Laboratory Safety Procedures

General Safety

It is your responsibility to make sure that you are sufficiently well informed to be able to work safely in a laboratory. You need to consider how your work affects not only yourself, but also the others working in the laboratory.

1. All laboratory operations should be planned carefully beforehand. Review and understand the written procedure for the experiment before coming to the laboratory. Your instructor will demonstrate how to use equipment and instruments, as well as, alert you to any special hazards and how to deal with them. Follow directions and pay close attention to instructions.
2. Your laboratory instructor will show you the location of the following items and instruct you how to use them. Be sure you know the location and proper use of the following:

Eye Wash Fountain	Fire Blanket
Safety Shower	First Aid Kit & Supplies
Fire Extinguisher	Emergency Exits
Nearest Phone	Sinks
Fume hood operation	Organic Waste disposal
Material Safety Data Sheets	Broken Glass Receptacle
Dispensing hood	Emergency Shut off (lab 218 only)

3. Consult your lab instructor if you are unsure of the hazards or proper use of any reagent or equipment. Ask questions about use, technique or disposal when you have any doubt as to the proper procedure. There are NO dumb safety questions.
4. Perform only the assigned experiments. Unauthorized experiments are strictly forbidden. Maintain a serious working atmosphere. Do not engage in horseplay.
5. Do not store equipment, backpacks, coats, chemicals or other materials on the floor or in other places where laboratory workers can trip or knock over the item, or in places that would block fire exits.
6. Do not work alone in laboratories.
7. Notify your instructor of any medical conditions that may require special attention.
8. Report any accidents, injuries or close calls to the lab instructor or teaching assistant immediately. To ensure appropriate treatment, report minor injuries, no matter how trivial they seem.

9. Do not consume or store any food or beverages in the laboratory. If the laboratory experiment involves food or beverages, clearly label them "Not for human consumption" or equivalent.
10. Keep your work area safe and clean.
11. After completing an experiment, turn off, unplug, and properly store all electrical equipment. Turn off all gas and water valves.
12. Wash hands, face, and arms thoroughly if contaminated and always wash your hands before leaving the building. Always wash before eating, drinking, smoking, or applying make-up after working in the laboratory. Shower after working with chemicals.
13. Get approval from the lab instructor if you intend to leave experiments unattended for a short time. Leave a note by the apparatus identifying the experimenter and back-up personnel.

Lab Attire & Protective Clothing

Proper attire and use of approved safety **goggles** are simple but effective ways of protecting oneself from laboratory hazards.

1. Wear approved protective **goggles** at all times when working in the laboratory. Eye protection must have "ANSI Z87" stamped on it. The use of contact lenses (especially soft contacts) in the laboratory is not recommended. Chemicals may become entrapped in, absorbed by or caught behind the lens, promoting eye irritation or causing damage to the eye.
2. Wear protective gloves as necessary. The type of glove selected should protect against the chemical that you are using, or against heat, cold, or sharp objects. Wear gloves for work with strong corrosives or with acutely toxic chemicals. Consult your lab instructor for proper glove selection.
3. Clothing should cover your shoulders to your toes (i.e. shorts and miniskirts are forbidden). Consider the flammability of the clothing fabric. The use of lab coats is strongly recommended. **Students wearing improper attire will be asked to leave the laboratory.**
4. Wear sturdy shoes that cover your feet. Sandals and open-toed shoes are dangerous footwear in the lab and are forbidden. Canvas shoes do not provide the necessary protection from laboratory accidents.
5. If you have long hair, tie it back to keep it away from flames and chemicals.
6. Have your personal clothing and protective equipment, such as lab coats, laundered or cleaned regularly. Immediately clean your personal clothing and protective equipment after contamination.

Working with Equipment & Glassware

A little precaution in the use of and assembly of equipment and glassware can prevent major accidents. Be careful with Bunsen burners, hot plates, hot plate-stirrers, and other hot objects.

