Organic Chemistry Laboratory Manual: Miniscale and Microscale Organic Experiments

Amar S. Tung, Ph.D.
Department of Chemistry and Physics
Lincoln University

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Acknowledgements

I wish to express my gratitude to my former students in CHE 203L (previously CHE 273) and CHE 204L (previously CHE 274) classes for their suggestions and their role in refining the experiments in this manual. I wish to express my sincere thanks and deep appreciation to Dr. Robert Langley for his leadership of our Chemistry Department, his inspiration and his support of each faculty member. And finally, I wish to acknowledge Lincoln University for supporting this work in part through a Faculty Development grant.

This manual is dedicated to Dr. Saligrama SubbaRao, a former but an esteemed member of Lincoln University Chemistry Department, for his wisdom, his guidance and valuable advice during my earlier years at Lincoln. I also dedicate this manual to all of my former and current students who came to the lab prepared, made good effort, asked pertinent questions, exhibited exemplary lab behavior and followed the organic chemistry code to be good citizens in the laboratory. I am indebted to all of these students and I shall, forever, be grateful to them.

Amar S. Tung

December 12, 2016
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Introduction to the Organic Chemistry Laboratory, Safety, Glassware and Apparatus

Introduction

The ability to obtain reliable and reproducible results in the laboratory depends on the techniques used in the laboratory. Proper laboratory etiquette in the chemistry laboratory is a very important component of the laboratory. The instructor will discuss the proper laboratory attire, laboratory safety rules and regulations, the proper disposal of chemicals and broken glassware, as well as the location of the safety showers, Eye Wash and First Aid Kit.

One of the most important aspects in the laboratory is the identification of proper glassware and equipment for use in an experiment. In this introduction to the chemistry laboratory, you must identify the glassware and apparatus shown and explain its use.

Laboratory Safety Rules and Regulations

Review the laboratory safety rules. You will be required to follow all of the laboratory rules and regulations.

1. Never work in the laboratory without proper supervision and never leave your experiment unattended.
2. Locate the eye wash station and shower and learn how they operate in case chemicals come in contact with your eyes or skin.
3. Food and drinks are not permitted in the laboratory.
4. Sitting or leaning on the countertops is strictly prohibited.
5. Protective safety glasses must be worn by everyone at all times.
6. Shorts and sandals should not be worn inside the laboratory.
7. Students are strongly encouraged to purchase and wear a lab jacket.
8. Always keep your laboratory area clean.
9. Do not “experiment” as a mad chemist. Unauthorized mixing of chemicals is strictly forbidden.
10. Do not smell or taste chemicals or compounds.
11. Do not throw solids into the drain. Ask the instructor how to properly dispose of any unused solid.
12. Always take the amount of stock solution you need for your experiment from the stock solution bottle, which will be under the hood.
13. Never pour unused stock solution back into the stock bottle. This will cause contamination of the stock solution.
14. Never pour unused liquids into the drain. Ask the instructor how to properly dispose of all liquids.
15. Report all chemical spills and accidents (does not matter how minor it may seem?) to the instructor immediately.
16. Whenever placing a glass stirring rod into a stopper, make sure that the stirring rod is properly lubricated.
17. Place all broken glass into the broken glass box. Do not place paper or plastic into the broken glass box.
18. Never pour water into “concentrated” acid and base solutions. Instead, slowly pour “concentrated” acids and bases into more dilute solutions.
19. Never place NaOH solutions into bottles with grounded tops.
20. Always clean your laboratory area and dispose of the chemicals properly after the experiment has been completed.
21. Always listen to the instructor as he/she describes the experiment and any additional laboratory safety rules and precautions for the experiment.
22. Never, ever heat a closed container.
23. When you are done determining the melting point of your substance in the MEL-TEMP Apparatus, turn the power dial to zero otherwise the thermometer can explode.
24. When working in the fume hood, never lean into the hood or place your elbows on the seat and do not put your face inside the fume hood. Make sure you and everyone around are wearing safety goggles.

**Student Health**
The laboratory experiments conducted in this course are designed with safety in mind. However, some students may have medical conditions that may increase sensitivity to the chemicals used in the laboratory. If you have any medical condition that may increase your risk, you should speak with your lab instructor and your physician so that proper arrangements can be made to ensure your safety.

**Safety Resources**
Safety Information Resources, Inc. Materials Safety Data Sheet (MSDS) Index:


Centers for Disease Control and Prevention (CDC), National Institute for Occupational Safety and Health (NIOSH) Pocket Guide to Chemical Hazards:

http://www.cdc.gov/niosh/npg/npg.html

Best Gloves chemical resistance

http://www.showabestglove.com/site/chemresi/

The following article appeared in LINCOLN UNIVERSITY Safety Bulletin (2007-08). This article was written for the benefit of our chemistry students and Lincoln University community.
LABORATORY SAFETY AT LINCOLN UNIVERSITY

Our motto is “Safety First, Last, and Always.”

The chemistry laboratory, especially organic chemistry, is potentially one of the most dangerous of undergraduate laboratories. That is why we have a set of safety guidelines. It is a very good idea to pay close attention to these rules, for one very good reason:

The penalties are only too real.

Disobeying safety rules is not at all like flouting many other rules. You can get seriously hurt.

1. **Wear your goggles.** Eye injuries are extremely serious and can be mitigated or eliminated if you keep your goggles on at all times. And we mean over your eyes, not on top of your head or around your neck. There are several types of eye protection available, some of it acceptable, some not, according to local, state and federal laws. We recommend the clear plastic goggles, UVEX safety eyewear, UVEX by Spherion S2500. Sure, they fog up a bit, but the protection is superb. Also think about getting chemicals or chemical fumes trapped under your contact lenses before you wear them to lab. Then don’t wear them to lab. Ever.

2. **Touch not thyself.** Not a biblical injunction, but a bit of advice. You may have just gotten chemicals on your hands in a concentration that is not noticeable, and, sure enough, up go the goggles for an eye wipe with the fingers. Enough said.

3. **There is no “away.”** Getting rid of chemicals is a very big problem. You throw them away from here, and they wind up poisoning someone else. (Our goals at Lincoln University are to create minimal hazardous waste). There are some laws to stop that from happening. The rules were really designed for industrial waste, where there are hundreds of gallons of waste that have the same composition. In a semester of organic chemistry lab, there will be much smaller amounts of different materials. Waste containers are provided for everything hazardous. If you don’t see the waste can you need, ask your instructor. When in doubt, ask.

4. **Students never work alone.** If you have a serious accident and you are all by yourself, you might not be able to get help before you get hurt seriously. At Lincoln University we don’t allow students to work alone. Professor Tung follows this for each laboratory class. He is the last one to leave the laboratory, follows the safety guidelines; lead by example and excel by performance.

5. **Don’t fool around.** Chemistry is serious business. Don’t be careless or clown around in the lab. You can hurt yourself or other people. You don’t have to be somber about it; just serious.

6. **Drive defensively.** Work in lab as if someone else were going to have an accident that might affect you. Keep the goggles on because someone else is going to point a
loaded, boiling test tube at you. Someone else may spill hot, concentrated acid on your body. Get the idea?

7. *Consumption* of any food or drink and playing are forbidden in the laboratory.

8. *Keep it clean.* Work neatly. Clean up spills. Turn off water or electrical equipment when you’re through with them. Close all chemical containers after you use them. Don’t leave a mess for someone else.

9. *Where it’s?* Learn the locations and proper use of the fire extinguishers, fire blankets, safety showers and eyewash stations.

10. *Making the best-dressed list.* Keep yourself covered from neck to the toes — no matter what the weather. That might include long-sleeved tops that cover the midsection. Is that too uncomfortable for you? How about a chemical burn to accompany your belly button, or an oddly shaped scar on your arm in lieu of a tattoo. Pants that come down to the shoes and cover any exposed ankles are probably a good idea as well. No open-toed shoes, sandals, or canvas-covered footwear. No loose fitting cuffs on the pants or the shirts. Nor are dresses appropriate for lab, guys. Keep the midsection covered. Tie back that long hair, and a small investment in a lab coat can pay off, projecting that extra professional touch. It gives a lot of protection too. Consider wearing disposable gloves. Gloves are not perfect protectors. Reagents like bromine can get through and cause severe burns.

11. *Not flames.* Many times you’ll be asked or told to heat something. In our organic chemistry laboratory, we *never* heat organic solvents over a hot plate or over a flame. Commonly used organic solvents (acetone, ethyl acetate, ethanol, and glacial acetic acid are flammable and volatile. Solvents must be used in the hoods

**NFPA (National Fire Protection Association) codes**

- **Red** - Flammability
- **Blue** - Health
- **Yellow** - Reactivity
- **White** - Special

**Health (Blue)**

<p>| 4 | Danger | May be fatal on short exposure. Specialized protective equipment required |</p>
<table>
<thead>
<tr>
<th></th>
<th>Warning</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>3</td>
<td></td>
<td>Corrosive or toxic. Avoid skin contact or inhalation</td>
</tr>
<tr>
<td>2</td>
<td></td>
<td>May be harmful if inhaled or absorbed</td>
</tr>
<tr>
<td>1</td>
<td>Caution</td>
<td>May be irritating</td>
</tr>
<tr>
<td>0</td>
<td></td>
<td>No unusual hazard</td>
</tr>
</tbody>
</table>

**Flammability (Red)**

<table>
<thead>
<tr>
<th></th>
<th>Danger</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td></td>
<td>Flammable gas or extremely flammable liquid</td>
</tr>
<tr>
<td>3</td>
<td>Warning</td>
<td>Flammable liquid flash point below 100° F</td>
</tr>
<tr>
<td>2</td>
<td>Warning</td>
<td>Combustible liquid flash point of 100° to 200° F</td>
</tr>
<tr>
<td>1</td>
<td>Caution</td>
<td>Combustible if heated</td>
</tr>
<tr>
<td>0</td>
<td></td>
<td>Not combustible</td>
</tr>
</tbody>
</table>

**Reactivity (Yellow)**

<table>
<thead>
<tr>
<th></th>
<th>Danger</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td></td>
<td>Explosive material at room temperature</td>
</tr>
<tr>
<td>3</td>
<td>Warning</td>
<td>May be explosive if shocked, heated under confinement or mixed with water</td>
</tr>
<tr>
<td>2</td>
<td>Warning</td>
<td>Unstable or may react violently if mixed with water</td>
</tr>
<tr>
<td>1</td>
<td>Caution</td>
<td>May react if heated or mixed with water but not violently</td>
</tr>
<tr>
<td>0</td>
<td></td>
<td>Not reactive when mixed with water</td>
</tr>
</tbody>
</table>

**Special Notice Key (White)**

<table>
<thead>
<tr>
<th></th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>W</td>
<td>Water Reactive</td>
</tr>
<tr>
<td>Oxy</td>
<td>Oxidizing Agent</td>
</tr>
</tbody>
</table>

**Material Safety Data Sheets**
Students are encouraged to look at **Material Safety Data Sheet** (MSDS) of common organic solvents and reagents used in the experiments. Our Material Safety Data Sheets are on the stockroom computer. The instructor will have these loaded in the file MSDS on the lab computer.

Amar S. Tung, Ph.D.
CHE-203 L (Organic Chemistry I Laboratory) & CHE-204 L (Organic Chemistry II Laboratory)

Guiding Rubric for Laboratory Reports

Completed laboratory reports and a laboratory notebook are required for the course, and it can be purchased at the bookstore. Your laboratory reports/notebook should be a complete record of every observation, procedure and operation that you carry out in the laboratory. It should be in ink not in pencil. Maintaining a neat and detailed description of each experiment will greatly facilitate the writing of your laboratory reports. Below is a guideline for the format of the Laboratory Reports/Notebook. Completed laboratory reports must be submitted electronically unless instructed otherwise.

It is required that that each student purchases “A laboratory notebook” at the beginning of the semester. This laboratory notebook should be a subject or composition notebook, not a ring binder or tear sheet; it should have numbered pages and you should leave the first three pages of the notebook for an index of the experiments. This notebook should be used for making data entries and important experimental observations. Each week update your index by adding date, the new experiment title and the respective page numbers. Again, your notebook should be a complete record of every observation. It should be in ink not in pencil. Date each page as you begin each experiment. Never tear a page from your laboratory notebook. At the end of each laboratory session, have your instructor initial your records. You will realize that keeping experimental records in a notebook will facilitate writing of your laboratory reports and better prepare you in laboratory exams. You can also use this notebook to write laboratory-based discussion questions and their answers.

Format for Investigative Type Experiments (35 to 40 pts)

The following information should be written as pre-lab or in the laboratory notebook before coming to the lab:

Final report points

DATE should be exact lab date when experiment was done 1 pt.

EXPERIMENT TITLE should be complete 2 pts

INTRODUCTION 5 pts

A very brief description of the experiment/technique that you are going to perform and the purpose of this technique.
TABLE OF COMPOUNDS (REAGENTS)  (4 pts for two compounds, 8 pts for three or more compounds)

<table>
<thead>
<tr>
<th>Compound</th>
<th>Structure</th>
<th>Molecular Weight</th>
<th>Physical Properties</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>(BP or MP, density for liquids)</td>
</tr>
</tbody>
</table>

Include the names, molecular formula, structural formulas, and pertinent physical constants for the major compounds used in the experiment.

The following information should be included during or after the laboratory session:

PROCEDURE and OBSERVATIONS  10 pts

This section should include a brief description of the procedure you performed in the lab and the apparatus used. The observations should describe all of the events that took place: the length of time the procedure took, appearance, smell, time took for color change of the chemicals (in the reaction vial or flask), the events affecting the outcome of the experiment, e.g. “The solution was heated too vigorously and some boiled over. Some product lost.” Observations need not be written in complete sentences but should contain all of the important information unique to your work.

RESULTS AND DISCUSSION  10 pts

This section should include the results of your experiment and any data that you collect such as weights of products, MP of products, BP of products, results of infrared spectroscopy, etc. If necessary, you can discuss why you obtained the results you did. (A discussion will not be necessary for all experiments). Read the laboratory handouts carefully to know what must be included in the lab report for earning maximum marks.

CONCLUSIONS  3-4 pts

A very brief section (one or two sentences) on the conclusions that you were able to draw from this experiment. If identifying an unknown, summarize the results of your findings here.

Format for Preparative Type Experiments  (50 pts)

The following information should be written in the laboratory notebook before coming to the lab:

DATE should be exact lab date when experiment was done  1 pt.

EXPERIMENT TITLE should be complete  2 pts

Final report points
INTRODUCTION

A very brief description of the reaction that you are going to perform and the proposed major product of this reaction.

MAIN REACTION and MECHANISM

Write the reaction, in equation form, that you will be conducting in the lab and include a detailed mechanism. Also, write the side reaction(s) that may take place.

TABLE OF REAGENTS

<table>
<thead>
<tr>
<th>Compound</th>
<th>Molecular Wt.</th>
<th>Amt. used (grams, ml, moles)</th>
<th>Physical Properties</th>
<th>Limiting reagent?</th>
</tr>
</thead>
</table>

Include the names, structural formulas, and pertinent physical constants for the major compounds used in the experiment. Clearly label the limiting reagent by placing an asterisk next to the compound or underlining the compound name.

TABLE OF PRODUCTS

| Compound | Molecular Wt. | Theoretical yield (grams & moles) | Physical Properties | Amount, isolated (BP or MP, and density if liquid) (grams & moles) |

Include the names, structural formulas, and pertinent information for the predicted product(s) of the reaction. Include the grams and moles of the theoretical amount of product. Show your calculations to obtain the theoretical yield!

EXPERIMENTAL PROCEDURE

This section should briefly describe what you plan to do in the lab.

The following information should be included during or after the laboratory session:

OBSERVATIONS

This section should include all of the notes and observations that you made during the experiment. This section is very important and useful if things do not go well. We can sometimes troubleshoot successfully just by carefully reviewing your observations.
METHOD OF PURIFICATION AND ISOLATION  
To ensure that you really understand what you are doing in lab and that you are not simply following a recipe blindly, explain in this section (briefly) how you were able to purify your product and isolate it in pure form. You may find a flowchart very helpful for this section.

