, Section 92640(a), it is the official policy of the University of California at Berkeley to permit any student to undergo a test or examination, without penalty, at a time when that activity would not violate the student's religious creed, unless administering the examination at an alternative time would impose an undue hardship which could not reasonably have been avoided. Please contact the Extension program office for more information.

Other Extension Policies:

Including Privacy, Nondiscrimination, Sexual Harassment, Safety and Security, Classroom Recording: http://extension.berkeley.edu/info/policies.html

SYLLABUS

Our goal in laboratory will be to present and explore some select techniques commonly used in organic synthesis and start you on the practice of organic synthesis. This semester we will focus on how to isolate and identify many of the families of organic materials you will be introduced to in the lecture companion course.

We will start with exploring how organic mixtures can be predicted to separate and how we can manipulate and benefit from that property using techniques like crystallization and extraction. We will expand on that discussion by introducing the practice of chromatography, both analytic applications (including thin layer chromatography) and preparatory (including column chromatography).

Techniques for isolating products from complex reaction mixtures and measuring the purity of those isolated samples will be explored. We will also discuss the challenges and opportunities of competitive reactions. Finally, we will begin a discussion of preparing new substances with organic synthesis. The techniques already presented will be tested as we look at how changing just a few atoms in a molecule radically alters it's properties. Students will introduce foundation functional groups to prepare, characterize and purify alkyl halides and olefins.

Then we'll bring it all together with the synthesis of a particularly useful substance: aspirin.

Topics:

Α.	Separations	C. Purification		
	1. Solubility		7. Purity	
	2. Crystallization		8. Distillation	
В.	3. Extraction		9. Characterization	
	Chromatography	D.	Preparation	
	4. Elution Mixtures		10. Alkyl Halides	
	5. Thin Layer Chromatography		11. Olefins	
	6. Column Chromatography		12. Aspirin	

These discussions and activities will require and assume students have knowledge presented in General Chemistry and Organic Chemistry Lecture I. (see course pre-requisites) But the lab is also a resource to support students in their related studies. Students are encouraged to ask questions and bring up concepts or challenges of chemistry they may be struggling with in their other chemistry classes.

Student Learning Outcomes

Upon successful completion of this class, a student should be able to:

- 1. Using molecular structure, predict whether a substance is more likely soluble in a polar or non-polar solvent.
- 2. Given a solution of organic substances, predict which component is most likely to crystalize.
- 3. Suggest a solvent to extract a single component from a given mixture.
- 4. Predict which of two elution mixtures is more likely to produce a clear separation between given substances.
- 5. Calculate the Rf of developed TLC plate.
- 6. Isolate a component from an mixture using column chromatography.
- 7. Purify a liquid by distillation.
- 8. Prepare an alkyl halide.
- 9. Predict the dehydration product of an alcohol.
- 10. Make and purify aspirin.

Disclaimer: Subject to change.





ORGANIC CHEMISTRY LABORATORY I

Снем х36 А1



(Subject to Change - 08.10.2016)

Week	Date	Theme Discussion Activity																			
1	9/1/16		Getting Started	Class Introduction Carbon Atoms and Organic Molecules Producing Lab Reports	Lab Tour Safety Rules, Contract & Quiz Locker Check-in																
2	9/8/16	s	Solubility	How molecular structure can be used to predict solubility and miscibility. Why some solutions will separate into a heterogenous sample.	Exp 2: Solubility																
3	9/15/16	Separation	Crystallization	Solutions at the limit of solubility can produce pure crystalline phases. How that effect can be predicted and utilized in the lab.	Exp 3A: Semimicroszcale Crystallization Exp 3C: Solvent Selection for Crystallization																
4	9/22/16		Extraction	Using solubility barriers to selectively pull a component from one solution into another.	Exp 4A: Extraction of Caffeine Exp 4B: Distribution of Solute in Immiscible Solvents																
5	9/29/16	aphy	Elution	How slight differences in solubility can produce big separations when solutions are passed through silica gel. How to tune this effect.	Exp 6A: Chromatography Exp 6B: Selecting Solvents for Elution																
6	10/6/16	Chromatogr	Thin Layer (TLC)	Using chromatography for analysis. Gas and thin layer techniques for identifying mixtures and their components.	Exp 12: TLC Identification of an Analgesic Drug																
7	10/13/16		0	0	0	0	0		0	0	0	0	0	0	Column Chromatography	The technique of column chromatography for isolating pure samples of other difficult to separate mixtures.	Exp 17: Chlorophyll & Carotenoid Pigments				
8	10/20/16	MIDTERM EXAM																			
9	10/27/16		Melting Point	Isolating and determining the purity of pure substances that compose common and valuable mixtures.	Exp 10: Isolation of Active Ingredients from Analgesic Drugs																
10	11/3/16	Purity	Sublimation	Taking advantage of other physical properties to isolate put materials. Exploring cold finger apparatus and sublimation.	Exp 13A: Isolation of Caffeine, p100																
11	11/10/16					-													Distillation	Using distillation to prepare pure samples and confirming their structure and purity with infrared spectroscopy.	Exp 15A: Essential Oils, Distillation of Oil of Cloves
12	11/17/16		Alkyl Halides	Preparing building block compounds from which more complicated substance can be constructed. Building alkyl halides.	Exp 23C: Prep of t-Pentyl Chloride																
13	11/24/16	Holiday - No Class Meetings																			
14	12/1/16	Prepar	Prepar	Prepa	Prepa	Prepa	Prepa	Prepa	Prepa	Prepa	Prepa	Prepa	Prepa	Prepa	Olefins	The dehydration of an alcohol to insert a foot hold, a double bond, in a carbon skeleton.	Exp 24: Prep of 4-Methyl Cyclohexene				
15	12/8/16		Synthesis	The story of willow bark. Designing, synthesizing, isolating and characterizing a better medicine by making a better molecule.	Exp 9: Synthesis of Aspirin																
16	12/15/16	FINAL EXAM -																			

Lab Textbook: A Microscale Approach to Organic Chemistry Laboratory Techniques (5th ed) by Pavia, Kriz, Lampman & Engel; Cengage Publishing ISBN: 978-1-133-10652-4 Lecture Textbook: Organic Chemistry by Brown, Iverson, Anslyn & Foote, Cengage Publishing, ISBN: 978-1-133-95284-8



Experiment 2: Solubility

How molecular structure can be used to predict solubility and miscibility. Why some mixtures will separate into heterogenous matter and others will form solutions.

We will be doing parts A through E of this experiment. We will not be doing part F.

Preparations

Read: Experiment 02 - Solubility (page 12) Technique 05 - Measurement Technique 10 - Solubility

- Do: Prepare your lab notebook:
 - · State experiment objectives (for each part).
 - · List materials used w/ properties (solvents used in previous experiments
 - Make a procedures bullet list (for each part).

Intended Learning Outcomes

- * Know that alkanes generally have the lowest mp, bp, viscosity and hardness of substances with equal molar mass.
- * Know that alkanes generally have the least solubility in water of substances with equal molar mass.
- * Know that the physical properties of alkanes are mostly due to their inability to participate in dipole-dipole and hydrogen bonding intermolecular forces.
- * Know alkanes and other substances unable to participate in dipole-dipole and hydrogen bonding intermolecular forces tend to be soluble (or miscible) in each other.
- * Know adding a carbonyl group to an alkane makes it a ketone or aldehyde,
- * Know carbonyl groups increase the polarity of a substance and allow it be a hydrogen bond receiver with substances (like water and alcohols) which are hydrogen bond donors.
- * Know adding a hydroxy group to an alkane makes it an alcohol.
- * Know hydroxy groups allow a substance to be both a hydrogen bond donor and receiver.
- * Predict the relative physical properties of substances (mp, bp, viscosity and hardness) of two substance by whether their molecular structures contains carbonyls or hydroxy groups.
- * Predict in which solvent a substance will have greater solubility (or miscibility) by whether the molecular structures of solvent and solute contains carbonyls or hydroxy groups.

Report

Prepare a report for this experiment according to this experiments report description for the parts we accomplished. Include the questions with answers for this experiment, except any your instructor tells you to omit.



Experiment 3: Crystallization

Solutions at the limit of solubility can produce pure crystalline phases. How that effect can be predicted and utilized in the lab.

We will be doing parts A and C of this experiment. We will not be doing part B.

Preparations

- Read: Experiment 03 Crystallization (page 22) Technique 08 - Filtration (section 8.3 and 8.5) Technique 09 - Physical Constants of Solids, Melting Point
- Do: Prepare your lab notebook:
 - · State experiment objectives (for each part).
 - · List materials used w/ properties (solvents used in previous experiments
 - Make a procedures bullet list (for each part).

Intended Learning Outcomes

- * Know crystallization produces a solid phase of a solute from solution.
- * Know chemists use crystallization as a way to get pure substances from reaction mixtures.
- * Know chemists accomplish crystallization by varying a property of a solution (temperature, polarity, pH or other) to slowly reduce the solubility of a particular solute.
- * Know the technique of crystallization requires finding a property which has a greater effect on the solubility of your desired solute, than it has on other solutes (impurities).
- * Know the melting point of a mixture is always less than the melting point of either of it's components when they are pure.
- * Know you can determine if two samples with similar melting points are the same substance by a mixed melting point test.
- * Know a mixed melting point test is accomplished by comparing the mixed melting point of the two samples to the pure melting points of both.
- * Predict the best solvent for crystallization of a particular substance based on solubility.
- * Predict the best solvent for crystallization of a particular substance based on how solubility changes with temperature.

Report

Prepare a report for this experiment according to this experiments report description for the parts we accomplished. Include the questions with answers for this experiment, except any your instructor tells you to omit.



Experiment 4: Extraction

Using immiscible mixtures to selectively pull a component from one phase into another. The same properties that cause a substance to crystalize, can cause it be pulled from one solution it is soluble in to another leaving undesired impurities behind.

We will be doing parts A and B of this experiment. We will not be doing parts C-E.

Preparations

Read: Experiment 04 - Extraction (page 34) Technique 12 - Extraction Essay - Caffeine

- Do: Prepare your lab notebook:
 - State experiment objectives (for each part).
 - · List materials used w/ properties (solvents used in previous experiments
 - Make a procedures bullet list (for each part).

Intended Learning Outcomes

- * Know extraction is the technique of separating a desired substance from a mixture using relative solubilities of two solvents.
- * Estimate which solvent a substance will likely be more soluble in by considering alkane/aryl groups, polar groups, hydrogen bonding groups, and acid-base groups of that substance.
- * Predict which of given immiscible solvent pairs are likely to be more useful for separating a given mixture.
- * Given the mass of solute recovered from a given volume of solvent, determine the mass volume concentration of that solution.
- * Know the distribution coefficient (also called factor or ratio) is the relative mass volume concentration of two immiscible phases at equilibrium.
- * Given the equilibrium concentration in mg/mL of a substance in two solvents, determine that substances distribution ratio between those solvents.
- * Use the distribution coefficient of a pair of immiscible solvents to determine how much of a given mass of solute can be extracted from a given volume of those solvents to a given volume of the other.

Report

Prepare a report for this experiment according to this experiments report description for the parts we accomplished. Include the questions with answers for this experiment, except any your instructor tells you to omit.