CHEM-223-Fall **2020-** Synchronous Online

VERY IMPORTANT: STUDENTS REGISTERED FOR THIS LABORATORY COURSE MUST ALSO BE REGISTERED FOR THE ACCOMPANYING RECITATION – CHEM 22301! Recitation class attendance is mandatory for any student enrolled in the lab and a significant portion of the CHEM 22300 lab grade comes from exams & quizzes given in the recitation class!

Table of Contents

(Will Meet Every Week: Online Synchronous via BB Collaborate)

Sequence of Experiments

13	Elimination – Synthesis of Alkenes
14	Student Power Point Presentations (My Favorite Organic Molecule)

Online Safety Quiz	10 points * 1 week	10 points
Online Pre-lab Quizzes	10 points * 13 weeks	130 points
Online Pre-lab Writeup	10 points * 7 weeks	70 points
Online Lab Participation (Kahoot Online Polling)	10 points * 13 weeks	130 points
Online Lab Participation (Synchronous Discussion BB Collaborate) OR (Discussion Boards)	10 points * 13 weeks	130 points
Lab Report for Expt- 1 – 7	30 points * 7 weeks	210 points
Online Assessment Expt- 8 – 13	20 points * 6 weeks	120 points
PP presentation (week 14)	30 points * 1 week	30 points
Recitation Exams & assessments	250 points	250 points
TOTAL		1080 points

Projected letter grade cut-offs: Grades will be based on the Hunter College grading scale.

A+: 97.5 - 100%
A: 92.5 - 97.4 %
A-: 90 - 92.4 %
B+: 87.5 - 89.9%
B: 82.5 - 87.4%
B-: 80 - 82.4%
C+: 77.5 - 79.9%
C: 70 - 77.4%
D: 60 - 69.9%
F: 0.0 - 59.9

Welcome to CHEM 22300 Lab – Introduction & Common Policies

COURSE DESCRIPTION & GRADING OVERVIEW

In this course, you will first learn how to separate and purify organic compounds. You will then synthesize a number of organic compounds yourself and employ the techniques that you have learned earlier (as well as learn some new ones) to separate, purify, and study your reaction products.

Your grade will be assigned on a 1080-point schedule. Your point totals will be based on pre-lab quizzes, your lab performance, written lab reports and recitation class assessments. Keep in mind that good **execution of laboratory techniques** (during discussion over BB Collaborate), **awareness and adherence to safe laboratory practices** (including cleanliness and proper disposal), the quality & quantity of the products you hand in (for in-person lab only), **the organization of your work (including how well you have planned your work beforehand) and how well you understand the chemical processes that occur are all factored into your grade**!

http://www.hunter.cuny.edu/onestop/repository/files/registrar/faculty-pdfs_reg/gradingsystem_reg.pdf

TEXT: Pavia, Kriz, Lampman, and Engel: A Small Scale Approach to Organic Laboratory Techniques, Fourth Edition. (This lab manual will refer to this textbook as "Pavia")

PLANNING AND EXECUTION

In the Organic Chemistry laboratory, you will plan and execute your work more independently than in previous laboratory courses that you have taken so far. You must do a lot of preparation work before you work on an experiment. In addition to reading the text, you must attend the lab recitation class. Take good notes in the lab recitation and review them carefully when planning each experiment. **The pre-laboratory assignments for each experiment must be completed BEFORE you come to lab and uploaded on BB – links will be posted by lab instructor!**

Your pre-lab preparation for Online Lab will help you actively participate during the Online Lab sessions.

Many of the experiments that you will complete this semester are not taken directly from the Pavia textbook though you may find many similarities. Part Six of the Pavia textbook (starting at page 548) should be especially helpful to you as it contains descriptions of the techniques you will use throughout the term. The importance of studying the recitation material and applying what you have learned cannot be exaggerated. The key to success is planning your work carefully before you enter the laboratory!

Participation during both Recitation and Online Lab is required for the ***Entire Session***
(Formative assessment using online Polling (eg. Kahoot) will be used along with other discussion assessments)

Please join the synchronous online lab sessions on time
(just like you would for in-person lab and stay for the entire lab session)
Points will be deduced for improper Netiquettes

Things to know (During In-person Lab):

It is essential that you start working promptly as soon as possible, rather than socialize with other students. You will be so busy in some experiments that you probably won't have time to talk at length to anyone. Sometimes, you will need to work on different parts of an experiment at the same time in order to finish on time. It is very important that you finish all work within the scheduled class time and within the total time allotted for the experiment (if more than one class session is dedicated for an experiment). **No work will be allowed outside the scheduled class time, including washing glassware and taking melting points. Everybody must physically leave the laboratory by the scheduled end of class time!** No additional time will be given to any student who falls behind on work.

You will work individually on some procedures, but there are also some procedures in an experiment that you will perform in pairs or as a small group. Your instructor will let you know about the working arrangement on the day of the experiment. One set of equipment will be issued to a pair of students – you and your assigned partner will be responsible for keeping them in good condition throughout the semester (even if the two of you don't necessarily work together on any experiment).

When you hold a flask in front of your instructor to ask a question about the contents, you must be able to describe exactly what you put in the flask, and the exact sequence of operations you have carried out in arriving at that point. Your lab instructor will not simply provide answers! You should always be prepared to intelligently discuss what you are doing and try to arrive at a solution to your own problem

LABORATORY SAFETY

During the first session, all students will be acquainted with safe laboratory practices, the safety features of the laboratory and the procedures to be followed in the case of an emergency. Students will also be provided with a copy of laboratory rules. The Safety Video and Copy of Laboratory Rules is posted online on BB.

Appendix I of this lab manual contains additional information on the hazardous properties of chemicals used in CHEM 223. You will not be allowed to proceed with the lab course until you are familiar with the rules and safety procedures.

You will also have an online safety quiz

Things to know (During In-person Lab):

For safety reasons, ALL STUDENTS must wait outside the laboratory until their instructor has entered and has given everyone permission to enter the room.

****** SAFETY GOGGLES and FACE MASKS MUST BE WORN AT ALL TIMES IN THE LABORATORY! *** Failure to comply with this regulation will result in deduction of points and/or ejection from the laboratory.***

SAFETY. Students are responsible for knowing the proper safety practices for every experiment, including safety information on all chemicals and procedures used in the experiment.

This information can be obtained by reviewing the safety video , from the laboratory text (Pavia, technique 1, p 548-565), and from the following handbooks available in the stockroom room 1414 north: Dangerous Properties of Industrials by Irving Sax; Handbook of Chemistry and Physics; Merck Index and Aldrich

Chemical Catalog; Webpage www.sigmaaldrich.com. For the safety and convenience of students taking Organic Chemistry I and II the chemicals in room 1404 north have been organized according to the individual experiment.

Please return chemicals to their correct positions.

You are to supply your own safety glasses, disposable gloves, paper towels and masks.

LAB CLEANLINESS

1. Make sure that the area around your workspace is clean while you're working on your experiment AND before you leave the laboratory. Your instructor will not clean up after you!
2. If you spill something or otherwise make a mess during a procedure, you must clean it up.
3. There are designated disposal containers for broken glass, chemicals (solid and liquid), gloves, etc. located throughout the room. If you are not sure where to dispose something, ask your lab instructor.

**** STUDENTS MAY NOT ATTEND ANY SECTION THEY'RE NOT REGISTERED IN****

MAKING UP A LAB

Due to the highly condensed nature of the Fall 2020 semester schedule, **make-up lab sessions are NOT available.**

THE LABORATORY NOTEBOOK (Required for pre-lab and taking notes during online session)

The notebook must have numbered, duplicate pages so that you can keep one copy of your lab report (other in turned in during in-person lab). For online lab you will upload the scanned copy or a picture of the pre-lab on BB for the experiments that need a pre-lab.

Suitable Organic Chemistry Laboratory Notebooks are sold at the Hunter College Bookstore or online.

The notebook is to be sufficiently complete and well organized so that anyone who reads it can know what has been done in each experiment and can repeat the procedures from what's written in it. This laboratory notebook has essentially the same requirements as a notebook used to record data in a research laboratory. All data are to be recorded at the time they are observed or obtained. This includes weights, boiling and melting points, observations of physical changes, results, and conclusions. Separate pieces of copy/loose leaf paper are **NOT** to be used for recording data to be transcribed later. Your laboratory instructor may check your notebooks at the end of each laboratory session to ensure that your data was properly recorded at the time when you conducted the experiment.