RESULTS AND DISCUSSION  
This section should include the results of your experiment and any data that you collect such as weights of products yield of products (SHOW CALCULATIONS), purity as determined by bp, mp, GC or spectroscopic analysis. Discuss if the reaction proceeded via the predicted mechanism, explain if you obtained the predicted major product and also explain the presence of any side products you may have synthesized and isolated.

CONCLUSIONS  
Very brief section (one or two sentences) on the conclusions that you were able to draw from this experiment.

Maintaining a laboratory record notebook requires a great deal of effort and preparation time. However, this is time well spent because if you follow the formats outlined here, you will spend much less time preparing your laboratory reports, and you will also decrease the amount of time that you are in the lab. Having the information at your fingertips, keeping the information organized, and acquiring an understanding of what you are doing will make things run much more smoothly and the likelihood of mistakes or failed experiments will decrease dramatically.

POST-LABORATORY QUESTIONS  
These questions help to evaluate your understanding of the concepts taught in the lab. You are required to answer all post lab questions. (10 points).

LABORATORY REPORT SUBMISSION
Laboratory reports must be submitted electronically as well as a printed version delivered to the instructor. Each report must have your name and also the complete name of your lab partner.

Lab reports/notebooks will be reviewed from time to time, during the laboratory period and they will also be collected for grading on a timely basis. For the first two lab experiments, students will be given the opportunity to resubmit the updated/corrected Lab reports to earn an improved grade. To earn an improved grade, previously submitted report must be done on a timely basis. Completed lab reports must be submitted one week after the last day of each lab and it must have your partner’s complete name.
CHE 203L

Organic Chemistry I Laboratory

EXPERIMENT #1

Title: Melting Point of Organic Solids.

Introduction

The melting point of a substance is the temperature at which a solid turns into a liquid. It is often used to identify organic compounds and also to assess their purity. It is not always possible to determine the melting point accurately but it is sufficient to determine the very narrow range of the temperature (1-2°C) in which the substance melts. The melting points of some of the representative organic compounds are presented in Table 1.

The MEL-TEMP apparatus consists of an electrically heated aluminum block that accommodates three capillary tubes. The sample is illuminated through the lower port and observed with a six power lens through the upper port. The heating rate can be controlled by a power or voltage setting on the rheostat.

Objective: To practice melting point determination technique and hence develop melting point determination skill.

Equipment/ materials

- MEL-TEMP Apparatus
- Capillary tubes
- Watch glass
- Dropping tubes for packing the solid

Only two students per group.

Note: From the list below, each group must do compounds 6 and 7 and any two of the organic compounds 1 to 5 and at least one out of 8, 9 and 10 and one of the two unknowns.

Organic Compounds: Provided to you on labeled watch glasses as finely ground powder.

1. Benzoic acid
2. Acetanilide
3. Fluorene
4. Ethyl-4-aminobenzoate (benzocaine)
5. 9-Fluorenone
6. trans-Cinnamic acid
7. Urea
8. 25% trans-Cinnamic acid + 75% Urea mixture
9. 50% trans-Cinnamic acid + 50% Urea mixture
10. 75% trans-Cinnamic acid + 25% Urea mixture
11. Unknown A
12. Unknown B

Students should follow the directions written on the whiteboard in Lab 317. Rate of temperature increase, on the thermometer, should be 1-2°C per minute from expected melting point minus 10 to 15°C. For example, if you expect the melting point of the compound to be 120°C, then from 105 to 110°C to the final melting point recording, the rate of temperature increase should be 1-2°C per minute.

Table 1.

Melting Point Temperature for Several Organic Compounds

<table>
<thead>
<tr>
<th>Compounds</th>
<th>Melting Point (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Biphenyl</td>
<td>70</td>
</tr>
<tr>
<td>Salicylic Acid</td>
<td>159</td>
</tr>
<tr>
<td>Acetylsalicylic Acid</td>
<td>130</td>
</tr>
<tr>
<td>Benzamide</td>
<td>130</td>
</tr>
<tr>
<td>Benzoic acid</td>
<td>121</td>
</tr>
<tr>
<td>Urea</td>
<td>132</td>
</tr>
<tr>
<td>trans-Cinnamic acid</td>
<td>132</td>
</tr>
<tr>
<td>Acetanilide</td>
<td>114</td>
</tr>
<tr>
<td>Fluorene</td>
<td>114</td>
</tr>
<tr>
<td>Vanillin</td>
<td>81</td>
</tr>
<tr>
<td>Stearic Acid</td>
<td>70</td>
</tr>
<tr>
<td>Succinic Acid</td>
<td>188</td>
</tr>
<tr>
<td>9- Fluorenone</td>
<td>83.5</td>
</tr>
<tr>
<td>Ethyl-4-aminobenzoate</td>
<td>89-90</td>
</tr>
</tbody>
</table>
Procedure

Follow the directions.

1) Place a small amount of the each substance (finely divided powder) on a clean dry watch glass. Push the open end of the capillary into the substances so that a small amount of the sample enters the capillary.
2) Hold the open end of the capillary tube above a dropping tube and drop the capillary tube. This allows the sample to settle down at the bottom of the capillary tube. The height of the sample in the capillary tube should be 2-3 mm (why?).
3) Place the capillary tube into the MEL-TEMP melting point apparatus. MEL-TEMP melting point apparatus has three slots. Set the power level from 3 to 6 and, determine the approximate melting point. Observe the melting point of the solid through the lens. Lower the power dial to zero (why?).
4) Fill another capillary tube with the unknown compound and place it in the MEL-TEMP. Maintain the **temperature increase at the rate of 1-2°C/ minute.** Record the temperature when the sample first begins to melt (liquefy) and again when the sample has melted completely. Repeat this one more time with the unknown.
5) Repeat steps 1 -4 for repeat determinations if necessary.

Note: Obtain the accurate melting point by controlling the heating rate more carefully. If you are continuing to use the same MEL-TEMP Apparatus, wait until the temperature drops at least 20°C below the approximate melting point).

**In discussion section of your lab report, for substances 8 to 10, discuss your results in the context of melting points of pure compounds.** Be sure to include the identification in the results, discussion and conclusion section.

**Wrap up.** Please discard the used capillaries in the cardboard container marked **Glass only.** Clean your bench space.

\[ \text{H}_2\text{N}-\text{O} \]

Benzocaine, an anesthetic

Please do not discard gloves, tissues in the cardboard container marked **Glass only.**
CHE 203L

Organic Chemistry I Laboratory

EXPERIMENT #2

Title: Purification of Acetanilide by Recrystallization

Introduction

In this experiment, you again will learn a common and very important technique used to purify organic solids, recrystallization. You will be given an impure sample of a well-organic solid called acetanilide and asked to purify it. You will determine the purity of the recrystallized product by recording its melting point. Recrystallization is routinely used to purify solids and is commonly used in pharmaceutical and chemical industry. Selection of a suitable solvent is of utmost importance in recrystallization.

Acetanilide: Although acetanilide has been used as an antipyretic and analgesic agent, it is more commonly used in the synthesis of complex medicines and dyes. For example, acetanilide was used to synthesize sulfanilamide, one of the first synthetic antibiotics which are still commonly used today to treat infections.

![Acetanilide structure](image)

Acetanilide mp: 113 - 115°C

Objective: To learn how to purify organic solids by recrystallization and recording its melting point.

All recrystallizations have 5 basic steps that you should remember:

1. Dissolve solid in hot solvent.
2. If some solid does not dissolve, do a hot gravity filtration to remove the insoluble impurities. Colored impurities are also removed with charcoal in this step.
3. Allow the solvent to cool slowly; crystals form. Complete crystallization in an ice bath.
4. Collect the crystals by vacuum filtration.
5. Allow the crystals to air dry.
Materials and Equipment:

- Impure Acetanilide
- Distilled water
- Hot plate
- Ice bucket
- 125 mL Erlenmeyer flasks
- 25 mL or 50 mL graduated cylinder
- Stemless funnel
- Filter paper
- Vacuum filtration apparatus
- Cloth gloves
- Mel TEMP melting point apparatus

Procedure:

1. Use a weighing paper or weighing boat, weigh close to 0.9g of impure acetanilide using a top loading balance. Record the exact weight in your notebook. Transfer as much of the sample as possible into the 125-mL Erlenmeyer flask. This sample is the weight of your starting material. Record in your notebook.

2. Heat approximately 70 mL of water to boiling in a separate clean 125-mL Erlenmeyer flask on a hot plate. Use appropriate heating knob setting. [CAUTION: Use cloth gloves to handle hot solutions.]

3. Using a 25 mL or 50 mL graduated cylinder, add 20 mL of the boiling water and several boiling stones (5 to 6 small or 1 or 2 large) to the acetanilide, swirl, and bring the mixture to a boil over a hot plate. Crude acetanilide may have a colored impurity that may not dissolve. Keep swirling the flask to help dissolve the solid and to prevent “bumping.”

4. Place one piece of fluted filter paper in a stemless glass funnel and put this on the top of the third Erlenmeyer flask.

5. Using a glass Pasteur pipette, carefully pour about 1-2 mL of boiling water (from the original 70 mL of boiling hot water) onto the fluted filter paper and let it drain into the clean Erlenmeyer flask. Place the flask on a hot plate. When the contents of the flask start to boil, immediately proceed with the hot filtration by carefully pouring your boiling acetanilide solution into the funnel.

6. After the filtration is complete, rinse the original flask containing the acetanilide with a small amount (1-2 mL) of boiling water and pour this through the filter paper.

7. The filtered solution should be clear and colorless at this point. If it is not, see your instructor. You might have had a problem with the hot filtration the first time, and you need to repeat that step.

8. If the volume of liquid in your flask exceeds 22 mL (how would you know the approximate volume), add 3 to 4 clean boiling stones and boil the solution briskly until the volume is reduced to about 20 to 22 mL.

9. Set the flask aside on your bench top, cover lightly with a clean cork, an inverted beaker, or a piece of filter paper, and allow the solution to cool to room temperature slowly.
While waiting for the flask to cool to room temperature, use your time efficiently and begin washing all dirty glassware and putting equipment back to its proper place.

10. After the flask has been at room temperature for approximately 5 to 10 minutes, place the flask in an ice bath for 10 minutes and then collect the crystals by vacuum filtration. Avoid disturbing the flask during the recrystallization step. **Why?** To transfer the last of the crystals from the Erlenmeyer flask to the Buchner funnel, use 1-2 mL of ice cold water. **DO NOT USE MORE THAN 1-2 mL OF COLD WATER – YOU WILL BEGIN TO DISSOLVE YOUR CRYSTALS!!** Your instructor will demonstrate the vacuum filtration step. Wash the packed crystals thoroughly with one mL of ice cold water.

11. Place the crystals in a **clean, dry, preweighed labeled** 50 or 100 mL clean beaker and allow the crystals to air dry in your lab drawer for two days. Determine the mass of your recrystallized acetaldehyde and its melting point. In order to determine the melting point of your recrystallized acetaldehyde **make a fine powder of small portion** and determine its melting point and that of impure acetaldehyde (starting material). (During the melting point determination, from **what temperature the rate of increase in temperature should be 1-2° C/min?**)

12. Compare the melting point of recrystallized acetaldehyde to that of impure acetaldehyde you started with.

13. Deposit your recrystallized acetaldehyde in a designated beaker.

14. Calculate percent yield as part of your lab report.

**Clean your bench space and the glassware before leaving the lab.**

**Place** Erlenmeyer flasks on spikes near the sink. Do not leave spatulas and glass rods in the sink. **Discard boiling stones in trash can.**

**Please do not discard your gloves in the cardboard container marked glass only.**
**Experiment #1 and Experiment #2**

**Post lab Questions:**

1. You have a crude preparation of acetonilide, which is dark brown in color. You have decided to recrystallize to increase its purity. List the five major steps of crystallization that you would use.

2. What are the two major properties of an ideal recrystallization solvent? (2 sentences)

3. What is the purpose of washing the crystals with about 2 ml of ice-cold solvent during the final vacuum filtration (1-2 sentences)?

4. Write down the structures of the 3 compounds you used for the recrystallization experiment.

5. In your melting-point experiment, the rise in temperature should be

   a) 0 – 1°C per minute
   b) 3 – 5°C per minute
   c) 2.5 – 3°C per minute
   d) 1 – 2°C per minute
   e) None of the above
CHE 203L

Organic Chemistry I Laboratory

EXPERIMENT #3

Title: Acid and Base Extractions: Separation of Benzoic Acid, Benzocaine (Ethyl-4-aminobenzoate) and Fluorene by Liquid-Liquid Extractions

Extraction is the drawing or pulling out of something from something else. Chemists extract compounds from solids or liquids using an aqueous or organic solvent. For example, every pot of coffee or cup of tea involves solid/liquid extraction, the extraction of organic compounds from solid ground coffee beans or tea leaves using hot water as the liquid. The lower molecular weight polar molecules such as caffeine dissolve in the hot water and are removed from the high molecular weight water-insoluble cellulose, protein, and lipid materials. In this lab, a mixture of an acidic organic compound (benzoic acid), a basic organic compound (benzocaine) and a neutral compound (fluorene) was provided. The purpose of this lab is to dissolve a known quantity of the mixture in ether and use acid-base chemistry to carry out liquid-liquid extractions

The Chemistry of Acid/Base Extractions for Separation

\[
\begin{align*}
H_2N &- \text{phenyl} \\
\text{O} &- \\
\end{align*}
\]

In strong acid, the amino group of ethyl-4-aminobenzoate is protonated to form its conjugate acid, an amine salt that is much more soluble in water than in dichloromethane. Therefore, the amine salt moves to the aqueous layer in the separatory funnel. When the acid layer is removed and neutralized, the neutral amine falls out of solution, since it is not soluble in water.

\[
\text{RNH}_2 + \text{HCl} \rightarrow \text{RNH}_3^+ + \text{Cl}^-
\]

Organic
Base
Anilinium salt (an organic salt)
(Water insoluble)

(Water soluble)

In strong base, like NaOH, benzoic acid is converted to its conjugate base, sodium benzoate, which is much more soluble in water than in ether. Therefore, the sodium benzoate moves to the
aqueous layer in the separatory funnel. When the base layer is removed and neutralized, the neutral carboxylic acid falls out of solution, since it is not soluble in water.

\[
\text{C}_6\text{H}_5\text{CO}_2\text{H} + \text{OH}^{\text{aq}} \rightarrow \text{C}_6\text{H}_5\text{CO}_2^- + \text{H}_2\text{O}
\]

A carboxylic acid
\(pK_a = 4\)
soluble in organic solvent

\[
\text{C}_6\text{H}_5\text{OH} + \text{OH}^{\text{aq}} \rightarrow \text{C}_6\text{H}_5\text{O}^- + \text{H}_2\text{O}
\]

A phenol
\(pK_a = 10\)
soluble in organic solvent

**Figure 4**

**Figure 5**

Solvent. (See Safety Alert).

**Organic solvents** used in extractions, of course, must be immiscible in water. They should also be relatively cheap, have a low boiling point (so they can be removed easily), chemically unreactive and hopefully relatively non-toxic. We will use dichloromethane (commonly called methylene chloride), which fulfills all of the above requirements, in this lab. Dichloromethane has a low bop \((39.6^\circ\text{C})\) and with a density of \(1.3266 \text{ g/mL}\), it is denser than water and other aqueous solutions, so it is always the bottom layer in an extraction.
SAFETY ALERT

1. Avoid letting any of the chemicals used in this experiment come in contact with your skin. If they do, immediately wash the affected areas with water. Wearing latex gloves would be a good idea.

2. Dichloromethane is extremely volatile. Be certain there are no flames in your vicinity when using it for extraction and when removing it from fluorene. NEVER HEAT Dichloromethane ON A HOT PLATE. USE A steam bath.

Materials, glassware and equipment:

- Beakers
- Spatula
- Top Loading Balance
- Vacuum Filtration System
- Sodium Hydroxide
- Sodium Chloride
- Fluorene
- Benzocaine
- Benzoic Acid
- Anhydrous Sodium Sulfate
- Retort stand
- Graduated cylinder
- Weighing paper
- Separatory funnel
- Funnel
- Erlenmeyer flasks (3)
- Weighing boat
- Hydrochloric Acid
- Dichloromethane
- Buchner funnel

Procedure:

AS ALWAYS FOLLOW LABORATORY SAFETY GUIDELINES PROVIDED to YOU. This is an important lab, you will learn a lot.