1. Whenever possible, use a broom and dustpan to handle broken glassware. Do not use damaged, cracked or broken glassware.
2. Dispose of broken glassware and sharp items such as syringe needles and razor blades in special containers as directed by your laboratory instructor. Do not place them in the regular trash. These items should be as free of chemical contamination as possible before disposal.
3. When inserting thermometers or glass tubing into stoppers or corks, lubricate them with water or glycerin and twist, using short strokes and minimum pressure. Covering the thermometer or tubing with a towel protects hands and fingers from injury in case the article being inserted breaks.
4. Treat a test tube as you would a gun. Never point a test tube at anyone, especially when it is being heated. Never look down into a test tube or flask in which an experiment is being conducted.
5. Never take equipment or glassware out of the chemistry laboratory.
6. Report broken thermometers to the lab supervisor. Mercury thermometers should not be used in this laboratory. If you find one, please inform your lab instructor.
7. All experiments are to be carried out in a fume-hood. The sash of the hood should be maintained at 18" or less for safe manipulation of dangerous and volatile chemicals. Whenever possible, keep the sash fully closed.
8. Do not attempt to catch falling glassware with your hands or feet; you may injure yourself.

Working Safely with Chemicals

Knowledge, caution and common sense add up to chemical safety. Assume any unfamiliar chemical is hazardous. Consider a mixture to be at least as hazardous as its most hazardous component. Know the hazards before you handle the material. Treat all chemicals with respect. Check your lab manual for special hazards. Follow all chemical safety instructions and procedures to the letter. Minimize chemical exposure by careful use and good housekeeping.

1. Laboratory operations that have the potential to produce hazardous levels of fumes, gases, or volatile solvent vapors in excess of recommended exposure levels require fume hoods.

2. Do not (a) use unlabeled chemicals, (b) taste a chemical, (c) pipet by mouth, (d) smell a chemical. Check odors only if instructed to do so. Check by gently wafting the vapor towards your nose with your hand. Avoid skin contact with reagents.
3. Never combine substances unless you have been explicitly instructed to do so.
4. Clean up any spilled reagents immediately, especially near the balances or reagent shelf. Before clean-up, neutralize acid spills with solid sodium bicarbonate, $NaHCO_3$; neutralize base spills with boric acid. You are responsible for neutralizing, if necessary, and cleaning up your own spills.
5. Read the chemical labels very carefully to ensure that you have the correct chemical. Read labels **three** times: when you pick it up; just before you use it; and after finishing. Match name, formula and concentration on the label to the experiment directions.
6. Use particular care to avoid skin and eye contact when working with corrosive agents such as acids and bases. If you spill any corrosive chemical on yourself, rinse the affected area with water for a minimum of 15 minutes. If the outside of a reagent bottle is contaminated, handle it with gloves, and rinse the bottle before using the reagent. Notify your instructor.
7. Always add acid to water. Always mix acids and water slowly and carefully.
8. Do not light Bunsen burners or strike matches unless instructed to do so. Do not risk electrical sparks when flammable vapors are present.
9. Do not insert pipets, spatulas, or medicine droppers from your own lab kit into the reagent bottle. Dispense solids by tapping out and take only what you need.
10. Never return unused reagents to the reagent bottle. Do not contaminate reagents by exchanging caps or stoppers or by laying stoppers on the desktop. Contact your lab instructor for proper disposal of excess reagent.
11. Do not place reagents directly on the balance pan. Use a tared weighing container or paper.
12. Clean up your work area and any common areas that you used prior to leaving the laboratory.
13. Notify your instructor of any medical conditions that may require special attention.

Working with Electrical Equipment

1. The use of electrical equipment always means there is a chance of shock or fire.
2. Do not use any electrically powered equipment that is not wired with a safety ground and 3-prong plug.
3. Report any shocks, defective or worn equipment, such as frayed wires, or undue heating to your laboratory instructor. Do not attempt to repair the equipment yourself.
4. Extension cords are not appropriate.

5. Never use electrical wires as supports.
6. Do not pull out live wires.
7. Do not touch electrical devices with wet hands or while standing on a wet floor.

Dealing with Injury

EYES: Flush with water using the eyewash for 15 minutes

INGESTION: Follow the label and MSDS instructions

SKIN CONTACT: Stand under the emergency shower and remove contaminated clothing immediately for major spills. For minor spills, flush with water for 15 minutes and remove contaminated clothing.