The notebook should be neat but this is less important than having it be a complete, original record. Copying data is a waste of time and leads to copying errors. The record made at the time of the observation is the important record. If changes or corrections are to be made, the material considered wrong is to be cancelled by drawing a line through it. The revised material is then to be added. It may be necessary to refer to the record to determine how an experiment might best be revised or interpreted.

You should have a Table of Contents on the first page of the notebook and all of the pages should be numbered. Start every experiment on a new page. Make all records in ink (DO NOT WRITE WITH A PENCIL!). Instead of copying details of a procedure verbatim, refer to the page in the lab manual (or other

sources) where the procedure is started. The notebook is a log of your work and should be dated regularly. As you conduct the experiment, you must write a short description of the actual procedure that you followed including all observations (this practice is difficult to mirror in online environment). The preliminary write-up, as indicated in each experiment, must be in your notebook **before** you begin the experiment (uploaded on BB – check links posted by instructor). All preliminary write-ups must include a list of hazards and toxicities of the compounds involved.

Experiments designed to develop familiarity of techniques can be recorded in terms of an introduction which states the objective; a description of the procedure, which may be identified by a reference to the manual; the observations; the conclusions (identify the unknown and state the supporting data and reasoning); answers to the question, and a discussion of the theory behind the experiment and its relationship to the observed results. If your own procedure is at all different from that in these notes (or manual), tell exactly how it differs. Data tables will also be used for the later preparative experiments (from Experiment 6 onward) and you will need to know how to carefully and accurately tabulate data to include all your results.

We must emphasize that your notebook should be up to date at all times during the laboratory period and your instructor will periodically examine it to ensure this. We repeat: **you must only use indelible ink and you are NOT permitted to use corrective fluid ("white-out") or tape.**

Your lab reports must only include detailed discussion of all the steps of lab (in past tense and third person- no use of I or we) and analysis of data that will be provided by your lab instructor. This can be typed and uploaded on BB.

Chemdraw should be used to draw structures. Your lab instructor will provide information on how you can download Chemdraw .

THE RECITATION (CHEM 22301)

Please remember that the recitation is the equivalent of a challenging one-credit course. Don't let yourself become one of the many students who receive a low grade for the entire course due to low scores on their recitation examinations! It is essential from a viewpoint of safety alone to attend all the recitations and attendance will be taken for that reason. However, the Recitation is also critical from the standpoint of your grade since your exam scores from that portion of the course will account for about 19% of the total possible points.

We would like to stress again the importance of studying and planning your work before you start the experiment! Students who really understand what they are doing in the lab will enjoy the work and might even look back on their organic chemistry laboratory as a really pleasurable learning experience. Those who do not understand the experiments will experience frustration and likely failure in addition to exposing themselves and others to the risk of a serious accident.

We will do our best to help you enjoy the course and achieve successful results, but if you don't do your homework and planning, no one will be able to help you. If an instructor determines that a student has not adequately prepared for an experiment, the student will be sent away from the laboratory and will not be allowed to do make-up work in another section.

PLEASE MAKE SURE THAT YOU ARE REGISTERED FOR THE RECITATION CHEM22301. YOU MAY BE DROPPED FROM CHEM22300 IF NOT.

Suggested Reading for Important Techniques (for different experiments): Pavia, 4th Edition (Listed from page 548 onwards)
Technique 1: Laboratory Safety
Technique 2: Laboratory Notebook
Technique 3: Laboratory Glass ware Care and Cleaning (pg 578-579 for names of glassware)
Technique 4 : How to find data for Compounds: Handbooks and Catalogs
Technique 5: Heating and Cooling
Technique 7: Assembling Reactions (pg 610-615) (different clamps & set up for distillation and reflux)
Technique 8: Filtration
Technique 9: Melting Points (pg 650-651)
Technique 10: Solubility
Technique 11: Crystallization- choosing solvent and drying (pg 664)
Technique 12: Extraction (separatory funnel and drying agents) (pg 668)
Technique 13: Physical Constants of Liquids: Boiling Point and Density Chart Part A. "Boiling Point and Temperature Corrections"
Technique 14: Simple Distillation
Technique 15: Fractional Distillation
Technique 25: Infrared Spectroscopy and Appendix 3 (pg 998)
Technique 29: Guide to Chemical Literature
First page has a list of common solvents
Essay-Aspirin (pg 47)
Essay- Analgesics (pg 53)
Essay- Caffeine (pg 67)
Essay: Green Chemistry (218-223)

Contact Information

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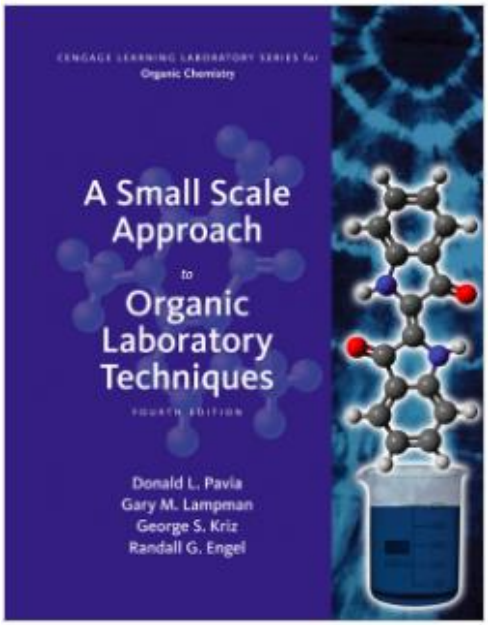
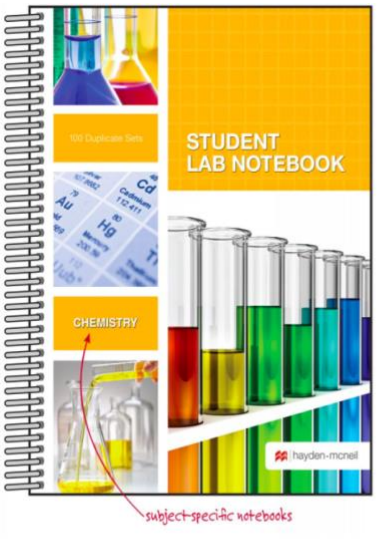
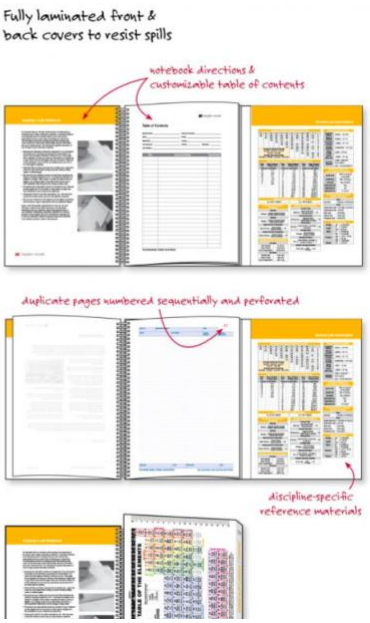
LABORATORY NOTEBOOK

All your lab pre-lab write-up are to be written in a dedicated laboratory notebook.
Pre-lab write up for Experiments (1 to 7) should be hand-written in ink (not pencil) on a lab notebook. You will upload a scanned copy or picture from Links provided by lab instructor.







Experiment 1:
Melting Point
WEEK 1: ONLINE VIA BB COLLABORATE DISCUSSIONS& VIDEOS

Reference: Pavia – Technique 9 – pages 645-654

A. INTRODUCTION

The melting point is the ultimate criterion of the purity of a solid since pure compounds melt within very precise temperature ranges. Your lab instructor will explain to you the use of the melting point apparatus as well as show you how to fill the melting point capillary. Please note that you need only a very small amount of compound (less than 0.1 g) for melting point determination.

Pre-lab Quiz (10 points)

Pre-lab Write-Up (10 points)

Follow the guidelines discussed earlier.