1. Get organic mixture from your instructor. Weigh a 1.5 g sample.
2. Transfer the entire solid to a clean 125 mL Erlenmeyer flask and add 50 mL of dichloromethane to this Erlenmeyer flask. Swirl the flask to dissolve all of the solid mixture.

3. Make sure that the stopcock on your separatory funnel is closed!! To avoid disaster, always have a beaker directly below the separatory funnel.

4. Place a funnel with a stem in your 125 mL separatory funnel. If you cannot get your entire solid to dissolve in the dichloromethane, then simply place a cotton plug in a stemmed funnel and filter the solution directly into a 125 mL separatory funnel with the aid of your stemmed funnel. The dichloromethane solution should be relatively clear.

Your instructor may provide you the organic mixture already dissolved in dichloromethane.

5. During the extraction procedure, you should use Erlenmeyer flasks at all times. Organic solvents/solutions should always be placed in Erlenmeyer flasks, not beakers. LABEL THESE FLASKS (and all subsequent glassware) WITH A SHARPIE MARKER.

6. Pour in 25 mL of 3M HCl solution to the separatory funnel. Two layers should form! Place the stopper on the separatory funnel and shake vigorously to allow the two layers to “mingle and mix.” Vent frequently. Your instructor will demonstrate this technique.

7. Place the separatory funnel back in the ring holder and remove the stopper. Allow the layers to separate for 1-2 minutes. Drain the bottom organic layer into a 125 mL Erlenmeyer flask labeled Organic. Drain the top aqueous layer into a 125 mL Erlenmeyer flask labeled ACID EXTRACTS.

8. Transfer the contents of Erlenmeyer flask labeled Organic to the separatory funnel. Add another 25 mL of 3 M HCl to the separatory funnel and shake and vent as before. Drain the bottom organic layer into a 125 mL Erlenmeyer flask labeled Organic back. Now drain the top aqueous layer into the Erlenmeyer labeled ACID EXTRACTS. This contains your basic compound. Set the ACID EXTRACTS E flask aside.

Now, the extraction with base:

9. Transfer the contents of Erlenmeyer flask labeled Organic back to the separatory funnel. Add 25 mL of 2M NaOH solution to the separatory funnel. Shake the funnel with frequent venting. Allow the layers to separate for 1-2 minutes, and then drain the bottom organic layer into a 125 mL Erlenmeyer flask labeled Organic.

10. Drain the top aqueous layer into a second 125 mL Erlenmeyer flask labeled BASE EXTRACTS. Repeat this procedure, washing the organic layer with another 25 mL of 2M NaOH solution and combine the two aqueous NaOH extracts.

11. Rinse the separatory funnel with about 5 mL of water and drain the flask completely into a separate beaker labeled WASTE. Why?
12. Transfer the contents of Erlenmeyer flask labeled Organic back to the separatory funnel. At this time, rinse the flask labeled Organic with about 5 mL of water and drain the flask completely into a separate beaker labeled WASTE. Why?

13. Add 25 mL of saturated NaCl to the separatory funnel. Stopper and shake the funnel with frequent venting. Allow the layers to separate for 1-2 minutes, and then drain the bottom organic layer into third 125 mL Erlenmeyer flask, marked ORGANIC. Saturated NaCl helps to dry the ether layer. (This will be explained in lab lecture).

14. You should now have three 125 mL Erlenmeyer flasks on your bench top:
   - ORGANIC (neutral fraction) in a beaker
   - ACID EXTRACTS
   - BASE EXTRACTS

15. Add ~4-6g of anhydrous sodium sulfate to the ORGANIC layer, gently swirl the flask, and let stand for 10-15 minutes. The sodium sulfate will act as a desiccant and will absorb traces of water that are remaining in the ether layer.

16. Cool the ACID EXTRACTS Erlenmeyer flask in an ice water bath and CAREFULLY and slowly add 6M NaOH until the solution is basic to litmus paper. The solution will turn cloudy. The product may oil out at first, but with swirling on ice, and scratching with a glass stir bar, the product will become solid. Allow the solid to form for at least 5 minutes on ice. Do not filter until you see solid.

17. Cool the BASE EXTRACTS Erlenmeyer flask in an ice water bath and CAREFULLY and slowly add 6 M HCl to this until the solution is acidic to litmus paper. You should observe a white precipitate at this point in time. All the crystals to form for at least 5 minutes on ice.

18. Filter both solutions separately by means of vacuum filtration. (See Kubrick 6th Edition, pp. 109-111; Gilbert and Martin (4th Edition) pp 70-71) [You can reuse your filter flask and Buchner or Hirsh funnel. Simply wash between uses.] Rinse each of the precipitated solids on the Buchner funnels with about 2 mL of ice cold water. If the amine is lumpy because it oiled out first, break up the lumps with a spatula before you wash the crystals with the cold water. Place the two solids in separate labeled beakers and allow them to air dry in your bench drawer over the next couple of days.

19. Now – back to the ORGANIC Erlenmeyer flask (contains neutral compound). Transfer the contents to a pre-weighed 25 or 50 mL clean beaker. Place it on a
steam bath. Ether will evaporate leaving your neutral product in the beaker. As the ether evaporates, you can observe the solid appearing on the walls of the beaker.

20. After the solid has dried, scrape the residue from the bottom and sides of the beaker, place the crystals in a vial, and record the weight and melting point for this solid. Record these values in your lab notebook as well.

21. Accurately weigh each of the other two solids from acid and base extractions, which you collected by vacuum filtration and record the melting point of each of these.

*Aqueous Wastes* from the filtrations of benzoic acid and benzocaine should be placed in the appropriate (ACID or BASE) AQUEOUS WASTE containers in the hood.

*Solid Waste* such as cotton plugs and Na₂SO₄ (sodium sulfate) should be discarded in the SOLID WASTE containers in the hood.

**CLEAN UP:**

Remember to clean up your lab space at the end of each laboratory period that means the bench top, the hood, area around sink, and the weighing balances.

Wash all glassware with soap and water and rinse with a small amount of acetone. Return all glassware and equipment to their original location.

To the student:


**REFERENCES.**

**POST LAB QUESTIONS**

1. Give the names and structures of two functional groups in organic chemistry that are acidic.
2. A mixture contains the following three compounds: benzoic acid \((\text{C}_6\text{H}_5\text{COOH})\), phenol \((\text{C}_6\text{H}_5\text{OH})\) and fluorine. An ether solution of this mixture is extracted with sodium bicarbonate solution to form aqueous layer A and organic layer B. The organic layer B is then extracted with sodium hydroxide solution to form aqueous layer C and organic layer D. Both solutions A and C are separately treated with hydrochloric acid to give solutions E and F respectively. Give the structure(s) of the organic solute(s) present in A, B, C, D, E and F. Explain and include a flow diagram for EC.

3. Write an expression for the acidity constant, \(K_a\) for acetic acid and benzoic acid\((\text{C}_6\text{H}_5\text{COOH})\). Which acid is stronger and why? Now write the \(pK_a\) values for both acids. What is the relationship between acid strength and \(pK_a\).

3. Suppose you do not know which layer in your separatory funnel is the aqueous layer, and you have no information about the density of the solvent, how could you determine which is the aqueous layer.
CHE 203L

Organic Chemistry I Laboratory

EXPERIMENT #4

Experiment Title: Preparation of 2-Methyl-2-Butene by Dehydration of 2-Methyl-2-Butanol

![Chemical Structure]

Introduction:

Alkenes can be prepared by two simple methods: i) by dehydrohalogenation of alkyl halides with a strong base (KOH) and ii) by dehydration of alcohols by using a strong acid (sulfuric acid or phosphoric acid).

Ease of dehydration: Tertiary alcohols are more easily dehydrated than secondary alcohols which in return are more easily dehydrated than primary alcohols. Why?

Objective:

In this lab you will use 2-methyl-2-butanol, a tertiary alcohol, for the preparation of 2-methyl-2-butene

Reagents:

1. 2-Methyl-2-butanol (tert- amyl alcohol)
2. 6.0 M Sulfuric acid
3. Anhydrous potassium carbonate

Test reagents:

1. 0.1 M Bromine (Br₂) in dichloromethane
2. Aqueous KMnO₄, 1%
Supplies:

1. Distillation set up glassware
2. Vials for product
3. 12x75 mm or 13x100 mm test tubes

Main Reaction:

\[ \text{2-methyl-2-butanol} \xrightarrow{\text{H}_2\text{SO}_4} \text{2-methyl-2-butene} + \text{2-methyl-1-butene} \]

MECHANISM:

The lone pair in the O attacks the H in H_2SO_4 to make the OH a better leaving group.

H_2O is a good leaving group, therefore, an E1 reaction occurs (because tertiary OH), leaving a carbocation intermediate.

The H_2O attacks the beta Hydrogens forming 2 isomers

Procedure:
Note: Your apparatus for distillation must be clean and dry. Wash your distilling head and condenser. Rinse with small (really small) volume of acetone and dry thoroughly.

Note: This lab involves distillation. All of distillation lab work will be done on the south lab bench. Why? This requires cooperation and resourcefulness on the part of each student. Do not lean on the bench.

Make a note of your important observations. These should be a part of your lab write-up.

In case sufficient heating elements are not available, a sand bath can be used as heat source. Appropriate precautions must be followed. Familiarize yourself with the Simple Distillation apparatus set up in the diagram, page 31.

1. Place 3-4 boiling chips in the 50 mL round bottom distillation flask.
2. Use a dedicated 10 mL graduated cylinder and transfer 10 ml of 2-methyl-2 butanol to the 50 mL round bottom flask. Check the ground glass joint # for connecting the still head and the West condenser. Make a careful note of all the connections. Never plug the electric heating element (Woven–glass heating mantle or Thermowell) directly into the power outlet. Always use a variable transformer.
3. Carefully add 7.2 ml of 6 M Sulfuric acid, and gently swirl the flask to mix.
4. Begin heating process of your simple distillation set-up. Record the pertinent observations as the reaction progresses.
5. Collect the material distilling at 45-48°C in a chilled 25 mL round bottom receiving flask. Continue to collect the distillate until 1-2 mL of the reaction mixture is left in the flask. How much distillate you will collect and how will you know how much you have collected?
6. Turn off the power supply. Remove the heating mantle, transfer the distillate into a 25 mL Erlenmeyer flask. Observe the presence of water in your product and make note of it.
7. Add a spatula-tip full of anhydrous potassium carbonate to dry and to neutralize any acid in your distillate. Record your observations. Add more as needed to dry your product but do not add too much which will lead to loss of your product.
8. Swirl the flask for 3-5 minutes to speed up the drying process. Transfer your dried product to a pre-weighed sample vial or to a 25 ml Erlenmeyer flask. Label it with your initials. Weigh our alkene product vial and determine the net alkene weight. Use this data to determine the percent yield in the lab write-up.

PROCEED TO TEST FOR DOUBLE BONDS
Test your alkene product (the distillate) for unsaturation (presence of double/triple bonds) using the following two methods:

A. Bromine in dichloromethane and
B. Baeyer test—KmnO₄ test.
A. Addition of Bromine:
Dissolve 2 drops of alkene product in ~ 0.5 mL of dichloromethane in a 13 x 100-mm test tube. In a dropwise fashion, add nearly 0.5 mL a 0.1 M solution of bromine dissolved in methylene chloride (dichloromethane) with gentle mixing. Record your observations. Rapid disappearance of red-orange color of bromine to give a colorless solution is a positive test for unsaturation.

Control- Repeat the above test with 2 drops of your starting alcohol reagent. Dissolve 2 drops of tert-amyl alcohol in 0.5 mL of dichloromethane in a 13 x 100-mm test tube. In a dropwise fashion, add nearly 0.5 mL of 0.1 M solution of bromine dissolved in methylene chloride (dichloromethane) with gentle mixing. Record your observation.

\[
\text{Alkene} \quad + \quad \text{Br}_2 \quad \quad \quad \quad \text{1, 2- dibromide}
\]

in dichloromethane

red-orange \quad \quad \quad \quad \quad \quad \text{colorless}

B. Baeyer test—\(\text{KMnO}_4\) test.
Dissolve 2 drops of alkene in ~1 mL of acetone in a 13 x 100-mm test tube. Add dropwise a 0.1 M solution of aqueous \(\text{KMnO}_4\) reagent. After each drop, mix the contents of the test tube and record your observations. Count the number of drops of \(\text{KMnO}_4\) reagent added for maximum solid (precipitate) formation. Persistence of purple color at the end indicates that the reaction has gone to completion. A positive test is the disappearance of purple permanganate color from the reagent leading to the formation of brown manganese oxides (solid). Note the chemistry of this reaction from the white board in the lab.

Control- Repeat the above test with 2 drops of your starting alcohol reagent dissolved in 1 mL of acetone in a 13 x 100-mm test tube. Add dropwise a 0.1 M solution of aqueous \(\text{KMnO}_4\) reagent. After each drop, mix the contents of the test tube and record your observations. Record your observations after each drop. Stop after the addition of three drops.
Baeyer Test for Unsaturation (Aqueous Potassium Permanganate Solution)

Alkene

\[
\text{Alkene} + \text{KMnO}_4 \xrightarrow{\text{H}_2\text{O}} \text{purple solution} \xrightarrow{\text{OH}} \text{brown solid}
\]

Alkyne

\[
\text{Alkyne} + \text{KMnO}_4 \xrightarrow{\text{H}_2\text{O}} \text{OH OH OH OH} + \text{MnO}_2 \text{brown solid}
\]

Wrapping it up. Dilute the residue remaining in the still pot (distillation flask) with slow addition of water, carefully neutralize it with sodium carbonate, and flush it down the drain with large quantities of water.

1. Place the Manganese dioxide from the Baeyer test for unsaturation in a waste container designated for "Heavy metals waste".
2. Place the bromine waste from the Bromine in dichloromethane test for unsaturation in a waste container designated for "Halide Waste".

Simple Distillation apparatus set up shown above
Experiment #4: Production of 2-methyl-2-butene by dehydration of 2-methyl-2-butanol

Prelab questions

1. What is the function of the acid catalyst in promoting the dehydration of alcohols?

2. In this lab you will use 6.0 M sulfuric acid. 40 to 50% phosphoric acid can also be used in place of sulfuric acid. Why would concentrated hydrochloric acid be an *inappropriate* catalyst for the dehydration of alcohols?

3. Why is there an upper limit to the temperature (the distillation temperature) at which the alkene(s) is (are) to be collected?

4. Write equations for the chemical reaction(s) that you will use to demonstrate the presence of alkene(s) in your distilled product.

5. Concentrated sulfuric acid is 18M. How would you make 300 mL of 6.0 M sulfuric acid? You have at your disposal graduated cylinders and beakers
CHE 203L

Organic Chemistry I Laboratory
EXPERIMENT #5

Title: Separation of Fluorene and 9-Fluorenone by Column Chromatography.

Objective

You will separate a ketone (9-Fluorenone) and a hydrocarbon (Fluorene) mixture by absorption chromatography and then check the efficiency of your separation by Thin Layer Chromatography.

Column Chromatography: Column chromatography is an extremely important technique in organic chemistry. It is a very versatile method used to separate and purify organic compounds. It can be used to purify both solids and liquids. Column chromatography is similar in principle to thin layer chromatography; compounds are separated based on their attraction to a solvent (mobile phase) and an adsorbent (stationary phase). Less polar compounds are less attracted to the adsorbent and elute (travel) more quickly than polar compounds. In this experiment, you will assemble a column from a Pasteur pipet and use it to separate the two components of a solid mixture of fluorene and fluorenone.

Required Reading:

For details, read section 6.3 (pp 183-190) Gilbert and Martin 4th Ed.

Special Safety Notes:

Alumina can be harmful to the lungs. Be careful to avoid inhalation.

Dichloromethane, acetone and hexane are volatile solvents; handle these safely. Do not discard these in the sink. Return the unused reagent to the appropriate bottle/flask in the hood.

Reagents:

- Dry alumina (Al₂O₃)
- Acetone
- 30% Acetone in Hexane
- 5% Dichloromethane in Hexane
- Hexane
- Dichloromethane
- Fluorene and 9-fluorenone mixture in 5% Dichloromethane in Hexane
Procedure:

Place 25 mg of the fluorene/fluorenone mixture in a conical vial. Add 0.3 mL of 5% dichloromethane in hexane. Stir to dissolve the solids. While the mixture is stirring, obtain 9 mL of hexane, 2 mL of 30% acetone in hexane and 2 mL of acetone in three separate 10 or 25 mL Erlenmeyer flasks. Place four test tubes in a rack and begin preparing the column.

