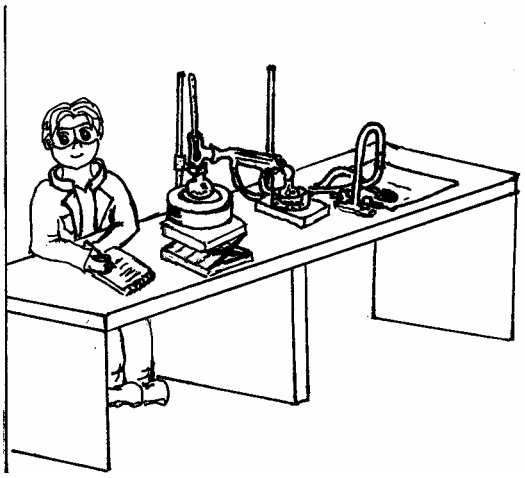
Chemistry 350

Organic Chemistry I

Report Book 2006-2008



Athabasca University

Athabasca University 

Course team

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CHEM 350 Report Book Contents

CHEM 350 Report Book Contents (cont.)

Welcome to Organic Chemistry 350's Laboratory Report Workbook

This report book, along with the 'Chemistry 350 Lab Manual', will help you prepare for a single weekend (~20h), or 3-4 days straight (~24-32h) of supervised lab instruction. All preparatory work in this report book (~12 h to finish, see list on page 3), may be completed and submitted to the Chemistry Lab Co-ordinator / Instructor prior to attending the labs, or just before the start of the Friday evening lab session.

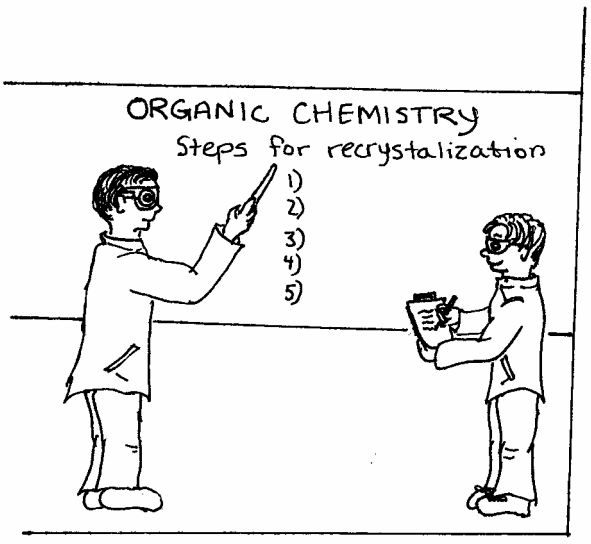
In order to successfully complete the laboratory component, please be aware of the following 4 step process of instruction. It is the intention of this CHEM350 Report book to provide you with the means of completing all four steps.

Step 1: First we tell you what you are going to do.

Find out by reading the lab manual, doing the pre-lab questions in this report book, and filling out the Table of Reagents etc., i.e., preparing for the labs at home. (By doing so you are able to work more efficiently in the lab and the over-all time spent in the supervised lab can be reduced to ~20 hours from the usually 32 hours.)

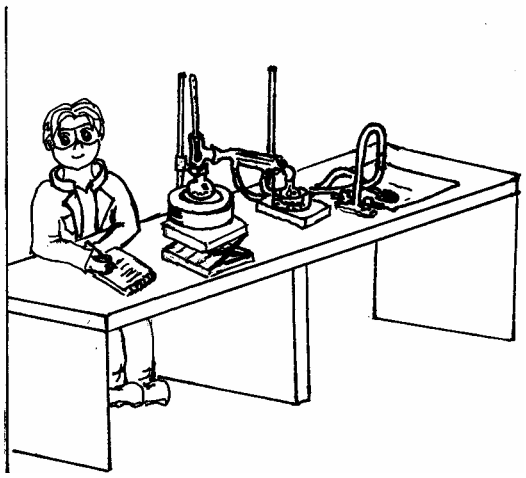
Step 2: Next we show you how to do it.

When you come to the lab, a lab instructor will give a safety orientation, followed by a series of mini lab lectures on each experiment. Various techniques will be demonstrated and you will be shown how to handle chemicals, dispose of hazardous waste, and operate the equipment.



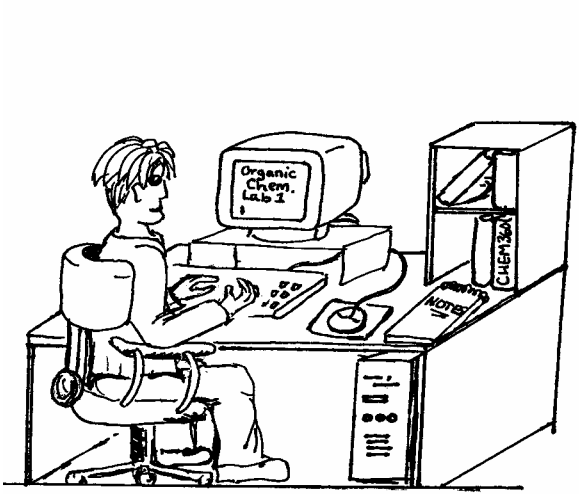
Step 3: Lab Time: Now you do what you've read, been told, and shown.

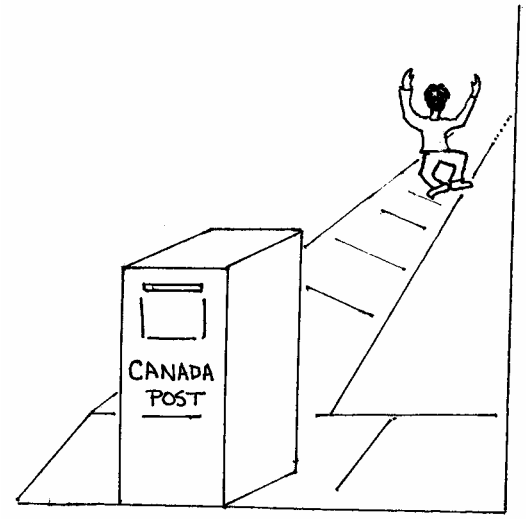
This is the time you spend in the lab performing your experiments, making your products, and recording your results in this report workbook.



Step 4: Finally, you tell us what you did.

This is the report writing stage. Actually most of your reports will have been written while in the lab. At home you will only have to do your calculations, write your discussion and conclusion and answer the questions at the end of each experiment.





Report Book Structure and How to Prepare for the Labs:

This CHEM350 Report Book is to used in conjunction with the CHEM350 Organic Chemistry I Lab Manual. It consists of an Introduction, Experiment Report Forms, Table of Reagents and a Course Evaluation. The reports are to be **completed one month** after of the lab session you attended. As a safety precaution, it is advisable to photocopy your reports before mailing them to your tutor for marking. Note: the marked reports are not returned to you.

How to best do the Report Book Exercises

1. First read through the lab manual introduction, and then answer the pre-lab questions for each experiment.
2. Complete the Objectives in the Experiment Report and begin to draft of your introduction.
3. Complete the Procedure (Refer to lab manual pages) and make a flowchart if necessary.
4. Complete the Table of Reagents for each experiment (detach a copy of the TOR to avoid flipping back and forth)
5. You are now ready to come to the lab and do the experimental work.

Note: Each experiment in the report book has the following headings:

Report Book Heading	Purpose and Use
1. Experiment Prelab Questions	Answer these questions to help you prepare and understand what you are doing in the lab. In order to answer these questions you will have to consult the CHEM350 Lab Manual, and to read the 'Introduction to Concept', and 'Background Information' sections of this manual. You may optionally submit these questions to the lab coordinator for review just before attending the lab.*
2. Objectives	Lists what you should learn from the lab. (see also lab manual). Use this information to fill in 'Objectives' in your Lab Write-up. When appropriate, write out any chemical reactions.
3. Introduction	Briefly state how the objectives of the experiment will be achieved and provide the relevant background information.
4. Procedure	Refer to the lab manual and only note any modifications or changes. Fill out the Table of Reagents**. Use the flowchart procedural step table to begin to record your work and observations.
The sections of your report shown below are completed while doing the experiment, or at home after the lab session.	
5. Results	While doing or immediately after your experiment, record your results in this section of the report.
6. Discussion an Conclusion	As soon after the lab as possible, discuss your results in light of the objectives, and make the appropriate conclusions. Remember to discuss sources of potential error and loss.
7. Post Lab Questions	Answer these questions to prove you understand what you did in the lab. To be completed after the experiment is finished. Submit your answers by mail along with your report and the Course Evaluation.

*CHEM350 Prelab questions are also available online at: http://science.pc.athabascau.ca/chem350.nsf
**CHEM350 Reagents are also available online at: http://science.pc.athabascau.ca/chem350.nsf

Acknowlegements:

The authors wish to thank Ms. Aimee Caouette for all the artwork. Athabasca University also wishes to thank Drs. K. Tanabe and T. Tamura and for all the IR Spectra used in this manual (pp. 65, 76, and 84). They were obtained from the SDBS web site: http://www.aist.go.jp/RIODB/SDBS/ (29-Sep-1999).

The following sources are also hereby acknowledged:

Laboratory Manual, Chemistry 320, Athabasca University, 1984.
Laboratory Manual, Chemistry 320, University of British Columbia, 1972-73.
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Lehman, J.W. 1999. *Operation Organic Chemistry*: A Problem-Solving Approach to the Laboratory Course, 3rd ed., Prentice Hall, New Jersey.

Mayo, D.W., R.M. Pike, and S.S. Butcher. 1989. *Microscale Organic Laboratory*, 2nd ed., John Wiley and Sons, Toronto, pp.229-232.

McMurry, J., 1992. *Organic Chemistry*, 3rd ed., Brooks/Cole Publishing Company, Pacific Grove, CA.

Weast, R.C. *et al*, 1974. *CRC Handbook of Chemistry and Physics*, 65th ed., CRC Press, Inc., Boca Raton, FL.

Each experiment has been modified and rewritten, keeping the particular needs of Athabasca University students in mind. The procedures described in this manual have been checked in our Athabasca laboratories by Jerry Pyrozko, Roger Klemm, Glen Conlin, and Robert Carmichael. Special thanks to Ms. Aimee Caouette for her help on the IR Tutorial (Summer 1999). The comments and suggestions received from the individuals mentioned above were greatly appreciated by the Course Co-ordinator.

Chem350 Experiment 1 Report Date:_____

Student Name:_____ ID Number:_____

Experiment 1 Prelab Questions:

1. Why do we need to know the melting point of a substance?
   a. To determine the exact time it takes for a sample to melt, and what color the compound becomes after heating.
   b. To determine the purity of a sample, 1-2 C range = pure, 3 C range or more = impure.
   c. To identify and then determine the crystal lattice structure of a compound.
   d. To identify the compound before using it in a reaction.

2. List the three steps used to prepare a melting point sample?
   a. (1) Mix the solid well before sampling. (2) Transfer a small amount of powder to a melting point tube. (3) Pack the sample to a height ~1 mm.
   b. (1) Transfer a small amount of powder to a melting point tube. (2) Pack the sample to a height ~1 mm. (3) Place the packed sample into the melting point apparatus and begin heating.
   c. (1) Crush the solid using a mortar and pestle. (2) Transfer a small amount of powder to a melting point tube. (3) Pack the sample to a height ~1 mm.
   d. (1) Crush the solid in a mortar. (2) Transfer a large amount of powder to a melting point tube. (3) Only pack the sample if the melting point tube is too full.

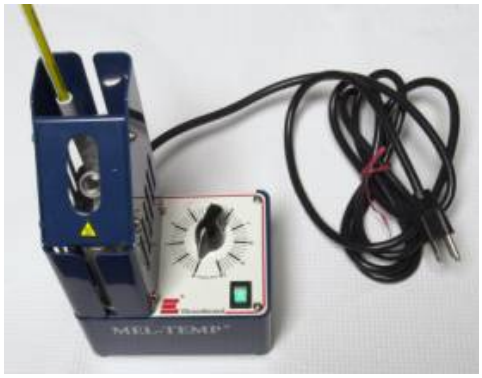
3. What are three main concerns regarding mercury filled melting point thermometers?
   a. Accuracy, precision, and fragility.
   b. Room temperature readings, accuracy, and spilt mercury disposal.
   c. Use for only mp determinations, they must be calibrated, and never heat above 250 C.
   d. Emergent stem error, paralax, and difficulty in finding a cheap supplier.

4. Define the temperatures recorded at the beginning and end of the melting point range.
   a. lower and upper limits of the melting point range.
   b. lower limit = when the first drop of liquid is seen; upper limit = when the sample is completely liquid.
   c. lower limit = when the sample is completely liquid; upper limit = when the sample has evaporated from the melting point tube.
   d. lower limit = when the sample begins to shrink/shrivel; upper limit = when the sample is completely liquid.

5. The CRC Handbook of Chemistry and Physics sometimes reports the melting point of a compound as a single number. What does this mean?
   a. It's the midpoint value between the upper and lower limit of the melting point range.
   b. It's the lower limit of the melting point range.
   c. It's the upper limit of the melting point range.
   d. It's the upper limit for the melting point range, corrected for barometric pressure effects.

6. The melting point apparatus should be heating at what rate (? C/min) as it approaches the melting point of the compound?
   a. The maximum rate of heating. Use the boost switch.
   b. 3
   c. 2
   d. 1

7. What is the name of the piece of laboratory equipment on the right?
   a. melting point apparatus
   b. pan balance
   c. spectrophotometer
   d. pH meter



8. The identification of a unknown solid will be achieved by mixed melting point determination.
   a. True
   b. False

Experiment 1 - Lab Safety

9. 'WHMIS' stand for?
   a. Workplace Hazardous Materials Information and Symbols
   b. Worker Hazardous Materials Information System
   c. Workplace Hazardous Materials Information System
   d. Worker Hazardous Materials Information Sheets

10. 'MSDS' stand for?
    a. Most Severe Data Sheet
    b. Material Sheet for Dangerous Supplies
    c. Material Safety Danger Sheet
    d. Material Safety Data Sheet

Chem350 Experiment 1 Report **Date:_____**

Student Name:_____ ID Number:_____

Title:

Objective(s):

Introduction: (definition and importance of mp, how one assesses purity using mp, mixed mp for ID, etc.)

Procedure: Ref. format: (author /surname, initials/, date. Title, publisher, page numbers)

Part A: Single melting point determination of unknown sample

Procedural Step	Observations
1. Record unknown code number 2. Record approximate melting point of the unknown 3. Prepare melting point tube i) Crush the sample using a mortar and pestle before loading the melting-point tube ii) iii) 4. Place tube in mp apparatus and heat sample 5. Record your experimentally determined melting point.	