B. EXPERIMENTAL PROCEDURE

Part A: Determine the melting point of the following compounds and observe the changes due to the presence of an "impurity."

a. trans-Cinnamic acid
b. Urea
c. a 1:1 mixture of both trans-Cinnamic acid and Urea.

Record your observations.
(For Online lab: This information be provided by the lab instructor)

HINT: There are three slots for inserting melting point capillary tubes inside the melting point apparatus. You should use up all available slots in the apparatus at one time to determine melting points for each sample instead of melting one sample at a time.

Part B: Determine the melting range of an unknown compound given by your instructor. The identity of your unknown compound will be one of the following:

Compound	Melting Range (°C)		Compound	Melting Range (°C)
Naphthalene	80-82		4-Methoxybenzoic acid	182-185
Anthracene	216-217		1-Naphthol	95-96
Benzoic acid	122-123		3-Nitroaniline	112-114
Benzophenone	49-51		4-Nitrophenol	112-114
p-Bromoacetanilide	165-169		3-Nitrobenzoic acid	140-143
Cholesterol	147-148		Salicylic acid	159-160
4-Chlorobenzoic acid	239-242		*o*-Toluic acid	103-105
Cinnamic acid	133-135		*p*-Toluic acid	180-181

Part C: Confirm the identity of the unknown compound from Part C by recording a mixed melting point measurement with a reference sample provided by your lab instructor.
(For Online lab: This information from unknown assigned to you will be provided by the lab instructor)

This is a routine part of any melting point procedure and should always be performed as long as a reference sample is available. You should try to remember this for later experiments!

C. WRITING YOUR LAB REPORT

Write your Lab Report following the format on page 34 and other ideas shared by lab instructor.

Follow Appendix- II (pg 33)

Discussion:
This should be a nice story in a logical order. Discuss all steps of the, discuss yield purity
Calculations
Summarize your results. Whenever possible, use a table format.
How the experiment concurs or disagrees with the theory. Sources of error.
(Use past tense and do not say "I" weighed)
You should comment on your yields, provide identification and the melting point of your recrystallized compounds. (Make sure that you clearly indicate the number of your unknown sample…
(No credit will be given for your work if you fail to include your unknown number!).

EXPERIMENT 2:
CRYSTALLIZATION
WEEK 2: ONLINE VIA BB COLLABORATE DISCUSSIONS & VIDEOS

Reference: Pavia - Techniques 8, 9, 10 and 11 - pages 632-682. Also read pages 548-587.

A. INTRODUCTION

The technique of crystallization is one of the most valuable available for the purification of solids. The basic idea of purification is easily understood and the manipulations are straightforward. In spite of this, crystallization remains more of an art than a science. A part of the trouble arises because a good crystallization frequently requires much patience. A more serious problem is that the best solvent to use cannot be chosen by a convenient magic rule, but must be found by trial and error. Since you have little or no experience, you will have to rely on our judgment.

The most fundamental property of a good solvent for crystallization of a solid is that the hot solvent must dissolve the substance readily while the cold solvent must dissolve it sparingly. This means that you would start your hunt for a good solvent by looking for one that gave borderline solubility. From here you would have to adjust the temperature range, the ratio of solid to solvent, or try combinations of solvents to find a mixture with just the right solvent properties.

In crystallization of a mixture of solids one is always faced with the practical problem of knowing how much solvent to use. If one of the components is poorly soluble in the chosen solvent one could go on adding the hot solvent for a long time before the mixture is dissolved completely. If you use too much solvent the desired compound will not come out of solution when you cool it. In practice, you probably will make this mistake many times and the only way to recover your compound is to concentrate the solution and see if the desired solid precipitates. Therefore, for best results, you must be able to judge whether any un-dissolved solid is the compound that you are trying to recrystallize or some poorly soluble impurity.

Recrystallization using a solvent mixture (solvent pair) is very useful for purification of certain compounds. In this technique one dissolves the compound in a warm solution of the solvent component in which the compound is more soluble, and then the second solvent (in which the compound is less soluble) is added until a slight turbidity appears (dropwise). The solution is reheated (or a drop of the first solvent is added) to obtain a clear solution. The solution is cooled to obtain the recrystallized compound.

Pre-lab Quiz (10 points)

Pre-lab Write-Up (10 points)

Follow the guidelines discussed earlier.

For this experiment, you will recrystallize an unknown compound using water. Your unknown compound could be: Benzoic acid, Salicylic acid or Sorbic acid.

Thermometers are delicate and must be handled gently!

You should pick a digital scale that you will use to measure weights for all the experiments. Since each balance has been calibrated differently, it is important that you use the same balance for every

weighing! It is your responsibility to make sure that your balance is kept clean. Please refrain from making any adjustments to the scale. Report to your instructor if there is a problem.

B. EXPERIMENTAL PROCEDURE

Recrystallization Of An Unknown From Water.

First, obtain a sample of your unknown compound from the lab instructor. Then, take a melting point of your impure sample. Set up all the apparatus that you will need for recrystallization **in the fume hood**. Boil approximately 150 mL of water. Pour 5-10 mL of this hot solvent into a flask. Place on a hot plate to warm up while you go on with your other preparations. Weigh out 2 g. of your impure unknown and place it in a 125 mL Erlenmeyer flask. Carefully add (in small amounts) the hot solvent.

Remember the importance of using the minimum amount of hot solvent. (You may have to decide whether the last traces of un-dissolved solid are samples or an insoluble impurity, so look carefully for changes in the amount of solid present). Swirl the flask between additions and when almost completely dissolved, add a boiling chip and bring back to boiling on the hot plate. Remove the boiling solution from the hotplate, add gradually a small amount of decolorizing carbon (Norite, caution-frothing), and swirl the solution gently. Heat the solution to boiling for approximately 5 minutes. Filter the hot solution using a heated funnel and fluted filter paper. (It is recommended that you place the funnel in a heated oven at the start of the lab in order to ensure that it is hot for the filtration process).

Cover the mouth of the flask containing the hot filtrate with a watch glass and allow to cool first to room temperature, then let it stand undisturbed in an ice bath. The more slowly a solution is allowed to cool, the better the quality and purity of the crystals you will obtain. When the product has crystallized completely, collect the crystals in a Buchner funnel. Rinse the Erlenmeyer flask with part of the filtrate to ensure complete transfer. Discontinue the suction when the crystals are still slightly moist. Wash the crystals with cold water. Apply suction again and press the crystals firmly with a clean glass stopper. Allow the crystals to dry in the air for a moment and then gently "bake" them in the oven.

HINT: While air drying the wet crystals is often the safest way to remove the solvent, it can also be very time consuming. Depending on the solvent that you have used (for example: water is fine for this), you may gently "bake" your wet crystals on a watch glass in the oven to evaporate the solvent faster. You must be careful to check the oven temperature and avoid overheating the crystals inside the oven!

(Think! At what temperature should you remove your crystals from the oven?)

Determine the weight, yield, and the melting point of the dried recrystallized material.
(For Online lab: This information from unknown assigned to you will be provided by the lab instructor)

Based on the melting point range and the table of melting points identify your unknown. To be certain of the identity of your compound, you must also take a mixed melting point with a reference sample of the known material. (YOU WILL PROPOSE THIS and WHAT YOU WILL LOOK FOR)

Submit your sample to your instructor (see next pg.)

Remember, your sample will be graded both on the quality of the crystals and the yield obtained. **(only for in-person labs)**

The yield in this case will be based on the mass of purified

unknown solid that you recover. The yield can also be expressed as a Percent Recovery, which is the mass of purified solid obtained is divided by the mass of impure solid that you started with times 100%. (For Online lab: Calculate yields based on data provided by the lab instructor)

(only for in-person labs)

After the experiment is completed, you should hand in a sample of your recrystallized compound to your instructor. (A proper container for solid samples is a small test tube or vial. If using a test tube, make sure to cover the top with a small piece of Parafilm). The container should bear a label that states:

(a) your name and unknown #
(b) the name of the substance and its melting point
(c) the page of your notebook describing the sample
(d) the weight of the sample and the tare weight. All weights must be in your notebook and recorded to two places past the decimal (in grams).

Results & Observations – For results, you should include all recorded weights (including actual yield of crystals), melting point, calculated percent yields, etc. Be sure to write down your in-class observations!