Preparing the column:

Place a very small plug of cotton into a 5.75" Pasteur pipet. Gently lodge the cotton plug into the narrowing region of the pipet using a length of wire. The cotton should be pressed enough to secure it without excessive compression. Add 1.25 g of alumina. Alumina to the pipet

and gently tap it on the bench to compact the solid.

Clamp the pipet vertically to a ring stand just above Tube #1. Using a pipet, add hexane, in portions, to the top of the column (about 3 mL). Allow the hexane to flow through the column. You will see the alumina begin to look "wet". Make certain that there is solvent above the alumina at all times. Do not allow the solvent to drain below the alumina. This can lead to formation of air bubbles and channels in the solid support. Continue to add hexane and allow it to flow through the column until the alumina looks homogeneous and free of dry patches or cracks.

Separating the Mixture:

When the solvent reaches the top of the alumina, add the fluorene/fluorenone solution using a Pasteur pipet. When the solution reaches the top of the alumina, add another 1 mL of hexane dropwise. Start eluting the column into Tube #2, adding hexane until the yellow band nears the bottom of the column. Nearly 5 mL hexane will be needed. When the hexane reaches the top of the column, switch the solvent to 30% acetone in hexane. Just before the yellow band reaches close to the bottom of the alumina column (not cotton plug), start eluting the eluent into Tube #3, adding 30% acetone in hexane until the yellow band elutes completely from the column.
TLC Analysis:

Obtain a 5 x 5 cm TLC plate and label it as shown using a pencil. Handle your TLC plate only from the edges. Using a 1 μL micropipette, spot Tube #2 and Tube #3 at the origin on lanes labeled #2 and #3 of the TLC plate. Spot standard solutions of fluorene and fluorenone on lanes labeled “ENE” and “ONE” of the TLC plate. Develop the plate in a covered beaker using hexanes: dichloromethane (1:1) as the eluting solvent, removing the plate when the solvent reaches the top line. Mark the solvent front. Allow the eluent to evaporate from the plate and examine it under UV light (254 nm). GENTLY circle each spot with a pencil. Draw the TLC plate to scale in your notebook, showing all spots or you can take a picture with your smartphone and attach or incorporate it in the body of your lab report.

things to keep in mind while writing your report:

For each plate calculate and tabulate Rf values for each spot (show your calculations). Identify each component and explain your reasoning.

References


Post lab Questions:

1. If a column fraction contained two compounds (two spots upon TLC analysis), what could be done to separate the two compounds from one another?

2. In this lab, why was the solvent changed in the middle of the column chromatography procedure?

3. If you had 2.0 g of material that needed to be purified, would you opt for using TLC or column chromatography to purify your material? Explain your answer.

4. 2-Naphthol can be treated with potassium hydroxide and the resulting product reacted with iodoethane to give a compound (Compound X) which is used as an orange blossom scent in perfumery.

   a) Give the structure of Compound A

   b) If the reaction did not go to completion and you had to separate Compound A from the starting 2-naphthol by column chromatography using a relatively non-polar solvent, which compound would elute from the column first? Why?
CHE 203L
Organic Chemistry I Laboratory

EXPERIMENT # 6

TITLE: SYNTHESIS OF DIPHENYLACETYLENE FROM STILBENE

Background

A standard method of synthesis of alkynes begins with the homologous alkene. Bromination followed by the dehydrohalogenation provides the desired alkyne. Today we will apply this 2-step method to the preparation of diphenylacetylene. While bromine is a liquid and could be used for bromination, we will instead generate it from pyridinium hydrobromide perbromide \((\text{C}_5\text{H}_6\text{NBr}_3)\), \(\text{MW} = 319.86\). The perbromide is a non-volatile, crystalline, odorless solid which, in the presence of an alkene decomposes to generate one mole of bromine per mole of perbromide. For small-scale experiments, an easily weighed, high molecular weight solid is more convenient to use than a corrosive, volatile liquid.

This experiment is a multistep preparation of diphenylacetylene form \textit{trans}-stilbene. The initial reaction involves the bromination of \textit{trans}-stilbene with pyridinium hydrobromide perbromide (synthetically equivalent bromine, but easier to handle) to form \textit{meso}-stilbene dibromide. The stilbene dibromide is then treated with a strong base (KOH), to induce a double dehydrohalogenation, resulting in the alkyne, diphenylacetylene.

\[
\begin{align*}
\text{trans-stilbene} & \rightarrow \text{(meso)-stilbene dibromide} & mp &= 236-237^\circ\text{C} \\
\text{(meso)-stilbene dibromide} & \rightarrow \text{diphenylacetylene} & mp &= 59-61^\circ\text{C}
\end{align*}
\]
Because bromine adds in an anti-fashion to \textit{trans} stilbene, the resulting dibromide is meso and nonresolvable. The meso dibromide melts at 273 – 278 °C while the \textit{d}, \textit{l} – isomer, a minor impurity in this reaction melts at 114°C. After the \textit{di} – dehydrohalogenation step, all stereochemistry is lost.

\textbf{Procedure:}

\textbf{Precautions:} Pyridinium hydrobromide perbromide is corrosive and a lachrymator. Potassium hydroxide is corrosive.

\textbf{A. Preparation of meso-stilbene dibromide}

In a 125mL Erlenmeyer flask dissolve 1g of \textit{trans}-stilbene in 20mL of acetic acid by heating on the steam bath, and then add the 2g of pyridinium hydrobromide perbromide. Mix by swirling, if necessary rinse crystals of reagent down the walls of the flask with a little acetic acid, and continue the heating for 1-2 mins longer. The dibromide separates almost at once in small plates. Cool the mixture to room temperature and then cool it in an ice bath. Collect the product by vacuum filtration and thoroughly wash your dibromide product with 2-3 mL ice cold methanol. (How to and why wash thoroughly?). After the product is \textbf{completely} dry, weigh it and record your results. Determine the melting point of your product and record it. \textbf{Hold back 100 mg of this product for an IR.}

\textbf{B. Preparation of Diphenylacetylene}

1. Weigh 1.0 g of your meso-stilbene dibromide product and transfer to a large test tube (17 x150 mm or larger)

2. Add 4 mL of triethylene glycol and then add 6 pellets of potassium hydroxide (KOH). How? Do not touch KOH pellets by your hands (KOH is strongly caustic).

3. Using a clamp attached to a stand, place your tube in a sand bath and heat the mixture by heating the sand bath. You will use a hot plate to heat the silicone sand bath to a temperature of 175-180°C. Record your observations when your mixture is being heated. \textbf{Do not touch the hot sand bath.}

4. Continue to heat your mixture at 175-180°C for 5 minutes and then cool to room temperature by placing the reaction test tube in a clean beaker. Wipe the outside of the glass tube with a clean paper towel.

5. Add 20 mL of water to the test tube containing your product with mixing. Mix thoroughly and place the test tube in an ice water bath for 5-10 minutes.
Diphenylacetylene should separate as nearly colorless granular solid. Collect your product by vacuum filtration. Wash the solid with 2-3 mL of water.

6. Transfer the product to a 25ml Erlenmeyer flask. Add 5mL of 95% ethanol mix and let it cool slowly. Leave it undisturbed for 10-20 minutes and watch for the appearance of large colorless crystals. Show your results to your instructor at each step of this process.

7. Collect your product by vacuum filtration by pouring your preparation at the center of filter paper placed in the Buchner funnel. Use a clean small glass rod to guide the transfer of poured solution to the middle of the filter paper.

8. Let it dry before removing the product.

9. **Weigh** your product, show it to your instructor and determine the melting point of your **dry product** and complete the following two tests for the presence of triple bond. The procedure for tests is very much the same procedure as tests for the presence of double bond in “Preparation of 2-Methyl-2-Butene by Dehydration of 2-Methyl-2-Butanol” (Lab #4). If required, take **IR spectrum of the diphenylacetylene**.

**PROCEED TO TEST FOR TRIPLE BONDS**: Tests for unsaturation. Recall your experience from the tests you carried out in alkene lab (**Preparation of 2-Methyl-2-Butene**).

A. Bromine in dichloromethane and
B. Baeyer test—K\textsubscript{MnO}_{4} test.

**A. Addition of Bromine:**

Dissolve about 50 mg of alkyne product in \(~0.5\text{ mL}\) of dichloromethane in a 13x100-mm test tube. In a dropwise fashion, add nearly 0.5 mL a 0.1M solution of bromine dissolved in methylene chloride (dichloromethane) with gentle mixing. Record your observations. Rapid disappearance of red-orange color of bromine to give a colorless solution is a positive test for unsaturation.

\[
\text{Alkyne} + \text{Br}_2\rightarrow\text{1, 1, 2, 2- tetrabromide}
\]

in Dichloromethane
B. **Baeyer test—KMnO₄ test.**

Dissolve about 50 mg of alkyne product in ~1 mL of acetone in a 13x100-mm test tube. Add dropwise a 0.1M solution of aqueous KMnO₄ reagent. After each drop, mix the contents of the test tube and record your observations. Count the number of drops of KMnO₄ reagent added for maximum solid (precipitate) formation. Persistence of purple color at the end indicates that the reaction has gone to completion. A positive test is the disappearance of purple permanganate color from the reagent leading to the formation of brown manganese oxides (solid). Note the chemistry of this reaction from the white board in the lab.

**Control- Repeat the above test with 2 drops of your starting alcohol reagent dissolved in 1 mL of acetone in a 13x100-mm test tube. Add dropwise a 0.1M solution of aqueous KMnO₄ reagent. After each drop, mix the contents of the test tube and record your observations. Record your observations after each drop. Stop after the addition of three drops.**

**Baeyer Test for Unsaturation (Aqueous Potassium Permanganate Solution)**

**Alkene**

\[
\text{CH}_2=\text{CH}_2 + \text{KMnO}_4 \xrightarrow{\text{H}_2\text{O}} \text{CH}_3\text{CH}_2\text{OH} + \text{MnO}_2
\]

**Alkyne**

\[
\text{C}_3\text{H}_4\text{CH}_3 + \text{KMnO}_4 \xrightarrow{\text{H}_2\text{O}} \text{CH}_3\text{CH}_2\text{OH} + \text{MnO}_2
\]
**Recommended reading for Synthesis of Diphenylacetylene from trans-Stilbene.**


**POST LAB QUESTIONS:**

1. All questions and answers shall be written into the lab notebook:
   a) Give the IUPAC name for trans-stilbene

   b) Knowing that Br₂ addition to double bonds is anti, draw a Newman projection for stilbene dibromide. Is this isomer chiral or achiral?

2. Would you come up with the same product is the starting material had been _cis_-stilbene?

3. If the starting material for this reaction had been cyclohexene, the final product would not have been an alkyne. Why not and what would the predicted product be?

4. Draw a reaction energy profile for the formation of both products in this sequence.
5. Why, when sodium amide in liquid ammonia is normally used for the dehydrohalogenation of 1, 2 dihalides to acetylenes, may a weaker base such as KOH be used to prepare 1,2-diphenylacetylene?

6. When performing chemical tests (Part B), it is useful to understand how (and if) test reagents react with certain functional groups.
   a) Write general equations for the bromination of alkynes and alkenes.

   In the acid permanganate test, you are oxidizing an alkene.

   b) Using the same general alkene as in a), draw an oxidized product.

7. Provide a mechanism to show why the major product of addition of bromine to trans-stilbene is the meso-dibromide.

8. A single dehydrohalogenation (E2: anti-elimination) on this meso dibromide would provide a bromoalkene rather than the alkyne. Can you predict its structure? Show your reasoning
CHE 203L

Organic Chemistry I Laboratory

EXPERIMENT # 7

Title: Nucleophilic Aliphatic Substitution: Synthesis of 2-chloro-2-methylbutane

INTRODUCTION

The conversion of a tertiary alcohol to the corresponding tertiary alkyl halide using concentrated hydrogen halide proceeds via an $S_N1$ mechanism. The first step in the reaction mechanism involves the protonation of the hydroxyl group of the alcohol, a Lewis acid-base reaction. In the second step, ionization occurs and a molecule of water is lost which leads to the formation of tertiary carbocation. Formation of the carbocation is the rate-determining step. In the final step, chloride ion attacks the intermediate carbocation to give 2-chloro-2-methylbutane. The purpose of this experiment is to synthesize 2-Chloro-2-methylbutane from 2-Methyl-2-butanol and concentrated hydrochloric acid. Perform two qualitative tests, Silver Nitrate and Sodium Iodide test to confirm the presence of alkyl halide as well as to identify the product as primary, secondary or tertiary halide.

Main Reaction and mechanism:

Synthesis of 2-Chloro-2-methylbutane from 2-methyl-2-butanol
Glassware:
Separatory funnel

Table of Reagents:

<table>
<thead>
<tr>
<th>Compound</th>
<th>Structure</th>
<th>Molecular Weight (g/mol)</th>
<th>Amount used, mL</th>
<th>Boiling point or B.P. (°C)</th>
<th>Melting Point (°C)</th>
<th>Density</th>
</tr>
</thead>
<tbody>
<tr>
<td>2-Methyl-2-Butanol</td>
<td>H₃C⁻OHCH₂CH₃⁻</td>
<td>88.1 g/mol</td>
<td>10.0</td>
<td>102.4 °C</td>
<td>-9.1 °C</td>
<td>0.805 g/mL at 25 °C</td>
</tr>
</tbody>
</table>

Reaction Apparatus set up:

---

Separatory Funnel
Retort Clamp

Erlenmeyer Flask

Platform for E Flask, not used as hot plate

Experimental Procedure:

1. Set up the reaction apparatus according to instructor’s instructions and demonstration. Recall extraction lab, use of a separatory funnel.

2. Using a dedicated graduated cylinder, transfer 10 mL of 2-methyl-2-butanol to the separatory funnel.

3. Again using a dedicated graduated cylinder, add 25 mL of concentrated HCl to the separatory funnel.

4. Without the stopper, gently swirl the contents in the funnel for 1-2 minutes.
5. With the stopper, carefully invert it, and release the excess pressure by opening the stopcock as you did in the extraction lab. Mix the contents in the funnel by shaking for additional 1-2 minutes. Allow the mixture to separate into 2 distinct layers.

6. Remove the aqueous layer into a 125 mL Erlenmeyer flask. Save it for the time being.

7. Wash the organic layer with 10 mL of saturated aqueous NaCl. Stopper the flask. Mix the contents by shaking. Drain the aqueous layer into a 125 mL Erlenmeyer flask.

8. Wash the organic layer with 10 mL of cold saturated NaHCO₃ to neutralize the acid. Initially, vigorous gas evolution will normally occur; gently swirl the unstoppered separatory funnel until it stops. Stopper the separatory funnel, carefully invert it, and release the excess pressure. Drain the aqueous layer into the 125 mL Erlenmeyer flask used in step 6.

9. Wash the organic layer with 10 mL of saturated aqueous NaCl. Drain the aqueous layer into the 125 mL Erlenmeyer flask used in step 7.

10. Collect the 2-chloro-2-methyl butane in a clean dry 25 mL Erlenmeyer flask. Observe the presence of water in your product.

11. Add one spatula tip full of anhydrous Na₂SO₄ and dry the product by gently swirling in the flask for about three minutes. If needed, continue the drying step.

12. Transfer the product into a preweighed vial and obtain weight of your product.

13. Perform the Silver Nitrate and Sodium Iodide tests on your product.

14. Label and turn in your product to the instructor.

**Chemistry of tests for halides:**

\[
\begin{align*}
R-X + NaI & \rightarrow R-I + NaX(s) \\
R-X + AgNO₃ + CH₃CH₂OH & \rightarrow \text{CH}_3\text{CH}_2\text{OH} \\
R-\text{OCH}_2\text{CH}_3 + AgX(s) + \text{HNO}_3 & \rightarrow
\end{align*}
\]
I. Test for halides

a. Silver Nitrate Test

1. Transfer 1ml of 0.1 M alcoholic AgNO3 to two 13x100 mm test tubes. Label the test tubes as 1 and 2.

2. Add one drop of your alkyl halide product to the AgNO3 containing test tube #1 and one drop of tert-amyl alcohol to test tube #2. Gently mix the contents.

b. Iodide Test

1. Transfer 1 ml of 1% sodium iodide to two 13x100 mm test tubes.

2. Add one drop of your alkyl halide product to the sodium iodide containing test tube #1 and drop of tert-amyl alcohol to test tube #2. Gently mix the contents. Allow to stand for 3 minutes.