Procedure (cont.):

Part B: Mixed melting point determination of an unknown sample

Procedural Step	Observations
1. Record unknown code number and suggested candidates 2. Literature Values of Unknown candidates 3. Prepare melting point tubes 4. Crush the sample using a mortar and pestle before loading the melting-point tube 5. Record your experimentally determined melting point.	

Table of Reagents for Exp. 1

Reagent	Formula	Mwt. (g/mol)	mp (°C)	bp (°C)	Hazardous Properties
benzoic acid	C_6H_5COOH				Irritant
3-chlorobenzoic acid	ClC_6H_4COOH				Irritant
biphenyl	$C_6H_5C_6H_5$				Irritant
salicylic acid	HOC_6H_4COOH				Toxic, Irritant
trans-cinnamic acid					Irritant
2-methylbenzoic acid	$CH_3C_6H_5COOH$				Irritant
4-nitrobenzaldehyde					Irritant
urea	NH_2CONH_2	60.06	133-135		Irritant
acetone (wash)	CH_3COCH_3			56.5	Flammable, Irritant

Results:

Part A

Melting point of sample # _____ = _____

Part B

Possible identity of unknown compound # _____:

1. _____ ; mp (Reference:)

2. _____ ; mp (Reference:)

Melting point of unknown compound # _____ = _____

Melting point obtained when unknown compound # _____ is mixed with

1. _____ = _____ (report range)

2. _____ = _____ (report range)

Conclusion:

(concluding statement, objectives achieved?)

The above results indicate that unknown compound #

_____ is probably _____.
(The structure of unknown _____ is drawn in the box.)

Structure of Unk.#_____

Experiment 1 Post Lab Questions:

Answers to these post lab questions must be submitted with your laboratory report.

1. In the lab manual introduction to this experiment, you were warned that heating the sample too quickly in the region of the melting point would result in the experimentally determined melting point being higher than the true value. Explain why this is so.

2. What is an eutectic mixture? How would you decide whether a given sample was a pure compound or an eutectic mixture of two compounds?

3. You are working in the lab, and you find an unlabelled vial with a white crystalline solid inside. In order to determine the identity of the compound, what would you have to do?

4. i) Give two reasons why you should calibrate your thermometer before using it of a melting point determination.

   ii) How do you properly 'cool off' a melting point thermometer?

Chem350 Experiment 2 Report Date:_____

Student Name:_____ ID Number:_____

Experiment 2 Prelab Questions:

1. Why does a chemist recrystallize an organic compound?
   a. To determine the solubility of the compound in a particular solvent.
   b. To convert the compound into a eutectic mixture.
   c. To identify the compound.
   d. To purify a compound prior to analysis or use in a reaction.

2. Which statement briefly explains how recrystallization increases the purity of a compound?

   a. Recrystallization is an art and it is by luck you get any pure crystals at all.
   b. Assuming that impurities are highly soluble in the selected solvent at all temperatures, by dissolving the desired impure compound in a minimum of hot solvent, and then cooling the solution, brand new crystals form while the impurities stay in solution, resulting in purer than original crystals.
   c. By dissolving the compound in hot solvent and then cooling the solution, brand new crystals form which are purer than the original crystals.
   d. Assuming that impurities are only highly soluble in the hot solvent, by dissolving the desired impure compound in a minimum of hot solvent, and then cooling the solution, brand new crystals form while the impurities stay in solution, resulting in purer than original crystals.

3. The following are the 5 steps of the recrystallization procedure:
   i. Select the recrystallation solvent.
   ii. Dissolve the crude solid in a minimum amount of hot solvent (= saturated solution).
   iii. Make a decision to hot gravity filter or not.
   iv. Slow cool and allow the crystals to form.
   v. Collect the crystals using vacuum filtration and wash with ice cold recrystallization solvent, and then allow the crystals to dry to a constant weight.
   a. True
   b. False

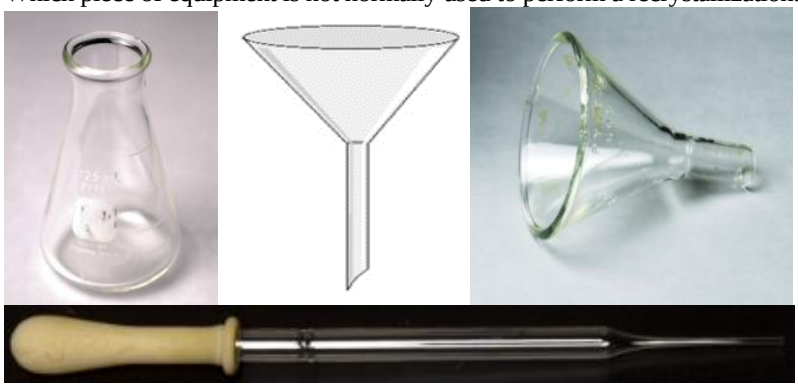
4. What are the criteria for selecting a solvent suitable for a single solvent recrystallization?
   a. soluble in hot solvent and soluble in cold solvent.
   b. insoluble in hot solvent and insoluble in cold solvent.
   c. insoluble in hot solvent and soluble in cold solvent.
   d. soluble in hot solvent and insoluble in cold solvent.

5. Boiling stones must be added to the recrystallization solvent prior to heating. Why (Note: there are two very good reasons for doing so)?
   a. In order to prevent the solution from 'bumping' in the first place.
   b. To avoid a sudden violent eruption of liquid should the solution be hot when the stones are added.
   c. a and b are correct.
   d. none of the above.

6. What are two situations where you are required to perform a hot gravity filtration.

a. colored impurities are present (therefore charcoal was added), and to remove soluble impurities.
b. soluble impurities are present (therefore charcoal was added), and to remove insoluble impurities.
c. colored impurities are present (therefore charcoal was added), and to remove insoluble impurities.
d. to remove boiling stones that were added, and to remove soluble impurities.

7. Which piece of equipment is not normally used to perform a recrystallization:



a. Erlenmeyer flask
b. Long narrow stemmed glass funnel
c. Short stemmed wide mouthed glass funnel
d. Pasteur pipette

8. If too much solvent is used when making a saturated solution, the yield of crystals at the end of the recrystallization will be reduced.
a. True
b. False

Experiment 2 - Lab Safety

9. Which of the four following statements is false?
a. Never heat a flammable solvent directly on a hot plate.
b. Never heat an Erlenmeyer flask filled more than 2/3rds full of liquid.
c. Always remember to use boiling stones when heating a solvent.
d. Acetanilide waste can be washed down the sink drain.

10. If you are unable to find any hazardous properties for a compound in the literature, you should
a. leave the Hazardous Properties column blank in the Table of Reagents in your CHEM350 Report Book.
b. assume the organic compound is safe; it has no hazardous properties.
c. assume that the organic compound is at least a 'suspected irritant'.

11. Acetanilide waste should be placed into the
a. General Organic Waste bottle in the fumehood.
b. Halogenated Organic Waste bottle in the fumehood.
c. Flushed down the drain with water.

Chem350 Experiment 2 Report

Date:_____

Student Name:_____ **ID Number:_____**

Title:

Objective(s):

Introduction:

Procedure:

(Ref:)

Single Solvent recrystallization of impure acetanilide

Procedural Step	Observations
1. Record appearance and amount of impure acetanilide weighed. Single Solvent Recrystallization Procedure (record appearance of solvent throughout and note any volume changes. Record elapsed time) 1. Select the solvent. 2 Heat volume of solvent to its bp. 3. 4. 5. Final Analyses	

Table 1. Table of Reagents for Exp. 2

Reagent	Formula	Mwt. (g/mol)	mp (°C)	bp (°C)	Hazardous Properties
acetanilide				NA	
sucrose	$C_{12}H_{22}O_{11}$			NA	
calcium carbonate	$CaCO_3$			NA	
silica	SiO_2			NA	
charcoal				NA	
water	H_2O		0	100	Burns when hot
acetone (wash)	CH_3COCH_3			56.5	Flammable liquid, irritant

NA= not applicable.

Experiment 2 Results:

Table 2. Table of Observations:

Procedural Step	Comment or Observation
Recrystallization solvent used:	
Volume of recrystallization solvent used:	
Appearance of solution after addition of charcoal	
Time allowed for crystals to form:	

Table 3. Table of Product Recrystallization Results

	Mass of Impure Acetanilide (g)	Mass of Pure Acetanilide Recovered (g)	Appearance of Crystals	% Recovery Yield	Melting Point (°C)
Impure acetanilide					
'Pure' acetanilide					
2nd crop 'Pure' acetanilide					

% recovery yield calculation:

Discussion:

Comments on and reasons for yield (high or low), and sources of error:

Conclusion:

Structure of Product

Experiment 2 Post Lab Questions:

Answers to these post lab questions should be submitted with your report.

1. The table below shows the solubility of a certain organic compound in water at five different temperatures.

Temperature (^oC)	Solubility of compound (in 100 mL of water)
0	1.5 g
20	3.0 g
40	6.5 g
60	11.0 g
80	17.0 g

   a) Plot a graph of the solubility of the compound versus temperature. Draw a smooth curve through the data points.

   b) If a student attempts to recrystallize a 0.5 g sample of this compound by heating it to 80^o C with 5.0 mL of water, would all of the sample dissolve? Briefly justify your answer.

   c) Assuming that the answer to part b is 'Yes', at what temperature will the crystals begin to appear when the student's solution begins to cool?

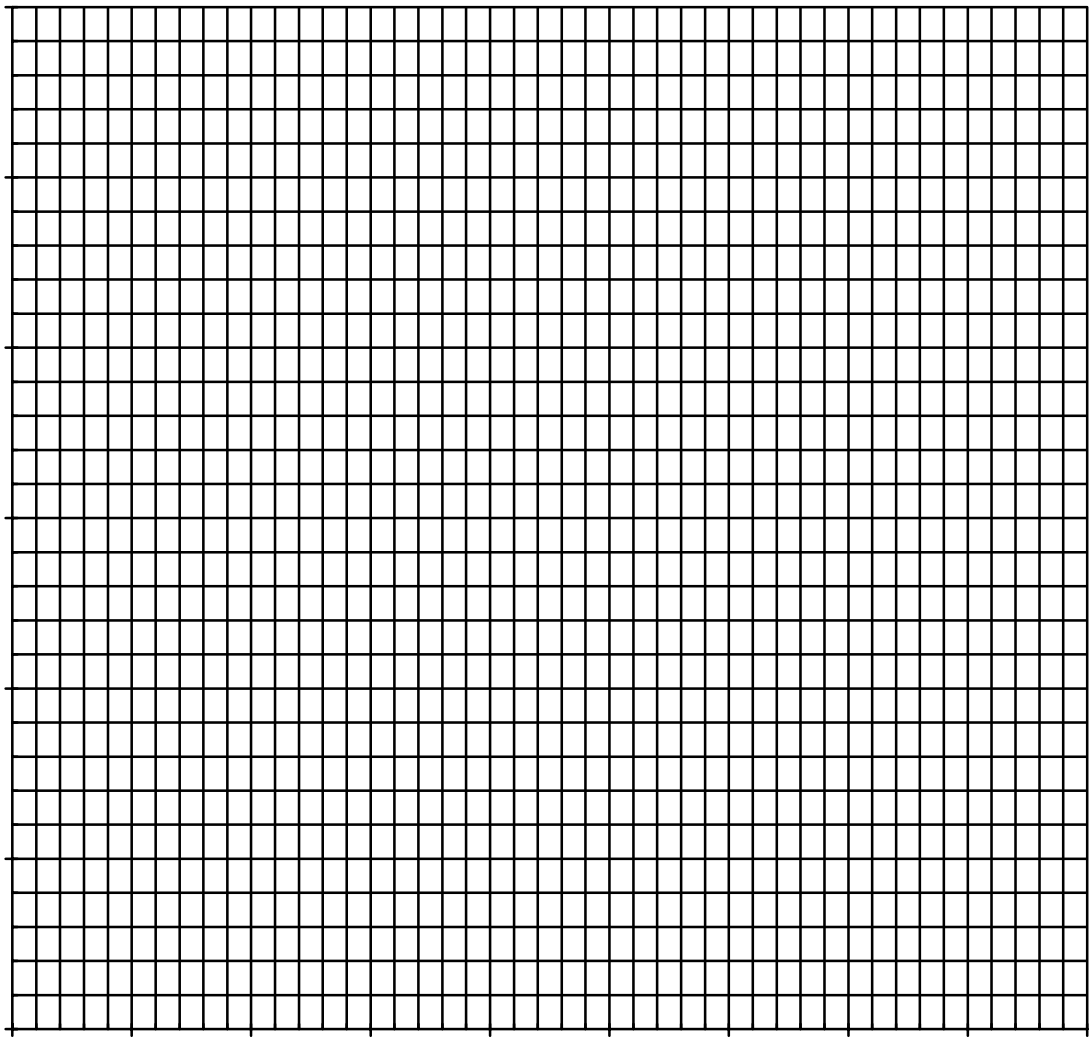
   d) If the student cooled the solution to 0^o C and filtered off the crystals, what is the maximum possible percentage recovery? What mass of the sample will remain in the filtrate?

2. Explain why you should slowly cool the filtered saturated solution obtained in step 3 of the recrystallization procedure?

3. During the last step of the recrystallization procedure, you collect the crystals by vacuum filtration. Why do you use ice cold recrystallization solvent to help transfer all the crystals to the Büchner funnel and wash the crystals?

4. Briefly explain the circumstances under which a mixed solvent recrystallization method would be used to recrystallize a given compound.

Graph paper insert



Chem350 Experiment 3 Report Date:_____

Student Name:_____ ID Number:_____

Experiment 3 Prelab Questions:

1. Why does a chemist distil an organic liquid?
   a. To determine the polarity of a particular solvent.
   b. To purify a compound prior to analysis or use in a reaction.
   c. To identify the compound.
   d. To convert the compound into its vapour form.

2. Which statement best explains how distillation purifies a compound?
   a. Distillation is used to concentrate a desired compound by removing undesired impurity vapours.
   b. Assuming that the impurity has a higher boiling point (must be greater than 25-30 ^{o}C different than the solvent), the desired solvent can be separated by heating it to its vapour phase and then collecting the pure vapours in a receiver flask.
   c. Distillation involves heating a liquid to its boiling point, at atmospheric or reduced pressure, to convert it to its vapour, and then condensing the vapour back into a liquid by cooling with a condensor.
   d. Distillation involves heating a liquid to its boiling point to convert it to its vapour, and then condensing the vapour back to liquid by icing the receiver flask.

3. The various heat sources available for a distillation, and when it is appropriate to use each heat source are:
   i. Bunsen burner (used only for aqueous non-flammable high boiling point (bp) solvents).
   ii. heating mantle (can be used for flammable solvents with bp up to 150 C).
   iii. steam bath (can be used for flammable solvents up to 85 C)
   iv. hot plate plus flat bottom dish/water bath (for low bp flammable solvents
   a. True
   b. False

4. Boiling stones must be added to the distillation flask prior to heating. Why?
   a. In order to prevent the solution from 'bumping'.
   b. To avoid a sudden violent eruption of liquid should the solution already be hot when the stones are added.
   c. To promote smooth boiling of the solvent.
   d. all of the above.