(For Online lab: This information from unknown assigned to you will be provided by the lab instructor)

C. WRITING YOUR LAB REPORT

Follow Appendix- II (pg 33)

In addition, your lab report should include the following at the appropriate section:

Discussion:
This should be a nice story in a logical order. Discuss all steps of the, discuss yield purity
Calculations
Summarize your results. Whenever possible, use a table format.
How the experiment concurs or disagrees with the theory. Sources of error.
(Use past tense and do not say "I" weighed)
You should comment on your yields, provide identification and the melting point of your recrystallized compounds. (Make sure that you clearly indicate the number of your unknown sample…
(No credit will be given for your work if you fail to include your unknown number!).

Experiment 3:
Simple & Fractional Distillation
WEEK 3: ONLINE VIA BB COLLABORATE DISCUSSIONS & VIDEOS

Reference: Pavia - Techniques 14 and 15 - pages 722-751.

A. INTRODUCTION

Distillation can be used as a method for purifying a single liquid and also as a means of separating a liquid from a dissolved solid or from a mixture of miscible liquids. The liquid (or mixture) is heated and when it boils, the vapors are condensed into a separate receiver. The resultant liquid (distillate) is collected in one or more fractions.

i) Purification of a Single Liquid:

A single liquid will begin to boil when its vapor pressure is equal to the vapor pressure of the atmosphere. For a pure liquid the boiling temperature should remain constant (within 2°C) for the duration of the distillation.

ii) Separation of a mixture:

When a solution of 2 miscible liquids is distilled, boiling will begin when the total pressure of the solution is equal to the atmospheric pressure. If the solution is 'ideal' (follows Raoult's Law), the total pressure will be the sum of the partial pressures of each liquid. These partial pressures are dependent upon the vapor pressure of the pure liquid and its mole fraction in the solution.

If the boiling points of liquids differ by more than 100°C, good separation can be obtained by simple distillation, as the partial pressures of the two liquids will be very different. However, if the boiling points are fairly close to each other (say within a 40°C difference), one cannot obtain sharp separation by simple distillation. A different method must be employed to increase the efficiency of the distillation. This is accomplished by the use of a fractionating column. Our column will consist of a condenser filled with a packing material.

iii) Fractional Distillation:

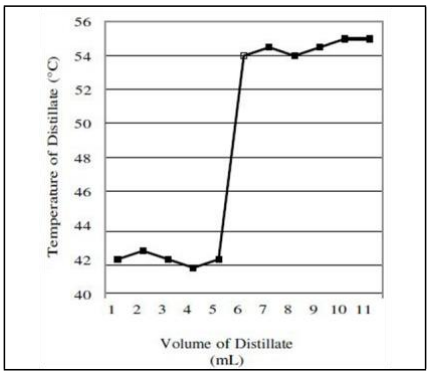
In order to understand the behavior of two liquids as they are distilled using a fractionating column, you must understand the liquid/vapor composition curves in Pavia on pages 723, 730, 732, 742 and 743.
You will have to spend a great deal of time studying Techniques 14 and 15 to understand distillation. Sections 14.2, 14.3, and 15.1-15.6 should be particularly helpful.

The column packing provides a surface for these multiple condensation and vaporizations to occur. In order for the column to function successfully, a temperature gradient must be maintained along its length (i.e.: the bottom of the column must be hotter than the top). This can be accomplished by heating the boiling flask very slowly and also by insulating the column with glass wool or cotton secured with aluminum foil. The mixture must move up the column slowly, thus ensuring an equilibrium of vapor and liquid all along the column. The temperature gradient is also dependent upon the heats of vaporization (HV) of the two liquids. As the difference between of the two components increase, a larger temperature differential is possible and the efficiency of the separation increases.

** If the flask is heated too quickly or too vigorously, poor separation will occur. **

Theoretically, if one performs a fractional distillation at maximum efficiency, a plot of temperature vs. volume of the distillate would resemble Figure 1 *(see next page)*.

Figure 1: Distillation of a Mixture



Pre-lab Quiz (10 points)

Pre-lab Write-Up (10 points)

Follow the guidelines discussed earlier.

B. EXPERIMENTAL PROCEDURE

You will work with a partner to distill a mixture of methanol-water, once with a simple distillation set- up and also by using a fractionating column. It is possible to run both set-ups at the same time. You should assemble both sets of apparatus at the beginning of the lab period, but you should focus on the fractional distillation first. After collecting about 15 mL of distillate, one of the partners in a pair should begin the simple distillation procedure. The results of both distillations will be compared.
(For Online lab: This information from unknown assigned to you will be provided by the lab instructor)

i) Apparatus

Simple Distillation: Follow figure 14.1 on Pavia page 723 with the following modifications: Use a heating mantle/regulator apparatus. Start with the regulator set at 50 and lower to approximately 30 immediately when you observe the liquid boiling. Using a 100 mL round bottom flask, secure both the flask and the heating mantle. Use a 10 mL graduated cylinder to collect your distillate. Have a beaker handy to empty the graduated cylinder as it fills up. You will be collecting more than 10 mL of distillate. Describe your apparatus in your notebook.

Fractional Distillation: The same basic set-up is required except that your fractionating column (filled with steel wool or glass beads) should be placed between the boiling flask and the still (see Pavia p.
735). Do not run water through your fractionating column. Use glass or cotton wool

to insulate the column and secure the insulation with aluminum foil. Again, describe your apparatus in your notebook.

ii) Procedure

Simple Distillation: Obtain approximately 60 mL of the methanol-water mixture assigned to you and pour it into the boiling flask. Add one or two Carborundum boiling stones. Make sure that all of the ground glass joints are securely fitted and that the water is flowing through your condenser properly (in the bottom, out the top). Have your instructor check your apparatus before you begin the distillation.
The mantle regulator should be set to maintain boiling in the flask. The distillate should come out at a rate of 1 drop every two seconds. Record the boiling point for each mL of distillate in your notebook (use tabular form).

Fractional Distillation: Carry out the distillation of a separate 60 mL sample of your unknown using the fractional distillation set-up. As soon as boiling starts turn down the regulator to keep the liquid boiling SLOWLY. As you heat slowly, a ring of condensate will rise slowly in the column. This rise should be gradual to allow for equilibration within the column. The ring of condensate should take at least several minutes to reach the top of the column. Once again, distill at a rate of 1 drop of distillate every 1-2 seconds. Once distillation actually begins maintain a constant rate by slowly increasing the heat as required. Record the boiling temperature for each mL of distillate in your notebook. If possible, make more frequent readings when the temperature starts to rise quickly. You may stop distilling after a second steady temperature is reached for 4-5 consecutive mL.

Results & Observations: For results, you should include the table of data collected (mL/temperature),

graphic display of your result on graph paper. Be sure to write down your in-class observations!

For your graph, plot mL of distillate (x-axis) vs. Temperature (y-axis) for your two distillations.

Make sure to plot both curves on the same paper using different symbols (or colors) for the 2 sets of data.
Draw the best curves for each of your data sets.
(For Online lab: This information from unknown assigned to you will be provided by the lab instructor)

C. WRITING YOUR LAB REPORT

Follow Appendix- II (pg 33)

In addition, your lab report should include the following at the appropriate section:

Discussion:
This should be a nice story in a logical order. Discuss all steps of the, discuss yield purity
Calculations

Summarize your results. Whenever possible, use a table format.
How the experiment concurs or disagrees with the theory. Sources of error.
(Use past tense and do not say "I" weighed)

In addition, your lab report should include the following at the appropriate section:

Discussion: In addition to your normal write-up, your discussion should cover all of these aspects:
1. Discuss the results you obtained for the 2 distillations. Include a discussion of your own observations during the distillations and also a discussion of the theoretical aspects of simple vs. fractional distillation.
2. What can you conclude from your own distillations?
3. If your fractional distillation showed no better separation than your simple distillation, give possible reasons for this result.
4. What are possible sources of error in this experiment?
5. Use your graph to estimate the volumes of methanol and water in the mixture. Calculate the percent composition of your mixture? Comment on the accuracy of this determination.