3. Note precipitate formation and record your results. If there is no change, place the test tubes in a 50 C water bath for 6 minutes.
CHE 203L

Organic Chemistry II Laboratory

EXPERIMENT # 8

Title: Identification of Organic Compounds by Qualitative Tests

Experiment to be done in pairs. Only two students per group. Wear safety goggles.

Background

Organic chemists must regularly identify the compounds that are synthesized via chemical reactions or isolated from natural sources. Over 11 million different organic compounds have been discovered, synthesized, and characterized. These are divided into categories on the basis of functional group present. To identity an unknown organic compound requires finding which functional groups it contains and then determining its molecular and three-dimensional structure. Both chemical and spectroscopic methods are used by practicing organic chemists. As you progress through organic chemistry, you will have opportunities to learn and use the techniques of organic qualitative analysis and spectroscopic methods. In this lab you will perform Qualitative Tests for some of the common functional groups you have encountered in Chapter 3 of your Organic Chemistry Text.

One of the goals of this experiment is to distinguish alcohols, aldehydes and ketones based on the Lucas test and Chromic Acid Test

Organic Compounds:

1. 1-Butanol
2. 2-Butanol
3. 2-Methyl-2-Propanol
4. 3-Methylbutanal (isovaleraldehyde)
5. p-Tolualdehyde
6. Cyclohexanone
7. Ethyl acetate

Materials:

1. Test tube racks
2. Small test tubes, 10x75mm or 12x75mm
3. Plastic pipettes with graduation marks
4. Timer
5. 

Test Reagents:

1. Lucas reagent (HCl-ZnCl2). Prepared with ZnCl2 in conc. HCl
2. Chromic acid reagent
3. 2,4- Dinitrophenylhydrazine reagent
Test Reagents:

1. Lucas Reagent (HCl-ZnCl₂): Distinguishes between primary, secondary and tertiary Alcohols. Tertiary alcohol reacts very fast with cloudy precipitation; secondary alcohol reacts slower than the tertiary alcohol and shows cloudy precipitation. Primary alcohol does not give any reaction.

2. Chromic Acid test for alcohols: Distinguishes primary, secondary and tertiary Alcohols: Primary alcohols are oxidized to carboxylic acids, secondary alcohols are oxidized to ketones, tertiary alcohols are resistant to oxidation.

3. Chromic Acid test for distinguishing aldehydes and ketones: Aldehydes are oxidized to carboxylic acids, ketones are resistant to oxidation.

4. 2, 4-Dinitrophenylhydrazine reagent: Formation of a colored precipitate is a positive test for aldehydes and ketones. Alcohols, esters, carboxylic acid show a negative reaction.

Chemical basis of Identification Tests:

1. Lucas reagent - Distinguishes between primary, secondary and tertiary alcohols.

Test compounds # 1, 2 and 3. (Chapter 11, S_N1 mechanism)

2. Chromic acid reagent (Cr⁶⁺ ion is the reactant here) - Distinguishes between primary, secondary and tertiary alcohols. Primary alcohols are oxidized to carboxylic acids; secondary alcohols are oxidized to ketones, tertiary alcohols are resistant to oxidation. THE OXIDIZING AGENT gets reduced.

Test compounds # 1, 2 and 3.

3. Chromic acid - Test - Also distinguishes aldehydes and ketones. Aldehydes are oxidized to carboxylic acids, ketones are resistant to oxidation. Why?

Test compounds # 4, 5 and 6

4. 2, 4-Dinitrophenylhydrazine reagent: formation of a colored precipitate is a positive test for aldehydes and ketones. Alcohols, esters, carboxylic acid show a negative reaction. If p-tolualdehyde is the aldehyde, the product is called p-tolualdehyde 2, 4- dinitrophenylhydrazone.

Test compounds # 1, 4, 6 and 7.

Reaction chemistry for oxidation:

A. Primary alcohols
B. Secondary alcohols

\[
\begin{align*}
\text{H} & \quad \text{R} - \text{C} - \text{O} - \text{H} \quad \text{oxid} \quad \text{R} - \text{C} - \text{O} \quad \text{oxid} \quad \text{R} - \text{C} - \text{O} \\
\text{H} & \quad \text{aldehyde} \quad \text{alcohol} \quad \text{acid} \\
\end{align*}
\]

With a mild oxidizing reagent  With a strong oxidizing reagent

Procedure:
In each test, use a separate test tube for each compound. Label your tubes with a marker.

I. Tests for Alcohols:

A. Lucas test for alcohols: Compounds 1, 2 and 3.

1. Use a separate test tube (10x75mm or 12x75mm) for each compound and add 0.1ml (2-3 drops) of each compound to the test tube.
2. Add dropwise, 4-10 drops of Lucas reagent (HCl-ZnCl₂) to the compound in a drop-wise fashion. **Mix each time upon the addition of each drop, note any change and record your observations** that you will need for writing your lab report.
   
   **Note:** Once you note a change, add only 1-2 additional drops of Lucas reagent. **Keep a record of # of drops added.**

3. Continue examining the contents of the tube for one minute. Record your observations.

B. Chromic acid test for alcohols: Compounds 1, 2 and 3

1. Dissolve one drop of alcohol reagent in 0.5 to 1ml of reagent grade acetone.
2. Add one drop of chromic acid reagent. Mix quickly.
3. Allow the solution to stand and observe the color change. Note the time for any change in appearance and color up to one minute.
II. Tests for Aldehydes and Ketones

A. 2,4-Dinitrophenylhydrazine test: Compounds 1, 4, 5, 6 and 7.

1. Add 2 drops of your aldehyde or ketone (50mg is a solid) to 0.5 ml of 95% ethanol in a test tube. Mix the contents. If compound is not soluble, see your instructor.
2. To the above test tube add 3 to 5 drops of the 2, 4-dinitrophenylhydrazine reagent in a drop-wise fashion and mix. Note the time and color of the precipitate. Formation of a colored precipitate is a positive test for aldehydes and ketones.

B. Chromic acid test for aldehydes. To distinguish aldehydes from ketones: Test Compounds 4, 5 and 6.

1. Dissolve one drop of aldehyde and ketone (10-20mg if solid) in 0.5 to 1ml of reagent grade acetone. (How will you do it?)
2. Add one drop of chromic acid reagent. Mix. Observe the color change that very second.
3. Allow the solution to stand for up to one minute and observe the color change. Note the time for any change in appearance and color. Note: For Chromic acid test, color change occurring after one minute is not a positive test.

Prelab question. Will be collected at the beginning of the lab period.

Post lab question

Write the mechanism for the Lucas (HCl-ZnCl₂) test with 1-butanol, 2- butanol and 2-methyl-2-propanol. Lucas reagent is ZnCl₂ in conc. HCl.
CHE 204L

Organic Chemistry II Laboratory

EXPERIMENT # 1

Title: Infrared (IR) Spectroscopy of Organic Compounds

Introduction

During the course of this semester we will study several different classes of compounds including alcohols, alkenes, aldehydes, ketones, esters, amides and carboxylic acids. Each of these classes is distinguished by the presence of a "Functional Group" in the molecule. For example, all alcohols contain an 'O-H' group attached to a sp³ hybridized carbon atom. Alkenes contain a carbon-carbon double bond (C=C), and carbonyl compounds contain a carbon-oxygen double bond (C=O). As you will learn, IR may actually be thought of as a Functional Group detector. The quickest and easiest way to determine the presence of one of these "Functional Groups" is to take the IR spectrum of the compound. The technique is simple and can often provide a definitive answer in less than ten minutes. Evidence provided by IR is widely respected. It is commonly used in judicial court proceedings as much as fingerprints are used. In fact, the IR of a pure compound bears the same relationship to that compound as fingerprints do to an individual. The focus in this lab exercise is not to discuss the theory of IR Spectroscopy in detail, but to focus on the interpretation of an IR Spectrum.

An IR instrument consists of an IR light source, a sample holder, a means of selecting individual wavelengths or frequencies of the light, some means of detecting the amount of incident light that the sample absorbs, and a device for plotting the amount of light absorbed as a function of wavelength or frequency. This plot is referred to as the 'IR Spectrum.' Since IR light is absorbed by most materials, the optics of an IR Spectrophotometer requires special materials. Most frequently they are built of NaCl or KBr — water soluble salts. This requires that the IR spectrophotometers be protected from moisture of any form. The instrument is usually sealed, but the windows, and the sampling devices require special handling. In this instrument, aqueous samples should never be used, and the sample holders must be cleaned with DRY organic solvents like reagent grade acetone. In this lab students will use Nicolet 6700 FT-IR instrument. FT-IR (Fourier Transform Infrared) is a method of obtaining infrared spectra by first collecting an interferogram of a sample signal using an interferometer, and then performing a Fourier Transform (FT) on the interferogram to obtain the spectrum. An FT-IR Spectrometer collects and digitizes the interferogram, performs the FT function, and displays the spectrum.

Sampling

The draw-backs with old model IR Spectrometers used to be is that the instruments were expensive and delicate cells were needed. IR spectra of solids and liquids were usually obtained by dissolving the sample in a relatively IR transparent solvent such as CCl₄ and using simple
liquid cells. A solid was ground to a fine paste with NUJOL™ (a mixture of highly purified hydrocarbons) and the resulting 'mull' studied directly. The NUJOL exhibited only a very few well defined peaks that can be ignored when examining the spectrum of the mull. Solid spectra may also be obtained by mixing the solid with dry KBr, grinding to a fine, well mixed powder, and then forming a disk of the mixture by applying high pressure in a specially designed device. Modern IR instruments avoid the use of NUJOL and need for making KBr pellets. Now IR spectra can be determined for solids, liquids, plastic films and gases with much ease. IR gas analysis is a common analytical tool for those involved in studies of atmospheric pollution and substance abuse e.g. alcohol vapor. The new FT-IR spectrometers avoid these tedious and laborious sample preparations and hence are easier to use. Excellent spectra can be obtained in a matter of a few minutes. This technique is called running a "neat" spectrum, meaning the spectrum is of the pure liquid only, without solvent.

Objective

1. To learn various functional groups encountered in Organic Chemistry
2. To learn of the important role of infrared spectroscopy in the study of structure of organic compounds
3. To develop skill in the recognition of characteristic absorption bands
4. To identify a compound by an interrogation of its infrared spectrum

Procedure:

The instructor will demonstrate you the procedure. You will take an IR spectrum of any TWO unknown compounds, one liquid and one solid and interpret the spectrum as part of this lab report. You should attempt to narrow the list of your unknown and make your best attempt to identify the compound as part of your lab report.

Note: After you have used the compound for your IR, please move the test tube containing the compound to a test tube rack marked "compounds used". This will allow each group/student use a different compound.

The following compounds will be provided to you. You will take an IR spectrum of TWO unknown compounds, one liquid and one solid and interpret the spectrum as part of this lab report.

- trans-Cinnamaldehyde
- trans-Cinnamic acid
- Cyclohexanol
- 9-Fluorenone
- 3-Methylbutanal
- Benzoic acid
- 4-Fluorobenzaldehyde
- Ethyl 4-aminobenzoate (benzocaine)
- 2-Fluorobenzaldehyde
Steps for obtaining an IR spectrum:

1. Click on Collect sample. It will prompt you to collect background spectra.
2. Take a background spectrum. After done, it will prompt you to collect sample but wait until you have loaded the sample.
3. Place your compound in the groove.
4. Rotate the coarse control knob in a clockwise to lower the diamond tip to come in close contact with the sample.
5. Click on collect sample spectrum, when done, click on analyze on top tool bar and click on identify. Identify the peaks. Save your spectra in a new window.
6. Print your spectrum by clicking the print in the tool bar on the left. Do not click on the horizontal print button.
7. Clean the diamond tip and sample plate thoroughly using minimal volume of acetone to make it ready for the next sample.

Interpret the key IR bands on your IR spectrum and submit it as part of lab report.

Reference Spectra

To the student: Attempt to interpret these IR spectra (specific IR band and functional group) and ask the instructor for help.

The Infrared spectra of thousands of compounds have been determined and compiled by several different companies. Two of the most popular collections are the Sadler Index of IR Spectra and the Aldrich Library of Infra-red Spectra. Both collections are easily accessible in 'hard copy' form in most major university libraries. They are also available in computer readable format for rapid searching and spectrum matching. All modern FT-IR spectrometers are controlled by computers. The operating system often has the capability of searching one or more databases of spectra and finding the spectra that most closely match the spectrum that was just run. It is always a good idea to compare your IR spectrum with an authentic spectrum of the material you think you have. ALL IR Spectra in this manual are reproduced with permission from: The Aldrich Library of FT-IR Spectra, Edition 1, Charles J. Pouchert, Volume 1, 1985.
Figure 1: Methyl formate - An ester
Figure 2: Isopentyl acetate - An ester
Figure 3: 3-Pentanone - A ketone
CH₃CH₂CH₂CH₂CH₂CH₂OH

**Figure 4:** 1-Hexanol - An alcohol

**Figure 5:** Isopropylbenzene (cumene) - An aromatic hydrocarbon (page 8)
Isopropylbenzene (cumene) (neat)

#11 AROMATIC HYDROCARBON

\[ \text{cyclohexane} \]

Resolution: 4,000

Scans: 4

% Transmittance

Wavenumbers (cm\(^{-1}\))

4000

3500

3000

2500

2000

1500

1000

500

61
Figure 6: 2-Methyl-2-propanol
CHE 204L

Organic Chemistry II Laboratory

EXPERIMENT # 2

Title: Diels-Alder Reaction: Preparation of 4-Cyclohexene-cis-1,2-dicarboxylic Acid Anhydride from Sulfolene and Maleic Anhydride

Objective:

1. Preparation of 4-cyclohexene-cis-1,2-dicarboxylic acid anhydride by using a microscale method
2. Familiarize with i) reflux condensation methodology and ii) the control of gaseous products.

Introduction: This experiment demonstrates the use of the Diels-Alder reaction in the preparation of six-membered carboxylic rings. The cyclic products are obtained by reaction of a conjugated diene with a dienophile (an alkene with electron-withdrawing group(s)). The illustration given here involves the treatment of 1,3-butadiene (generated in situ) with maleic anhydride to form the corresponding Diels-Alder product. These addition products are often called adducts.

The Diels-alder reaction is an organic chemical reaction between a conjugated diene and a substituted alkene, commonly termed the dienophile to form a substituted cyclohexane system or a six-membered ring in a single step. A diene is a molecule containing two carbon-carbon double bonds. Dienophiles are molecules with double bonds that react with conjugated dienes in Diels-Alder reactions. They normally have an electron-withdrawing group attached. In this reaction, three pi bonds; two in the diene and one in the dienophile reorganize to give a six-membered ring containing one pi bond and two sigma bonds. All of these bonds move in a concerted manner.

Reaction Mechanism

\[
\text{Sulfolene} \xrightarrow{\text{heat}} \text{Maleic Anhydride}
\]

The diene produced from 3-sulfolene reacts with maleic anhydride (dienophile) as follows:
Table of Reagents:

<table>
<thead>
<tr>
<th>Compound</th>
<th>Structure</th>
<th>Molecular Mass</th>
<th>Molecular Formula</th>
<th>Physical Properties</th>
</tr>
</thead>
<tbody>
<tr>
<td>3-Sufolene</td>
<td><img src="image" alt="Structure" /></td>
<td>118.15 g/mol</td>
<td>$C_4H_6O_2S$</td>
<td>MP: 66°C&lt;br&gt; Density: 1.3 g/cm$^3$</td>
</tr>
<tr>
<td>Maleic Anhydride</td>
<td><img src="image" alt="Structure" /></td>
<td>98.06 g/mol</td>
<td>$C_4H_2O_3$</td>
<td>Decomposes upon heating&lt;br&gt;Density: 1.48g/cm$^3$</td>
</tr>
<tr>
<td>Xylene</td>
<td><img src="image" alt="Structure" /></td>
<td>106.16 g/mol</td>
<td>$C_8H_10$</td>
<td>Density 0.862 g/mL</td>
</tr>
</tbody>
</table>

MATERIALS AND EQUIPMENTS:
1. Weighing balance
2. Conical vial
3. Air condenser
4. Sand bath
5. Buchner funnel
6. Filter paper
7. Glass rod
8. Scooper
9. Thermometer
10. Ice bath
11. Vacuum filtration apparatus
Procedure:

1. Weigh and place 340 mg of 3-sulfolene, 180 mg of maleic anhydride and 160 μL of xylene in a 5.0 mL clean conical reaction vial. Add a boiling stone.
2. Properly attach the conical reaction vial with an air condenser protected with a calcium chloride drying tube.
3. Heat the reaction mixture at reflux, using a sand bath at 170°C for 20 min.