INHALATION: Move victim to fresh air and get prompt medical attention.

l. If you become overexposed to a hazardous substance, inform your laboratory instructor and get medical attention. For first-aid instructions, check the First Aid Manual (Meyer Hall Room 266), or the MSDS information found in the South East corner of Meyer Hall.
2. Remove any clothing on which a chemical has been spilled.
3. Treat burns (heat or chemical) immediately by placing the burned area under cold water for at least 15 minutes. Cold water markedly reduces the subsequent pain and blisters.
4. If an individual's clothing or hair is on fire, roll the individual on the floor and use a blanket or coat to smother the fire—running/walking to the safety shower can fan the flames. Get medical help promptly for any burns.
5. Report allergies or sensitivities to your lab supervisor.

Dealing with Fire

l. Suffocate a small fire in a vessel by covering it. Do not pick up the vessel. Remove nearby flammable objects. Inform the instructor or TA immediately.
2. If it is not practical to cover the fire, but the fire is small and does not involve metals, then a CO_2 or dry chemical (ABC) extinguisher can be used to put out the fire. Fight the fire from a position from which you can escape, and only if, you are confident that it can be extinguished. Remember that it is easy to underestimate a fire.
3. If the fire cannot be extinguished quickly and simply, all persons should evacuate the laboratory, close the doors, pull the fire alarm in the hall, and leave the building using the stairs.

Check-in/Check-out Procedures

Check-In

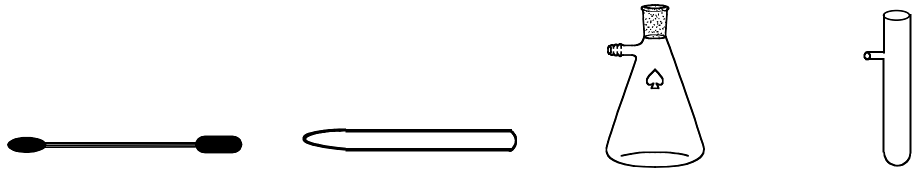
1. You are required to have goggles for check-in. You will be given a cloth towel for use throughout the term.
2. According to the check-in sheet, check the availability of all items in your drawer (see the next page for drawings of unfamiliar glassware). Replace missing and damaged items. Remove the items not listed in the check-in sheet.
3. Wash the dirty glassware with a brush and soap then rinse with distilled water. Persistent stains may be washed with acetone or another solvent recommended by your instructor.
4. Record the lock combination in your notebook
5. Relay any relevant medical information to your instructor.
6. Return the signed check-in sheet to your TA.
7. After each lab period, lock your drawer; remove all items from your hood except for the hot plate and the two ring stands with clamps; return other items including tubing, cork rings, thermometers, stir-bars, etc.; wipe down the hood as necessary.

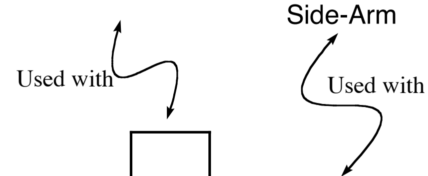
Check-Out

1. According to the check-in sheet, check the availability and cleanliness of all items in your drawer. Replace missing and damaged items. Remove extra items and the items not listed in the check-in sheet. This includes your safety goggles, towels, synthesized products, items from common drawers, magnetic stir-bars, etc.
2. Wash the dirty glassware with a brush and soap then rinse with distilled water. Persistent stains may be washed with acetone or another solvent recommended by your instructor. Clean the plastic tub.
3. Have your TA or instructor check your drawer and sign the check-in/out sheet.
4. Lock your drawer or put it back in the cabinet.
5. Wipe down your hood

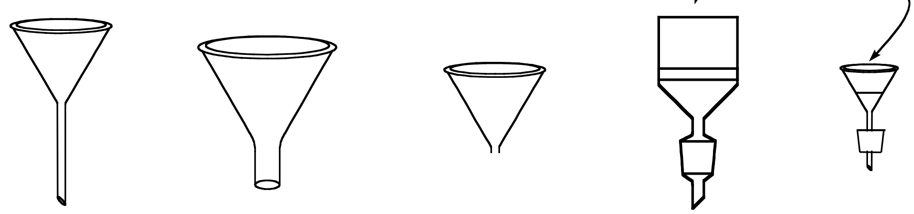
Laboratory Glassware

General Items:

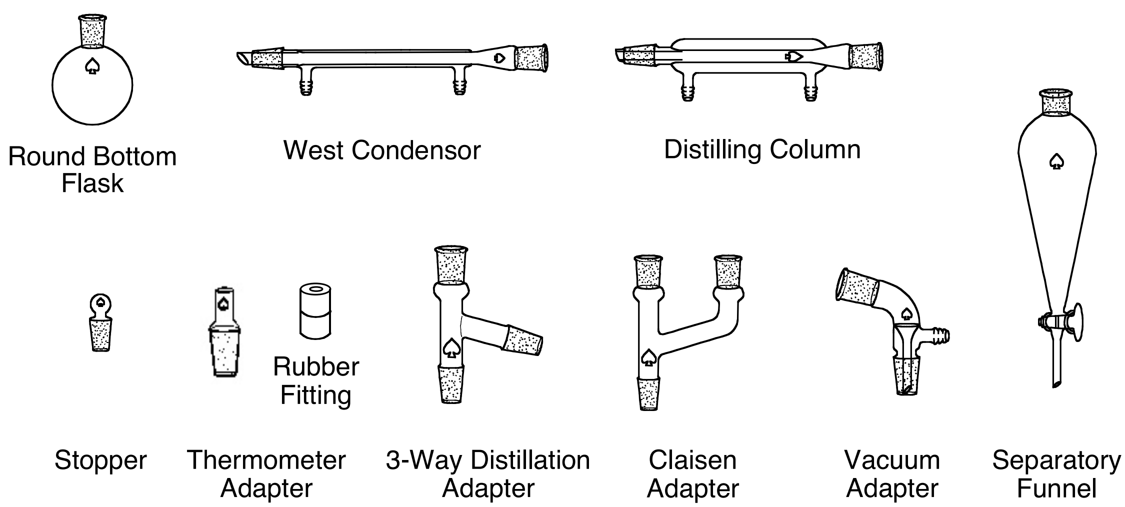




Funnels:



19/22 Standard Taper Glassware:



Cleaning your glassware: Wash with warm soapy water using a test tube brush to remove difficult material, and rinse with distilled water. The glassware should be left to dry in your drawer until the next lab period. Sometimes hard to remove materials can be removed by washing with acetone, or with an acid rinse followed by tap water then distilled water.

Laboratory Notebooks

Lab records in **Organic 1 & 2** are to be written in a bound, quadrille-ruled carbonless copy lab notebook, which is kept up to date. The records that you write must be sufficiently detailed to allow a competent second person to repeat exactly what you have done without your being present to explain. There are two principal reasons for keeping this record. First, in the event that your work produces an unusual result (better or worse than ordinary) you and your instructor will need to know why. If an accurate record of the procedure is not kept there is no way to repeat the procedure or to figure out why the result was out of the ordinary. Both physical and biological scientists are required by the terms of their employment to maintain accurate lab records in order to ensure repeatability and establish legal priority. The only way to get used to keeping such records is to get in the habit now. Second, doing organic chemistry in the lab is not like following a recipe in a cookbook. Good results will come only when you thoroughly plan your experiment and understand the reason for each step.

You should set up your notebook as follows

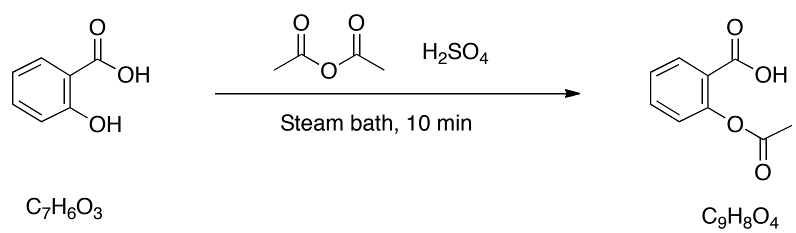
1. The first few pages of the volume are reserved for a table of contents and entries with references to page numbers are made in the table as each experiment/procedure is recorded.
2. The notebook is the **ONLY** thing you will write in/on when performing the experiment. All entries are made in black or blue ballpoint pen. Errors are to be crossed out with a single stroke, not erased. White-out is not to be used. Writing should be tidy and legible.
3. In this lab, you will be using a carbonless copy notebook. You will be asked to turn in the top page to your instructor and you can use the bottom copy for your reference. Thus, you should press firmly to make a good carbonless copy for your records.
4. Before you come to lab, format your notebook as shown below.
   - Include your name, date and reference at the top of the page
   - Include a short appropriate title for the experiment.
   - Show the reaction being performed.
   - Include all reagents used in the reaction and any reagents used for recrystallization. You do not need to include drying agents and aqueous solutions used in workup procedures.
   - Include all the columns of information, in the order shown.
   - The theoretical yield of the reaction needs to be calculated; however, you will likely need to adjust this value once you know you know exactly how much of the limiting reagent you used in the reaction.
5. Although clear handwriting is important, you should <u>**never**</u> rewrite the lab procedure you have previously written in your lab notebook just to make it look neat. Spend your time making sure you have the appropriate details and just lineout (do not scratch out) mistakes.

6. You must record your results after the procedure and before you sign the notebook. This includes all calculations needed to determine the results such as the weight, moles, and percent yield of your product obtained. Also, include all observations such as color of the material, evolution of vapor or gas, production of heat, etc. (see the example below)
7. Answer **the post-lab questions** from the lab manual **in your lab notebook**. Pre-lab questions should be answered in your lecture notebook.
8. Your lab notebook sheets will be collected and graded after each lab. Credit will be given for an appropriately documented experiment and correct answers to the post-lab questions.

Sample Notebook (to be completed prior to lab)

Reference: ONU Lab Notebook 2006-7 edition pg XXX Date

Preparation of Acetylsalicylic Acid



In this experiment, aspirin is prepared from salicyclic acid using an acylation reaction catalyzed with sulfuric acid.

Compound	Amount	d	MW	mmol	mol. equiv.	Other data
salicyclic acid (*o*-hydroxybenzene)	2.0 g	–	138.12	14.5	1	mp 158–161 °C; irritant
acetic anhydride	5 mL	1.082	102.09	53	3.7	bp 138–140 °C; corrosive
sulfuric acid (cat)	0.1 mL	1.840	98.08	1.9	0.1	catalyst; extremely corrosive
acetylsalicylic acid	T.Y.= 2.76 g	–	180.16	14.5	1	mp 138–140 °C

The above table/reaction should be hand written in your notebook before arriving to lab.

Data (d, bp, mp, etc) for the above table can be obtained from the web or books on reserve in the library.

Sample Notebook (to be completed during the lab: note the short statements)

- 2.12 g of salicylic acid was placed in a 125 mL Erlenmeyer flask
- 5 mL acetic acid was added
- 0.1 mL $H_2SO_4$was added
- Heated on steam bath for 12 min
- Cooled to rt
- Cooled in an ice bath and the flask was scratched to induce crystal formation
  note: small white crystals precipitated only after scratching
- 50 mL of distilled water was added
- The crystals were collected by vacuum filtration
- 1.83 g of white crystals were obtained (mp = 137.2 – 139.4 °C)

The Product is a yellow solid. IR spectra (1745 and 1735 cm^{-1}), NMR, m.p. and TLC confirm the product.

theoretical yield = 2.12 g salicylic acid ÷ 138.12 g/mol x 1 mol pdt/ 1mol sm x 180.16 g/mol = 2.76 g

% yield = 1.83 ÷ 2.76g x 100 = 66.3 %

Turning in Synthetic Products

A component of the course grade includes yields and purity of the materials obtained (This typically amounts to 20–30 of 200 points for the 4–6 synthetic products prepared). Thus, for each lab experiment where you make a product, you are required to record/submit the amount of material obtained, the percent yield, and physical data (m.p, b.p, or spectra) to your instructor. Your instructor will then check the amount obtained and the purity of the material (this may be done randomly, but it is done!). Academic dishonesty in the reporting of yields and physical data can result in dismissal from the course.

You will turn in your products in a labeled plastic sample bag or glass vial. Bags and vials should have a label that has your name, section, the **STRUCTURE** of the compound in the container, the date, and tare weight of the bag (or the vial plus the cap). The tare weight is the weight of the empty container.

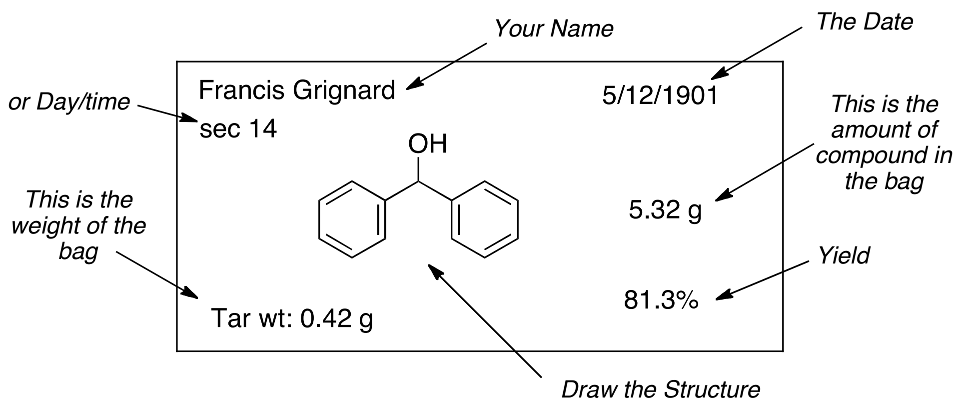


Figure 1. Vial/sample bag labels should contain the above information.

Graphing Data

Graphs are used to show trends in data.

To make a good graph, consider the following points:

A) A graph can be done by hand on graph paper or in a program like Excel. In either case, use as much of an 8 x 11 piece of paper as possible. Turn it sideways (landscape) if necessary.

B) Typically the independent variable, *x*, runs long ways at the bottom of the page, and the measured variable, *y*, runs vertically.

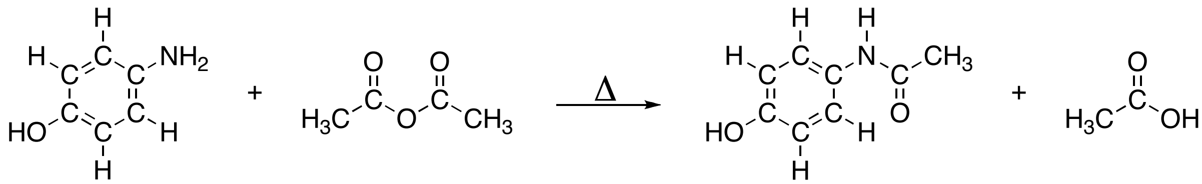
C) The gridlines should be in small increments so that values can easily and accurately be extracted from the graph.

D) Add points to the graph, and include a legend especially if two data sets are being plotted on the same graph (such is the case in the distillation lab)

E) Provide a best-fit line to the data points—This is not "connect the dots."

1 Synthesis of Acetaminophen

Reaction



1	2	3	4
4-Aminophenol	**Acetic Anhydride**	**Acetaminophen**	**Acetic Acid**
MW 109.13	MW 102.09	MW 151.17	
mp 188 – 190 ˚C	bp 138 – 140˚ C	mp 169 – 171˚ C	

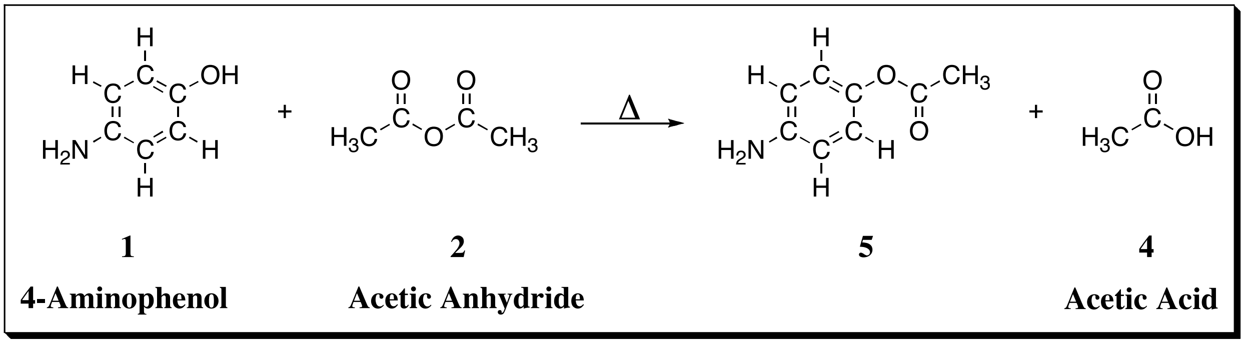
Learning Objectives/Outcomes

- Students will learn how to properly set up a lab notebook, including writing organic reactions, calculating theoretical yield, percent yield, mmol, and molar equivalents.
- Students will be able to identify the laboratory safety equipment and explain the appropriate use of that equipment.
- Students will be able to identify basic laboratory equipment and its proper location.
- Students will be able to execute a one-step organic synthesis.
- Students will demonstrate their ability to setup and carry out a vacuum filtration to isolate their synthetic product.

Discussion

The equation above shows the reaction of 4-aminophenol (**1**) with acetic anhydride to produce acetaminophen (**3**), a widely used over-the-counter analgesic. This is a straightforward organic reaction with a 1:1 reactant to product stoichiometry, yet there are some practical differences between typical reactions encountered in your freshman chemistry labs and the organic reaction shown above.

One major difference between the reactions you used in freshman lab and those you will encounter this year is that organic reactants, due to their complex structures, often have more than one reaction path available to them under a given set of conditions. This means that products other than the one desired can form and use up reactant, thus reducing the molar efficiency (% yield) of the reaction. For example, in this reaction we want the primary amine functional group to do a substitution reaction with the acid anhydride functional group of reactant **2**, giving us an amide functional group. However, a similar substitution reaction between the OH group of reactant **1** and the anhydride group of compound **2** can give ester **5** (**Scheme 1**.)



Scheme 1: A Possible Side Reaction

Adding water to the reaction mixture apparently creates a reverse reaction that converts the ester back into 4-aminophenol and prevents this side reaction from becoming a serious diversion, but nonetheless it does occur to a small extent. Thus, we see that side reactions can reduce the efficiency of a desired reaction even when the equilibrium constant for the desired reaction is very favorable, because the equilibrium constant for the side reaction might also be large.

It is also true that many organic reactions have $K_{eq} < 100$. This means that appreciable amounts of unreacted starting materials will remain in the reaction vessel even after the reaction is complete. This unreacted starting material will reduce the amount of pure product recovered in two ways; first, 1.00 mole of reactant will give perhaps 0.90 mol of product if 0.10 mole of the starting material remains unreacted, and second, the product which is recovered will have to be made free from unreacted starting material in some way, and such purification processes always remove some of the good product in addition to the impurities.

Finally, since the cost of organic chemicals rises exponentially with their purity, the reactants employed in organic lab often have 1–5% of various impurities in them. Not all of the reactant you add to your reaction mixture is really the correct starting material! Once again, these impurities in your starting material reduce the amount of product you will eventually get.

The starting material for acetaminophen, 4-aminophenol, is pale yellow when pure. However, when you weigh it out it may be black because its electron-rich benzene ring is easily oxidized, and although little of the oxidation products are present in commercially produced **1**, the oxidized products are strongly colored and mask the color of pure **1**.

Procedure

Before beginning this experiment, everyone needs to clean their Buchner funnel with acetone to ensure all colored organic compounds are completely rinsed away. Place 2.0 g of 4-aminophenol in 15 mL of deionized water in a 125 mL Erlenmeyer flask. Warm the solution for ten minutes on a hotplate set on 4 (the solid may not all dissolve). Remove the flask from the hotplate and add 3.4 mL of acetic anhydride about 10 drops at a time while swirling the flask. After addition of the acetic anhydride is complete, add a boiling stick, and heat the mixture to boiling for about five minutes (DO NOT ALLOW WATER TO EVAPORATE, set the hot plate at 4). Cool to room temperature then in an ice bath and induce crystallization. When the mixture is at approximately 10 °C (a thermometer is not needed--it should be cool to the touch) and crystallization has ceased, filter through the large (clean) Büchner funnel (**see picture on left**) **Note:** **<u>Always</u> clamp your filter flask before attaching the vacuum hose and Büchner funnel.** Once your filtration is complete, turn off the vacuum, remove the tubing, and place the Büchner funnel containing the wet crystals in your drawer. You will use the acetaminophen you made today in several labs throughout this semester.

Waste Disposal

The filtrate may be disposed of down the drain. Filter papers should go in the regular waste receptacle and broken glass should be placed in the glass waste boxes. Acetone washings, small amounts of excess acetic anhydride, and 4-aminophenol should be added to the non-halogenated waste container. Excess amounts (>10g) of "wasted" starting materials should be bottled separately for disposal.

Post-Lab Questions

1) What is the limiting reagent in your experiment?
2) Calculate the theoretical yield for the synthesis of acetaminophen based on your limiting reagent.
3) Calculate your percent yield for acetaminophen based on the mass of dry product obtained in this experiment. (Your solid will need to dry until the next lab period before measuring its mass.)
4) Construct a data table following the guidelines on page 12.