5. Which is the name of the piece of glassware on the right?
   a. Claisen adaptor
   b. three way connector or still head
   c. vacuum take off adaptor
   d. thermometer adaptor

6. Simple distillations can be performed on a mixture of two solvents only if the boiling points are different by more than 25-30 C. Fractional distillations are performed on a mixture of two solvents if the boiling points are different by less than 25-30 C.
   a. True
   b. False

Experiment 3 Lab Safety

7. Which of the four following statements is false?
   a. Never heat a distillation flask to dryness.
   b. Never heat a round bottom flask filled more than 1/2 full of liquid.
   c. Always remember to use boiling stones when heating a solvent.
   d. Ensure you have a open system before you begin heating.
   e. Once your distillation apparatus is fully assembled, then insert your thermometer down through the thermometer adapter.

8. When setting up the condensor, adjust the cooling water flow so that:
   a. it flows in the bottom and out the top of the condensor, at a maximum flow rate.
   b. it flows in the top and out the bottom of the condensor, at a maximum flow rate.
   c. it flows in the bottom and out the top of the condensor, at a minimum flow rate.

Chem350 Experiment 3 Report Date:_____

Student Name:_____ ID Number:_____

Title:

Objective(s):

Introduction:

Procedure:

(Ref:)
Changes/Modification:

Part A: Distillation of impure cyclohexanol

Procedural Step	Observations
3. Record amount of impure cyclohexanol used. Distillation Procedure 1. 2. 3. 4. 5. 6. Volume of Forerun Boiling point range of forerun Barometric Pressure Boiling point of product	

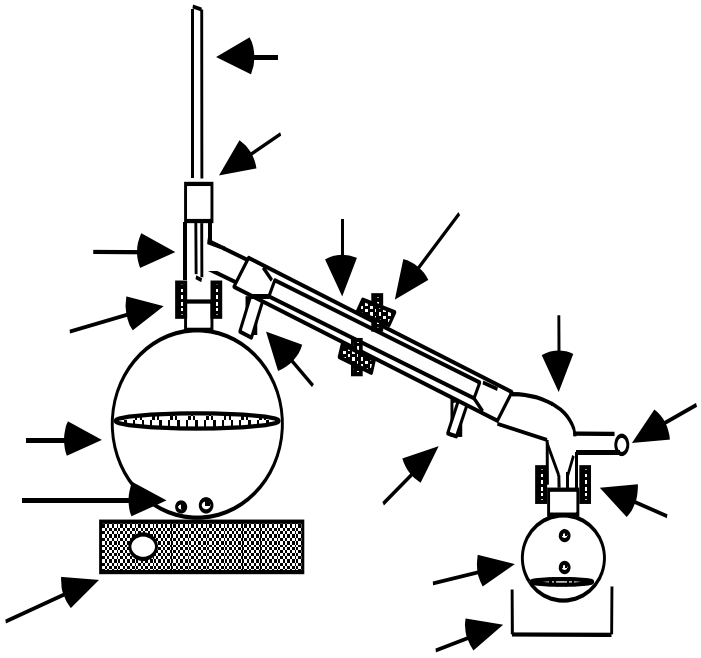
Part B: Fractional distillation of 50:50 mixture of cyclohexane:toluene.

Procedural Step	Observations
1. Record appearance and amount of 50:50 cyclohexane:toluene used. Fractional Distillation Procedure 1. 2. 3. 4. 5. 6.	

Table 1. Table of Reagents for Exp. 3

Reagent	Formula	Mwt. (g/mol)	mp (°C)	bp (°C)	Hazardous Properties
cyclohexanol					
toluene					
cyclohexane					
acetone (wash)	CH_3COCH_3			56.5	Flammable liq., irritant

Sketch for the assembly of a simple distillation apparatus (fill in labels).



Labels to place on sketch:

distillation flask	condensor
receiving flask	water in
heating mantle	water out
boiling stones	ice bath
thermometer	clamps (3)
three-way connector (still head)	vacuum adapter
thermometer adapter	'open to air'

Experiment 3 Results:

Table 2. Table of Observations:

Procedural Step	Comment or Observation

Table 3. **Part A** Table of Product Simple Distillation Results

	Volume (mL)	Appearance of Liquid	% Recovery Yield	Boiling Range (°C)/Pressure	Press. Corrected Boiling Range(°C)
Impure Cyclohexanol					
Forerun					
'Pure' cyclohexanol					

Table 4. **Part B** Table of Product Fractional Distillation Results

	Volume (mL)	Appearance of Liquid	% Recovery Yield	Boiling Range (°C)/Pressure	Press. Corrected Boiling Range(°C)
50:50 cyclohexane:toluene					
Forerun					
Fraction 1					
Fraction 2					
Fraction 3					

Discussion:

Comments on and reasons for yield (high or low), sources of error (uncalibrated thermometer, atmospheric pressure effects):

Conclusion:

Structure of Products		

Experiment 3 Post Lab Questions:

Answers to these post lab questions should be submitted with your report.

1. A student who was performing a distillation for the first time failed to position the thermometer correctly. The bulb was set too high. What effect would this have on the observed boiling point of the liquid being distilled?

2. Under perfect conditions, the number of theoretical plates required to separate an ideal mixture of two components of boiling points T_A and T_B is given by the relationship:

$$\text{Number of theoretical plates needed} = \frac{120}{T_A - T_B}$$

On this basis, how many theoretical plates are needed to separate a mixture of cyclohexane and toluene? (Note: In practice, the actual number of theoretical plates required may be as high as double the number predicted by this equation!)

3. You suddenly notice you have forgotten to add boiling stones to your round bottomed distillation flask but the distillation is now in progress. What should you do?

4. What is the purpose of the condensor during a distillation?

Chem350 Experiment 4 Report Date:_____

Student Name:_____ ID Number:_____

Experiment 4 Prelab Questions

1. The two common ways to assess the purity of a liquid organic sample are:
   a. thin layer chromatography, and determine its refractive index.
   b. distil the liquid and determine its density by determining the mass and volume of product.
   c. convert the compound into its vapour form and condense it back to a liquid.
   d. fractional distillation of the liquid, followed by refractive index analysis.

2. The refractive index of a liquid is fundamentally based on the change of the speed of
   a. flowing water.
   b. gaseous molecules.
   c. light.

3. The refractive index is dependent upon which two key factors?
   a. temperature of the sample and the wavelength of the incident light.
   b. the operator of the refractometer and the amount of sample used.
   c. density of the sample and the wavelength of the incident light.

4. Which of the following sequences describes the correct order of the steps needed to measure a refractive index?
   a. Turn on refractometer, apply sample, adjust side hand wheel, adjust thumb wheel for chromatic aberration, readjust side hand wheel, read meter.
   b. Turn on refractometer, apply sample, adjust thumb wheel for chromatic aberration, adjust side hand wheel, readjust side hand wheel, read meter.
   c. Turn on refractometer, adjust thumb wheel for chromatic aberration, adjust side hand wheel, apply sample, read meter
   d. All of the above (the order does not matter).

5. From the formulae provided below, choose the one which describes the correct method to calculate the percentage error in a refractive index measurement.

   a) $= \frac{|\text{actual value} - \text{theoretical value}|}{\text{theoretical value}} \times 100\% =$

   b) $= \frac{\text{actual value}}{\text{theoretical value}} \times 100\% =$

   c) $= \frac{|\text{theoretical value} - \text{actual value}|}{\text{actual value}} \times 100\% =$

Experiment 4 Lab Safety

6. What is the major safety concern regarding this experiment?
   a) Handling flammable and toxic solvents.
   b) the sodium lamp in the refractometer.
   c) disposal of waste sample.
   d) the temperature of the sample during refractometer readings.

Chem350 Experiment 4 Report Date:_____

Student Name:_____ ID Number:_____

Title:

Objective(s):

Introduction:

Procedure:

(Ref:)
Changes/Modification:

Part A: Refractive index (*n*) of cyclohexanol

Procedural Step	Observations
Table 4. Record *n* of purified cyclohexanol. 2. Record *n* of starting impure cyclohexanol (optional)	

Part B: Refractive Index (*n*) of fraction/mixtures of cyclohexane:toluene.

Procedural Step	Observations
2. Record *n* of fractionated cyclohexane:toluene mixtures.	

Table 1. Table of Reagents for Exp. 4

Reagent	Formula	Mwt. (g/mol)	mp (°C)	bp (°C)	Hazardous Properties
cylcohexanol					
toluene					
cyclohexane					
acetone (wash)	CH_3COCH_3			56.5	Flammable liq., irritant

Experiment 4 Results:

Table 2. **Part A** Table of Product Simple Distillation Results

	Observed n_D	Temperature (°C)	Corrected n_D^{20}	% Error *n* Cyclohexanol
Impure Cyclohexanol				
'Pure' cyclohexanol				

*Literature Value of cyclohexanol n_D^{20} =

Table 3. **Part B** Table of Refractive Index (*n*) of Fractional Distillation Samples

	Observed n_D	Temperature (°C)	Corrected n_D^{20}	% Error
50:50 Cyclohexane:Toluene				
Fraction 1				
Fraction 2				
Fraction 3				

*Literature Value of cyclohexane n_D^{20} =
*Literature Value of toluene n_D^{20} =

Table 4. **Part B** Table of Product Fractional Distillation Results

	Mol% Cyclohexane	Mol% Toluene
50:50 Cyclohexane:Toluene		
Fraction 1		
Fraction 2		
Fraction 3		

Calculation of the percent mole fractions:

Discussion:

Comments on and reasons for high or low readings, %error, mole fraction results, assessment of the efficiency of the separation achieved in your fractional distillation, and sources of error:

Conclusion:

Experiment 4 Post Lab Questions:

Answers are to be included with your report.

1. Look up the boiling points of cyclohexanol, cyclohexane and toluene in a suitable reference book and report your findings. Don't forget that when you quote a boiling point, melting point, or similar physical property you should always cite the source. Example:

   1,3-Butadiene; b.p. = - 44 ^{o}C (*Handbook of Chemistry and Physics*, 47th ed. Cleveland, Ohio: The Chemical Rubber Co., 1966)

2. Suggest a reason why the boiling point of cyclohexanol is so much higher than those of cyclohexane and toluene.

3. Suggest a reason why the refractive index of cyclohexanol is higher than that of water.

4. To reduce the percentage error in the n_D reading of your purified cyclohexanol (compared to the literature value), what should you do?

Chem350 Experiment 5 Report Date:_____

Student Name:_____ ID #:_____

Experiment 5 Prelab Questions:

1. What is the easiest way to separate two immiscible liquids?
   a. use a ultracentrifuge.
   b. use a Büchner funnel.
   c. use a separatory funnel.

2. Fifty milliters of 5% sodium hydroxide and dichloromethane were added to a separatory funnel. What would you observe?
   a. a homogeneous, clear, and colourless solution.
   b. two layers of liquid, both clear and colourless.
   c. two layers of liquid, fizzing, and the separatory funnel would have to be immediately vented.

3. Given K = ([solute] in solvent A, g . L^{-1}) / ([solute] in solvent B, g . L^{-1})
   The distribution coefficient for a compound in a two solvent extraction system is 2.0. If you are given 4.0 g of compound dissolved in 100 mL of solvent B, is the following the correct answer for how much compound will be extracted, if you use 50 mL of solvent A for the extraction.

$$K = 2.0 = \frac{(x\ /0.05L)}{(4-x)/0.1L)}, \text{rearrange to solve for x,} = \frac{(8-2x)}{0.1L} = \frac{x}{0.05L} \text{ or } 0.1\,x = 0.05\,(8 - 2x), \text{ therefore, } 0.2x = 0.4 \text{ or } x = 2g$$

   a. Yes
   b. No

4. Why do we add 5% NaOH to extract the organic acid from the organic mixture?
   a. The strong, aqueous, inorganic base (NaOH) will react with the organic acid and convert the organic acid to its water insoluble, salt form (R-COO-Na+).
   b. The strong, aqueous, inorganic base (NaOH) will react with the organic acid and convert the organic acid to its water soluble, salt form (R-COO-Na+).
   c. The weak, aqueous, inorganic base (NaOH) will not react with the organic acid and not convert the organic acid to its water soluble, salt form (R-COOH + NaOH --- no reaction).

5. Why do we add 1.5 M HCl to extract the organic base from the organic mixture?
   a. The strong, aqueous, inorganic acid (HCl) will react with the organic base and convert the organic base to its water insoluble, salt form (R-NH3+Cl-).
   b. The strong, aqueous, inorganic acid (HCl) will react with the organic base and convert the organic base to its water soluble, salt form (R-NH3+Cl-).
   c. The weak, aqueous, inorganic acid (HCl) will not react with the organic base and not convert the organic base to its water soluble, salt form (R-NH2 + HCl --- no reaction).

6. Why do we add concentrated hydrochloric acid (6 or 12 M) to the separated, aqueous salt of the organic acid ($R\text{-}COO^-Na^+(aq) + HCl(aq) \rightarrow ?$).
   a. The strong, concentrated, aqueous, inorganic acid (6 or 12 M HCl) will react with the salt of the organic acid, and reconvert it to its water insoluble, salt form ($R\text{-}COO^- H^+$).
   b. The strong, concentrated, aqueous, inorganic acid (6 or 12 M HCl) will lower the pH of the organic salt solution, but will not react with the organic acid salt.
   c. The strong, concentrated, aqueous, inorganic acid (6 or 12 M HCl) will react with the salt of the organic acid, and reconvert it to its water insoluble form (R-COOH).

7. Why do we add concentrated sodium hydroxide (6 M) to the separated, aqueous salt of the organic base ($R\text{-}NH3^+Cl^-$).
   a. The strong, concentrated, aqueous, inorganic base (6M NaOH) will react with the salt of the organic base, and reconvert it to its water insoluble, salt form ($R\text{-}NH2^+ Cl^-$).
   b. The strong, concentrated, aqueous, inorganic base (6M NaOH) will raise the pH of the organic salt solution, but will not react with the organic base salt.
   c. The strong, concentrated, aqueous, inorganic base (6M NaOH) will react with the salt of the organic base, and reconvert it to its water insoluble form (R-NH2).

Experiment 5 Lab Safety

8. What is/are the safety concern(s) regarding this experiment?
   a) A leaking separatory funnel.
   b) dichloromethane is toxic and readily absorbed through the skin.
   c) hot glassware during the recrystallizations.
   d) all the above.

Chem350 Experiment 5 Report

Date:_____

Student Name:_____ **ID Number:_____**

Title:

Objective(s):

Introduction:

General Reaction Equations:

Reaction 1: Reaction of Organic acid with dilute sodium hydroxide:

Reaction 2: Reaction of Organic base with dilute hydrochloric acid:

Reaction 3: Reaction of the salt of the organic acid with strong acid:

Reaction 4: Reaction of the salt of the organic base with strong base:

Procedure:

(Ref:)
Changes/Modification:

Part A: Extraction of the organic acid through salt formation.