CAUTION: Avoid overheating. Sulfur dioxide is evolved in the process and adequate ventilation should be provided.

Preparation of 4-cyclohexene-cis-1, 2-dicarboxylic acid anhydride

Procedure:

4. Weigh and place 340 mg of 3-sulfolene, 180 mg of maleic anhydride and 160 μL of xylene in a 5.0 mL clean conical reaction vial. Add a small boiling stone.
5. Properly attach the conical reaction vial with an air condenser protected with a calcium chloride drying tube as shown in Fig. 3.1
6. Heat the reaction mixture at reflux, using a sand bath at 170°C for 20 min.

CAUTION: The reaction is exothermic. Avoid overheating. Sulfur dioxide is evolved in the process and adequate ventilation should be provided.

Isolation of Product

1. Add 1.0 mL of toluene to the cooled solution in the original conical reaction vial, and then add petroleum ether (BP, 60-80°C) drop wise with mixing until a slight cloudiness persists. Roughly 0.50-0.70 mL of petroleum ether will be needed.
2. Reheat the solution in the sand bath until it becomes clear and then cool it in an ice bath. During the recrystallization step, the sides of the vial may have to be scratched with a glass rod to induce crystallization. Wait for recrystallization of your product.

Vacuum filtration:

3. Place the filter paper in the Buchner or Hirsch funnel. With vacuum on, transfer a few drops of ice cold petroleum ether to have a complete seal. By tapping the conical vial, transfer its contents to the center of filter paper in the Buchner or Hirsch funnel. Collect the crystalline product by vacuum filtration, rinse the conical flask with minimal amount of cold petroleum ether and thoroughly wash the filter cake on the Buchner or Hirsch funnel with 1.00 mL of ice cold petroleum ether as demonstrated by the instructor in previous labs.
CAUTION: Do not wash with a warm or an excess of the petroleum ether; a loss of product will result.

Characterization

1. Weigh your cycloaddition product, calculate the moles of product and calculate the percent yield.

2. Determine the melting point of a finely divided sample and compare your result with the value found in the literature.

3. Obtain an IR spectrum of your product. Interpret IR spectrum of your product(s) as part of your lab report.
CHE 204L (Organic Laboratory II)

EXPERIMENT # 3

Title: Relative Rates of Electrophilic Aromatic Bromination

Introduction:

An Electrophilic aromatic substitution is one in which a hydrogen atom on an aromatic ring is replaced by a strong electrophile. It is general knowledge that the substituents on the ring affect the rate of reaction as well as the orientation of the electrophile with respect to the existing substituents. Activators, which generally have a free pair of electrons at the benzylic position, allow further substitution to occur at ortho and para positions. Activators also increase the rate of the reaction compared to that of un-substituted benzene. For instance, the presence of the –OH substituent makes the ring a million times more reactive for aromatic nitration than benzene. Deactivators other than halogens favor the Meta position. When an aromatic ring that has two substituents i.e. an activating group and a deactivating group, it is the activating group that dictates the further substitution. Preferences for a given orientation of attack are explained by resonance effects and inductive effects. This is related to the degrees to which the positively charged intermediate is stabilized by the nature of the existing substituents.

The purpose of this experiment is to use data obtained from the bromination of a family of similar compounds to explore the relationship between structure and reactivity. The reactivity of aromatic ring can be affected by the substituent attached to it. The substituent on the aromatic ring do this by increasing and decreasing the electron density of the delocalized ring of electron around the carbon atoms, either resonance or inductive effects. Inductive effects occur when electrons are withdrawn or donated to the benzene ring via sigma bonds. Resonance effects occur when a substituent(s) withdraw or donate electrons to the ring via overlapping pi orbitals. As such substituents are grouped as ortho- and para-directing activators, ortho- and para-directing deactivators and meta-directing deactivators.

![Figure 1](image)

Fig. 1

Objectives:

1. Ascertain the relative reaction rates of bromination of various aromatic compounds
2. Determine how rings substituents affect the rate of bromination of aromatic compounds
AS ALWAYS FOLLOW LABORATORY SAFETY GUIDELINES PROVIDED to YOU. This is a simple lab but you can learn a lot from it. This lab reinforces subject matter you learn in chapter 16.

**Aromatic compounds:**
The following compounds will be used for electrophilic aromatic bromination:

1. Phenol

2. 1-Naphthol

3. Anisole

4. N-Methylaniline

5. Acetanilide

6. *para*-Hydroxybenzoic acid

7. Toluene

**Reagents:**

1. 15 M acetic acid
2. 0.05 M Br₂ solution in 15 M acetic acid

*Unless labeled otherwise, all of the above aromatic compounds are provided as 0.2M solutions in 15 M acetic acid.*

**PROCEDURE:**

1. Obtain 7 large test tubes (18 x 150mm) and label each with the name of the aromatic compound. Place the test tubes in a rack.

2. Using a glass Pasteur pipette, transfer 1.5mL of the aromatic compound in to each test tube and place it in the rack. (Use Pasteur pipettes for delivery)

3. Obtain a digital timer to accurately measure the reaction time. (Place test tube racks over a white background)

4. Working in the fume hood, use a dedicated 50 mL graduated cylinder to carefully transfer approximately 20 mL of 0.05 M Br₂ solution (prepared in 15 M acetic acid) into a 125 mL
Erlenmeyer flask. Cover this Erlenmeyer flask with a clean cork stopper but do not stopper the Erlenmeyer flask. Keep the Br₂ solution in the fume hood.

6. Use a pre-marked Pasteur pipette to deliver 1.5mL of 0.05 M Br₂ solution in 15 M acetic acid and add quickly to one of the tubes. Continuously mix the contents of the test tube.

Use a stop watch and carefully record the time when Br₂ color disappears.

7. Repeat steps for all the aromatic compounds

8. If more than one substrate reacts instantaneously at room temperature repeat the reaction at 0°C in an ice bath

9. If the times for more than one substrate are close to one another, run the reactions side by side adding Br₂ to each at the same time.
CHE 204L (Organic Chemistry II Laboratory)

EXPERIMENT #5

Title: Electrophilic Aromatic Substitution- Friedel Crafts Alkylation: Preparation of para-tert-Butylphenol (4-tert-Butylphenol)

Introduction:

Something chemically remarkable happens when three conjugated double bonds are incorporated into a six-membered ring to produce a so-called aromatic compound. In sharp contrast to the addition reaction that typify alkenes and alkynes, the chemistry of aromatic molecules is characterized by substitutions, that is, transformations in which a portion on the ring is replaced by some other atom or functional groups. We will be exploring one of these types of fascinating and very useful reactions. You will have the opportunity not only to execute reactions in which functional groups are introduced onto the aromatic ring but also to study both qualitatively and quantitatively the effects that substituents on the ring have on rates at which these substitutions occur. This experiment should help you to understand the remarkable chemistry of aromatic compounds.

Electrophilic aromatic substitution is an important reaction in organic chemistry that allows the introduction of many different functional groups onto an aromatic ring. A general form of the reaction is given by Equation 4.1, where Ar-H is an aromatic compound, an aromatic compound, an arene, and $E^+$ represents the electrophile formed in the first step and it is this very electrophile that is replaces an hydrogen on the ring. This equation is oversimplified because the electrophile is usually generated during the reaction, and a Lewis base assists in the removal of $H^+$. The general reaction form is shown as:

$$\text{Ar} - \text{H} \quad + \quad E^+ \quad \rightarrow \quad \text{Ar} - \text{E} \quad + \quad H^+$$

An arene \hspace{1cm} An electrophile \hspace{1cm} A substituted arene

Main Reaction:

$$\text{catalyst}$$

$$\text{Phenol(s) + tert-Butyl chloride (l) } \quad \rightarrow \quad p\text{-tert-Butyl phenol(s)} \quad + \quad \text{HCl (g)}$$
The general reaction form is shown as:

\[ \text{E}^+ + \text{Ar} \xrightarrow{\text{slow}} \text{E} \xrightarrow{\text{ArCl}} \text{E}^- + \text{H}^+ \]

**Mechanism:** Has three steps as shown below. These are a) formation of a strong electrophile, b) formation of cyclohexadienyl cation (areniion ion) intermediate, and c) the formation aromatic compound.

The Friedel-Crafts alkylation reaction using alkyl halides is a classic example of electrophilic aromatic substitution. As a versatile method for directly attaching alkyl groups to aromatic ring, it is a process of great industrial importance. The three main limitation to this reaction as a synthetic tool are: (1) the substitution fails if the aromatic ring carries strongly ring-deactivating groups, such as NO₂, R3N⁺, C(O)R, and CN; (2) more than one alkyl groups may be introduced onto the aromatic ring, a process termed polyaalkylation, and (3) mixtures of isomeric products may be formed if the alkyl group rearranges. The instructor may already have covered these topics during the CHE 204 lecture class. You are encouraged to ask questions during the lab; this is a very fascinating and also very important aspect of organic chemistry.

The rates of electrophilic aromatic substitutions are generally second-order overall, being first order in both the aromatic component and the electrophile as shown below.

\[ \text{Rate} = K_2 \left(\text{Ar-H}\right) \left(\text{E}^+\right) \]  

**Equation 4.2**
Aromatic compounds undergo via a substitution mechanism rather than an addition. This happens because of the increased stability of the aromatic ring, it is energetically favorable for the substitution mechanism to take place. Many different substituents can be introduced to the aromatic ring halogens, hydroxyl, carboxylic groups, acyl groups and alkyl groups. The Friedel Crafts reactions are a set of reactions developed by Charles Friedel and James Crafts in 1877 to attach substituents to an aromatic ring. Friedel–Crafts alkylation involves the alkylation of an aromatic ring with an alkyl halide using a strong Lewis acid catalyst, AlCl₃ in this experiment.

**Objective:** To prepare \( p\text{-}\text{tert}\)-butylphenol (4-\( \text{tert}\)-Butylphenol) using Friedel Crafts Alkylation.

**Table of reagents:**

<table>
<thead>
<tr>
<th>Name, Molecular wt.</th>
<th>Structure</th>
<th>State at Room temperature</th>
<th>Melting point(°C)</th>
<th>Density g/mL</th>
<th>Solubility</th>
<th>Molecular wt.</th>
</tr>
</thead>
<tbody>
<tr>
<td>tert-butyl chloride</td>
<td><img src="image" alt="Structure" /></td>
<td>liquid</td>
<td>51</td>
<td>0.84</td>
<td>sparingly</td>
<td>92.57</td>
</tr>
<tr>
<td>Phenol</td>
<td><img src="image" alt="Structure" /></td>
<td>solid</td>
<td>40.5</td>
<td>1.07</td>
<td>yes</td>
<td>94.11</td>
</tr>
</tbody>
</table>

**Procedure:**

1. Place 2.0 mL of \( p\text{-}\text{tert}\)-butyl chloride in a 25 or 50mL Erlenmeyer flask.
2. Carefully add 1.45g of phenol into the flask, and stir with glass rod (or metal spatula) until the phenol is completely dissolved.
3. PLACE THE FLASK IN THE HOOD.
4. With continuous stirring with a clean and dry glass rod, slowly and carefully add in parts at a time, a total of 0.15g of granular anhydrous aluminum chloride.
5. Stir the contents for 5 more minutes.
6. Place the flask in the hood with glass rod.
7. The contents of the flask will solidify in 20-30 minutes.
8. When the reaction mixture has solidified, add 8mL of purified water and break up the solid with a metal spatula until it is finely divided.
9. Check the pH using a glass rod, it should be acidic. If it is not, add 0.6 mL of concentrated HCl.
10. When all the clumps are broken, vacuum filter to collect the solid and dry as much as possible.
11. Transfer the product into a 25mL clean beaker.

**Recrystallization of para-tert-Butylphenol**

1. Using a Pasteur pipette, add 0.9 ml of boiling ether (from the steam bath) to the dissolve the product. Keep the beaker on the top of steam bath during this step.

2. Cool to room temperature and then place the beaker in an ice water bath.

3. Collect the recrystallized product by vacuum filtration. Wash the filter cake thoroughly with 2 mL of ice cold water.

4. Air dry the product in a lab drawer; determine the weight and the melting point of the product of dry product. Compare your result with the value found in the literature.

5. Calculate the percent yield of your product. Obtain an IR spectrum of your product. Interpret IR spectrum of your product(s) as part of your lab report.
CHE 204L (Organic Chemistry II Laboratory)

EXPERIMENT #5

Title: Identification of Aldehydes and Ketones by Derivatization with 2,4-Dinitrophenylhydrazine Reagent

Background

Aldehydes and ketones are organic compounds which incorporate a carbonyl functional group, C=O. The carbon atom of this group has two remaining bonds that may be occupied by hydrogen or alkyl or aryl substituents. If at least one of these substituents is hydrogen, the compound is an aldehyde. If neither is hydrogen, the compound is a ketone. In this laboratory you will prepare a 2, 4-dinitrophenylhydrazone derivative of your unknown aldehyde or ketone, determine the melting point of your dry derivative and identify your unknown by comparing the published melting points tables provided to you.

Reaction with amines:

The reaction of aldehydes and ketones with ammonia or 1°-amines forms imine derivatives, also known as Schiff bases, (compounds having a C=N function). Reaction with 2, 4-Dinitrophenylhydrazine reagent leads to the formation of a colored precipitate which serves as a positive test for aldehydes and ketones. Alcohols, esters, carboxylic acid show a negative reaction. This reaction is shown as follows.

With the exception of unsubstituted hydrazones, these derivatives are easily prepared and are often crystalline solids - even when the parent aldehyde or ketone is a liquid. Since melting points can be determined more quickly and precisely than boiling points, derivatives such as these are useful for comparison and identification of carbonyl compounds. If the aromatic ring of phenylhydrazine is substituted with nitro groups at the 2- & 4-positions, the resulting reagent and the hydrazone derivatives it gives are strongly colored, making them easy to identify.

The rate at which these imine-like compounds are formed is generally highest at a pH of 5, and drops at pH higher than 5 and at pH lower than 5. This agrees with a general acid catalysis in which the conjugate acid of the carbonyl reactant combines with a free amino group, as shown in the above animation. At high pH there will be a vanishingly low concentration of the carbonyl conjugate acid, and at low pH most of the amine reactant will be tied up as its ammonium conjugate acid. With the exception of imine formation itself, most of these derivatization reactions do not require active removal of water (not shown as a product in the previous equations). The reactions are reversible, but equilibrium is not established instantaneously and the products often precipitate from solution as they are formed.
Formation of 2, 4- dinitrophenylhydrazone derivative with acetone:

Acetone in ethanol solvent

+ 

2, 4- dinitrophenylhydrazine reagent

Acetone 2, 4- dinitrophenylhydrazone, mp 126°C

Procedure:

1. Add 4 drops your liquid or 100- 200mg of solid unknown to a clean test tube containing 2 mL of ethanol. Dissolve the solid.

2. Add 1.0 mL of 2, 4- dinitrophenylhydrazine reagent drop wise with continuous mixing. Record your observations.

b. As a control add 2drops of botanic acid to a test tube. Add 2 to 3 drops of 2, 4- dinitrophenylhydrazine reagent drop wise with continuous mixing. Record your observations.

3. Place your test tube in a water bath at 60°C for 5-10 minutes. Cool the test tube to RT and then place in an ice water bath.

