Organic Chemistry Laboratory Techniques I
CHM 2211L Syllabus and Rubric

Fall 2016

Welcome to Organic Chemistry Laboratory Techniques I. Your instructor is enthusiastic about working with you to meet the course objectives. This syllabus contains information that will be useful to you throughout the semester. Please return here as needed.

Instructor:

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Office Hours: By appointment only

GTA:

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Room: CHM 321 Chemistry Tutoring Center

Course Description (obtained from my.ucf.edu): The first semester of organic chemistry laboratory work used to provide an introduction to laboratory techniques used in organic chemistry laboratory settings. These techniques lead to two specific types of experiments to be performed: Investigative and Preparative.

Instructor's Specific Course Objectives:

In addition to teaching you organic chemistry, our goal is to teach you how to become independent scientists that are able to perform experiments, make conclusions based on data obtained and how that correlates or deviates from theory. After successful completion of this class you should be able to read dense journal articles, interpret data and visualize experiments allowing you the ability to change experimental parameters (in your chosen field of study) and see the broader impact on these experiments.

Required Materials:

- Required Text: Introduction to Organic Laboratory Techniques – A Small Scale Approach.Authors: Pavia, Lampman, Kriz, and Engel. 3rd Edition ISBN: 1-4390-4932-7.
- Organic Chemistry Lab Manual (Edward Kluger)
- Required Materials for the Lab: Bound notebook (composition), pen, **Safety glasses and Laboratory Coat!** No sandals or open-toed shoes are allowed.

Recommended Materials:

- Donald L. Pavia, Gary M. Lampman, George S. Kriz, James A. Vyvyan *Introduction to Spectroscopy, 4th Ed.* [Paperback] Cengage, ©2009 (ISBN: 9780495114789)
- Robert M. Silverstein, Francis X. Webster, David Kiemle *Spectrometric Identification of Organic Compounds, 7th Ed.* Wiley, ©2005 (ISBN: 978-0-471-39362-7)
  Quick Lab Techniques Guide
- The Organic Chemistry Laboratory Survival Guide, J. W. Zubrick ISBN: 9780470129326.
  Organic Basics
- Organic Chemistry as a Second Language, David Klein ISBN: 9780470284414.

Grading Scale/Assignment Breakdown:

		Grading Scale
Lab Reports for Experiments:	40%	**90-100% = A**
Quizzes:	20%	**80-89% = B**
Notebooks:	20%	**70-79% = C**
Final Exam:	20%	**60-69% = D**
Total Possible Percent:	100%	**Below 60% = F**

Late Policy: Assignments may be submitted up to one day late for a 20% penalty. Assignments submitted after the three-day period will not be accepted.

***ATTENDANCE IS MANDATORY!** Due to time and space limitations, formal documentation will be required to make up a lab at a later time.

Types of Experiments to be Performed:

- **Preparative Experiments:** These types of experiments involve converting one or more starting compounds into one or more products. Most of the experiments performed will be preparative experiments.
- **Investigative Experiments:** These types of experiments involve making observations and learning techniques that are common to laboratory work in organic chemistry but do not involve converting a starting material into a final product.

Grading Guidelines:

I. **Laboratory Reports: (40% of your grade)**
Seven lab reports will be assigned throughout the summer semester. **All will count** for the determination of final grades. That is to say there is no dropped lab report.

Only electronic submissions will be made through turnitin.com.
Reports must be typed documents and should include the following sections. For some sections there are examples provided.

A. **Introduction:** (18 points)

This section allows you to "put all of your cards on the table," by outlining all experimental goals and the theory that gives reason to the ability to do the experiment. The introduction should include the following points:

i. State specific goals to be accomplished through performing the experiment.
ii. Discuss the products and starting materials that will be obtained/utilized in the experiment.
iii. Discuss the theory that will allow the experiment to be a success (mechanism, properties of materials, etc.)
iv. Any skills or setups will be utilized in order to perform the experiment.
v. Any theory that is necessary in order to completely explain the results obtained through performing the experiment.

B. **Procedure:** (20 points)

This section is a complete description of the experimental procedure that was utilized in order to complete the experiment. Within this section, there should also be a description of any special observations made throughout the experiment performed (color changes, precipitations, etc.). This section should be written in the **third person passive past tense** and follow the format used in The Journal of American Chemical Society. This section can also include a flow chart of separation schemes in order to clearly outline what was done if the student deems it necessary.

Procedure Example in JOC format:

"A mixture of 9,9-diethylfluorene (1.11 g, 5.0 mmol),paraformaldehyde (0.33 g, 11.0 mmol), and 33% HBr solution in acetic acid (10 mL) was heated at 60-70 °C for 20 h. Upon cooling, the precipitates were collected by filtration, carefully washed with water, and dried in vacuo, affording 1.47 g of pale white solid (72%). Recrystallization from toluene/hexane gave an analytically pure sample. Mp 148-150°C"

Procedure Example in JACS format:

"A mixture of the primary amine 7 (9.9 g, 42.7 mmol), sodium iodide (19.4 g, 129 mmol), K_2CO_3 (17.8 g, 129 mmol), and 4-bromo-1-butene (6.5 mL,64 mmol) in DMF (150 mL) was heated to 100ºC for 10 h, cooled to room temperature, and poured into a separatory funnel containing Et_2O (200 mL)and water (200 mL). The organic layer was washed with water (2 x100 mL) and brine (100 mL), dried over $MgSO_4$, and filtered, and the solvents were removed using a rotary evaporator. The product was purified by flash chromatography (10:1 and then 4:1 hexane/ethyl acetate) to give 9.0 g (74%) of a colorless oil"

C. **Results:** (15 points)
This section includes all values, spectra, and calculations pertinent to the starting materials and products utilized/obtained in the experiment. The results section should include:

i. Calculations (input into equation builder) and values obtained for both starting materials and products (actual yield, theoretical yield, percent yield).
ii. Values important for the overall characterization of the product (boiling point, melting point, and any other relevant physical characteristics).
iii. Be sure to identify any limiting reagents and excess reagents in any chemical reaction occurring.
iv. Obtain literature standard spectra (SDBS) in order to compare it with obtained product or starting materials. Cite your sources!!!
v. ****IR spectra** must be described as follows: First, write a **table** with the most important peaks that you obtained including wavenumber (cm^{-1}), functional group, type of vibration, intensity (see table at the end of your textbook); then label on the spectrum exact assignments for peaks.
Hint: **Resist the urge to discuss or make conclusions here, save that for the discussion section.**

Results Example:

- Formula for **molar calculations:**

$$(mL\ of\ compound)\times(density\ (\frac{g}{mL}))\times\left(\frac{1}{molecular\ weight\ \left(\frac{g}{mol}\right)}\right)=moles\ of\ compound$$

  - Example: Anisole

$$(8.1513\ mL\ Anisole)\times\frac{0.995g}{1\ mL}\times\left(\frac{1\ mol}{108.14\ g}\right)$$

$$=7.5*10^{-2}moles\ of\ Anisole(Limiting\ reagent)$$

- Formula for **Percent (%) Yield:**

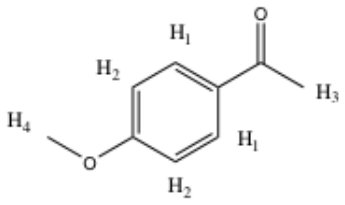
$$\left(\frac{Actual\ Yield\ (g)}{Theorectical\ Yield\ (g)}\right)\times100=Percent\ Yield\ (\%)$$

  - Example: Percent yield calculation of product (4-methoxyacetophenone)—

$$\left(\frac{3.89\ g}{11.26\ g}\right)\times100=34.5\ \%\ Percent\ Yield$$

					methoxyacetophenone*		
^{1}H-NMR (Experimental) in $CDCl_3$					^{1}H-NMR (SDBS) in $CDCl_3$		
Assignment	Chemical Shift region (ppm):	Peak (ppm)	Multiplicity	Integration	H number :	Peak (ppm) :	Multiplicity :
H_1	Aromatic (6.5-8.2 ppm)	7.827-7.809 ppm	doublet	1.89	H_1	7.929 ppm	doublet
H_2	H's on Aromatic ring (6.5-8.5 ppm)	6.827-6.809 ppm	doublet	1.86	H_2	6.929 ppm	doublet
H_4	$-CH_3$ (up-field, 3-4 ppm)	3.741 ppm	singlet	3.02	H_3	3.863	singlet
H_3	Methyl ketone (2-3 ppm)	2.556-2.041 ppm	singlet	3.00	H_4	2.564	singlet

Table 4: ^{1}H-NMR Spectroscopy values for product 4-methoxyacetophenone and values for the most likely compound identity predicted (SDBS[2]). *Also structure of 4-methoxyacetophenone with assigned labels for this table.



D. **Discussion:** (20 points)

This section is where everything from the experiment is tied together and all results are explained using: chemical theory, physical observations, and peer-reviewed information available through textbooks and the UCF Database. Some aspects to consider when writing this section are:

i. What is the theoretical yield compared to the actual yield obtained? Can lower than ideal yields be accounted for? What is causing this to occur?

ii. How does the results compare to the theory learned? All of the theory that was used in the beginning of the lab report is meant to be used to explain results and findings. Be sure to use that theory to explain results completely and compare to a known standard (i.e. SDBS of organic compounds).

iii. Was there error that occurred during the experiment or characterization of the product? (The answer to this question is always yes) What type of error was encountered (Systematic vs random error)?

iv. Suggest ideas and methods that could be used instead of what was done in order to account for errors that were encountered during the lab. In other words, what could be fixed to obtain better yields?

v. What conclusions can you draw from the experiment performed?

Discussion Example:

"An aldol reaction is a useful reaction for forming new carbon-carbon bonds. The α-hydrogen of the carbonyl is removed forming a nucleophilic enolate that is in equilibrium with another carbonyl containing compound[1]. The new carbon-carbon bond is formed between the alpha carbon of one molecule and the carbon of the carbonyl of the second molecule. This forms a beta hydroxyl carbonyl compound, known as an aldol. The elimination of water of this product under basic or acidic conditions through an E1cb elimination reaction is called an aldol condensation reaction forming an α,β-unsaturated aldehyde or ketone. In this experiment, two lab groups attempted to produce 2-propylcinnamaldehyde. The ^{1}HNMR was obtained for both products. It is evident that the desired product was formed but containing trace impurities (integration values less than 0.2) and starting material shown in Figures 3 and 4. Both spectra show the expected aromatic peaks at δ 7-8 ppm as well as the identifiable aldehyde hydrogen shift around 9 ppm. Both spectra exhibit a single peak near 4 ppm. The IR spectrum for Group Green (Figure 2) has the characteristic broad medium beak for an alcohol functional group. This is indicative of an alcohol functional group. *This infers that the elimination step was not completed.* The distillation step of the procedure called for the temperature to reach 120°C which was not accomplished. The first fraction from this was removed. The next step called for the temperature to reach 150°C, which was also never accomplished. Water was removed quite extensively via distillation and is supported by the spectrum In figure 1; the IR lacks a characteristic water broad O-H stretching peak above 3000 cm^{-1} and the O-H bending that is seen between 1500-1400 cm^{-1}. A proton NMR stacked spectra is presented in figure 5 and shows that the products from both groups are the same. It shows that both spectra contain the same significant peaks in the aromatic region, the aldehyde, and the OH peak around 3 ppm. Neither groups were able to bring about the eliminating step. It is noteworthy to mention that the elimination step should have been carried out quite easily as the formation of more conjugated system of 2-propycinnamaldehyde is energetically more favorable[1]. The product was weighed after completion of the reaction and 1.31 g with a 16% yield was recovered. Much of the product was lost during the distillation purification step, as the process never reached the optimum temperature for the reaction to go to completion. The final product was identified as 2-[hydroxy(phenyl)methyl]pentanal using predictive HNMR software MNOVA. The predicted ^{1}HNMR for 2-propylcinnamaldehyde and 2-[hydroxy(phenyl)methyl]pentanal has been provided in figures 6 and 7 to show the difference between the desired and the proposed product."

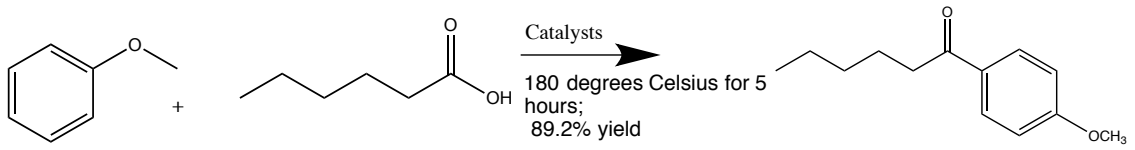
E. **Evaluation/Synthesis:** (15 points)

For Preparative Labs

1) Include the first page of the journal article of a synthesis method that increases % yield, utilizes more environmentally friendly reagents, etc., or modifies the procedure in some way.
2) In your own words, describe the broader impact and technique
3) Draw the reaction scheme for the reaction
4) Compare the performed synthesis of the given compound to a more recent method of synthesis for the same or similar compound by using the *Journal of Organic Chemistry*, *Journal of American Chemical Society*, or some other journal article equivalent. Hint: It helps to use scientific databases to find these articles (i.e. Scifinder).

   For investigative labs: Follow steps 1-4, however, adjust the focus to include: Using your given/determined compound find a synthetic route for the same or similar compound by using the *Journal of Organic Chemistry*, *Journal of American Chemical Society*, or some other journal article equivalent. Hint: It helps to use scientific databases to find these articles (i.e. Scifinder).

Evaluation and Synthesis Example:



This journal article discuses the efficient and more complex study of synthesizing 4-methoxy phenyl hexyl ketone from the acylation of anisole by using a more efficient and green catalyst (Hb zeolite-supported tungstophosphoric acid catalyst (HPW/ Hb)). The yield of 4-methoxy phenyl hexyl ketone was quite high at 89.2 % under the conditions suggested and mentioned above. The complimentary effects between HPW and Hb zeolite lead to sites that are more acidic, making it a very efficient catalyst. As mentioned previously this catalyst is also a more green option to those toxic catalysts normally used in the lab or industry because it can be recycled for four times while maintain a high yield of 70%. In Experiment 59 the acylation of anisole lead to the synthesis of 4-methoxyacetophenone. While experiment 59 allowed for a 60% yield using anhydrous aluminum chloride, dichloromethane, and acetyl chloride in the presence of concentrated hydrochloric acid, this experiment uses a much more efficient and stable catalyst leading to a higher yield. A smaller quantity of catalyst was used and it's ability to be

recycled and still function so efficiently could lead to a cut in cost for labs in research and industry in the future.

F. **Bonus Points:** (3 points)
You will always obtain bonus points if your lab report is less than three pages long. Concise writing in my book is worth more than fluffy writing, so in turn you will be awarded bonus points.

G. **References:** (2 points)
This section is worth 2 points and is required for submission. If this section is not included, your lab report will not be eligible for any points. You must properly reference all resources used. Many incidents of plagiarism result from students' lack of understanding about what constitutes plagiarism. However, you are expected to familiarize yourself with UCF's policy on plagiarism. All work you submit must be your own scholarly and creative efforts. UCF's Golden Rule defines plagiarism as follows: "**whereby another's work is used or appropriated without any indication of the source, thereby attempting to convey the impression that such work is the student's own.**"

H. Overall Writing Style and Formatting **(10 pts)**

II. Laboratory Final Exam: (20% of your grade)
The final exam will be comprehensive and will count as 20% of the final grade for the organic laboratory. The final will cover aspects from each experiment with a range of topics including: Theory, Technique, and Application. A final review may be conducted during the last laboratory period if time permits.

III. Quizzes: (20% of your grade)
Quizzes will be taken through webcourses and is due BEFORE the start of each lab meeting. The quizzes will include questions about the experiment from the previous week and the current week's experiment. There will be no make-up for missed quizzes as you have a week to do them. There are NO DROP QUIZZES.

IV. Laboratory Notebook: (20% of your grade)
All scientists, no matter from which field, are required to keep a laboratory notebook that others with little to no knowledge could follow and replicate your procedure. The laboratory notebook must be brought to all lab periods and all matters to the laboratory must be recorded in it with ink pen. There are some sections of the laboratory notebook that must be completed before the lab period, admission to the lab will be denied if these sections are not completed prior to lab.
(see website for more details and an example)

Note: The instructor reserves the right to add to and/or change the schedule and other policies and information in the syllabus at any time.

Important Dates:

Event	Date	Notes
Labor Day	Monday September 5	no labs
Veterans Day	Friday November 11	no labs
Thanksgiving	Thurs Nov 24-Friday Nov 25	no labs
Study Day	Monday December 5	no classes

Schedule CHM 2211L Fall 2016		
The Week of	Lab Number	Lab Title
Aug. 22	Lab Orientation	Check In/Lab Orientation
Aug. 29	**Labs 21B**	**Safety/t-pentyl chloride**
Sept. 5	Lab 9	Aspirin
Sept. 12	**CP Technique 20**	**Chromatography**
Sept. 19	Lab 22	4-methylcyclohexene
Sept. 26	**Lab 44**	**methyl orange**
Oct. 3	**Lab 13**	**Eugenol**
Oct. 10	**CP**	**Soap**
Oct. 17	Lab 12	Isopentyl acetate
Oct. 24	Lab 32A	Benzoin
Oct. 31	Lab 32B	Benzil
Nov. 7	**CP**	**Tetracyclone* (includes all data from benzoin and benzil labs)**
Nov. 14	CP	polystyrene
Nov. 21	NONE	Thanksgiving ☺
Nov. 28	**CP**	**Unknown Lab**
Dec. 6	----------	Final Exam/Check Out

CP = course pack

Bold = Seven lab reports that need to be turned in