EXPERIMENT 4:
THIN LAYER CHROMATOGRAPHY
WEEK 4: ONLINE VIA BB COLLABORATE DISCUSSIONS & VIDEOS

Reference: Pavia - Techniques 19 and 20 - pages 780 - 815.

A. INTRODUCTION

Separation of a mixture into its pure components is an essential part of organic chemistry. For example, a chemist may want to purify the crude extract of a medicinal plant, isolate the pure product(s) of a chemical reaction from the reaction mixture, or identify foreign compounds in a urine sample.
Experiments 2-7 in this course cover basic separation and purification techniques: filtration, recrystallization, distillation, chromatography, and extraction. These techniques can be distinguished by the important physical properties involved: solubility, boiling point or polarity. Essentially, the purification of a mixture takes advantage of the way any physical property varies between the components of a mixture.

i) Background Information

Chromatography is one of the most ubiquitous methods of analyzing and purifying organic compounds. Flash column chromatography separates large quantities of compounds under air pressure while TLC (thin layer chromatography) is more useful for qualitative and small-scale separations or the identification of compounds in a mixture.

The fundamental principle of chromatography is the distribution equilibrium that forms when a compound is either dissolved in a mobile phase or adsorbed on a stationary phase.
When a compound is dissolved in the mobile phase, it is carried along the direction of flow. But when it is adsorbed on the stationary phase, it does not move. If compound B spends more time in the mobile phase than compound A, B will move further along the direction of flow than A, and will eventually be separated in space from A. The longer the mobile phase travels, the better the separation between A and B. Stationary phases are usually very polar, while mobile phases vary widely in polarity, but are less

Pre-lab Quiz (10 points)

Pre-lab Write-Up (10 points)

Follow the guidelines discussed earlier.

EXPERIMENTAL PROCEDURE

i) Experimental Outline

TLC will be used to determine R_f values and an appropriate solvent system for the separation of four organic compounds (anthracene, cholesterol, 1,4-naphthoquinone, and para-nitroaniline).

i) Procedure

TLC Plate:

The goal of this experiment is to determine a solvent system in which all four spots will move up the plate, and to calculate the Rf -values for the four compounds. First, with a pencil, lightly mark a baseline on a TLC plate, about 1 cm from the bottom. **Do not touch the silica face of the TLC plate with your fingers, and never use a pen to mark your TLC plate because the ink will also migrate with the solvent!** On the very top of the TLC plate, label the spots in pencil according to what is being spotted (e.g. A = anthracene).

Obtain a small amount of the four compounds and dissolve each separately (in a small test tube, or on a watch glass) in a small amount of methylene chloride (dichloromethane). A small amount of **compound is considered a trace amount on the small ends of your spatula tips. In preparing sample solutions, the concentration must be adjusted so that isolable, discrete spots can be developed. A solution that is too low in concentration results in very faint spots, which can be difficult to visualize, while streaking and poor separation is observed when the concentration is too high.**

HINT: Methylene chloride (dichloromethane) evaporates **VERY QUICKLY**! You must work very quickly as soon as you pour the methylene chloride from its glass container to the watch glass.

Spot the plate, by dipping either end of the capillary tube into the solution. The solution will be drawn up by capillary action. You then empty the capillary tube by touching it **lightly** on the surface of the TLC plate. This will transfer the solution to the plate as a small spot. You should only hold the micropipette in contact with the plate very briefly, otherwise, the entire contents may be delivered to the plate and your spot will be too large. It may be a good idea to gently blow on the plate as the sample is applied. This will help the solvent to evaporate quickly, keeping the spot small.

Developing the TLC Plate

Choose a solvent or solvent mixture (preferably a mixture of ethyl acetate, $CH_3CO_2C_2H_5$, and hexane. Petroleum ether can be used as a substitute for hexane) and prepare the developing chamber as shown in the illustration on page 13. Start by developing one plate with pure petroleum ether and another with pure ethyl acetate. Then use a one to one mixture of ethyl acetate and hexane. By examining these TLC plates, you can decide whether to increase or decrease the polarity as needed by adding more of either solvent. Be sure that you know what proportions of the solvents are used as you adjust the polarity of the mixture. The level of the solvent in the jar must be below the level of the spots, and the atmosphere in the jar should be saturated with solvent vapors. (If the jar is not saturated with solvent vapors, the solvent will not run all the way up the plate!). When the solvent front is near the top of the plate, immediately remove the plate from the beaker with forceps, and mark the solvent front with a pencil, before the solvent completely evaporates.

Visualizing the Spots on the TLC Plate

Allow the TLC plates to dry. First, check your plate with the UV lamp (short-wave). Lightly outline the spots which you observe with a pencil, and make a sketch of the TLC plate in your

notebook. Note any differences in the appearance of the spots. **CAUTION:** Do not look directly at the UV lamp, or shine it at anyone else! If the spots are not visible under UV light, place the slide inside an iodine chamber and allow it to sit until the subliming iodine coats the TLC plate. Mark any new spots that become visible.

Calculating the R_f Values

Next, measure the position of the original spotting to the spot (baseline), and of the solvent front. Calculate the R_f values. For each compound record in tabular form, the solvent (or solvent mixture) and the R_f value.

(For Online lab: This information from unknown assigned to you will be provided by the lab instructor)

WRITING YOUR LAB REPORT

Follow Appendix- II (pg 33)
In addition, your lab report should include the following at the appropriate section:

Discussion:
This should be a nice story in a logical order. Discuss all steps of the, discuss yield purity Calculations
Summarize your results. Whenever possible, use a table format.
How the experiment concurs or disagrees with the theory. Sources of error.
(Use past tense and do not say "I" weighed)

In addition, your lab report should include the following at the appropriate section:

Results & Observations: Table of R_f data for both parts. Be sure to write down your in-class observations and attach a photocopy of your TLC plates produced from both parts!

Discussion: In addition to your normal write-up, your discussion should cover all of these aspects:

(1) Relative polarity of all the compounds and how this correlates with their structures
(2) Nature of silica gel.

EXPERIMENT 5:
COLUMN CHROMATOGRAPHY
WEEK 5: ONLINE VIA BB COLLABORATE DISCUSSIONS & VIDEOS

Reference: Pavia - Techniques 19 and 20 - pages 780 - 815.

Column Chromatography

This technique is performed by packing a glass tube with an adsorbent as shown on the next page. There are many different types of adsorbents (solid phase) that are used in column chromatography, and the choice of adsorbent depends on the types of compounds to be separated. The most common adsorbents used are: silica gel and alumina.

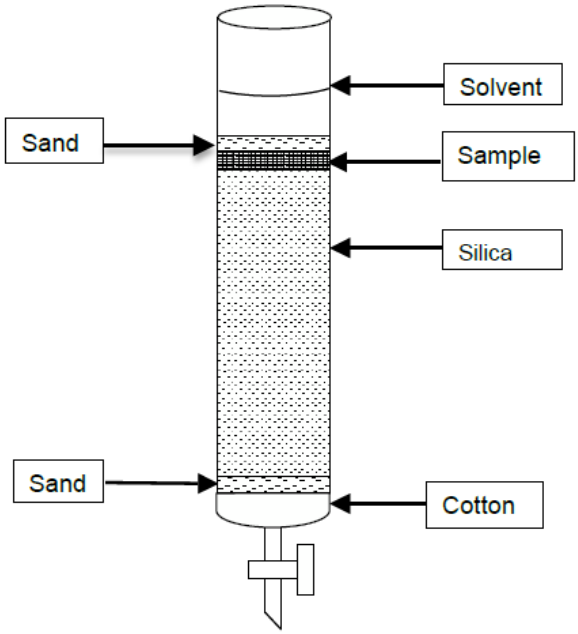
Silica gel is used to separate a wide variety of functional groups such as hydrocarbons, alcohols, ketones, esters, acids, azo compounds and amines. Alumina is also used extensively, and comes in three forms: acidic, basic, and neutral. Acidic alumina is used for separating acidic materials such as carboxylic acids and amino acids. Basic alumina is used to separate amines, while neutral alumina can be used to separate non-acidic and non-basic compounds. Likewise, cellulose, starch, and sugars are used to separate natural products, and magnesium silicate is used in the separation of acetylated sugars, steroids, and essential oils.

A column may be packed 'wet' by mixing together a slurry of the solvent and adsorbent and pouring it into the tube. Alternatively, it can be filled with the dry adsorbent. The mixture to be purified is then dissolved in a small amount of the appropriate solvent and added carefully to the top of the solid adsorbent. It is added carefully to ensure that the packing is not disturbed. You develop the column by adding more of the solvent to the top, and then collecting the fractions of eluent that come out at the bottom. For 'flash' column chromatography, moderate air pressure is used to push the solvent through the column. The success of the separation and the contents of the fractions can be determined by spotting the fractions along with the initial mixture on TLC.

Pre-lab Quiz (10 points)

Pre-lab Write-Up (10 points)

Follow the guidelines discussed earlier.



A column can be developed with a single solvent, or a **solvent gradient** (a solvent system which gradually increases in polarity). For example, a column may be developed first with a low-polarity solvent, such as hexane, and as fractions are collected the developing solvent is changed to 10:1, 5:1, and 1:1 hexane-methylene chloride. The solvent is changed by adding it as soon as the previous solvent is level with the silica gel and before the top of the column to runs dry. A polarity gradient is used for mixtures of compounds with very different polarities.

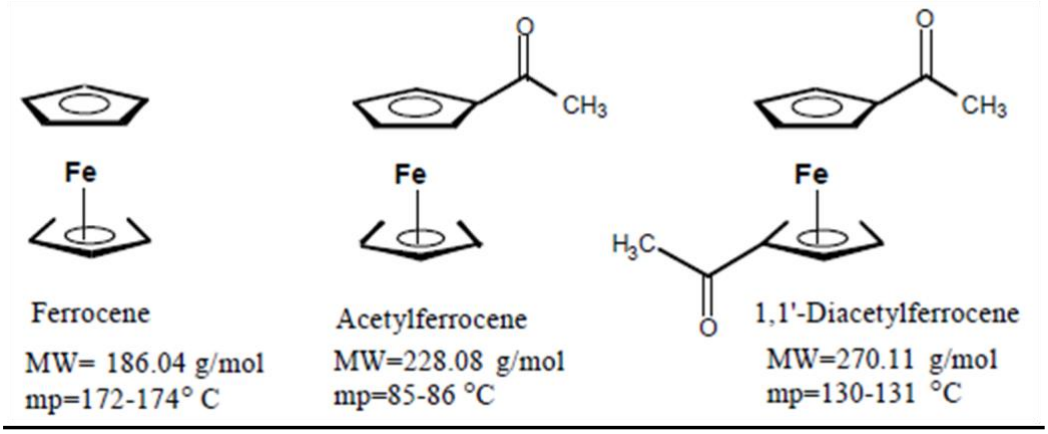
Non-polar compounds adsorb less readily to the polar stationary phase, and consequently will travel more along with the mobile phase. Since polar compounds are better adsorbed onto the polar stationary phase, they tend to travel more slowly. A polar solvent can best compete with the stationary phase to attract more polar analytic, thus carrying it along with the mobile phase. So, the best mobile- phase/solvent system will be sufficiently polar to compete with the stationary phase so that the analyze is carried far enough down the column, (or away from the baseline) but is still sufficiently attracted to the stationary phase so that the compound will not travel all the way down the column (or along the solvent front).

Solvents: A common non-polar solvent for chromatography is hexane. It can be used with a variety of polar solvents. The following solvents are listed in approximate order of increasing polarity: cyclohexane, petroleum ether, pentane, carbon tetrachloride, benzene, toluene, chloroform, ethyl ether, ethyl acetate, ethanol, acetone, acetic acid, methanol, and water.

Elution sequence: The order of elution for common compounds from fastest (moves with a non-polar solvent) to the slowest (where a more polar solvent is necessary) is as follows: hydrocarbons, olefins, ethers, halocarbons, aromatics, ketones, aldehydes, esters, alcohols, amines, and acids, strong bases.

Small-scale column chromatography will be used to separate three *organometallic* compounds: ferrocene, acetylferrocene, and diacetylferrocene. The separation will be on the microscale level, using a Pasteur pipette as your column. Prior to the column separation, TLC will be used to demonstrate the efficiency of separation in different solvent mixtures. Thus, you will be using TLC to determine the appropriate solvent system for running your column. As a

general guideline, an R_f value of about 0.4 in a TLC is best for eluting a particular compound from a column.



Column Chromatography

Packing the Micro-column

Silica gel should always be transferred inside the hood, since the small particle size makes it very hazardous to the respiratory system if it is breathed in. Prepare a column from a Pasteur pipette by carefully pushing a small piece of cotton down to the narrow part. Clamp the pipette with a thermometer clamp and add about 1/4 inch of sand (use weighing paper as a funnel). Then add about 2-1/2 inches of silica gel. Get a small quantity (just a spatula tip full) of the mixture of ferrocenes and add it directly to the top of the silica gel in your column. Then add another quarter of an inch of sand.

HINT: Save some chemicals and time! If you add too much of the ferrocene mixture in your column, you will have to use more solvent and spend more time to separate your mixture…

Separation of Ferrocene, Acetylferrocene, and 1,1-Diacetylferrocene

Begin eluting the column by first adding 100 % hexane (b.p. 68-70°C) with another Pasteur pipette, in order to move the non-polar compound down the column. After completely eluting with 100% hexane, you should gradually increase the solvent polarity in order to force the more polar compounds down the column. Increase the polarity of the solution by utilizing a 20:80 mixture of ethyl acetate and hexane, and finally work your way up to a 50:50 mixture of ethyl acetate to hexane. (Notice the different colored bands and determine which band belongs to which compound and why.)

HINT: Solvents you pour down the column will take a LONG time to travel the column by gravity alone. Attach a pipette bulb at the top of your column and gently squeeze between solvent pours to help push the liquid down faster. Ask your instructor to demonstrate this technique before attempting it!

To ensure a clean separation of the three metallocenes, you should completely elute each band before adding the increasingly polar solvent mixture. You should also collect the solvent between each band in a separate test tube. (Diluting the components with excess solvent will cause ill-defined spots during the final TLC separation.) **NOTE**: For best results, DO NOT let

the top of the column run dry until you are finished!

Collect (1 milliliter fractions per test tube) in labeled test tubes and run a TLC plate to check the effectiveness of column separation using a 60:40 mixture of hexane to ethyl acetate.
(For Online lab: This information from unknown assigned to you will be provided by the lab instructor)

WRITING YOUR LAB REPORT

Follow Appendix- II (pg 33)
In addition, your lab report should include the following at the appropriate section:

Discussion:
This should be a nice story in a logical order. Discuss all steps of the, discuss yield purity Calculations
Summarize your results. Whenever possible, use a table format.
How the experiment concurs or disagrees with the theory. Sources of error.
(Use past tense and do not say "I" weighed)

In addition, your lab report should include the following at the appropriate section:

Results & Observations: Table of R_f data. Be sure to write down your in-class observations and attach a photocopy of your TLC plates produced from both parts!

Discussion: In addition to your normal write-up, your discussion should cover all of these aspects:
(1) Relative polarity of all the compounds and how this correlates with their structures
(2) Nature of silica gel.

Experiment 6:
Identification Of An Unknown Mixture By TLC
(for acid-base extraction)
WEEK 6: ONLINE VIA BB COLLABORATE DISCUSSIONS & VIDEOS

TLC to Identify Unknown Mixture To Be Used In Experiment 7 for acid-base extraction

Your instructor will assign you an "unknown" mixture that you will use in Experiment 7. **Make sure that you record the sample number!**

Your unknown mixture contains TWO of the three compounds: acetylsalicylic acid (aspirin), phenacetin or caffeine. You will determine which two compounds are present in your mixture, and then separate the components from each other to obtain pure samples of each.

You must develop a TLC plate with your unknown in order to determine the identity of compounds that are in your mixture. Your mixture contains TWO of the following compounds: acetylsalicylic acid (aspirin), phenacetin and caffeine.

Prepare a TLC plate by marking its baseline and labeling the top with "A," "P," "C," and "Mx" (for the three compounds mentioned earlier and the unknown mixture respectively).

After you have marked your TLC plate, obtain and dissolve a small amount of your unknown mixture with dichloromethane in a clean small test tube / watch glass. Spot it with a clean capillary tube at the appropriate position on your TLC plate. Repeat the dissolving/spotting process with the three reference compounds. Be careful not to mix up your four spots (A, P, C, and Mx)!

Develop your TLC plate with the following solvent mixture: 95% ethyl acetate, 5% acetic acid (you may use a communal solvent chamber with your classmates). Be careful to quickly mark the solvent front.

Mark the developed spots first with the aid of the UV light. If necessary, you may place your developed TLC plate in the iodine chamber (allow 5-10 min for the I_2 to absorb).

At this point, you should know what compounds your unknown mixture is made up of. You will need this information for Experiment 7.

Your pre-lab write-up for that experiment should be tailored to the specific combination of compounds that you have in your unknown.

(For Online lab: This information from unknown assigned to you will be provided by the lab instructor)

WRITING YOUR LAB REPORT

Follow Appendix- II (pg 33)
In addition, your lab report should include the following at the appropriate section:

Discussion:
This should be a nice story in a logical order

Results & Observations: Table of R_f data. Be sure to write down your in-class observations and attach a photocopy of your TLC plates produced from both parts!

Calculate the $\mathbf{R_f}$ values for each spot on your TLC plate(s). Tabulate your data as shown below.

TLC of Mixture - Stationary Phase: Silica Gel; Mobile Phase: 95% ethyl acetate / 5% acetic acid

TLC Results - R_f of Spots MIXTURE UNKNOWN # _____			
Mixture Spot 1: X/Y = _____	Aspirin X/Y = _____	Phenacetin X/Y = _____	Caffeine X/Y = _____

Discussion: In addition to your normal write-up, your discussion should cover all of these aspects:

(1) Relative polarity of all the compounds and how this correlates with their structures
(2) Nature of silica gel.
(3) Describe using flow chart your method of acid-base extraction

Your lab report MUST have a proper flowchart that provides ALL necessary steps to properly separate your mixture. Use the structures from your unknown in the flow chart. (You will NOT get full credit for a generic or incomplete flowchart that omits steps). In addition, your lab report should include the following at the appropriate section:

Results & Observations: Be sure to write down your in-class observations and attach a photocopy of your TLC plates produced from both parts! Use the tables below as a model for tabulating your data and include them (only the portions relevant to your work in the laboratory) in your lab report.

Explain the results of the TLC errors: a) using too much sample; b) using too little sample; c) using a too-polar solvent; d) trying to elute a spot of crystalline material which is insoluble in the eluent.

Experiment 7:
Separation Of AMixture Using Acid-Base Extraction
WEEK 7: ONLINE VIA BB COLLABORATE DISCUSSIONS & VIDEOS

Reference: Pavia - Technique 12 - pages 683-710. Read the Essays on Aspirin (p. 47), Analgesics (p. 53), Identification of Drugs (p. 60), Caffeine (p. 67), and any others of interest such as Local Anesthetics (p. 354). In the above reading assignments you must pay special attention to the principles behind the separation techniques employed (pages 683-704). Pay attention to sections on extraction (sections 12.1-12.8) and drying agents (section 12.9).

It is also essential to understand the fundamental principles of acidity and basicity, and the application of these principles in organic chemistry. You must spend a substantial amount of time studying your **lecture textbook** to develop this understanding.

A. INTRODUCTION

The separation of acids and bases from neutral compounds by extraction is routinely employed in research laboratories. Aspirin (acetylsalicylic acid) readily donates a proton to hydroxide, carbonate, or bicarbonate ion. Its resulting conjugate base (an anion) is more soluble in water solutions of these bases than in common organic solvents. Caffeine is basic and readily accepts a proton in aqueous acid. Its conjugate acid (a cation) is more soluble in water than in the relatively non-polar common organic solvents. Phenacetin is neutral and does not easily accept or donate a proton. All three compounds are pressed together with a starch binder to form APC tablets. Please note that phenacetin has been found to cause kidney damage and is no longer in common medicinal use.

Your unknown mixture contains TWO of the three compounds: acetylsalicylic acid (aspirin), phenacetin or caffeine. You will determine which two compounds are present in your mixture, and then separate the components from each other to obtain pure samples of each. The compounds in your mixture are industrial chemicals and are not intended for human consumption! After separating the two compounds, you will use thin layer chromatography to test the completeness of your separation.

Success in this experiment requires a particularly large amount of planning and careful preparation!!

Pre-lab Quiz (10 points)

Pre-lab Write-Up (10 points)

Follow the guidelines discussed earlier.

B. EXPERIMENTAL PROCEDURE

Based on the chemical composition of your assigned unknown mixture, you must plan a suitable separation process for your unknown. Some general hints on separation technique:

Weigh out exactly 3.00 g of the unknown mixture. (You will experience solubility problems later if you use more than this!). Dissolve the unknown mixture in approx. 30 mL of dichloromethane in a small Erlenmeyer flask. Transfer the resulting solution to a small separatory funnel (Make sure that the stopcock of the funnel is closed!). Rinse the flask with an additional 5 mL of dichloromethane and add this to the separatory funnel to insure complete transfer of the unknown mixture. If you observe some crystals in the solution, do not be concerned. This could be phenacetin, which will go into solution when you begin the extraction steps.

At this point, you must decide which reagent to use for your separation of components by extraction of the dichloromethane layer. Since you know (from TLC results) what the components of your mixture are, you should be able to decide upon a separation scheme. Remember that your goal is to use an

extraction method that will cause one of the components of your mixture to become soluble in the aqueous phase.

The aqueous phase is separable from the dichloromethane phase, so the material that has become soluble in the aqueous phase can be separated from the material that remains in the dichloromethane. When you add your extracting reagent, two phases will appear. Remember to save all your phases, properly labeled in flasks, until you have recovered all the components of your mixture.

SAFETY NOTE: Although dichloromethane is much less toxic than the other common chlorinated hydrocarbon solvents, keep containers stoppered as far as possible, to minimize your exposure.

When draining your separatory funnel, you should consider inserting a narrow strip of paper between the stopper and funnel rather than removing the stopper altogether. Air must enter the funnel as it drains off. (Otherwise, a vacuum is created within the funnel and your liquid will stop draining out).
Dichloromethane is denser than water.

Swirl the 2-phase mixture well before you stopper the funnel. Relieve the pressure frequently by inverting the funnel and opening the stopcock. Point it away from people!

Additional Notes

1. CAUTION: A great deal of gas is evolved when bicarbonate is used. (What is it?)
2. Acetylsalicylic acid (aspirin) slowly hydrolyses in water. It should be isolated in the same lab period and not left standing in water for a long time.
3. Caffeine has enough water solubility that it does not precipitate from water easily. It must therefore be removed from aqueous layers by extraction with dichloromethane.

4. The best way to acidify bicarbonate washes is to add 10% HCl slowly to them until the acid is in excess, rather than the inverse procedure. Check to make sure the final pH is acidic.

5. The best way to neutralize HCl extracts is to add 10% NaOH to the extract slowly. Make sure the final pH is basic.

6. Dichloromethane layers must be dried with anhydrous Na_2SO_4 before evaporating them to recover components of your unknown.

7. The dichloromethane layers, once over Na_2SO_4 may stand over the week in your lab kit.

8. Remember to tare your flasks before evaporation of dichloromethane so you can get a weight of your unknown.

9. Determine the weight, the mp and the TLC of each component.

10. Recrystallize each component, re-measure each mp and perform TLC.

11. Caffeine may be recrystallized from acetone.

12. Phenacetin may be recrystallized from 95% ethanol-water.

13. Aspirin may be recrystallized by dissolving it in a minimal amount of 95% ethanol and then adding more water to the resulting solution

C. WRITING YOUR LAB REPORT

Follow Appendix- II (pg 33)
In addition, your lab report should include the following at the appropriate section:

Discussion:
This should be a nice story in a logical order

(For Online lab: This information from unknown assigned to you will be provided by the lab instructor)

Part I. Calculate the $\mathbf{R_f}$ values for each spot on your TLC plate(s). Tabulate your data as shown below.

TLC of Mixture - Stationary Phase: Silica Gel; Mobile Phase: 95% ethyl acetate / 5% acetic acid

TLC Results - R_f of Spots MIXTURE UNKNOWN # _____			
Mixture Spot 1: X/Y = _____	Aspirin X/Y = _____	Phenacetin X/Y = _____	Caffeine X/Y = _____

Part II. Tabulate your data as shown below. When calculating % yield, **assume that you begin**

with equal weights of each component (e.g.: 6 g of mixture = 3 g of compound A + 3 g of compound B)

Separation of Unknown Mixture (Assume that you begin with equal weights of each component)

Extraction / Recrystallization Results **MIXTURE UNKNOWN #** _____	
Name of Component 1 Crude Weight:_____g % Yield:_____% M.P.:_____°C R_f of Spot: X / Y = _____	**Name of Component 2** Crude Weight:_____g % Yield:_____% M.P.:_____°C R_f of Spot: X / Y = _____
Name of Component 1 (Recrystallized) Recrystallized Weight:_____g Recrystallized % Yield:_____% M.P.:_____°C R_f of Spot: X / Y = _____	**Name of Component 2 (Recrystallized)** Recrystallized Weight:_____g Recrystallized % Yield:_____% M.P.:_____°C R_f of Spot: X / Y = _____

Discussion: In addition to your normal write-up, where you discuss the entire acid-base extraction with reactions (arrows) your discussion should cover all of these aspects:
(i) a conclusion about the composition of your unknown mixture; **(ii)** a statement on the purity of your isolated compounds; **(iii)** an analysis of relative polarities of your compounds and how their structure contributes to its relative polarity; **(iv)** sources of error in the experiment (e.g: Why are the R_f's of identical materials not identical); **(v)** If you had any particular problems in the separation experiment, discuss them along with relevant observations (e.g.: low yields or yields over 100% , poor separation, inconsistent R_f, mp's). You may do this by referring to your flow chart.

APPENDIX I

Pre-Lab Write-Up Format:

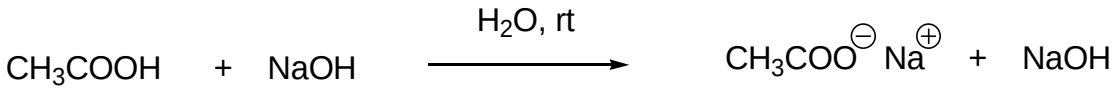
For each preparative reaction, you are to carry out a separate preliminary write-up which must be entered in your notebook before you start. **Points for pre-lab write-up will be awarded at the start of each experiment (they should be uploaded on BB via link posted by your lab instructor).** All equations must be balanced!

Here is a somewhat trivial example for illustration:

1. TITLE OF EXPERIMENT: Neutralization of Aqueous Acetic Acid

2. **OBJECTIVE**: BRIEFLY describe or list the aim(s) of the experiment.
(For synthesis labs write reactions and table of reagents shown below)

I. Main Reaction



II. Side Reactions
(Balanced equations for any known side reactions.)

3. TABLE OF SAFETY HAZARDS & PHYSICAL CONSTANTS

III. Table of Reactants and Products. Solvent Data. Hazards

Compound	Mol. Weight.	Grams Used	Moles Used	Mole Theor.	Mole Used / Mole Theor	Hazards/ Physical Props
CH_3CO_2H	60.0	6.50 g	0.11	0.10	1.1	List hazards & relevant physical Properties
NaOH	40.0	4.0	0.10	0.10	1	As above
CH_3CO_2Na	82.0	--	--	0.10	--	As above
H_2O		--	--		--	As above

Solvent: H_2O, bp 100°C; mp 0°C; density 1.00 g/ml, nontoxic.

IV. Procedure

Steps required for the experiment or flow chart (should demonstrate you have critically thought about the experiment, calculated the reagents you will need, have listed the equipments and supplies that will be required, know the reaction conditions

Theoretical Yield: (show all calculations)

#1-4 & other specific prelab exercises must be completed before starting the experiment. Prelab preparation will be considered in determination of your final grade.

V. Observations
(to be entered during the lab period or online discussions) along with any changes you may have made to the above procedure.

METHODS / PROCEDURE: Describe any changes to the procedure as outlined in the lab handout. **There is no need to rewrite the procedure in the handout or textbook. Cite the relevant pages.**

RESULTS & OBSERVATIONS: Record your observations in a clear fashion, and in sufficient detail such that it can be easily understood by anyone. Tabulation of data is usually the most efficient way of doing this. **The carbon copies of the original data from your lab note book must be attached as an appendix to your write up.**

TREATMENT OF RESULTS: Calculations, Graphs, etc. Summarize your results. Whenever possible, use a table format. This will be very helpful in planning your discussion.

Please note that you should enter "1" for the limiting reagent in both Mole Ratio columns. For liquid reactants it is convenient to enter both the mass (g) and volume (mL) in the table using the densities which you also enter in the table.

APPENDIX II

REPORT FORMAT: GENERAL WRITE-UP FORMAT FOR SYNTHESIS EXPERIMENTS

DATE:

1. TITLE OF EXPERIMENT

2. OBJECTIVE: BRIEFLY describe or list the aim(s) of the experiment.
(include reactions)

3. INTRODUCTION: CONCISELY describe the theory behind the experiment.

4. DISCUSSION/CONCLUSION:
This should be a nice story in a logical order.
Discuss all steps of the reaction, show mechanisms, discuss yield purity
Calculations, Graphs, etc.
Summarize your results. Whenever possible, use a table format.
How the experiment concurs or disagrees with the theory. Sources of error.
(Use past tense and do not say "I" weighed)

Late notebooks will be penalized. Organize and format your report so that it is easy to read. Leave adequate space between sections. You may use a word processing program to prepare your report but the carbon copies of the original data from your notebook must be submitted as an appendix. **Enquire with your instructor if he/she has any other specific write-up requirements.**

LABORATORY NOTEBOOK

Your notebook should contain in the very least #1-4, listed for the write-up. along with For notebook keeping, a convenient practice is to use the left side of your notebook as a worksheet for initial recording of observations/results, calculations, TLC plates etc, and the right hand side for more complete documentation of procedures and results. Do not use loose papers for recording data. All entries must be recorded in ink. The use of white-out is strictly prohibited. If you make an error, strike it out with a single line such that this action cannot be construed as an attempt to falsify data.

Lab reports & Pre-lab write up should be uploaded on BB via link posted by your instructor and check using safe-assign for plagerism.

Cheating will be reported to the course coordinator and the Dean of Students.

Appendix III
Hazardous Properties of Some of the Chemicals used in the CHEM 223 Laboratory

Reference: N. l. Sax, Dangerous Properties of Industrial Materials (Also see CRC Handbook, under "Toxicity")

U = Unknown
3 = May cause death or permanent injury 2 = May cause temporary damage
1 = Fairly safe

- Benzoic acid-l
- p-Nitroaniline- Acute Local- U; Ingested -3; Skin absorption -3; Headache, nausea, vomiting, stupor
- Ethanol - moderate fire hazard
- Methanol - Acute local -1; Ingested -3; Inhalation -2; skin absorption A cumulative poison. Fire hazard - moderate.
- CH_2Cl_2 - Highly irritating to the eyes! Acute: local irritant - 2; Systemic: Ingestion -2; Skin absorption -2; Inhalation -3; Chronic: Local -U; Systemic -1. Fire Hazard -none. For comparison, note the maximum allowed air concentrations in ppm: Benzene -10; CCl_4-10; $CHCl_3$- 50; CH_2Cl_2-500 (CRC Handbook)
- Benzene - a recognized carcinogen. Acute -2; Chronic -3; a cumulative poison: People who work with benzene routinely over a period of years must take particular care to minimize exposure. Fire hazard - high.
- Cyclohexane - Chronic -U; Acute -2. Fire hazard – high
- Chloroform - Chronic -U; Acute systemic -3 Considered more toxic than CH_2Cl_1 but less toxic than CCl_4 and benzene. Fire hazard -none.
- Ethyl Acetate - Acute -2; Chronic -1; Fire hazard - high
- Dimethyl ether - Acute -2; Chronic -2; Fire hazard - extremely high!
- Iodine - Acute -3; Chronic -3; Extremely irritating to the lungs.