4. Collect the crystals of your product by vacuum filtration by rinsing the tube with about 1-2 mL of cold ethanol. Wash the crystals with ice cold 95% ethanol

5. Recrystallize the crystals from aqueous ethanol. How? Collect the crystals of your product by vacuum filtration by rinsing the tube with about 1-2 mL of cold ethanol. Wash the crystals with ice cold 95% ethanol

6. Dry the crystals as much as possible and carefully determine the melting point as accurately as possible.

7. Compare the melting points from the tables and identify your unknown.
CHE 204L (Organic Chemistry II Laboratory)

EXPERIMENT #7

Title: The Cannizzaro Reaction with 4-Chlorobenzaldehyde: Preparation of 4-Chlorobenzoic Acid and 4-Chlorobenzyl Alcohol

Introduction

The carbonyl group of aldehydes represents the intermediate stage of oxidation between an alcohol and a carboxylic acid. Cannizzaro reaction involves the base-induced disproportionation of an aldehyde lacking a hydrogen atom in the alpha position. In the presence of hydroxide ion, aldehydes that lack acidic alpha hydrogen atoms undergo a self-oxidation-reduction reaction. Under the influence of a strong base, one molecule of the aldehyde reduces a second molecule of aldehyde to the primary alcohol. This process is the oxidation of a corresponding carboxylate anion. Aldehydes with alpha-hydrogen atoms do not undergo this reaction, because the presence of a base results in the formation of an enolate ion.

The purpose of this lab is to demonstrate the simultaneous oxidation and reduction of 4-chlorobenzaldehyde, an aromatic aldehyde. These simultaneous processes shows the preparation of benzoic acid and benzyl alcohol. The experiment also shows the techniques used for separation of carboxylic acid from a neutral alcohol. The first step in the Cannizzaro reaction is the nucleophile attack of the hydroxide anion on the carbonyl group of the aldehyde. This attack is followed by the transfer of a hydrogen atom with its pair of electrons (a hydride ion) to the carbonyl group of an aldehyde.

Main Reaction

![Chemical Reaction Diagram]
### Table of Reagents:

<table>
<thead>
<tr>
<th>Compound</th>
<th>Structure</th>
<th>Molecular Weight (g/mol)</th>
<th>Melting Point (°C)</th>
<th>Boiling Point (°C)</th>
<th>Density</th>
</tr>
</thead>
<tbody>
<tr>
<td>4-Chlorobenzaldehyde</td>
<td><img src="image" alt="Structure" /></td>
<td>140.57 g/mol</td>
<td>47.5 °C</td>
<td>60°C</td>
<td>1.196 g/cm³</td>
</tr>
<tr>
<td>Methanol</td>
<td><img src="image" alt="Structure" /></td>
<td>32.04 g/mol</td>
<td>-97.6 °C</td>
<td>148.5 °C</td>
<td>0.792 g/cm³</td>
</tr>
<tr>
<td>Potassium Hydroxide</td>
<td><img src="image" alt="Structure" /></td>
<td>56.11 g/mol</td>
<td>406 °C</td>
<td>1,327 °C</td>
<td>2.12 g/cm³</td>
</tr>
</tbody>
</table>

### Materials

- 5 ml conical vial
- Magnetic spin vane
- Reflux condenser
- Automatic delivery pipette

### Procedure

1. Weigh 0.15 grams of 4-chlorobenzaldehyde and add to a 5 ml conical vial. Place a magnetic spin vane in the conical vial.
2. Using an automatic delivery pipette transfer .4 ml of methanol to the 5 ml conical vial.
3. With gentle swirling of the conical vial, add 0.55 mL of 11 M aqueous solution of potassium hydroxide to the conical vial; do not let any KOH solution on the ground-glass joint. Equip the conical vial with a water cooled reflux condenser and completely assemble the apparatus for heating under reflux as demonstrated by your instructor. Reflux the reaction mixture in a sand bath at 80 °C for 1 hour.
4. Cool the reaction mixture to room temperature and add 3.0 ml of ice chilled distilled water.
5. Extract the resulting solution with three 0.5 mL portions of dichloromethane. Three extractions are carried out as follows: after addition of 0.5 mL methylene chloride, cap the vial, shake gently, and then carefully vent by loosening the cap. Tighten the cap gently and vortex. Let the layers separate. The bottom layer is methylene chloride layer.
6. Transfer the methylene chloride layer using a Pasteur pipette and save this pipette for reuse for the remaining two extractions. The instructor will demonstrate this technique.
7. Complete the three extractions and pool the three extractions and label as methylene chloride extract.
8. Save both the methylene chloride extract and alkaline phase for further work-up. The reaction phase is complete.

Isolation of 4 - Chlorobenzyl Alcohol

1. Wash and combine methylene chloride extracts with two .25 ml portions of saturated sodium bicarbonate solution followed by one .5 ml portion of distilled water.
2. Remove the aqueous upper phase with a Pasteur filter pipette for each washing step and save this combined material in a separate 10 ml Erlenmeyer flask.
3. Add 0.28g of granular anhydrous sodium sulfate to the methylene chloride layer to dry the organic solvent.
4. After drying the solution, transfer the liquid by using a Pasteur filter pipette to a tared 3.0 ml conical vial. Be particularly careful not to let any granules of the hydrated sodium sulfate adhere to the surface of the pipette and become transferred with the dried organic phase.
5. Rinse the sodium sulfate drying agent with 0.3 ml of fresh methylene chloride and combine the rinse with the dried organic phase. Evaporate the methylene chloride using a stream of dry nitrogen gas in a warm sand bath in the hood. Crude 4-Chlorobenzyl alcohol will remain in the vial after removal of the solvent.
6. If time permits, purify the crude alcohol as follows: Recrystallize the solid from the conical vial with 0.25 mL of 4% acetone in hexane. Collect the product under reduced pressure using a Hirsh funnel, and wash the filter cake with 0.2 mL of ice-cold hexane. Air-dry the product on a porous clay plate or on a filter paper.
7. Weigh the 4-chlorobenzyl alcohol and calculate the percent yield. Determine the melting point and compare your value with that found in the literature.

Isolation of 4 - Chlorobenzoic acid

1. Dilute the alkaline phase obtained and saved during the original extraction procedure by adding 2.0 ml of water.
2. Acidify the dilute aqueous phase by the addition of 0.4 ml of concentrated hydrochloric acid.
3. Collect the voluminous white precipitate of the product that forms by use of a Hirsch or Buchner funnel. Rinse the filter cake with 2.0 ml of distilled ice cold water.
4. Air dry the products in a lab drawer; when dry, determine the weight and the melting point of each product. Compare your result with the value found in the literature.
5. Calculate the percent yield of each product.

Obtain an IR spectrum of each product. Interpret IR spectrum as part of your lab report.
CHE 204L

Organic Chemistry II Laboratory

EXPERIMENT #

TITLE: ISOLATION AND IDENTIFICATION OF CASEIN

INTRODUCTION

Casein is the most important protein in milk. It functions as a storage protein, fulfilling nutritional requirements in mammals. Casein can be isolated from milk by acidification to bring it to its isoelectric point. At its isoelectric point, the number of positive charges on the protein is equal to the number of negative charges. Proteins are least soluble in water at their isoelectric points because they tend to aggregate by electrostatic interaction. The positive end of one protein molecule attracts the negative end of another protein molecule, and the aggregates precipitate out of solution. On the other hand if a protein molecule has a net positive charge (at low pH or acidic condition) or a net negative charge (at high pH condition), its solubility in water is increased.

In this experiment which is carried out in two parts, casein was isolated from milk under isoelectric elections, and some chemical tests were performed to identify proteins. First, casein was isolated from milk which has a pH of about 7. Casein is isolated as in insoluble precipitate by acidification of the milk to its isoelectric point (pI of casein is 4.6). The fats that precipitate along with the casein are removed by washing the wet solid with ethanol and with ethanol ether mixture. In the second part of the experiment, the casein product is identified as a protein using the following tests: the biuret test, the ninhydrin test, the heavy metal test, and the xanthoprotein test.

At its isoelectric point (where net charge is zero), casein molecules have minimum solubility; Hence these aggregate out of the solution due to the electrostatic interactions between molecules; the positive end of one protein attracts the negative end of another protein. When a protein has a net positive charge in acidic conditions, or has a net negative charge in basic conditions, it has a higher solubility in water.

The pH of milk is in the range of 6.4-6.8. In this experiment, casein will be isolated from milk. This will be accomplished by acidification of milk to its isoelectric point (pI) of pH 4.6 by the gradual addition of acetic acid. The fats from milk will also precipitate with the casein and these will be removed by dissolving these in 95% alcohol followed by washing the solid casein with 1:1 mixture of diethyl ether-ethanol. You will perform four tests to prove the substance you isolated is indeed a protein. These tests are: the biuret test, ninhydrin test, heavy metal ions test, and xanthoprotein test.

CHEMICAL ANALYSIS OF CASEIN PROTEIN:
1. The **biuret test** is a test for the presence of amide (peptide) bonds; it is one of the most general tests for proteins. When a protein reacts with copper (II) sulfate, a positive test is the formation of a copper complex that results in a violet color.

![Biuret Test Reaction](image)

2. In the **ninhydrin test**, amino acids with a free –NH₂ group and proteins containing free amino acid groups react with ninhydrin to give a purple-blue complex.

![Ninhydrin Test Reaction](image)

3. **Heavy metal ions** precipitate proteins from solutions. The ions that are most commonly used for protein precipitation are Zn²⁺, Fe³⁺, Cu²⁺, Sb³⁺, Ag⁺, Cd²⁺, and Pb²⁺. The precipitation occurs because proteins become cross-linked by heavy metals.

![Heavy Metal Precipitation](image)

4. The **xanthoprotein test** is a characteristic reaction of proteins that contain phenyl groups. Concentrated nitric acid reacts with the phenyl ring, reaction called aromatic nitration to give a yellow-colored aromatic nitro compound.
Materials and Equipment:

1. Hot plate  
2. 25 mL, 50 mL or 100 mL graduated cylinders  
3. Buchner funnel  
4. 1000 mL filter flask  
5. Filter paper  
6. Cheese cloth  
7. Rubber band  
8. 95% ethanol  
9. Diethyl ether-ethanol mixture  
10. Regular milk  
11. Glacial acid  
12. Concentrated nitric acid  
13. 2% albumin  
14. 2% glycine  
15. 2% gelatin  
16. 1% tyrosine  
17. 5% copper (II) sulfate  
18. 5% lead (II) nitrate  
19. Ninhydrin reagent  
20. 10% sodium hydroxide

Objectives:

1. To precipitate the casein from milk under isoelectric conditions and isolate it.  
2. To perform chemical/biochemical tests to identify the common functional groups of proteins.

Procedure

1. Use a 50 mL graduated cylinder and add nearly 50.00 mL of milk, record the exact mass of milk and heat the flask in a water bath. Stir the solution constantly with a clean stirring glass rod. When the bath temperature has reached about 40 to 45°C, remove the
flask from the water bath, and add about 10 drops of glacial acetic acid while stirring. Observe the formation of a precipitate. Gently swirl the flask for about 5 minutes.

2. Filter the mixture into a 100-mL clean beaker by pouring it through a cheese cloth, which is fastened with a rubber band over the mouth of the beaker. Remove most of the liquid from the precipitate by squeezing the cloth gently. Discard the filtrate from the beaker in the sink. Using a spatula, scrape the precipitate from the cheese cloth into a clean 125 mL E flask.

3. Add 25 mL of 95% ethanol to the flask. Stir the mixture with a clean glass rod for nearly 3 minutes and allow the solid to settle. Carefully decant the liquid that contains fats into an E flask labeled ethanol wash.

4. To the solid residue, add 25 mL of a 1:1 mixture of diethyl ether-ethanol. Stir the mixture with the same glass rod for about three minutes and allow the solid to settle. Collect the solid by vacuum filtration.

5. Spread the casein on a clean paper towel and let it dry. Determine the mass of the dried casein. Use this mass to calculate the percentage of casein in the milk.

**Chemical Analysis of Proteins**

1. The biuret test.
2. The ninhydrin test.
3. The heavy metals ions test.
4. The xanthoprotein test.

*Note to the student:* Use caution as to not to cross contaminate the reagents by moving transfer pipets from one reagent flask/bottle to another.

**CHEMICAL ANALYSIS OF PROTEINS**

1. **THE BIURET TEST**

   Place 15 drops of each of the following solutions or as indicated in five labeled 13x100mm clean labeled test tubes.
   a) 2% glycine
   b) 2% gelatin
   c) 2% albumin. Use only 5 drops
   d) One quarter of a full spatula of Casein prepared in part A in 15 drops of distilled water
   e) 1% tyrosine

   To each test tube, while swirling add 5 drops of 10% NaOH solution and 2 drops of dilute CuSO₄ solution. Record your results and observations.
<table>
<thead>
<tr>
<th>SUBSTANCE</th>
<th>COLOR FORMED, Observations</th>
</tr>
</thead>
<tbody>
<tr>
<td>2% glycine</td>
<td></td>
</tr>
<tr>
<td>2% gelatin</td>
<td></td>
</tr>
<tr>
<td>2% albumin</td>
<td></td>
</tr>
<tr>
<td>Casein + H₂O</td>
<td></td>
</tr>
<tr>
<td>1% tyrosine</td>
<td></td>
</tr>
</tbody>
</table>

1. **THE NINHYDRIN TEST**

Place 15 drops of each of the following solutions were in five labeled 13x100mm clean labeled test tubes.

a) 2% glycine
b) 2% gelatin
c) 1% albumin, **use only 5 drops**
d) One quarter of a full spatula of Casein prepared in part A in 15 drops of distilled water
e) 1% tyrosine

To each test tube, while swirling add 5 drops of ninhydrin reagent and place the test tubes in boiling water bath for about 5 minutes. Record your results and observations.
<table>
<thead>
<tr>
<th>SUBSTANCE</th>
<th>COLOR FORMED, Observations</th>
</tr>
</thead>
<tbody>
<tr>
<td>2% glycine</td>
<td></td>
</tr>
<tr>
<td>2% gelatin</td>
<td></td>
</tr>
<tr>
<td>2% albumin</td>
<td></td>
</tr>
<tr>
<td>Casein + H₂O</td>
<td></td>
</tr>
<tr>
<td>1% tyrosine</td>
<td></td>
</tr>
</tbody>
</table>

2. **HEAVY METAL IONS TEST**

Transfer 2 mL of milk in each of two clean, labeled 13x100mm or 12x75 test tubes. Add a few drops of each of the following metal ions to the corresponding test tubes:

a) Pb²⁺ as aqueous Pb(NO₃)₂ solution
b) Na⁺ as aqueous NaNO₃ solution

<table>
<thead>
<tr>
<th>REAGENT</th>
<th>PRECIPITATE FORMED, Observations</th>
</tr>
</thead>
<tbody>
<tr>
<td>aqueous Pb(NO₃)₂</td>
<td></td>
</tr>
<tr>
<td>aqueous NaNO₃</td>
<td></td>
</tr>
</tbody>
</table>

3. **THE XANTHOPROTEIN TEST**

84
Place 15 drops of each of the following solutions were in five labeled 13x100mm clean labeled test tubes 2% glycine

a) 2% gelatin
b) 2% albumin, use only 5 drops
c) One quarter of a full spatula of Casein prepared in part A in 15 drops of distilled water
d) 1% tyrosine

In a FUME HOOD, while gently swirling, add 10 drops of concentrated HNO₃ dropwise to each test tube. Heat the test tubes were in a boiling water bath for about 5 minutes. Record your results and observations in the following chart.

<table>
<thead>
<tr>
<th>SUBSTANCE</th>
<th>COLOR FORMED, Observations</th>
</tr>
</thead>
<tbody>
<tr>
<td>2% glycine</td>
<td></td>
</tr>
<tr>
<td>2% gelatin</td>
<td></td>
</tr>
<tr>
<td>2% albumin</td>
<td></td>
</tr>
<tr>
<td>Casein + H₂O</td>
<td></td>
</tr>
<tr>
<td>1% tyrosine</td>
<td></td>
</tr>
</tbody>
</table>
APPENDIX A: Recycling codes

Recycling codes are used to identify the material from which an item is made, to facilitate easier recycling or other reprocessing. Such symbols have been defined for batteries, biomatter/organic material, glass, metals, paper, and plastics.