Procedural Step	Observations
Record Unknown Code:	

Part B: Extraction of the organic base through salt formation.

Procedural Step	Observations

Part C: Recovery of the organic acid from its salt.

Procedural Step	Observations

Sample Calculation of volume of 12 M HCl to add:

Part D: Recovery of the organic base from its salt.

Procedural Step	Observations

Sample Calculation of volume of 6 M NaOH to add:

Table 1. Table of Reagents for Exp. 5

Reagent	Formula	Mwt. (g/mol)	mp (°C)	bp (°C)	Hazardous Properties
dichloromethane					
benzoic acid	C_6H_5COOH				
2-methylbenzoic acid					
4-methylbenzoic acid					
4-chlorobenzoic acid					
salicylic acid					
3-nitroaniline					
4-chloroaniline					
naphthalene					
5% NaOH	NaOH				
1.5 M HCl	HCl				
12 M HCl (conc.)	HCl				
6 M NaOH	NaOH				
distilled water	H_2O				
methanol	CH_3OH				
ethanol	CH_3CH_2OH				
ethyl acetate					
hexanes					
acetone (wash)	CH_3COCH_3			56.5	Flammable liq., irritant

Experiment 5 Results:

Table 2. Table Summarizing Observations:

Procedural Step	Comment or Observation

Table 3. Yield and Characterization of Unknown #_____

	Yield (g)	Appearance of Crystals	Melting Point (°C)	Tentative Identification of Unkown	Melting Point of Known* (°C)	Mixed Melting Point (°C)
Organic Acid						
Organic Base						
Neutral Compound						

*Reference : The Handbook of Chemistry and Physics, _____ ed., Cleveland, Ohio, The Chemical Rubber Co., _____.

Discussion:

Reaction equations with your identified unknowns. Comments on and reasons for yield (high or low), sources of error, etc.:

Conclusion:

Structure of Products		

Experiment 5 Post Lab Questions:

Answers to be submitted with report.

1. When extracting an organic compound from an aqueous solution into an organic solvent, e.g., diethyl ether, a chemist will sometimes add sodium chloride to the aqueous solution. What is the purpose of such an addition, and what is the procedure called?

2. Why is the procedure used in this experiment called liquid-liquid extraction?

3. A CHEM350 student was working on her yield determination of her recrystallized *p*-aminobenzoic acid, when some naphthalene was inadvertently spilt into her crystals. You happen along the scene, and offer the following advice to the distraught student:
   a) Redissolve all the solid in dichloromethane, extract with dilute aqueous acid, re-isolate the organic compound by precipitating the salt of the base with strong base, and recrystallize your *p*-aminobenzoic acid again.
   b) Redissolve all the solid in dichloromethane, extract with dilute aqueous base, re-isolate the organic compound by precipitating the salt of the acid with strong acid and recrystallize *p*-aminobenzoic acid again.
   c) Do either a or b.
   d) Discard everything into the hazardous waste container. Nothing can be done.

4. When an aqueous solution of an organic compound is shaken with an immiscible organic solvent, such as diethyl ether, the solute distributes itself between the two phases. When the two phases separate into two distinct layers, an equilibrium will have been established such that the ratio of the concentrations of the solute in each solvent defines a constant, K, called the distribution coefficient (or partition coefficient).

   $$K = \frac{\text{concentration of solute in solvent A, e.g., diethyl ether } (g \cdot L^{-1})}{\text{concentration of solute in solvent B, e.g., water } (g \cdot L^{-1})}$$

   The distribution coefficient for compound X in the diethyl ether/water system is 3.0.
   If you were given a solution containing 8.0 g of X in 500 mL of water, and wanted to extract compound X into diethyl ether, show that it would be more effective to extract X using three 50 mL aliquots of diethyl ether rather than a single 150 mL aliquot. (HINT: Determine how much of X would remain in the aqueous solution in each case.)

Chem350 Experiment 6 Report Date:_____

Student Name:_____ ID Number:_____

Experiment 6 Prelab Questions

1. What does the Bromine Test detect?
   a. all types of unsaturation in a molecule.
   b. double bonds only.
   c. triple bonds only.
   d. differentiates between alkanes and molecules with bond unsaturation.

2. What does the Baeyer Test detect?
   a. differentiates between alkanes and molecules with all types of bond unsaturation.
   b. double bonds only.
   c. differentiates between alkanes and molecules containing double and triple bonds.
   d. triple bonds only.

3. What does the Ammoniacal Silver Test detect?
   a. differentiates between alkanes and molecules with all types of bond unsaturation.
   b. double bonds only.
   c. differentiates between alkanes and molecules containing double and triple bonds.
   d. triple bonds only.

4. If a compound gives a positive reaction in all four tests it is most likely to be an _____.
   a. aldehyde
   b. alkene
   c. alkane
   d. alkyne

5. If the compound does not react in any of the four tests, the compound could be a(n) _____.
   a. alkene
   b. carboxylic acid
   c. alkane
   d. alkyne

6. The sulfuric acid test is also a test used for determining an organic compounds solubility.
   a. True.
   b. False.

Experiment 6 Lab Safety

7. Which reagent used in the functional group tests must be specially handled before discarding, and why?
   a. bromine, as it must be discarded in the halogenated organic waste bottle in the fumehood.
   b. ammoniacal silver test reagent, must be decomposed by the addition of nitric acid in order to prevent the formation of explosive silver acetylides.
   c. potassium permanganate, as it must be neutralized before discarding.

Chem350 Experiment 6 Report Date:_____

Student Name:_____ ID Number:_____

Title:

Objective(s):

Introduction:

Procedure:

(Ref:)
Changes/Modification:

Table 1. Table of Reagents for Experiment 6.

Reagent	Formula	Mwt. (g/mol)	Mp (°C)	Bp (°C)	Hazardous Properties
pentane					
cyclohexene					
phenylacetylene					
biphenyl					
toluene					
bromine					
dichloromethane					
Baeyer Reagent					
Ammoniacal Silver					
sulfuric acid (conc.)	H_2SO_4				
acetone (wash)	CH_3COCH_3			56.5	Flammable liq., irritant

Experiment 6 Part A Results:

Bromine Test			
Test Substance	**Observation**	**Inference**	**Equation**
Pentane			
Cyclohexene			
Phenylacetylene			
Biphenyl			
Toluene			
Unkown			

Baeyer Test			
Test Substance	**Observation**	**Inference**	**Equation**
Pentane			
Cyclohexene			
Phenylacetylene			
Biphenyl			
Toluene			
Unknown			

Ammoniacal Silver Test			
Test Substance	**Observation**	**Inference**	**Equation**
Pentane			
Cyclohexene			
Phenylacetylene			
Biphenyl			
Toluene			
Unkown			

Sulfuric Acid Test			
Test Substance	**Observation**	**Inference**	**Equation**
Pentane			
Cyclohexene			
Phenylacetylene			
Biphenyl			
Toluene			
Unkown			

Discussion:

Comments on tests, sources of error, and false positives/negatives:

Conclusion:

Instructor Led Group Infrared Analysis Problems

Use the tables below to record your results of the Infrared Spectral Analyses for the following compounds (IR spectra on CHEM350 Lab Manual pages 122-128. Label the diagnostic absorption bands on the spectra.

Cyclohexanol	Absorption Band#	Wavenumber (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or weak)	Functional Group Indicated

Functional Group absent:

2-methyl-3-butyn-2-ol	Absorption Band#	Wavenumber (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or weak)	Functional Group Indicated

Functional Group absent:

3-buten-2-ol	Absorption Band#	Wavenumber (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or weak)	Functional Group Indicated

Functional Group absent:

benzhydrol	Absorption Band#	Wavenumber (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or weak)	Functional Group Indicated

Functional Group absent:

Instructor Led Group Infrared Analysis Problems (cont.)

benzaldehyde	Absorption Band#	Wavenumber (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or weak)	Functional Group Indicated

Functional Group absent:

acetic acid	Absorption Band#	Wavenumber (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or weak)	Functional Group Indicated

Functional Group absent:

dibutylamine	Absorption Band#	Wavenumber (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or weak)	Functional Group Indicated

Functional Group absent:

Infrared Analysis Practice Problems:
Use the tables below to record your results of the Infrared Spectral Analyses of the provided known spectra on CHEM350 Lab Manual pages 131-138.

cyclohexanone	Absorption Band#	Wavenumber (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or weak)	Functional Group Indicated

Functional Group(s) absent:

benzaldehyde	Absorption Band#	Wavenumber (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or weak)	Functional Group Indicated

Functional Group(s) absent:

ethyl benzoate	Absorption Band#	Wavenumber (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or weak)	Functional Group Indicated

Functional Group(s) absent:

benzoic acid	Absorption Band#	Wavenumber (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or weak)	Functional Group Indicated

Functional Group(s) absent:

Infrared Analysis Practice Problems (cont.):
Use the tables below to record your results of the Infrared Spectral Analyses of the provided known spectra.

phenylacetylene	Absorption Band#	Wavenumber (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or weak)	Functional Group Indicated

Functional Group(s) absent:

benzonitrile	Absorption Band#	Wavenumber (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or ,weak)	Functional Group Indicated

Functional Group(s) absent:

styrene	Absorption Band#	Wavenumber (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or weak)	Functional Group Indicated

Functional Group(s) absent:

diethyl ether	Absorption Band#	Wavenumber (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or weak)	Functional Group Indicated

Functional Group(s) absent:

Infrared Unknowns:
Use the tables below to record your results of the Infrared Spectral Analyses for the unknowns (see handouts). Please remember to attach to the report, the unknown spectra with the diagnostic absorption bands identified.

Code: Name:	Absorption Band#	Wavenumber (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or weak)	Functional Group Indicated

Functional Group absent:

Code: Name:	Absorption Band#	Wavenumber (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or weak)	Functional Group Indicated

Functional Group absent:

Code: Name:	Absorption Band#	Wavenumber (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or weak)	Functional Group Indicated

Functional Group absent:

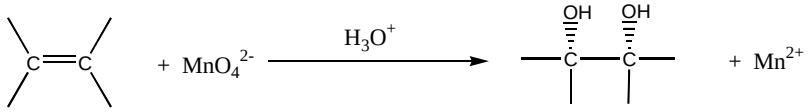
Code: Name:	Absorption Band#	Wavenumber (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or weak)	Functional Group Indicated

Functional Group absent:

Experiment 6 Post Lab Questions:

Answers are to be submitted with your lab report.

1. The reaction of an alkene with acidic potassium permanganate is an example of a redox reaction. Use the method that you learned in a General Chemistry course to write out a balanced equation for the reaction below.



a C_2H_4 + b $KMnO_4$ + c H^+ <------> d $C_2H_6O_2$ + e K^+ + f Mn^{+2}

Half Rxns.

reduction: ___MnO_4^- + ___H^+ + ___e^- ------> ___Mn^{2+} + ___H_2O

oxidation: ___C_2H_4 + ___H_2O ----------> ___$C_2H_6O_2$ + ___ e^- + ___H^+

Bal. Equation: _____

2. The reaction of an alkene with potassium permanganate can also occur in a basic medium, in which case the inorganic product is a brown precipitate of manganese (IV) oxide. (The organic product is again the diol). Write a balanced redox equation for the reaction of an alkene with alkaline potassium permanganate.

a C_2H_4 + b $KMnO_4$ + **?** OH^- <------> d $C_2H_6O_2$ + e K^+ + f $MnO_{2\,(s)}$ + **?** OH^-

Half Rxns.

reduction: ___MnO_4^- + ___H_2O + ___e^- ------> ___$MnO_{2\,(s)}$ + ___OH^-

oxidation: ___ C_2H_4 + ___OH^- ----------> ___ $C_2H_6O_2$ + ___ e^-

Bal. Equation: _____

3. What are the major differences you would see in the infrared spectra of an alkane, alkene, and alkyne?

Chem350 Experiment 7 Report Date:_____

Student Name:_____ ID Number:_____

Experiment 7 Prelab Questions

1. Which of the following compounds is optically active?
   a. ultra pure water.
   b. acetone.
   c. tetrahydrofuran.
   d. dichloromethane.
   e. none of the above.

2. The measured optical activity of a solid compound is affected by three major factors. They are:
   a. concentration and temperature of the solution, and length of sample tube.
   b. size of the molecule, natural source of chemical, and solubility.
   c. density and temperature of compound, and length of sample tube.

3. If 0.8000 g compound was dissolved in 50.00 mL of solvent, and the solution was placed in a 2 dm long sample tube, and gave an (observed rotation) of +3.2 degrees, the specific rotation would be:
   a) $+50^{o}$
   b) $+100^{o}$
   c) $+1000^{o}$

4. During the solid-liquid extraction of the lichen with acetone, the lab manual (Exp. 7 Procedure Part A of lab manual) suggests we extract for 30 minutes. What would happen if the extraction went longer than 30 minutes?
   a. The usnic acid being leached out of the lichen would begin to denature.
   b. Nothing. A maximum amount of usnic acid has been extracted and further extraction time does no harm.
   c. The extraction solvent, acetone, will begin to evaporate and thereby diminish the overall yield of usnic acid.
   d. The experiment will have to be terminated because there will now be insufficient time to complete it.

5. Why is all the 'acetone extraction solvent' removed prior to beginning the recrystallization part of the procedure?
   a. So that a saturated solution can be made using reagent grade acetone.
   b. The acetone must be removed in order to isolate and characterize the crude usnic acid.
   c. Acetone is not a suitable recrystallization solvent.
   d. Tetrahydrofuran and acetone are miscible solvents so acetone must be removed prior to using the polarimeter.

Experiment 7 Lab Safety

6. Which reagent(s) used in this experiment must be specially handled, and why?
   a. acetone, as it is highly flammable.
   b. tetrahydrofuran, as it is highly toxic and flammable.
   c. a and b are correct.

Chem350 Experiment 7 Report

Date:_____

Student Name:_____ **ID Number:_____**

Title:

Objective(s):

Introduction:

Procedure:

(Ref:)
Changes/Modification:

Procedure for extraction of usnic acid from lichen.

Procedural Step	Observations
Record appearance and amount of lichen used. Extraction: Gravity Filtration Solvent Removal Recrystallization Product Analysis	

Table 1. Table of Reagents for Experiment 7.

Reagent	Formula	Mwt. (g/mol)	Mp (°C)	Bp (°C)	Hazardous Properties
lichen					
acetone					
ethanol					
L- tartaric acid					
water					
tetrahydrofuran					
usnic acid					

Experiment 7 Results:

Table 2. Table Summarizing Observations:

Procedural Step	Comment or Observation

Table 3. Part A-C. Table of Product, Usnic acid Extraction from Lichen

Table 3 shows a summary of the extraction results for the experiment. The calculations for % Composition of Lichen (w/w) is shown below the table.

	Mass Lichen (g)	Product Yield (g)	Appearance of Crystals	Melting Pt. (°C)	Mixed Melting Pt. (°C)	Reference Melting Pt. (°C)	% Lichen (w/w)
( ) Usnic acid							

% Weight of Lichen Calculation:

Table 4. Part D-E. Results of Polarimetry Measurements for Unknown and Usnic Acid.

Table 4 shows a summary of the polarimetry results of the experiment. The calculations for specific rotation and optical purity are shown beneath the table.

	Mass (g)	[Solution] (g/mL)	Observed Rotation (α)*	Corrected Observed Rotation (α–blank)	**Specific Rotation*** n_D	Reference Rotation n_D^{20}	Optical Purity
Unknown (L-tartaric acid)							
( ) Usnic acid							

*At the temperature of solution during optical rotation determination:

Specific Rotation Calculations:

Optical Purity of ( ) Usnic acid product: (O.P.= actual n_D^{20}/theoretical n_D^{20}) x 100%)

Discussion:

Comments on and reasons for yield (high or low), specific rotations, optical purity, and sources of error:

Conclusion:

Structure of Product

Experiment 7 Post Lab Questions:

1. Define the difference between diastereomers and enantiomers. Choose a specific example (eg. glucose/fructose) to help explain your answer.

2. Draw a Fischer projection and line/wedge diagrams for the two enantiomers of usnic acid. (see Figure 7.1 in Chem350 Lab Manual)

Chem350 Experiment 8 Report Date:_____

Student Name:_____ ID Number:_____

Experiment 8 Prelab Questions

1. The preparing of cyclohexene from cyclohexanol is an example of a widely used method of converting an alcohol functional group into an _____ functional group?
   a. alkene
   b. alkane
   c. non-reactive.
   d. reactive.

2. The purpose of adding sodium chloride to the aqueous layer in Step 6 of the procedure is to:
   a) to make a salt of the organic acid.
   b) to 'salt' out the water from the organic layer.
   c) to preserve the product

3. The purpose of adding phosphoric acid to the reaction vessel containing cyclohexanol is:
   a. to neutralize any contaminating base.
   b. to act as a catalyst in the reaction.
   c. to slow the reaction rate and thereby increase the yield.

4. How do you separate the aqueous and the cyclohexene organic layer?
   a. distillation.
   b. reflux.
   c. using a separatory funnel.
   d. extraction.

5. The purpose of adding saturated sodium chloride (brine) to the aqueous layer in Step 6 of the procedure is to:
   a. to make a salt of the organic acid.
   b. to make the product less soluble in the water and to 'salt out' the water from the organic layer.
   c. to preserve the product.
   d. to add water to the organic layer.

6. Which of the following ways would characterize your final product and thereby help prove that you have converted cyclohexanol to cyclohexene:
   a. infrared spectroscopy.
   b. nuclear magnetic resonance spectroscopy.
   c. refractive index.
   d. density.
   e. all of the above.
   f. only a and b are correct.

7. What is the first step called in the mechanism for an acid catalyzed dehydration?
   a. protonation.
   b. elimination.
   c. carbocation intermediate formation.
   d. substitution.

8. Alexander Zaitzev's rule for elimination reactions states:
   a. "in the addition of HX to an alkene, the more highly substituted carbocation is formed as the intermediate rather than the less highly substitued one".
   b. "Base-induced elimination reactions generally give the more highly substituted (more stable) alkene product".
   c. "The structure of a transition state resembles the structure of the nearest stable species. Exergonic reaction steps resemble reactants and Endergonic reaction steps resemble products".

Experiment 8 Lab Safety

9. Which reagent(s) used in this experiment must be specially handled, and why?
   a. saturated sodium chloride, as it is highly corrosive.
   b. cyclohexanol, as it is a toxic starting reagent.
   c. phosphoric acid, as it is highly corrosive.
   d. both b and c.

Chem350 Experiment 8 Report Date:_____

Student Name:_____ ID Number:_____

Title: Preparation of Cyclohexene from Cyclohexanol

Objective(s):

Reaction equation:

Introduction:

Procedure:

(Ref:)
Changes/Modification:

Procedure for the acid-catalyzed dehydration of cyclohexanol to form cyclohexene.

Procedural Step	Observations
Record amount of pure cyclohexanol used. Setup Reaction Reaction Work-up Final Distillation Procedure 1. 2. 3. 4. 5. 6. Volume of Forerun Boiling point range of forerun Boiling point of product	

Table 1. Table of Reagents for Experiment 8

Reagent	Formula	Mwt. (g/mol)	Mp (°C)	Bp (°C)	Hazardous Properties
cylcohexanol					
phosphoric acid					
cyclohexene					
sodium chloride	NaCl				
sodium carbonate					
calcium chloride	$CaCl_2$				
acetone (wash)	CH_3COCH_3			56.5	Flammable liq., irritant

Experiment 8 Results:

Table 2. Table Summarizing Observations:

Procedural Step	Comment or Observation

Table 3. Properties of the Acid-Catalyzed Dehydration Product, Cyclohexene

Table 3. shows a summary of the results of the experiment. The calculations for theoretical yield and percent yield should be shown below the table. Note: _____ was the limiting reagent, since the only other reagent involved in the reaction, phosphoric acid, served as a catalyst.

	Mass (g)	Appearance of Liquid	Boiling Pt. (°C) (/Pressure)	Theoretical Yield (g)	% Yield
Cyclohexene					

Boiling Point Pressure Correction:

Theoretical Yield Calculation:

% Yield Calculation:

Table 4. Tabulation of Characteristic Infrared Absorptions for cyclohexanol and cyclohexene.

Table 4 contains the results of the Infrared Spectral Analyses for cyclohexanol and cyclohexene. See also attached labelled spectra for peak numbering and identification.

cyclohexanol	Peak#	Wavenumber (cm-1)	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or ,weak)	Functional Group Indicated

cyclohexene	Peak#	Wavenumber (cm-1)	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or ,weak)	Functional Group Indicated

Discussion:

Comments on reasons for yield (high or low), purity (high or low), sources of error, etc.:

Conclusion:

Structure of Product

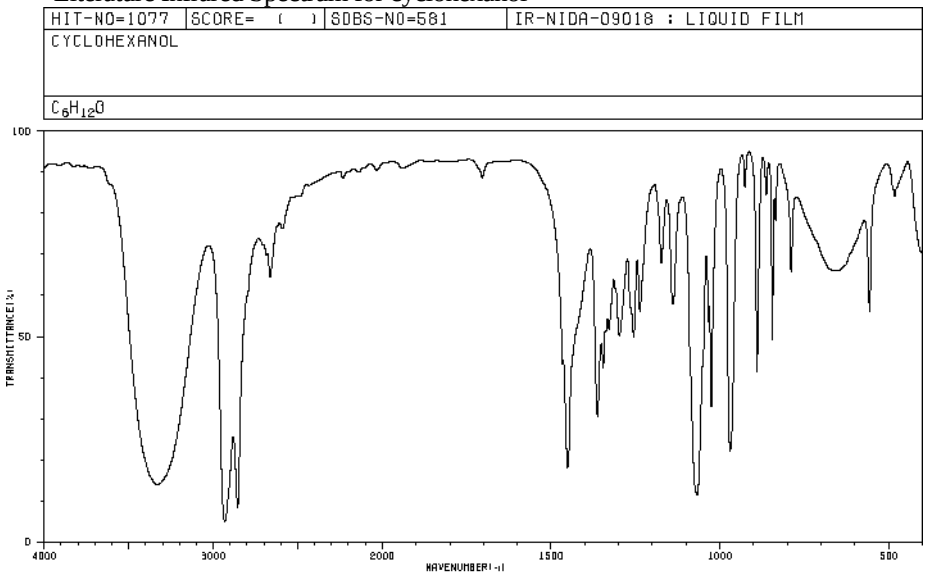
Experiment 8 Post Lab Questions:

Answers to be submitted with report.

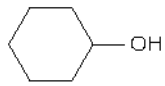
1. What is the purpose of adding 10% sodium carbonate solution to the distillate in step 7 of the procedure?

2. Identify two possible by-products that could be formed from cyclohexanol in this experiment. (HINT: See lab manual Ex.8 Introduction. You may also want to search through your textbook to find what other reactions can occur between an alcohol and a concentrated mineral acid (e.g. phosphoric acid).

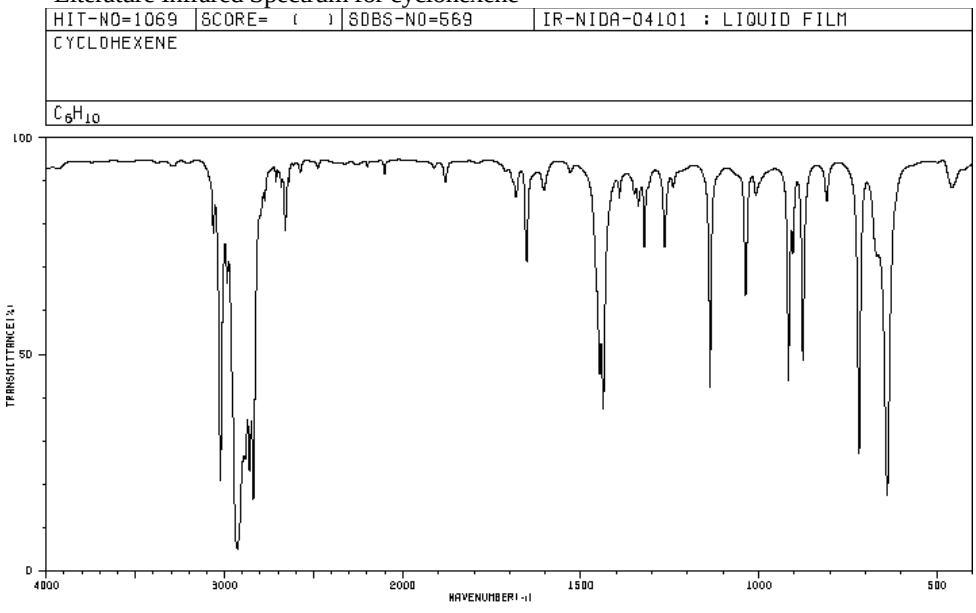
Literature Infrared Spectrum for cyclohexanol



3331	13	1704	86	1266	47	970	21	667	64
2932	4	1467	42	1238	53	926	84	557	53
2855	8	1452	17	1174	66	890	39	482	61
2686	68	1363	29	1140	55	863	81		
2666	62	1346	41	1068	11	845	47		
2586	74	1329	50	1034	52	835	74		
2233	84	1298	49	1025	32	789	64		



Literature Infrared Spectrum for cyclohexene



3063	74	2711	86	1652	68	1330	84	917	42
3023	20	2682	84	1604	84	1322	72	904	70
2985	84	2659	74	1447	45	1265	72	877	46
2927	4	2639	86	1438	36	1241	84	810	81
2860	21	1880	86	1392	84	1137	41	719	26
2836	15	1691	86	1351	84	1037	60	640	16
2776	81	1682	84	1339	81	1009	84	457	84

Chem350 Experiment 8 (Alternate) Report Date:_____

Student Name:_____ ID Number:_____

Experiment 8 (Alternate) Prelab Questions

1. The preparing of methylpentenes from 4-methyl-2-pentanol is an example of a widely used method of converting an alcohol functional group into an _____ functional group?
   a. alkene
   b. alkane
   c. non-reactive.
   d. reactive.

2. The purpose of adding sulfuric acid to the reaction vessel containing 4-methyl-2-pentanol is:
   a. to neutralize any contaminating base.
   b. to act as a catalyst in the reaction.
   c. to slow the reaction rate and thereby increase the yield.

3. How do you separate the aqueous and the methylpentenes organic layer?
   a. distillation.
   b. reflux.
   c. using a separatory funnel.
   d. extraction.

4. The purpose of adding saturated sodium chloride (brine) to the aqueous layer in Step 8 of the procedure is to:
   a. to make a salt of the organic acid.
   b. to make the product less soluble in the water and to 'salt out' the water from the organic layer.
   c. to preserve the product.
   d. to add water to the organic layer.

5. Which of the following ways would characterize your final product and thereby help prove that you have converted 4-methyl-2-pentanol to methylpentenes:
   a. infrared spectroscopy.
   b. nuclear magnetic resonance spectroscopy.
   c. refractive index.
   d. density.
   e. all of the above.
   f. only a and b are correct.

6. What is the first step called in the mechanism for an acid catalyzed dehydration?
   a. protonation.
   b. elimination.
   c. carbocation intermediate formation.
   d. substitution.

7. Alexander Zaitzev's rule for elimination reactions states:
   a. "in the addition of HX to an alkene, the more highly substituted carbocation is formed as the intermediate rather than the less highly substitued one".
   b. "Base-induced elimination reactions generally give the more highly substituted (more stable) alkene product".
   c. "The structure of a transition state resembles the structure of the nearest stable species. Exergonic reaction steps resemble reactants and Endergonic reaction steps resemble products".

Experiment 8 Lab Safety

8. Which reagent(s) used in this experiment must be specially handled, and why?
   a. saturated sodium chloride, as it is highly corrosive.
   b. 4-methyl-2-pentanol, as it is the starting reagent.
   c. hot sulfuric acid, as it is highly corrosive.

Chem350 Experiment 8 (Alternate) Report Date:_____

Student Name:_____ ID Number:_____

Title:

Objective(s):

Reaction equation:

Introduction:

Procedure:

(Ref:)
Changes/Modification:

Procedure for the acid-catalyzed dehydration of 4-methyl-2-pentanol to form methylpentenes.

Procedural Step	Observations
Record amount of pure 4-methyl-2-pentanol used. Setup Reaction Reaction Work-up Final Distillation Procedure 1. 2. 3. 4. 5. 6. Volume of Forerun Boiling point range of forerun Boiling point of product	

Table 1. Table of Reagents for Optional Experiment 8

Reagent	Formula	Mwt. (g/mol)	Mp (°C)	Bp (°C)	Hazardous Properties
4-methyl-2-pentanol	$C_6H_{14}O$	102.18		132	
sulfuric acid	H_2SO_4				
sodium hydroxide (10%)	NaOH				
sodium chloride	NaCl				
calcium chloride	$CaCl_2$				
acetone (wash)	CH_3COCH_3			56.5	Flammable liquid, irritant
1-pentene, 2-methyl	C_6H_{12}	84.16		62	Flammable liquid, irritant
1-pentene, 4-methyl	C_6H_{12}	84.16		53-54	Flammable liquid, irritant
2-pentene, 2-methyl	C_6H_{12}	84.16		67	Flammable liquid, irritant
2-pentene, 3-methyl				69	Flammable liquid, irritant
2-pentene, 4-methyl				57-58	Flammable liquid, irritant

Optional Experiment 8 Results:

Table 2. Table Summarizing Observations:

Procedural Step	Comment or Observation

Table 3. Properties of the Acid-Catalyzed Dehydration Products, Methylpentenes

Table 3. shows a summary of the results of the experiment. The calculations for theoretical yield and percent yield should be shown below the table. Note: _____ was the limiting reagent, since the only other reagent involved in the reaction, sulfuric acid, served as a catalyst.

	Mass (g)	Appearance of Liquid	Boiling Pt. (°C) (/Pressure)	Theoretical Yield (g)	% Yield
Methylpentenes					

Boiling Point Pressure Correction:

Theoretical Yield Calculation:

% Yield Calculation:

Table 4. Tabulation of Characteristic Infrared Absorptions for 4-methyl-2-pentanol and methylpentenes.

Table 4 contains the (hypothetical) results of the Infrared Spectral Analyses for 4-methyl-2-pentanol and methylpentenes.

See also attached labelled spectra for peak numbering and identification.

4-methyl-2-pentanol	Peak#	Wavenumber (cm-1)	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or ,weak)	Functional Group Indicated

methylpentenes	Peak#	Wavenumber (cm-1)	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or ,weak)	Functional Group Indicated

Tabulation of GC methylpentenes results (http://www.remotelab.ca)

Table 1 Concentration (%v/v) of Isomers determined by Gas Chromatography

Component	%(v/v)
4-methyl-1-pentene	
cis and *trans*-4-methyl-2-pentene	
2-methyl-1-pentene	
2-methyl-2-pentene	
cis and *trans*-3-methyl-2-pentene	

(attach online printed report to your lab report)

Discussion:

Comments on reasons for yield (high or low), purity (high or low), % isomers, sources of error, etc.:

Conclusion:

Structure of Products

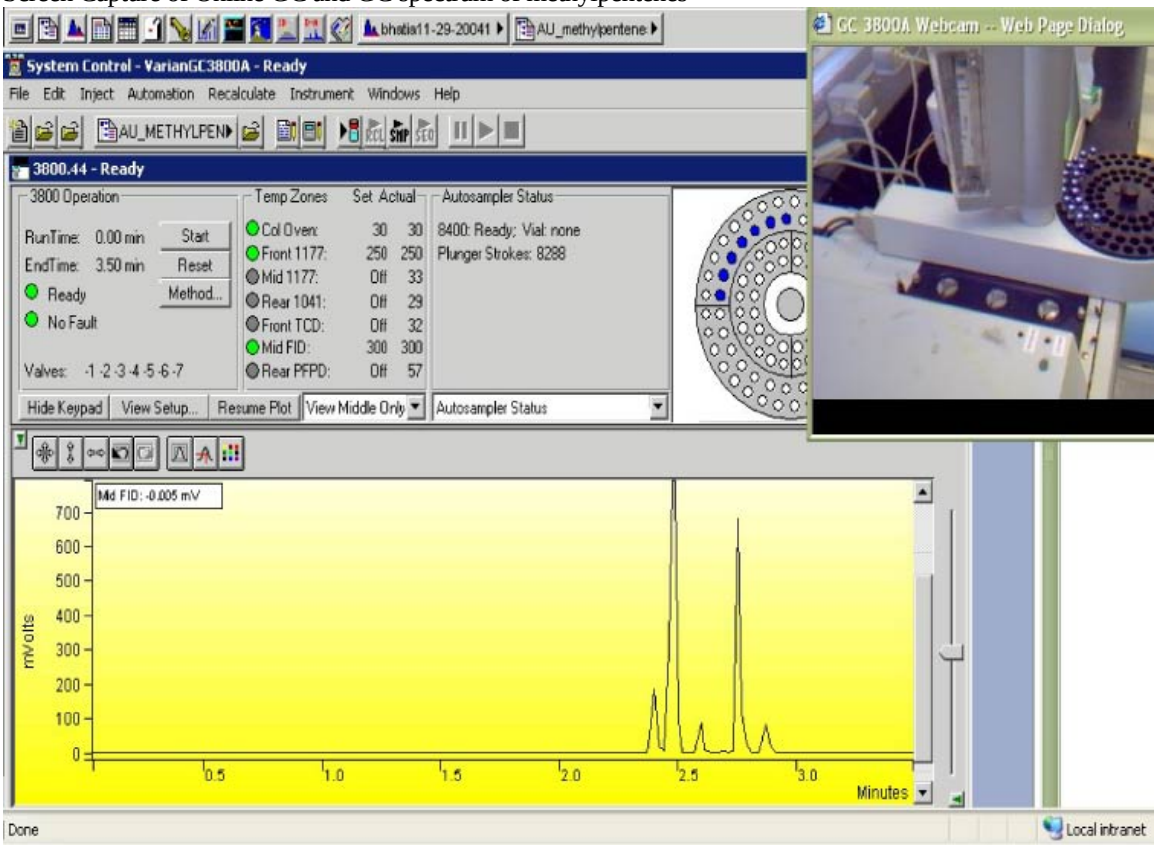
Experiment 8 (Alternate/Optional) Post Lab Questions:

Answers to be submitted with report.

1. What is the purpose of adding 10% sodium hydroxide solution to the distillate in step 6 of the procedure?

2. Would infrared spectroscopy be useful in identifying the products of the reaction performed in this experiment? Briefly explain your answer.

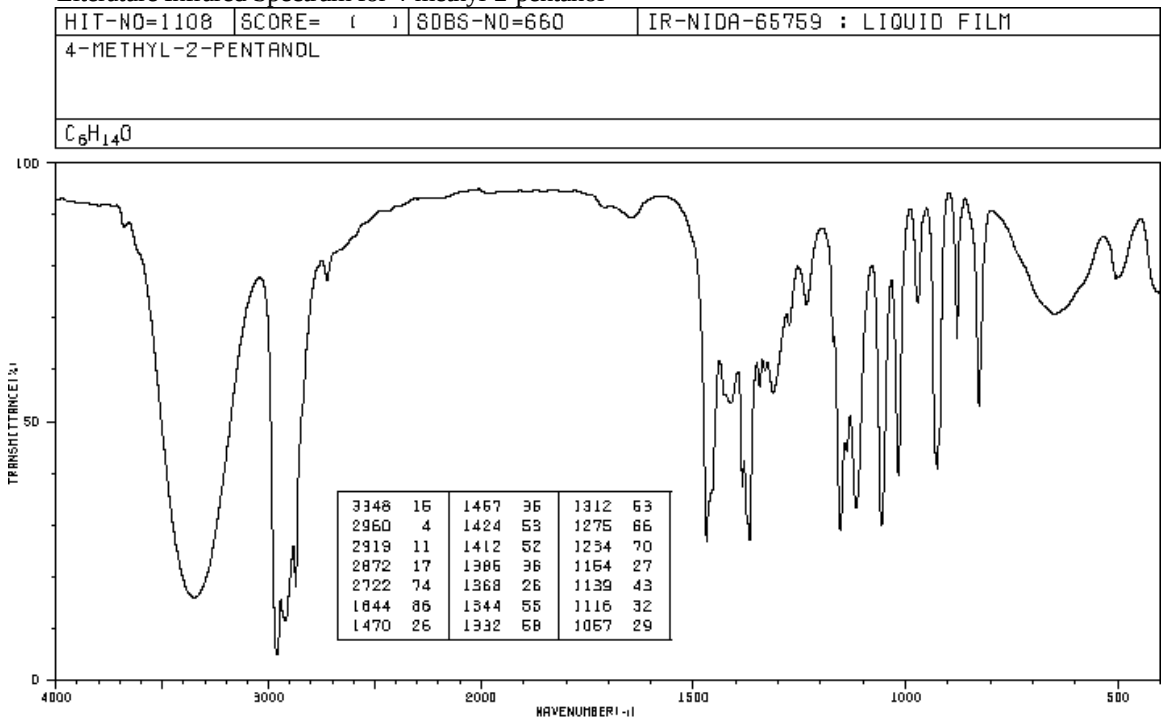
Screen Capture of Online GC and GC spectrum of methylpentenes



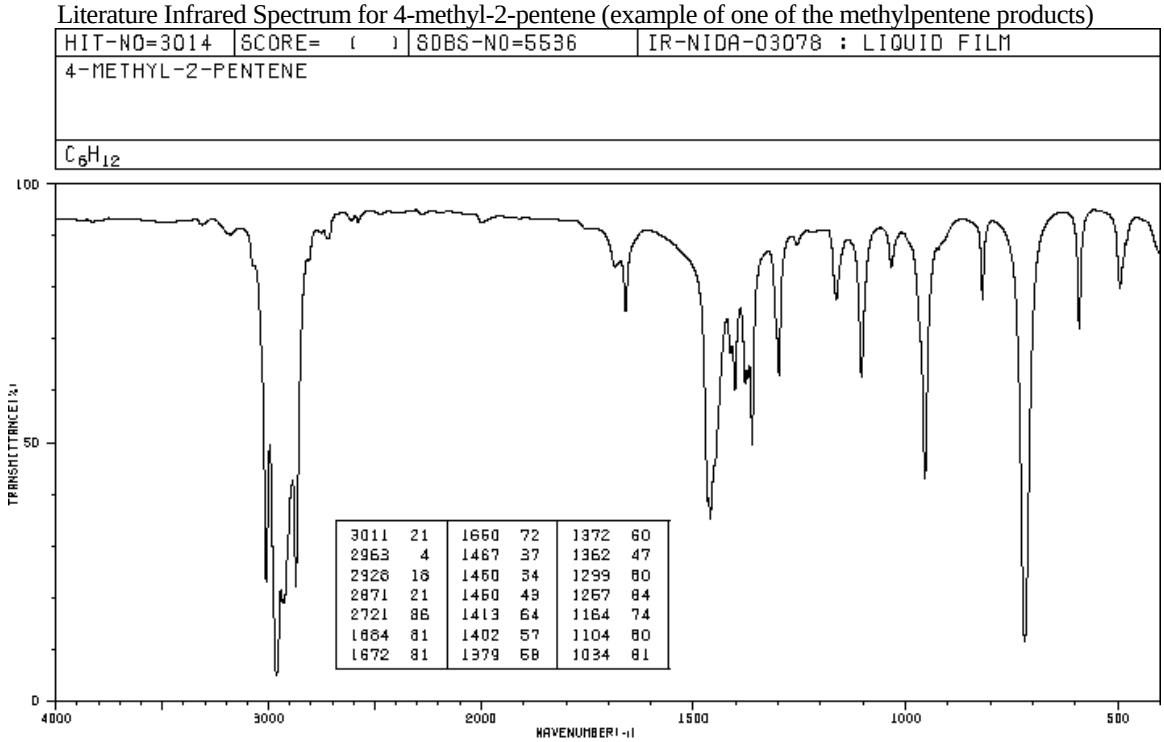
Experiment 8 (Alternate) Student Data for GC Analysis of methylpentenes product

Table1. Concentration (%v/v) of Isomers		**n=17**
Component	**Avg %(v/v)**	**Std Dev %(v/v)**
4-methyl-1-pentene	5.326	0.099
4-methyl-2-pentene (cis/trans)	65.329	1.129
2-methyl-1-pentene	4.697	0.133
2-methyl-2-pentene	22.181	0.808
3-methyl-2-pentene (cis/trans)	2.467	0.591
Average Total % Isomers	100.000	

Literature Infrared Spectrum for 4-methyl-2-pentanol



Literature Infrared Spectrum for 4-methyl-2-pentene (example of one of the methylpentene products)



Chem350 Experiment 9 Report Date:_____

Student Name:_____ ID #:_____

Experiment 9 Prelab Questions

1. What is the purpose of dissolving the acetanilide in glacial acetic acid prior to beginning the nitration reaction?
   a. So that the acetanilide is 'in solution' when the sulfuric acid is added.
   b. So that the acetanilide is 'in solution' when the nitrating mixture is added.
   c. To stabilize the acetanilide prior to the addition of sulfuric acid.
   d. To prevent the acetanilide from reacting to quickly when the nitrating mixture is added.

2. What happens when you mix sulfuric acid with nitric acid?
   a. The sulfuric acid is the weaker acid so nitric acid converts it to sulfate ion.
   b. Nitronium ion is formed.
   c. a slow endothermic reaction occurs.

3. What is the name of the electrophile used in this experiment?
   a. sulfuric acid.
   b. nitirc acid.
   c. acetanilide.
   d. nitronium ion.

4. What acts as the nucleophile in this experiment?
   a. sulfuric acid.
   b. nitirc acid.
   c. acetanilide.
   d. nitronium ion.

5. Why do you wash the product several times (Procedure Steps 8-10) with ice-cold water?
   a. to rinse away all unreacted acetanilide.
   b. to rinse away excess acid.
   c. a and b are correct.

6. How is the product characterized in this experiment?
   a. melting point.
   b. melting point and infrared spectroscopy.
   c. yield, melting point and infrared spectroscopy.
   d. none of the above.

7. What major differences in absorption bands would you expect to see in the infrared spectra of acetanilide and *p*-nitroacetanilide?
   a. amide carbonyl adsorption at 1680 cm^{-1} for both and only a nitro ~1500 cm^{-1} adsorption at for the starting reagent.
   b. the absorption due to the introduced nitro group in the product (1600-1500 and 1400-1300 cm^{-1}).
   c. the absorption due to the introduced amide group in the product (1600-1500 and 1400-1300 cm^{-1}).

Experiment 9 Lab Safety

8. Which reagent(s) used in this experiment must be specially handled, and why?
   a. nitric acid, it is highly corrosive.
   b. glacial acetic acid, it is highly corrosive.
   c. sulfuric acid, it is highly corrosive.
   d. all of the above.

Chem350 Experiment 9 Report Date:_____

Student Name:_____ ID Number:_____

Title:

Objective(s):

Equation(s):

Introduction:

Procedure:

(Ref:)
Changes/Modification:

Proc. For the electrophilic aromatic substitution of acetanilide to form *p*-nitroacetanilide.

Procedural Step	Observations
Record amount of pure acetanilide used.	

Table 1. Table of Reagents for Experiment 9

Reagent	Formula	Mwt. (g/mol)	Mp (°C)	Bp (°C)	Hazardous Properties
acetanilide					
acetone (wash)					

Experiment 9 Results:

Table 2. Table Summarizing Observations:

Procedural Step	Comment or Observation

Table 3. Table of *p*-nitroacetanilide, Nitration Product.

Table 3. presents the summary of the results of the experiment. The calculations for limiting reagent, theoretical yield and percent yield are shown below the table. Note: _____ was found to be the limiting reagent.

Name of product	Mass (g)	Appearance of Crystals	Melting Pt. (°C)	Theoretical Yield (g)	% Yield

Limiting Reagent and Theoretical Yield Calculation:

% Yield Calculation:

Table 4. Tabulation of Characteristic Infrared Absorptions for acetanilide and *p*-nitroacetanilide.

Table 4 contains the results of the Infrared Spectral Analyses for acetanilide and *p*-nitroacetanilide. See also attached labelled spectra for peak numbering and identification.

Acetanilide	Peak#	Wavenumber (cm-1)	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or ,weak)	Functional Group Indicated

***p*-nitroacetanilide**	Peak#	Wavenumber (cm-1)	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or ,weak)	Functional Group Indicated

Discussion:

Comments on and give reasons for yield (high or low), purity, sources of error, and infrared spectrum results, etc.:

Conclusion:

Structure of Product

Experiment 9 Post Lab Questions:

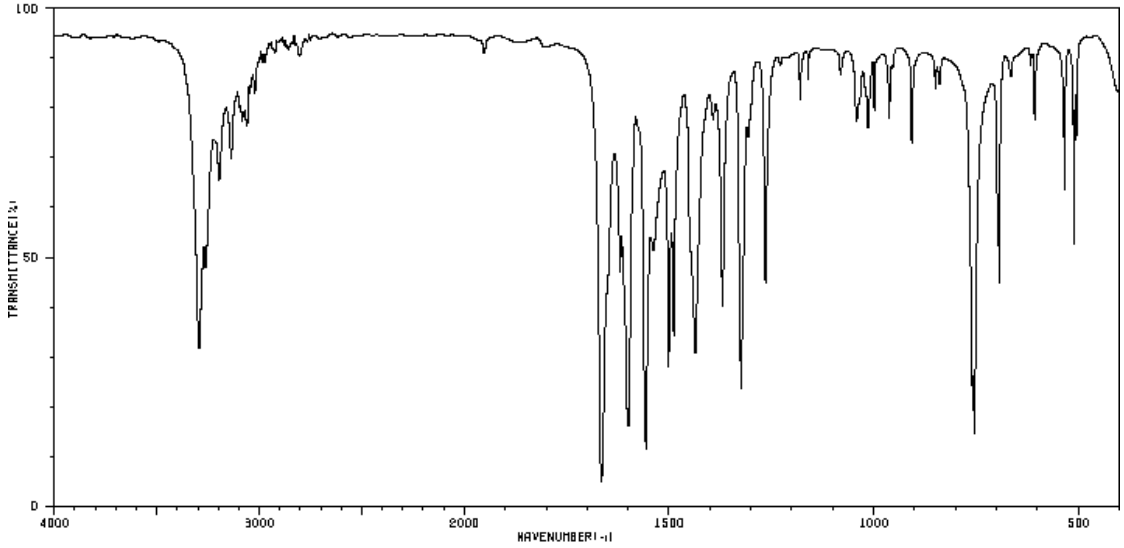
Answers to be submitted with your lab report.

1. During the nitration of acetanilide (Step 4 of the procedure), care is taken to keep the reaction mixture cool. What do you think might be the consequences of allowing the reaction mixture to become too warm?

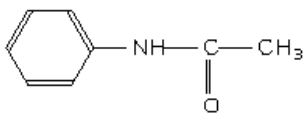
2. What organic compound (other than ethanol) would you reasonably expect to isolate from the ethanol/water mixture that was used to recrystallize your 4-nitroacetanilide?

Literature Infrared Spectrum of acetanilide

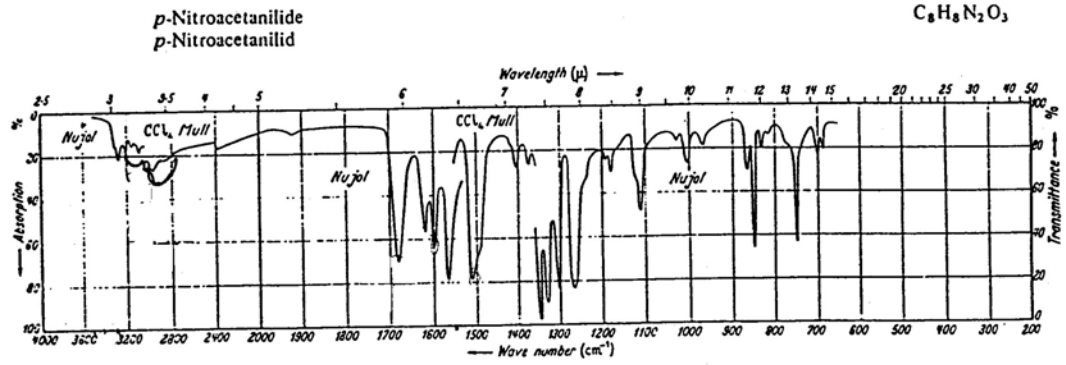
HIT-NO=1095	SCORE= ( )	SDBS-NO=729	IR-NIDA-61373 : KBR DISC
ACETANILIDE			
C_8H_9NO			



3294	30	3022	78	1489	33	1180	78	761	23
3261	46	1665	4	1436	29	1042	74	754	13
3196	82	1620	44	1393	74	1014	72	694	43
3137	66	1598	16	1369	38	999	77	607	74
3083	74	1557	10	1324	22	962	74	534	60
3059	74	1538	48	1307	72	908	70	511	50
3045	79	1601	26	1266	43	768	62	506	70



Literature Spectrum of *p*-nitroacetanilide (nujol mull)-from Sigma Aldrich IR



Compound Name	Chemical Formula	Solid (S) or Liquid (L)	Formula Weight	MP or BP (°C)	Density (g/mL)	Refract. Index	Hazardous Properties*
acetanilide	$CH_3CONHC_6H_5$	S	135.17	113-115			Toxic, irritant
acetanilide,4-methyl	$CH_3CONHC_6H_4CH_3$	S	149.19	149-151			Irritant
acetanilide, *p*-nitro	$CH_3CONHC_6H_4NO_2$	S	180.16	216			Irritant
acetanilide, *o*-nitro	$CH_3CONHC_6H_4NO_2$	S	180.16	94			Irritant
acetanilide, *m*-nitro	$CH_3CONHC_6H_4NO_2$	S	180.16	154-156			Irritant
acetic acid, glacial (17.4 M)	CH_3CO_2H	L	60.05	118.1	1.049		Corrosive, hygroscopic
acetic acid, *p*-ethoxyphenyl	$C_2H_5OC_6H_4CH_2CO_2H$	S	180.2	87-90			Irritant
acetic anhydride	$(CH_3CO)_2O$	L	102.09	140	1.082	1.3900	Corrosive, lachrymator
acetone	CH_3COCH_3	L	58.08	56.5	0.7899	1.3590	Flammable, irritant
acetone, diethylamino	$(C_2H_5)_2NCH_2COCH_3$	L	129.2	64/16mm	0.832	1.4250	Irritant
acetophenone	$C_6H_5COCH_3$	L	120.15	202	1.030	1.5325	Irritant
activated carbon		S					(see charcoal)
allyl alcohol (2-propen-1-ol)	$CH_2=CHCH_2OH$	L	58.08	96-98	0.854	1.4120	Highly Toxic, flammable
ammonia (14.8 M)	NH_3	L	17.03		0.90		Corrosive, lachrymator
ammonium hydroxide (14.8 M)	NH_4OH	L	35.05		0.90		Corrosive, lachrymator
aniline	$C_6H_5NH_2$	L	93.13	184	1.022	1.5860	Highly toxic, irritant
aniline, 4-bromo	$BrC_6H_4NH_2$	S	172.03	62-64			Toxic, irritant
aniline, 4-chloro	$ClC_6H_4NH_2$	S	127.57	72.5			Highly toxic, irritant
aniline, *o*-ethyl	$CH_3CH_2C_6H_4NH_2$	L	121.18	210		1.5590	Toxic, irritant
aniline, 2-ethoxy	$CH_3CH_2OC_6H_4NH_2$	L	137.18	231-233	1.051	1.5550	Irritant, light sensitive
aniline, 4-methyl	$CH_3C_6H_4NH_2$	L	107.16	196	0.989	1.5700	Toxic, irritant
aniline, 3-nitro	$NO_2C_6H_4NH_2$	S	138.13	114			Highly toxic, irritant
aspirin (see salicylic acid, acetate)	$CH_3CO_2C_6H_4CO_2H$	S	180.16	138-140			Irritant, toxic
benzaldehyde	C_6H_5CHO	L	106.12	179.5	1.044	1.5450	Hi.toxic, cancer susp.agent
benzaldehyde, 4-methyl	$CH_3C_6H_4CHO$	L	120.15	204-205	1.019	1.5454	Irritant (*p*-tolualdehyde)
benzaldehyde,4-methoxy	$CH_3OC_6H_4CHO$	L	136.15	248	1.119	1.5730	Irritant, (anisaldehyde)
benzaldehyde, 4-nitro	$NO_2C_6H_4CHO$	S	151.12	106			Irritant
benzene	C_6H_6	L	81.14	80.1	0.908	1.4990	Flamm., cancer susp.agent
benzene, bromo	C_6H_5Br	L	157.02	155-156	1.491	1.5590	Irritant
benzene, chloro	C_6H_5Cl	L	112.56	132	1.107	1.5240	Flammable, irritant
benzoate, ethyl	$C_6H_5CO_2C_2H_5$	L	150.18	212.6	1.051	1.5050	Irritant
benzoate, methyl	$C_6H_5CO_2CH_3$	L	136.15	198-199	1.094	1.5170	Irritant
benzocaine, 4-aminobenzoic acid, ethyl ester,	$H_2NC_6H_4CO_2C_2H_5$	S	165.19	88-92			Irritant
benzoic acid	$C_6H_5CO_2H$	S	122.12	122.4			Irritant
benzoic acid, 4-acetamido	$CH_3CONHC_6H_4CO_2H$	S	179.18	256.5			Irritant
benzoic acid, 4-amino	$H_2NC_6H_4CO_2H$	S	137.14	188-189	1.374		Irritant
benzoic acid, 3-chloro	$ClC_6H_4CO_2H$	S	156.57	158			Irritant
benzoic acid, 4-chloro	$ClC_6H_4CO_2H$	S	156.57	243			Irritant
benzoic acid, 3-hydroxy	$HOC_6H_4CO_2H$	S	138.12	210-203			Irritant
benzoic acid, 4-hydroxy	$HOC_6H_4CO_2H$	S	138.12	215-217			Irritant
benzoic acid, 2-methyl	$CH_3C_6H_4CO_2H$	S	136.15	103-105			See also *o*-toluic acid
benzoic acid, 4-methyl	$CH_3C_6H_4CO_2H$	S	136.15	180-182			See also *p*-toluic acid
benzoic acid, 4-nitro	$O_2NC_6H_4CO_2H$	S	167.12	239-241			Irritant
benzonitrile	C_6H_5CN	L	103.12	191	1.010	1.5280	Irritant
benzophenone	$(C_6H_5)_2CO$	S	182.22	49-51			Irritant
benzoyl chloride	C_6H_5COCl	L	140.57	198	1.211	1.5530	Corrosive, toxic
benzyl alcohol	$C_6H_5CH_2OH$	L	108.14	205	1.045	1.5400	Irritant, hygroscopic
benzyl amine	$C_6H_5CH_2NH_2$	L	107.16	184-185	0.981	1.5430	Corrosive, lachrymator
benzyl chloride	$C_6H_5CH_2Cl$	L	126.59	179	1.1002		Hi.toxic, cancer susp.agent
biphenyl	$C_6H_5C_6H_5$	S	154.21	69-71	0.992		Irritant
boric acid	H_3BO_3	S	61.83		1.435		Irritant, hygroscopic
Brady's Reagent	$(NO_2)_2C_6H_3NHNH_2$	L	See hydrazine, 2,4-dinitrophenyl				
bromine	Br_2	L	159.82	58.8	3.102		Highly toxic, oxidizer
butanal	$CH_3CH_2CH_2CHO$	L	72.11	75			Flammable, corrosive
1,3-butadiene, E,E-1,4-diphenyl	$C_6H_5C_4H_4C_6H_5$	S	206.29	153			Irritant
butane, 1-bromo	$CH_3CH_2CH_2CH_2Br$	L	137.03	101.3	1.276	1.4390	Flammable, irritant
butane, 2-bromo	$CH_3CH_2CHBrCH_3$	L	137.03	91.3	1.255	1.4369	Flammable, irritant

Compound Name	Chemical Formula	Solid (S) or Liquid (L)	Formula Weight	MP or BP (°C)	Density (g/mL)	Refract. Index	Hazardous Properties*
butane, 1-chloro	$CH_3CH_2CH_2CH_2Cl$	L	92.57	78.4	0.886	1.4024	Flammable liquid
butane, 2-chloro	$CH_3CH_2CHClCH_3$	L	92.57	68.2	0.873	1.3960	Flammable liquid
1-butanol	$CH_3CH_2CH_2CH_2OH$	L	74.12	117-118	0.810	1.3990	Flammable, irritant
2-butanol	$CH_3CH_2CHOHCH_3$	L	74.12	99.5-100	0.807	1.3970	Flammable, irritant
2-butanone	$CH_3CH_2COCH_3$	L	72.11	80	0.805	1.3790	Flammable, irritant
2-butanone, 3-hydroxy-3-methyl	$(CH_3)_2C(OH)COCH_3$	L	102.13	140-141	0.971	1.4150	Irritant
1-butene, 3-chloro-	$CH_3CH(Cl)CH=CH_2$	L	90.55	62-65	0.900	1.4155	Flammable, lachrymator
3-buten-2-ol	$CH_2=CHCH(OH)CH_3$	L	72.11	96-97	0.832	1.4150	Flammable, irritant
n-butyl butyrate	$C_3H_7CO_2C_4H_9$	L	144.21	164-165	0.871	1.4060	Irritant
3-butyn-2-ol, 2-methyl	$CH{\equiv}CC(CH_3)_2OH$	L	84.12	104	0.868	1.4200	Flammable, toxic
calcium carbonate	$CaCO_3$	S	100.99		2.930		Irritant, hygroscopic
calcium chloride, anhydr.	$CaCl_2$	S	110.99		2.150		Irritant, hygroscopic
camphor (1R, +)	$C_{10}H_{16}O$	S	152.24	179-181	0.990	1.5462	Flamm., irritant
carbon dioxide, solid	CO_2	S	44.01	-78.5(subl.)			Frost bite burns
carbon tetrachloride	CCl_4	L	153.82	76	1.594		Susp. Cancer agent
charcoal (Norit)		S	Decolourizing agent, used in recrystallizations				Irritant
chloroform	$CHCl_3$	L	119.38	61.3	1.500		Highly toxic
cinnamaldehyde, *trans*	$C_6H_5CH=CHCHO$	L	132.16	246(decomp)	1.048	1.6220	Irritant
cinnamic acid, trans	$C_6H_5CH=CHCO_2H$	S	148.16	135-136			Irritant
crotonaldehyde	$CH_3CH=CHCHO$	L	70.09	102.4	0.846	1.4365	Highly toxic, flammable.
Cyclohexane	C_6H_{12}	L	84.16	80.7	0.779	1.4260	Flammable, irritant
cyclohexane, bromo	$C_6H_{11}Br$	L	163.06	166.2	1.324	1.4950	Flammable, irritant
cyclohexane, methyl	$C_6H_{11}CH_3$	L	98.19	101	0.770	1.4220	Flammable, irritant
cyclohexene	C_6H_{10}	L	82.15	83	0.811	1.4460	Flammable, irritant
cyclohexanol	$C_6H_{11}OH$	L	100.16	161.1	0.963	1.4650	Irritant, hygroscopic
cyclohexanone	$C_6H_{10}(=O)$	L	98.15	155.6	0.947	1.4500	Corrosive, toxic
cyclohexanone, 4-methyl	$CH_3C_6H_9(=O)$	L	112.17	170	0.914	1.4460	Corrosive, toxic
cyclopentane	C_5H_{10}	L	70.14	49.5	0.751	1.4000	Flammable, irritant
cyclopentane, bromo	C_5H_9Br	L	149.04	137-138	1.390	1.4881	Flammable
cyclopentanone	$C_5H_8(=O)$	L	84.12	130.6	0.951	1.4370	Flammable, irritant
dichloromethane	CH_2Cl_2	L	84.93	40.1	1.325	1.4240	Toxic, irritant
diethyl ether (see ethyl ether)	$C_2H_5OC_2H_5$	L	74.12	34.6	0.708	1.3530	Flammable, toxic
1,4-dioxane	$C_4H_8O_2$	L	88.11	100-102	1.034	1.4220	Flamm., cancer susp.agent
diphenylmethanol	$(C_6H_5)_2CH(OH)$	S	184.24	65-67			Irritant
ethyl acetate	$CH_3CO_2C_2H_5$	L	88.11	76-77	0.902	1.3720	Flammable, irritant
ethyl alcohol, anhydrous	CH_3CH_2OH	L	46.07	78.5	0.785	1.3600	Flammable, poison
ethyl ether, absolute	$CH_3CH_2OCH_2CH_3$	L	74.12	34.6	0.708	1.3530	Flammable, irritant
fluorene	$C_{13}H_{10}$	S	166.22	114-116			Irritant
formaldehyde (sol'n)	$HCHO$	L	30.03	96	1.083	1.3765	suspect. Cancer agent
formamide, N,N-dimethyl	$HCON(CH_3)_2$	L	73.10	149-156	0.9487	1.4310	suspect. Cancer agent
furfuryl amine	$(C_4H_3O)CH_2NH_2$	L	97.12	145-146	1.099	1.4900	Irritant
gold	Au	S	196.97	1064	19.28		Expensive/valuable
***n*-hexane**	$CH_3(CH_2)_4CH_3$	L	86.18	69	0.659	1.3750	Flammable, irritant
hydrazine, 2,4-dinitrophenyl	$(NO_2)_2C_6H_3NHNH_2$	70% soln	198.14				Flammable, irritant
hexanes	C_6H_{14}	L	86.18	68-70	0.672	1.3790	Flammable, irritant
hydrochloric acid, conc. 12 M	HCl	L	36.46		1.20		Corrosive, highly toxic
iodine	I_2	S	253.81	133	4.930		Corrosive, highly toxic
lichen		S					Allergin
ligroin (high bp petrol. Ether)	C_6-C_7 (light naphtha)	L		60-80	0.656	1.3760	Flammable, irritant
Lucas Reagent		Solution	of hydrochloric acid/zinc chloride (from zinc dust)				Toxic, irritant
magnesium (metal)	Mg	S	24.31	651	1.75		Flammable
magnesium oxide	MgO	S	40.31		3.58		Moist. Sens., irritant
magnesium sulfate, anhydrous	$MgSO_4$	S	120.37		2.660		Hygroscopic
magnesium sulfate, 7-hydrate	$MgSO_4.7H_2O$	S	246.48		1.670		(psom salt)
manganese dioxide	MnO_2	S	86.94	535 (dec.)	5.026		Oxidizer, irritant
methanol, anhyd.	CH_3OH	L	32.04	64.5	0.791	1.3290	High. Toxic, flammable
methanol, diphenyl	$(C_6H_5)_2CH(OH)$	S	184.24	69			Irritant
methanol, triphenyl	$(C_6H_5)_3C(OH)$	S	260.34	164.2			Irritant

Compound Name	Chemical Formula	Solid (S) or Liquid (L)	Formula Weight	MP or BP (°C)	Density (g/mL)	Refract. Index	Hazardous Properties*
methylene chloride	CH_2Cl_2	L	84.93	40.1	1.325	1.4230	See dichlormethane
mineral spirits (light kerosene)	C_{12}-C_{14}	L		179-210	0.752	1.4240	Flammable, irritant
naphthalene	$C_{10}H_8$	S	128.17	80.5			Flamm., susp.cancer agent
nitric acid (conc. 15.4 M)	HNO_3	L	63.01		1.400		Corrosive, oxidizer
2-octanone	$CH_3(CH_2)_5COCH_3$	L	128.22	173	0.819	1.4150	Irritant
pentane	C_5H_{12}	L	72.15	36.1	0.626	1.3580	Flammable, irritant
2-pentanol, 4-methyl	$C_6H_{14}O$	L	102.18	132	0.802	1.4110	Irritant
3-pentanol	$C_2H_5CH(OH)C_2H_5$	L	88.15	115/749mm	0.815	1.4100	Flammable, irritant
3-penten-2-one, 4-methyl	$(CH_3)_2C=CHCOCH_3$	L	98.15	129	0.858	1.4450	Flammable, lachrymator
1-pentene, 2-methyl	C_6H_{12}	L	84.16	62	0.682	1.3920	Flammable, irritant
1-pentene, 4-methyl	C_6H_{12}	L	84.16	53-54	0.665	1.3820	Flammable, irritant
2-pentene, 2-methyl	C_6H_{12}	L	84.16	67	0.690	1.400	Flammable, irritant
2-pentene, 3-methyl	C_6H_{12}	L	84.16	69	0.698	1.4040	Flammable, irritant
2-pentene, 4-methyl	C_6H_{12}	L	84.16	57-58	0.671	1.3880	Flammable, irritant
petroleum ether, (Skelly B)	Mixt. of C_5-C_6	L		35-60	0.640		Flammable, toxic
petroleum ether, hi bp (ligroin)	Mixt. of C_6-C_7	L		60-80	0.656	1.3760	Flammable, toxic
phenethyl alcohol	$C_6H_5CH_2CH_2OH$	L	122.17	221/750mm	1.023	1.5320	Toxic, irritant
phenol	C_6H_5OH	S	94.11	40-42	1.071		Highly toxic, corrosive
phenol, 2,4-dimethyl	$(CH_3)_2C_6H_3OH$	S	122.17	22-23	1.011	1.5380	Corrosive, toxic
phenol, 2,5-dimethyl	$(CH_3)_2C_6H_3OH$	S	122.17	75-77	0.971		Corrosive, toxic
phenylacetylene	$C_6H_5C\equiv CH$	L	102.14	142-144	0.930	1.5490	Flamm., cancer susp.agent
phenylmagnesium bromide	C_6H_5MgBr	L	181.33		1.134		Flammable, moist.sensit.
phosphoric acid (85%, 14.7 M)	H_3PO_4	L	98.00		1.685		Corrosive
potassium chromate	K_2CrO_4	S	194.20	968	2.732		Canc.susp.agent, oxidizer
potassium dichromate	$K_2Cr_2O_7$	S	294.19	398			Hi.toxic, canc.susp.agent
potassium hydroxide	KOH	S	56.11				Corrosive, toxic
potassium iodide	KI	S	166.01	681	3.130		Moist.sens., irritant
potassium permanganate	$KMnO_4$	S	158.04	d<240	2.703		Oxidizer, corrosive
propane, 2-chloro, 2-methyl	$(CH_3)_3CCl$	L	92.57	50	0.851	1.3848	Flammable
propane, 2-nitro	$(CH_3)_2CHNO_2$	L	89.09	120	0.992	1.3940	Canc.susp.agent, flamm.
2-propanol, 2-methyl-	$(CH_3)_3COH$	L	74.12	82.3	0.7887		Flammable, irritant
propionate, ethyl	$C_2H_5CO_2C_2H_5$	L	102.13	99	0.891	1.3840	Flammable, irritant
propionic acid	$C_2H_5CO_2H$	L	74.08	141	0.993	1.3860	Corrosive, toxic
rosaniline hydrochloride	$C_{20}H_{14}(NH_2)_3Cl$	Solution	337.86	250 (dec)			Susp. cancer agent
salicylic acid	$HOC_6H_4CO_2H$	S	138.12	158-160			Toxic, irritant
salicylic acid, acetate ester	$CH_3CO_2C_6H_4CO_2H$	S	180.16	138-140			Irritant, toxic
Schiff's Reagent		Solution	of roseaniline hydrochloride & sulfur dioxide				Toxic
silane, tetramethyl	$Si(CH_3)_4$	L	88.23	26-28	0.648	1.3580	Flammable, hygroscopic
silica, sand	SiO_2	S	60.09	NA			abrasive
silver nitrate	$AgNO_3$	S	169.88	212	4.352		Highly toxic, oxidizer
sodium acetate	CH_3CO_2Na	S	82.03				hygroscopic
sodium bisulfite	$NaHSO_3$	S			1.480		Severe irritant
sodium borohydride	$NaBH_4$	S	37.38	400			Flam. solid, corrosive
sodium bicarbonate	$NaHCO_3$	S	84.01		2.159		Moist. sensitive
sodium carbonate	Na_2CO_3	S	105.99	851	2.532		Irritant, hygroscopic
sodium chloride	NaCl	S	58.44	801	2.165		Irritant, hygroscopic
sodium dichromate, dihydrate	$Na_2Cr_2O_7.2H_2O$	S	298.00		2.350		Hi.toxic, cancer susp.agent
sodium hydrogen carbonate	$NaHCO_3$	S	84.01		2.159		See sodium bicarbonate
sodium hydroxide	NaOH	S	40.00				Corrosive, toxic
sodium iodide	NaI	S	149.89	661	3.670		Moist.sens., irritant
sodium metabisulfite	$Na_2S_2O_5$	S	190.10		1.480		Moist.sens., toxic
sodium methoxide	$NaOCH_3$	S	54.02				Flam. solid, corrosive
sodium sulfate	Na_2SO_4	S	142.04	884	2.680		Irritant, hygroscopic
styrene	$C_6H_5CH=CH_2$	L	104.15	146	0.909		Flammable
styrene, β-bromo	$C_6H_5CH=CHBr$	L	183.05	112/20mm	1.427	1.6070	Irritant
sucrose	$C_{12}H_{22}O_{11}$	S	342.30	185-187			Tooth Decay!
sulfur dioxide	SO_2	Gas	64.06	-10 bp			Nonflamm, corrosive

Compound Name	Chemical Formula	Solid (S) or Liquid (L)	Formula Weight	MP or BP (°C)	Density (g/mL)	Refract. Index	Hazardous Properties*
sulfuric acid (conc. 18 M)	H_2SO_4	L	98.08		1.840		Corrosive, oxidizer
sulfurous acid	H_2SO_3	L	82.08		1.030		Corrosive, toxic
L-tartaric acid	$HO_2CC_2H_2(OH)_2CO_2H$	S	150.09	171-174			Irritant
tetrahydrofuran	C_4H_8O	L	72.11	65-67	0.889	1.4070	Flammable, irritant
tetramethylsilane	$Si(CH_3)_4$	L	88.23	26-28	0.648	1.3580	Flammable, hygroscopic
tin	Sn	S	118.69		7.310		Flammable solid, moist.sens.
Tollen's Reagent		L	See ammonia + silver nitrate				
toluene	$C_6H_5CH_3$	L	92.14	110.6	0.867	1.4960	Flammable, toxic
toluene, 4-nitro	$NO_2C_6H_4CH_3$	S	137.14	52-54	1.392		Hi.toxic, irritant
o- or 2-toluic acid	$CH_3C_6H_4CO_2H$	S	136.15	103-105			Probable irritant
p- or 4-toluic acid	$CH_3C_6H_4CO_2H$	S	136.15	180-182			Probable irritant
triethylphosphite	$(C_2H_5O)_3P$	L	166.16	156	0.969	1.4130	Moist. sens., irritant
triphenylmethanol	$(C_6H_5)_3C(OH)$	S	260.34	160-163			Probable irritant
urea	NH_2CONH_2	S	60.06	135	1.335		Irritant
(-) usnic acid	$C_{18}H_{16}O_7$	S	344.32	198			Toxic
(+) usnic acid	$C_{18}H_{16}O_7$	S	344.32	201-203			Toxic
water	H_2O	L	18.02	100		1.33	Will burn skin when hot
water, ice	H_2O	S/L	18.02	0	1.00		Frostbite, hypothermia
xylenes	$CH_3C_6H_4CH_3$	L	106.17	137-144	0.860	1.4970	Flammable, irritant
zinc dust	Zn	S	65.37	419.5			Flammable, moist.sens.

*Be sure to consult the chemical's MSDS for more specific detail on hazardous properties.

CHEM350 Prelab Questions (Feb. 2006)

Exp.1 Prelab Question Answers:
Q1-b; Q2-c; Q3-a; Q4-b; Q5-c; Q6-d; Q7-a; Q8-a; Q9-c; Q10-d

Exp.2 Prelab Question Answers:
Q1-d; Q2-b; Q3-a; Q4-d; Q5-c; Q6-c; Q7-b; Q8-a; Q9-d; Q10-c; Q11-a

Exp.3 Prelab Question Answers:
Q1-b; Q2-b; Q3-a; Q4-d; Q5-c; Q6-b; Q7-e; Q8-c

Exp.4 Prelab Question Answers:
Q1-a; Q2-c; Q3-a; Q4-a; Q5-a; Q6-a

Exp.5 Prelab Question Answers:
Q1-c; Q2-b; Q3-a; Q4-b; Q5-b; Q6-c; Q7-c; Q8-d

Exp.6 Prelab Question Answers:
Q1-d; Q2-c; Q3-d; Q4-d; Q5-b; Q6-a; Q7-b

Exp.7 Prelab Question Answers:
Q1-e; Q2-a; Q3-b; Q4-b; Q5-a; Q6-c

Exp.8 Prelab Question Answers:
Q1-a; Q2-b; Q3-b; Q4-c; Q5-b; Q6-e; Q7-a; Q8-b; Q9-d

Exp.8 Alternate Prelab Question Answers:
Q1-a; Q2-b; Q3-c; Q4-b; Q5-e; Q6-a; Q7-b; Q8-c

Exp.9 Prelab Question Answers:
Q1-c; Q2-b; Q3-d; Q4-c; Q5-b; Q6-c; Q7-b; Q8-d