

## Projects for Organic Laboratory

### Introduction

For two lab periods in November, you will work on a project that requires you to design experiments, carry them out and interpret the results in order to reach some conclusions. For this project, your Teaching Assistant will assign you to work with a partner. Two weeks before the start of the project sessions, each pair will have an opportunity to choose the project topic they wish to pursue based on the descriptions provided. You and your partner will then work together to design the necessary experiments for the project. An experimental design and a list of materials needed will be handed in to the TA one week before the project sessions start. In designing the experiments for the project, refer to experiments that you performed this semester as well as to Parts 1, 2, 3 and 4 (chapters 1-19) of the book *Techniques in Organic Chemistry*, by J. R. Mohrig et al. and the accompanying videos. Use the knowledge and lab techniques that you have learned throughout the semester to complete the project.

One of the purposes of this project is to help you develop both problem solving and interpersonal skills. You may find that you and your partner are unsure of how to proceed initially with the project and spend some time “floundering”. Don't panic! This is a natural part of the process. Listen to each others' ideas and work together to devise and modify your plan of attack. You will be evaluated on how you plan and carry out your experiments, how you analyze and interpret your results and on the quality of the reports.

#### *Tips for working with a partner*

- Contribute to the group effort (ask questions, clarify concepts, suggest procedures, modify plans, etc.).
- Be prepared for meetings and lab periods (read material beforehand, work on calculations, etc.).
- Meet when planned (let your partner know if you cannot attend).
- Do not distract your partner from the task at hand.
- Follow through on tasks that you agree to complete.

### Schedule and assignments for the project sessions

Projects will be assigned to the pairs based on lists of preferences submitted by each pair to the TA. Partners will then meet to devise an experimental plan for the project. The experimental plan, plus two copies of a list of materials needed, will be submitted to the TA one week before work on the project is to begin.

As usual, your TA will collect the yellow copies of your data and observations sheets at the end of each lab period during the project sessions. In addition, after each lab period, *each student* will write a “progress report”. The final lab report is due on the last lab period of the semester, and *each student* will write his or her own final report. During this last lab period, each *pair* will also present their results and conclusions in the form of a short oral report.

You may find as you work on the project that modifications to the original procedure must be made. If you require additional items for the second lab period that were not listed in the initial request for chemicals and equipment, submit a new request to your TA before the second lab period.

### Schedule for the Project Sessions

2 weeks prior to Session 1	Choose project
1 week prior to Session 1	Submit list of materials needed Submit project design
Session 1	Begin work on project
Session 2	Each student submits progress report for Session 1 Work on project continues
Session 3	Each student submits progress report for Session 2 Each student submits final written report Pair presents oral report

### Grading policies

The project assignment will be worth a total of 10 points, two times that of a regular lab assignment. The distribution of the weighting factors will be as follows:

<b>Project design</b> .....	0.15
<b>Progress reports</b>	
for Session 1 .....	0.25
for Session 2 .....	0.25
<b>Final report</b>	
Introduction .....	0.15
Results and calcs. ....	0.15
Discussion .....	0.45
<b>Oral report</b>	
Individual's presentation .....	0.30
Overall presentation .....	0.30
<b>Total</b> .....	2.0

For most sections of the assignment, students will be graded on an individual basis. The exceptions are the grades for the “Project design” and the “Overall presentation”, both of which will be “pair” grades. Each partner will receive the same grades for these two sections.

As usual, the TA will give a numerical grade from 1-5 for each section, and each section of the assignment will be multiplied by the appropriate weighting factor according to the numbers listed above. Thus, a final numerical grade out of 10 ( $2.0 \times 5$ ) for the project will be calculated based on the section scores and their weighting factors.

### Performance grade

The final performance grade for the semester will be based, in part, on your performance during the weeks of the project. Your TA will receive feedback on your performance via a *peer evaluation* rating from your partner. The rating should be based on a student's effort and cooperation, creativity and contribution to the project, effort outside of the laboratory and attendance at meetings. This peer evaluation will be strictly confidential. Fill out and bring p. 8 to the last period and hand it in to your TA. The TA will arrive at a final performance grade for each student based on these peer evaluations as well as his/her own insight on a student's performance.

## Make-up policy

If a student misses one or more lab periods during the weeks of the project, the two partners can decide how that student's work should be made up. He/she might be assigned to do extra data analysis, computer work, library research or other assignments that may be completed outside of the laboratory. In other cases, she/he may need to come to a different lab period to complete experimental work. If a student needs to make up work during a different lab period, s/he must make arrangements for this through the Laboratory Director, Prof. O'Connell, Merkert 125D.

## Project design

The project design should include the following:

1. A statement of the purpose of the project and the chemical principle(s) being studied.
2. An outline or description of the proposed procedures. When designing the project, use the following questions as a guide:
  - a. What techniques will be used?
  - b. What should be the sample size?
  - c. If solutions are needed, what concentrations should be used?
  - d. How will substances be identified (if applicable)?
  - e. How will substances be purified?
  - f. How will the purity of the substances be determined?
  - g. How will the percent recoveries or percent yields be determined?
3. Discuss significant hazards that will be encountered and describe special precautions which will be taken to avoid them. See p. 6 of this handout for information regarding safety references.
4. Fill out and attach 2 copies of the "Materials needed" sheet (p. 7).

## Progress reports

The progress reports will describe the procedures that were carried out and the data and observations that were obtained during each lab period. They should include the following:

1. *Details of the experiments that were performed.* Provide step by step instructions of the procedures which were followed using the past tense and the third person, passive voice. Examples follow:

**correct:** A 50 mL portion of 6 M hydrochloric acid was added to the solution.  
**incorrect:** I added 50 mL of 6 M hydrochloric acid to the solution.  
**incorrect:** Add 50 mL of 6 M hydrochloric acid to the solution.
2. *Modifications to the procedure.* You may find as you proceed that changes to the original procedure must be made. These modifications and the reasons for them should be noted in the appropriate progress reports.
3. *Data and observations obtained (carbon copies of notebook pages).* As usual, your TA will collect the yellow copies of your laboratory notebook sheets, and these

will constitute part of the progress report. The data on these sheets should be presented in appropriate forms (lists, tables, etc.). Include all observations that were made during the course of the experiments.

Each progress report should be about one page in length, not including the data and observation sheets from the notebook. Each student will hand in two progress reports. The progress reports should not attempt to analyze or interpret the data obtained and thus should not unnecessarily repeat information which will be presented in the final report.

### **Final written report**

The final report should include the following three sections. Write the appropriate heading at the beginning of each section (Introduction, Results and calculations, or Discussion).

**Introduction** Introduce the reader to the problem that you and your partner set out to solve. Explain what chemical principles were being investigated. Describe the approach that you chose to take in tackling the problem and why this approach was chosen.

**Results and calculations** Report relevant numerical data (melting or boiling points,  $R_f$ 's, retention times, percent recoveries and yields, etc.) and any calculations and graphs that show how the data was analyzed.

**Discussion** Provide an interpretation of the results and draw conclusions. Discuss what the experimental data proved or failed to prove and why. Review the chemical principles involved and explain whether or not your results fit expected results. Describe problems encountered in performing the experiments and how these problems may have affected the final results. List any limitations of the experimental procedures and discuss sources of error. Discuss ways in which the procedures could be improved.

The final report must be typed. Use double spacing, a one inch margin and a 12 point font size. The length of the Introduction and Discussion sections *together* should not exceed three pages. The final report should not discuss details of the procedures followed in lab and thus should not unnecessarily repeat information that was presented in either of the progress reports.

As with all laboratory reports, the progress and final reports must be written *in your own words*. If identical reports are handed in by two or more members of a group, these reports will be given scores of zero for all the identical sections.

### **Oral report**

The oral presentation is a "pair" effort, and the material should be divided up so that each person speaks for 5-8 minutes. A possible division of topics follows:

1. Introduction, background information, an explanation of the rationale behind the plan of attack.
2. Experimental procedures and set-ups. Changes which were made to the procedures as the project progressed.
3. Observations and results that were obtained.

4. Conclusions and discussion of the results. Were the results what you expected? What would you do next if you could continue the project?

The above is just a suggestion and may be modified as you see fit.

You can use visual aids in your oral report. A portable LCD projector will be available if you would like to give a Power Point presentation; in this case, you must bring your own laptop computer and any necessary adapters. Alternatively (or additionally), you can print text or paste enlarged diagrams on poster boards which can be held up during the presentation. A blackboard will be available. Some groups may wish to provide handouts for members of their audience.

The presentation must not be longer than 15 minutes. Your audience will consist of your lab TA and your laboratory classmates. The audience will have an opportunity to ask questions when the presentation is over.

The pair will be graded on the overall organization of the presentation as well as on the content. The material should be explained in a clear and interesting fashion. Individuals will be graded on their comprehension and enthusiasm.

### Materials available in the laboratory

The following chemicals and equipment will be available in the labs at all times during the weeks scheduled for the projects. You must dilute these “stock” solutions to prepare solutions of varying concentrations.

6 M HCl soln	saturated NaCl soln	ethanol	TLC equipment
conc. HCl soln	CaCl <sub>2</sub> (solid)	diethyl ether	GC equipment
6 M NaOH soln	MgSO <sub>4</sub> (solid)	mortars/pestles	
sat'd NaHCO <sub>3</sub> soln	Na <sub>2</sub> SO <sub>4</sub> (solid)	alkacid paper	
conc. H <sub>2</sub> SO <sub>4</sub> soln	methanol	syringe + pipets	

If you use the GC, be sure to record the following parameters for each chromatogram obtained:

- column temperature (gas chromatograph)
- column type (gas chromatograph)
- attenuation (gas chromatograph)
- mV setting (chart recorder)
- chart speed (chart recorder)
- injection size (in  $\mu\text{L}$ )

Also, be aware of the fact that CH<sub>2</sub>Cl<sub>2</sub> is used to clean the syringe between injections, and that a small peak near the beginning of the GC trace can often be attributed to this solvent.

### Safety precautions

1. Know the hazards of the chemicals that you are using. Look them up in the resource material listed on the next page.
2. The acid and base solutions and inorganic salts which are provided during these lab periods (see list above) may be disposed of down the sink *if they do not contain any organic compounds*. All organic chemicals and solvents must be disposed of in an appropriate **Laboratory Byproducts** jar. The following Byproducts jars will be available:

1. *Non-halogenated liquids*: ethanol, methanol, diethyl ether, isooctane, toluene, anisole
2. *Halogenated liquids*: 1-chlorobutane
3. Semicarbazones (solids)
4. Acidic solids: 2-naphthol, *o*-toluic acid
5. Solid *p*-toluidine
6. Liquid esters

If organic byproduct material is mixed with either strong acid or base solution, you *must* neutralize the mixture before pouring it into a Byproducts jar.

**Speak with your Teaching Assistant if you are unsure of the proper way to dispose of *any* substance.**

3. **Never** light a match or a Bunsen burner in an organic laboratory! Many organic solvents and liquids are flammable. Do **not** use a hot plate to heat the following **flammable** liquids: ethanol, methanol, diethyl ether, toluene, isooctane, and 1-chlorobutane. Use a steam bath instead (a thermowell may be used if the liquid is contained in a round-bottom flask).  
**Note!** Diethyl ether is especially flammable. Do not use this solvent in the vicinity of a hot plate.

### Safety references

A vendor is required by law to provide a Material Safety Data Sheet (MSDS) for every chemical sold. These sheets impart information regarding the hazards associated with a chemical, the precautions to take when working with the chemical, as well as some physical data. You will find a list of sites with MSDS's on the following webpage:

<http://www.ilpi.com/msds/#Internet>

You should check the MSDS's for a few chemicals with which you are familiar to get an idea of the kind of information that is provided. For instance, find the MSDS's for sodium chloride, hydrochloric acid and acetone.

The Boston College library has created a Chemical Laboratory Safety and Methods website:

<http://libguides.bc.edu/labsafety>

as well as a page for Chemical and Physical Property Data:

<http://libguides.bc.edu/chemdata>

Names of partners \_\_\_\_\_

Lab period \_\_\_\_\_ TA \_\_\_\_\_

### **Materials needed for the project**

List the reagents and solutions needed.

_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

List the equipment that you will require (other than equipment provided in your drawer).

_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

**Each pair must hand in two copies of this list to their instructor.**

## Peer Evaluation Form for the Performance Grade

Rate your partner using a whole number from 1–5 on overall performance during the project. **This form will be kept strictly confidential and will not be shown to anyone.** Bring this sheet with you to the last lab period of the semester and give it to your TA.

Partner's name

Rating

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----- Fold here and staple or tape closed.-----

## Project Topics

Background information and hints for the projects are provided on the following pages. Each pair in a laboratory will work on a different project. For this reason, the TA will obtain from each pair a list of the three topics in order of preference. After reviewing the lists, the TA will assign the topics and specific projects.

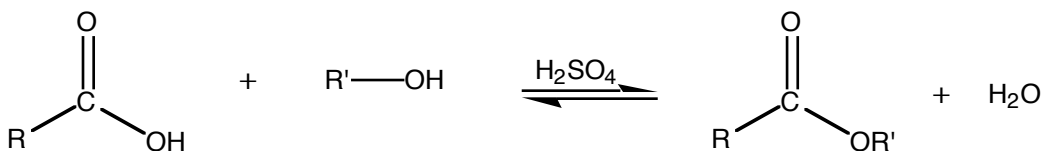
**Topic 1:** Ester Syntheses

**Topic 2:** Separation of a Mixture

**Topic 3:** Isolation and Derivatization of a Natural Product

## Projects 1, 2 and 3. Ester Syntheses

Esters can be synthesized by refluxing a carboxylic acid with an alcohol in the presence of an acid catalyst:



You will explore this reaction by varying a reaction condition and examining the effect that it has on the *yield and purity* of the product. You can change one of the following conditions:

- 1) mole ratio of carboxylic acid to alcohol
- 2) amount of catalyst

Pick one of the above conditions to explore.

The alcohol and carboxylic acid reactants are liquids. Use on the order of 10-15 mL of the limiting reactant. Do not use more than 20 mL of either reactant for any reaction that you run. Do not use more than 3 mL of concentrated sulfuric acid as the catalyst.

**Caution! Concentrated sulfuric acid is extremely corrosive. Wear gloves when using this reagent.** Reflux the mixture for 30 minutes. Refer to *TOC*, pp 59-60, for instructions on how to set up a refluxing reaction mixture at the miniscale level.

After the reflux is completed, you must perform an extraction to remove the sulfuric acid catalyst and any unreacted starting materials. First, wash the reaction mixture with distilled water. Next, wash the mixture with an appropriate solution to remove the acids. How can you check to make sure all of the acidic compounds have been removed from the organic layer? Take care to remove all the water from the organic layer.

Your ester product will be a liquid that must be purified. Provide data which indicates the purity of the product.

The carboxylic acid and alcohol combinations are:

- 1-pentanol plus acetic acid
- isobutyl alcohol plus propionic acid
- 1-propanol plus propionic acid

## Projects 4, 5 and 6. Separation of a Mixture

You will be given a mixture of three different compounds. You must devise a plan to separate the components of the mixture.

Each mixture contains **two** of the following liquids:

isooctane  
anisole  
toluene  
1-chlorobutane

You must determine which two liquids your mixture contains.

Each mixture also contains **one** of the following solid compounds:

*p*-toluidine (this compound sublimes slowly at room temperature)  
2-naphthol  
*o*-toluic acid

You must determine which solid your mixture contains. All of these solids are soluble in the liquids that are listed above.

Once the three components of the mixture are separated from each other, the solid compound must be recrystallized.

Determine the mass percent composition of the mixture. Assess the purity of each compound that you isolate.

First, perform the separation using miniscale (30-50 mL of the mixture) techniques. Perform the separation using microscale (1-5 mL of the mixture) techniques as well. Refer to *TOC* for information on the appropriate microscale techniques. Discuss the advantages and disadvantages of each method.