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Code</th>
<th>Description</th>
<th>Examples</th>
</tr>
</thead>
<tbody>
<tr>
<td>Plastics (see resin identification code[^1])</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>#1 PET(E)</td>
<td>Polyethylene terephthalate</td>
<td>Polyester fibers, soft drink bottles</td>
<td></td>
</tr>
<tr>
<td>#2 PEHD or HDPE</td>
<td>High-density polyethylene</td>
<td>Plastic bottles, plastic bags, trash cans, imitation wood</td>
<td></td>
</tr>
<tr>
<td>#3 PVC</td>
<td>Polyvinyl chloride</td>
<td>Window frames, bottles for chemicals, flooring, plumbing pipes</td>
<td></td>
</tr>
<tr>
<td>#4 PELD or LDPE</td>
<td>Low-density polyethylene</td>
<td>Plastic bags, buckets, soap dispenser bottles, plastic tubes</td>
<td></td>
</tr>
<tr>
<td>#5 PP</td>
<td>Polypropylene</td>
<td>Bumpers, car interior trim, industrial fibers, carry-out beverage cups</td>
<td></td>
</tr>
<tr>
<td>#6 PS</td>
<td>Polystyrene</td>
<td>Toys, flower pots, video cassettes, ashtrays, trunks, beverage/food coolers, beer cups, wine and champagne cups, carry-out food containers, Styrofoam</td>
<td></td>
</tr>
<tr>
<td>#7 O (OTHER)</td>
<td>All other plastics</td>
<td>Polycarbonate (PC), polyamide (PA), styrene, acrylonitrile (SAN), acrylic plastics/polyacrylonitrile (PAN), bioplastics</td>
<td></td>
</tr>
<tr>
<td>#9 or ABS</td>
<td>Acrylonitrile butadiene styrene</td>
<td>Monitor/TV cases, coffee makers, cell phones, most computer plastic</td>
<td></td>
</tr>
</tbody>
</table>
APPENDIX B: Calculation of Percent yield for Preparative labs

CHE 203L, CHE 204L
Percent yield:

For Preparative labs e.g. Synthesis of 2-Methyl-2-butene, Synthesis of 2-Chloro-2-Methylbutane, Diels-Alder lab, Preparation of tert-Butylphenol and other Preparative labs you need to include detailed reaction mechanism and a separate section under results on percent yield. Percent yield is calculated on the basis of reaction stoichiometry of the main reaction.

The formula below represents the percent yield, which is the ratio of the actual yield to the theoretical yield expressed as a percent. The limiting reagent determines the theoretical yield (maximum amount of product that you can obtain on the basis of reaction stoichiometry). You start with calculating the moles of each reactant that is involved in the reaction and then determine the limiting reagent.

- The theoretical yield represents the maximum amount of product in g that can be formed from given amounts of reactants. In most of our organic chemistry labs (alkene prep lab, alkyl halide lab, Diels-Alder lab, tert-Butylphenol lab, reaction stoichiometry of limiting reactant reagent to product is one to one. Therefore,

\[ x \text{ Moles of limiting reagent} = x \text{ Moles of product.} \]

Theoretical yield, \( g \) of product = number of moles of product multiplied by molar mass of product.

- The experimental yield is the amount of the product resulting from your experiment i.e. the amount of product that you actually obtained in the laboratory. It is also called actual yield.

\[
\text{Percent yield} = \frac{g \text{ of product obtained}}{g \text{ of theoretical yield}} \times 100
\]

Or

You will also get identical results if you use the following equation:

\[
\text{Percent yield} = \frac{\text{experimental yield in moles}}{\text{theoretical yield in moles}} \times 100
\]
APPENDIX C: Table of Acid and Base Strength

Table of Acid and Base Strength

(Acids of interest to an organic chemistry student are shown in bold)

<table>
<thead>
<tr>
<th>pKa or Ka</th>
<th>Acid</th>
<th>Name</th>
<th>Formula</th>
<th>Conjugate Base</th>
<th>Name</th>
</tr>
</thead>
<tbody>
<tr>
<td>Large</td>
<td></td>
<td>Perchloric acid</td>
<td>HClO4</td>
<td>Perchlorate ion</td>
<td>Perchloric acid</td>
</tr>
<tr>
<td>3.2 * 10^9</td>
<td>Hydroiodic acid</td>
<td>HI</td>
<td>I^-</td>
<td>Iodide ion</td>
<td></td>
</tr>
<tr>
<td>1.0 * 10^9</td>
<td>Hydrobromic acid</td>
<td>HBr</td>
<td>Br^-</td>
<td>Bromide ion</td>
<td></td>
</tr>
<tr>
<td>pKa= -7</td>
<td>Hydrochloric acid</td>
<td>HCl</td>
<td>Cl^-</td>
<td>Chloride ion</td>
<td></td>
</tr>
<tr>
<td>1.0 * 10^4</td>
<td>Sulfuric acid</td>
<td>H2SO4</td>
<td>HSO4^-</td>
<td>Hydrogen sulfate ion</td>
<td></td>
</tr>
<tr>
<td>2.4 * 10^1</td>
<td>Nitric acid</td>
<td>HNO3</td>
<td>NO3^-</td>
<td>Nitrate ion</td>
<td></td>
</tr>
<tr>
<td>pKa= -1.8</td>
<td>Hydronium ion</td>
<td>H3O+</td>
<td>H2O</td>
<td>Water</td>
<td></td>
</tr>
<tr>
<td>5.4 * 10^-2</td>
<td>Oxalic acid</td>
<td>HO2C-CO2H</td>
<td>HO2-CO2^-</td>
<td>Hydrogen oxalate ion</td>
<td></td>
</tr>
<tr>
<td>7.1 * 10^-3</td>
<td>Phosphoric acid</td>
<td>H3PO4</td>
<td>H2PO4^-</td>
<td>Dihydrogen phosphate ion</td>
<td></td>
</tr>
<tr>
<td>pKa= 3.2</td>
<td>Hydrofluoric acid</td>
<td>HF</td>
<td>F^-</td>
<td>Fluoride ion</td>
<td></td>
</tr>
<tr>
<td>1.8 * 10^-4</td>
<td>Methanoic acid</td>
<td>HCO2H</td>
<td>HCO2^-</td>
<td>Methanoate ion</td>
<td></td>
</tr>
<tr>
<td>pKa= 4.2</td>
<td>Benzoic acid</td>
<td>C6H5COOH</td>
<td>C6H5COO-</td>
<td>Benzoate ion</td>
<td></td>
</tr>
<tr>
<td>1.8 * 10^-5</td>
<td>Ethanoic acid (acetic acid)</td>
<td>CH3COOH</td>
<td>CH3COO</td>
<td>Ethanoate (acetate) ion</td>
<td></td>
</tr>
<tr>
<td>pKa= 4.75</td>
<td>Ethanoic acid</td>
<td>CH3COOH</td>
<td>CH3COO</td>
<td>Ethanoate (acetate) ion</td>
<td></td>
</tr>
<tr>
<td>4.4 * 10^-7</td>
<td>Carbonic acid</td>
<td>H2CO3</td>
<td>HCO3^-</td>
<td>Hydrogen carbonate ion</td>
<td></td>
</tr>
<tr>
<td>pKa= 6.2</td>
<td>Hydrosulfuric acid</td>
<td>H2S</td>
<td>HS^-</td>
<td>Hydrogen sulfide ion</td>
<td></td>
</tr>
<tr>
<td>pKa= 9.89</td>
<td>Phenol</td>
<td>C6H5OH</td>
<td>Phenoxide ion</td>
<td>Hydrogen phosphate ion</td>
<td></td>
</tr>
<tr>
<td>6.2 * 10^-10</td>
<td>Hydrocyanic acid</td>
<td>HCN</td>
<td>CN^-</td>
<td>Cyanide ion</td>
<td></td>
</tr>
<tr>
<td>5.8 * 10^-10</td>
<td>Ammonium ion</td>
<td>NH4+</td>
<td>NH3</td>
<td>Ammonia</td>
<td></td>
</tr>
<tr>
<td>pKa= 15.74</td>
<td>Water</td>
<td>H2O</td>
<td>OH^-</td>
<td>Hydroxide ion</td>
<td></td>
</tr>
<tr>
<td>pKa= 16</td>
<td>Ethanol</td>
<td>C2H5OH</td>
<td>C2H5O^-</td>
<td>Ethoxide ion</td>
<td></td>
</tr>
<tr>
<td>pKa= 25</td>
<td>Ethyne</td>
<td>C2H2</td>
<td>C2H^-</td>
<td>Acetylide ion</td>
<td></td>
</tr>
<tr>
<td>pKa= 36-38</td>
<td>Ammonia</td>
<td>NH3</td>
<td>NH4^-</td>
<td>Amide ion</td>
<td></td>
</tr>
</tbody>
</table>
Summer 2018 - Application to Faculty Development Program – Funding for Summer Work (3/22/18)

Laboratory Manual Revisions for Physical Science & Physical Chemistry

Principal Investigator: Whelton A. Miller III, Ph.D.
Assistant Professor
Department of Chemistry, Physics & Engineering
College of Science and Technology
Lincoln University

Salary Supplements for Projects Requiring Summer Work

<table>
<thead>
<tr>
<th>Title</th>
<th>Description</th>
<th>Requested Funding Amounts</th>
<th>Competition Date</th>
</tr>
</thead>
<tbody>
<tr>
<td>A. Development of a Physical Science Laboratory Manual</td>
<td>Easy to follow, prepare, and manage laboratory manual for Physical Science I &amp; II (GSC 101L &amp; 102L) Laboratory</td>
<td>$1750/month</td>
<td>8/14/18</td>
</tr>
<tr>
<td>B. Development of a Physical Chemistry Laboratory Manual</td>
<td>Easy to follow, prepare, and manage laboratory manual for Physical Science I &amp; II (GSC 101L &amp; 102L) Laboratory</td>
<td>$1750/month</td>
<td>8/14/18</td>
</tr>
</tbody>
</table>

Total Award Amount Requested $3,500.00

Abstract

General physical sciences, such as chemistry and physics, are becoming increasingly interconnected with today’s biomedical research laboratories, by providing tools and general expertise to conduct research in biological sciences and biomedical engineering. The minimum skill sets are key cross-disciplinary concepts, involving a combination of knowledge and proficiency from the fields of biology, chemistry, and physics. Therefore, it is essential that students and new graduates have a basic understanding of physical science concepts. The following proposes revisions to the laboratory manuals for Physical Science (GSC 101 & 102) and Physical Chemistry (CHE 300 & 301). Support for this revision project will advance the general science and chemistry programs at Lincoln.
I. Introduction
The ability to think algorithmically and to apply mathematical concepts to develop more efficient solutions to scientific problems is essential to science today. However, there is a shortage of overlap with lecture and laboratory coursework in the physical sciences. Currently, there are at least 3 sections of physical science laboratory, and only 1 section of physical chemistry laboratory per semester for parts I & II of both courses. Both Physical Science and Physical Chemistry require updated laboratory manuals, which are easy to follow for faculty, the lab management team, as well as the students. To address this Dr. Miller intends to consolidate and update 2 laboratory manuals for four courses, Physical Science I & II, and Physical Chemistry I & II.

II. Rationale for Summer Funding
As previously mentioned, it is necessary to revise laboratory manuals for both Physical Science (GSC 101 & 102) and Physical Chemistry (CHE 300 & 301). It is anticipated that total revisions of both manuals, excluding review, will take approximately 1 month (or equivalent). The best time to make revisions to course material is during summer break. Therefore, funding is requested to support revisions of both laboratory manuals over the equivalent of one month during summer 2018.

III. Timeline: Expected Deliverables
The anticipated total time to update, disseminate, and publish the new laboratory manuals is approximately summer session (or 1 semester equivalent) in total, comprised of the end of Spring semester 2018 to the end of Summer session 2018 (Tab.1). Starting the revision process during summer also allows time for review of material per faculty requests, and laboratory manager review of manuals by at least the beginning of Fall 2018. Therefore, the expected deliverables for this project include the following objectives: (1) Review of Existing Physical Science and Physical Chemistry manuals. (2) Sorting of relevant (selected) experiments. (3) Redesign (revision) of Physical Science & Physical Chemistry manuals. (4) Laboratory manager (& student laboratory managers) review of manuals. (5) Implementation of new laboratory manuals by Fall 2018. (6) Presentation & review of manuals by faculty and laboratory manager.

<table>
<thead>
<tr>
<th>Timeline</th>
<th>May</th>
<th>Jun</th>
<th>July</th>
<th>Aug</th>
<th>Sept</th>
<th>Oct</th>
<th>Nov</th>
<th>Dec</th>
<th>Jan</th>
</tr>
</thead>
<tbody>
<tr>
<td>Review of Existing Physical Science &amp; Physical Chemistry material</td>
<td></td>
<td></td>
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<td></td>
<td></td>
<td></td>
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<tr>
<td>Sorting of relevant experiments</td>
<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td>Redesign of Physical Science &amp; Physical Chemistry manuals</td>
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<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Laboratory Manager (&amp; Student Laboratory Manager Team) Review of Manuals</td>
<td></td>
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<tr>
<td>Implementation of New Laboratory Manuals</td>
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<td></td>
<td></td>
</tr>
<tr>
<td>Reevaluation &amp; Review of Manuals by Faculty and Laboratory Manager</td>
<td></td>
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</tbody>
</table>

Table 1. Estimated timeline for project milestones (project timeline).

W. A. Miller
IV. How the project will enhance the University Community
One effective learning process is to promote active participation (hands-on), problem-solving, and self-directed learning. Having organized and synchronized laboratory manuals for all physical science and physical chemistry sections is important for student learning and course standardization. This will also be a valuable “go-to” source for new faculty in the Department of Chemistry, Physics & Engineering. It can also serve as a template for future standardization of laboratory manuals in the department.

V. Project Milestones & Dissemination (Shared Outcomes)
The major goal of this project is to create two laboratory manuals for Physical Science I & II, and Physical Chemistry I & II. Since laboratory courses historically do not start until ~ 2 weeks into the semester, the time for dissemination and review of the project will be adequate. Dissemination of the project will include review by appropriate department faculty before the beginning of Fall semester 2018. Cataloging of manuals (via electronic storage) for access by any department faculty, as well as any university committees, e.g. General Education Committee. User feedback can be solicited by email, and possible web-based questionnaires (for students), as well as student course evaluations by the end of Fall semester 2018. Finally, grades of students using this new resource in the laboratory will be compared with those from previous years.
VI. Appendix

Appendix I. Brief Autobiography
Dr. Miller received a B.S. in Biochemistry from University of Delaware in 2001 where he worked under the supervision of Dr. Douglass F. Taber. After graduation, he took a job working in industry as a synthetic organic chemist for a pharmaceutical company. After over 3 years of industrial experience, he returned to school to complete a Ph.D. in Theoretical/Computational Chemistry from the University of the Sciences in Philadelphia in 2012. After graduate school, he was given a unique opportunity through the University of Pennsylvania’s (Penn) - Postdoctoral Opportunities in Research and Teaching (PENN-PORT) program, an NIH sponsored, Institutional Research and Academic Career Development Award (IRACDA) postdoctoral fellowship. In addition to Dr. Miller’s responsibilities through the Penn-PORT program, he served on the Biomedical Postdoctoral Council (BPC), as well as chair of the Engineering PostDoc Association (EpoD). He has worked closely with the Physician Scientist Training Program (PSTP) as a mentor to a high school student, as well as a program guest speaker. This allowed Dr. Miller to be a Postdoctoral Research Fellow in the Department of Bioengineering at Penn, as well as serve as a Visiting Professor in the Department of Chemistry at Lincoln University. After accepting a position at Lincoln University as an Assistant Professor, as well as a Visiting Assistant Professor in the Department of Chemical & Biomolecular Engineering position at Penn, Dr. Miller continues to working on collaborative research projects and include colleagues at Instituto Tecnológico de Santo Domingo, University of Pennsylvania, and University of the Science. Furthermore; initial equipment for the C1MM and projects have been partially funded by the NIH through the Penn-PORT program. Dr. Miller is always looking forward to more opportunities for minority student development and enrichment in the STEM-related disciplines.

Appendix II. Brief Description of current Research
Dr. Miller’s current research involves using computational chemistry techniques for theoretical design and study of organometallic and inorganic compounds, protein ligand interactions, and structural electronic effects. Dr. Miller plans to use his area of expertise to identify and predict electronic and structural properties and interactions for molecular design.

Appendix III. References: