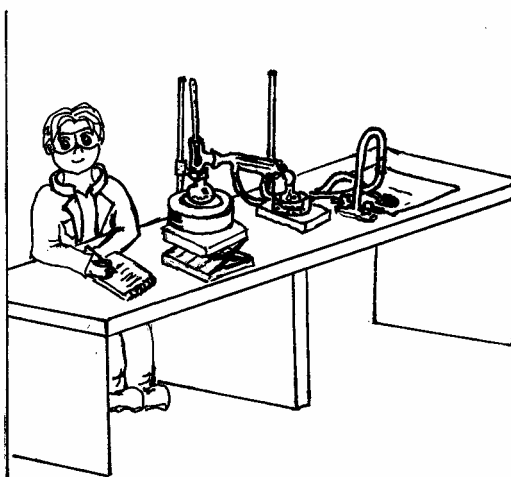


# Chemistry 350

Organic Chemistry I

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Report Book 2004-2005



## **Course team**

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# CHEM 350 Report Book Contents

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<b>General Introduction</b>	1
Report Book Structure	3
Acknowledgements	4
<b>Experiment 1</b> Melting-point determinations	5
Pre-lab Questions	5
Experiment 1 Lab Report	6-9
<b>Experiment 2</b> Recrystallization	11
Pre-lab Questions	11
Experiment 2 Lab Report	12-15
<b>Experiment 3</b> Distillation-simple and fractional	17
Pre-lab Questions	17
Experiment 3 Lab Report	18-22
<b>Experiment 4</b> Refractive index	23
Pre-lab Questions	23
Experiment 4 Lab Report	24-28
<b>Experiment 5</b> Extraction, separation, drying agents	29
Pre-lab Questions	29
Experiment 5 Lab Report	30-36
<b>Experiment 6</b> The reactions of common functional groups and infrared spectroscopy tutorial	37
Part 1: Hydrocarbons	
Pre-lab Questions	37
Experiment 6 Lab Report	38-48

## CHEM 350 Report Book Contents (cont.)

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<b>Experiment 7</b> Extraction of usnic acid	49
Pre-lab Questions	49
Experiment 7 Lab Report	50-54
<b>Experiment 8</b> Prep. of cyclohexene from cyclohexanol	55
Pre-lab Questions	55
Experiment 8 Lab Report	56-60
<b>Experiment 8 (Optional)</b> Prep. of methylpentenes	61
Pre-lab Questions	61
Experiment 8 Lab Report	62-68
<b>Experiment 9</b> The nitration of acetanilide	69
Pre-lab Questions	69
Experiment 9 Lab Report	70-74
<b>Table of Reagents</b>	75
CHEM350 Lab Course Questionnaire	79

## Welcome to Organic Chemistry 350's Laboratory Report Workbook

This report book, along with the 'Chemistry 350 Lab Manual', will help you prepare for the single weekend (~20h) of supervised lab instruction. All preparatory work in this report book (~12 h to finish, see list on page 3), must 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 Workbook to provide you with the means of completing all four steps.

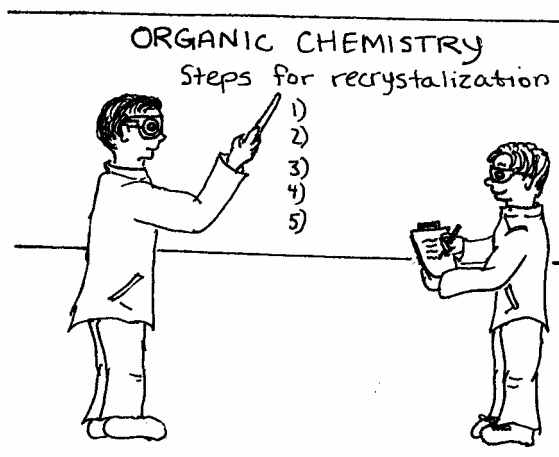
### Step 1: First we tell you what you are going to do.

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.

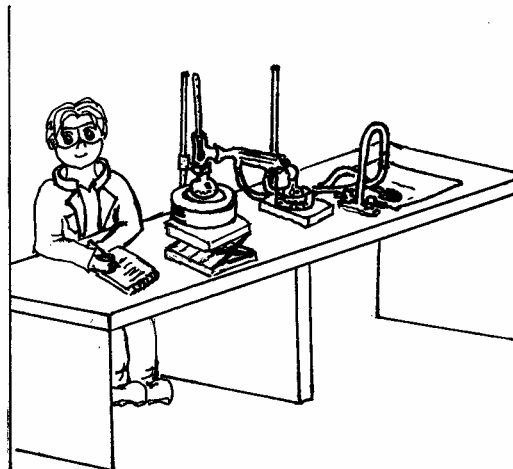
When you come to the lab, a lab instructor will give a safety orientation, followed by a series of mini lab lectures on each experiment. Various techniques will be demonstrated and you will be shown how to handle chemicals, dispose of hazardous waste, and operate the equipment.



## Introduction

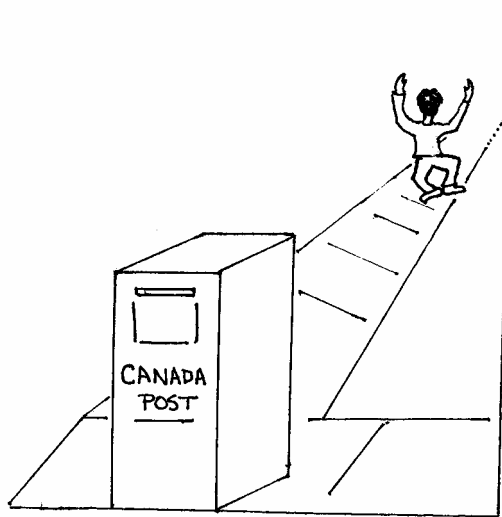
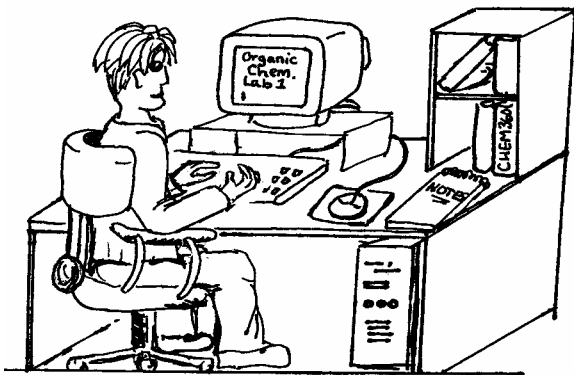
### Step 3: Lab Time: Now you do what you've read, been told, and shown.

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 be 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. Complete all the questions, and submit 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 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 and 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.

**Acknowledgements:**

The grateful authors wish to especially 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. 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.

*Laboratory Manual, Chemistry 240*, Dalhousie University, 1973.

*Laboratory Manual, Chemistry 240A/B*, Sir Wilfred Grenfell College, 1982-83.

*Laboratory Manual, Chemistry 240*, Memorial University of Newfoundland, 1976-77.

L.M. Browne, 1998. *Laboratory Manual, Chemistry 161*, University of Alberta.

L.M. Browne, 1998. *Laboratory Manual, Chemistry 163*, University of Alberta.

L.M. Browne, 1993. *Laboratory Manual, Chemistry 361*, University of Alberta.

Lehman, J.W. 1999. *Operation Organic Chemistry: A Problem-Solving Approach to the Laboratory Course*, 3<sup>rd</sup> 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*, 3<sup>rd</sup> ed., Brooks/Cole Publishing Company, Pacific Grove, CA.

Weast, R.C. *et al*, 1974. *CRC Handbook of Chemistry and Physics*, 65<sup>th</sup> 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.
  - b. To determine the purity of a sample, and its identity using the mixed melting point technique.
  - c. To identify and then determine the crystal lattice structure of a compound.
  
2. List the steps to prepare a melting point sample?
  - i)
  - ii)
  - iii)
  
3. What are three main concerns regarding 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.
  
4. Define the temperatures recorded at the beginning and end of the melting point range.
  
  
5. In a CRC Handbook of Chemistry and Physics, the melting point of a compound is sometimes reported 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.
  
6. The melting point apparatus should be heating at what rate (?°C/min) as it approaches the melting point of the compound?

**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**

<b>Procedural Step</b>	<b>Observations</b>
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 <sub>6</sub> H <sub>5</sub> COOH				Irritant
3-chlorobenzoic	ClC <sub>6</sub> H <sub>4</sub> COOH				Irritant
biphenyl	C <sub>6</sub> H <sub>5</sub> C <sub>6</sub> H <sub>5</sub>				Irritant
salicylic acid	HOC <sub>6</sub> H <sub>4</sub> COOH				Toxic, Irritant
<i>trans</i> -cinnamic acid					Irritant
2-methylbenzoic	CH <sub>3</sub> C <sub>6</sub> H <sub>5</sub> COOH				Irritant
4-nitrobenzaldehyde					Irritant
urea	NH <sub>2</sub> CONH <sub>2</sub>	60.06	133-135		Irritant
acetone (wash)	CH <sub>3</sub> COCH <sub>3</sub>			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.# \_\_\_\_\_

Structure of Unk.# _____

**Experiment 1 Questions:**

Answers to these questions should 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?
  
2. Briefly explain how recrystallization increases the purity of a compound.
  
3. What are the 5 steps of the recrystallization procedure.
  - i)
  - ii)
  - iii)
  - iv)
  - v)
  
4. What are the criteria for selecting a solvent suitable for recrystallization?
  
  
  
  
  
  
  
  
  
  
5. Boiling stones must be added to the recrystallization solvent prior to heating. Why (Note: there are two very good reasons for doing so)?
  
  
  
  
  
  
  
  
  
  
6. Give two situations where you are required to perform a hot gravity filtration?

Exp.2

CHEM350 Report Book 2004-05

**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 <sub>12</sub> H <sub>22</sub> O <sub>11</sub>			NA	
calcium carbonate	CaCO <sub>3</sub>			NA	
silica	SiO <sub>2</sub>			NA	
charcoal				NA	
water	H <sub>2</sub> O		0	100	Burns when hot
acetone (wash)	CH <sub>3</sub> COCH <sub>3</sub>			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					
2 <sup>nd</sup> 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 Questions:**

Answers to these 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.

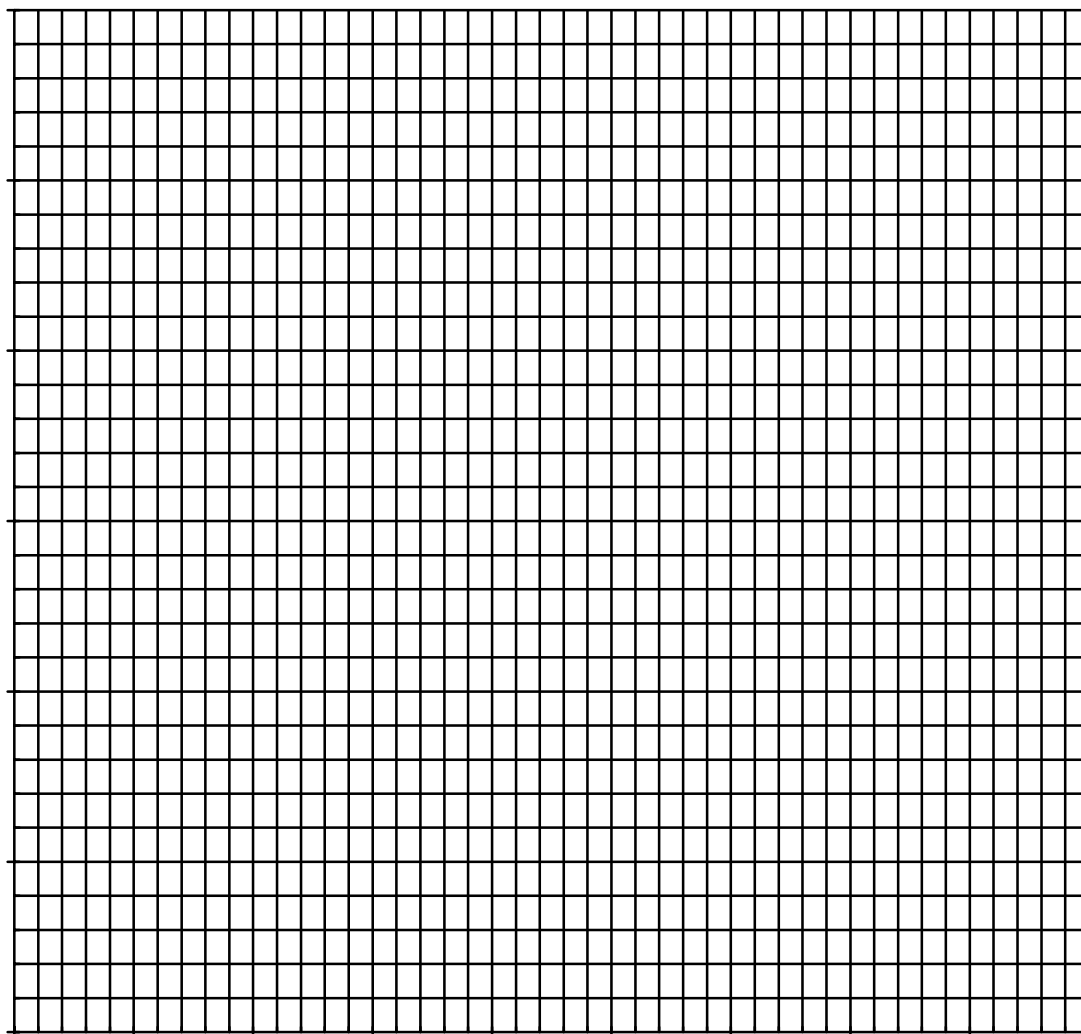
Temperature (°C)	Solubility of compound (in 100 mL of water)
0	1.5 g
20	3.0 g
40	6.5 g
60	11.0 g
80	17.0 g

- 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° 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° 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.

Exp.2

CHEM350 Report Book 2004-05

Graph paper insert





Exp.3

CHEM350 Report Book 2004-05

**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	

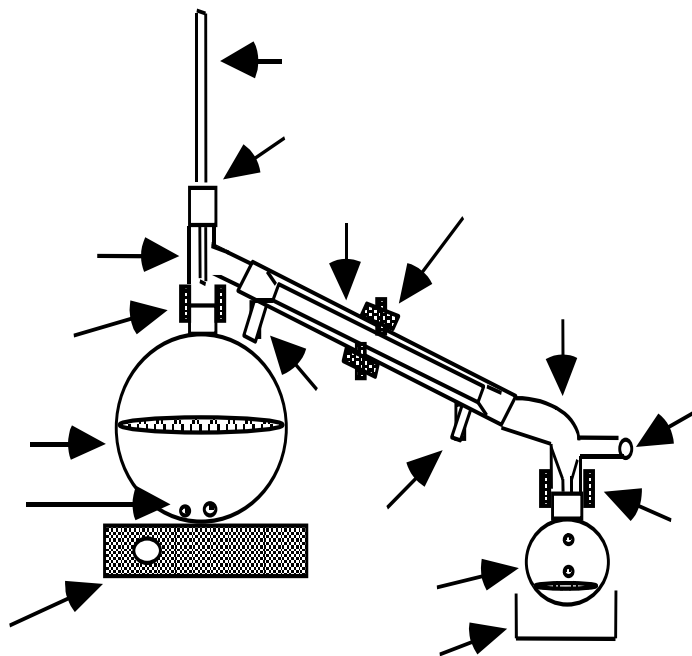
**Part B:** Fractional distillation of 50:50 mixture of cyclohexane:toluene.

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 <sub>3</sub> COCH <sub>3</sub>			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 Questions:**

Answers to these 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. Name two ways to assess the purity of a liquid organic sample.
  - i)
  
  - ii)
  
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

\_\_\_\_\_ as it passes from air into the liquid medium
  
3. The refractive index is dependent upon which two key factors?
  - i)
  
  - ii)
  
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, 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
  
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)  $= \frac{|\text{theoretical value} - \text{actual value}|}{\text{actual value}} \times 100\% =$

Exp.4

CHEM350 Report Book 2004-05

**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
cyclohexanol					
toluene					
cyclohexane					
acetone (wash)	CH <sub>3</sub> COCH <sub>3</sub>			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 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 °C (*Handbook of Chemistry and Physics*, 47<sup>th</sup> 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 milliliters 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  $K = \frac{\text{concentration of solute in solvent A, e.g., (g} \cdot \text{L}^{-1})}{\text{concentration of solute in solvent B, e.g., (g} \cdot \text{L}^{-1})}$

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 answer correct for how much compound will be extracted, if you use 50 mL of solvent A for the extraction:

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

4. Why do we add 5% NaOH to extract the organic acid from the organic mixture?
5. Why do we add 1.5 M HCl to extract the organic base from the organic mixture?

Exp.5

CHEM350 Report Book 2004-05

**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 Unknown	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 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).

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

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?
2. What does the Baeyer Test detect?
3. What does the Ammoniacal Silver Test detect?
4. If a compound gives a positive reaction in all four tests it is most likely to be a
  - a) aldehyde
  - b) alkene
  - c) alkane
  - d) alkyne
5. If the compound does not react in any of the four tests, the compound is most likely to be :
  - 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 class (True or False).
7. Which reagent used in the functional group tests must be specially handled before discarding, and why?

Exp.6

CHEM350 Report Book 2004-05

**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 Reagent					
sulfuric acid (conc.)	H <sub>2</sub> SO <sub>4</sub>				
acetone (wash)	CH <sub>3</sub> COCH <sub>3</sub>			56.5	Flammable liq., irritant

**Experiment 6 Part A Results:**

<b>Bromine Test</b>			
<b>Test Substance</b>	<b>Observation</b>	<b>Inference</b>	<b>Equation</b>
Pentane			
Cyclohexene			
Phenylacetylene			
Biphenyl			
Toluene			

<b>Baeyer Test</b>			
<b>Test Substance</b>	<b>Observation</b>	<b>Inference</b>	<b>Equation</b>
Pentane			
Cyclohexene			
Phenylacetylene			
Biphenyl			
Toluene			

<b>Ammoniacal Silver Test</b>			
<b>Test Substance</b>	<b>Observation</b>	<b>Inference</b>	<b>Equation</b>
Pentane			
Cyclohexene			
Phenylacetylene			
Biphenyl			
Toluene			

<b>Sulfuric Acid Test</b>			
<b>Test Substance</b>	<b>Observation</b>	<b>Inference</b>	<b>Equation</b>
Pentane			
Cyclohexene			
Phenylacetylene			
Biphenyl			
Toluene			

**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.

<b>Cyclohexanol</b>	Absorption Band#	Wavenumber (cm <sup>-1</sup> )	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or weak)	Functional Group Indicated

Functional Group absent:

<b>2-methyl-3-butyn-2-ol</b>	Absorption Band#	Wavenumber (cm <sup>-1</sup> )	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or weak)	Functional Group Indicated

Functional Group absent:

<b>3-buten-2-ol</b>	Absorption Band#	Wavenumber (cm <sup>-1</sup> )	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or weak)	Functional Group Indicated

Functional Group absent:

<b>benzhydrol</b>	Absorption Band#	Wavenumber (cm <sup>-1</sup> )	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or weak)	Functional Group Indicated

Functional Group absent:

## Instructor Led Group Infrared Analysis Problems (cont.)

<b>benzaldehyde</b>	Absorption Band#	Wavenumber (cm <sup>-1</sup> )	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or weak)	Functional Group Indicated

Functional Group absent:

<b>acetic acid</b>	Absorption Band#	Wavenumber (cm <sup>-1</sup> )	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or weak)	Functional Group Indicated

Functional Group absent:

<b>dibutylamine</b>	Absorption Band#	Wavenumber (cm <sup>-1</sup> )	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.

<b>cyclohexanone</b>	Absorption Band#	Wavenumber (cm <sup>-1</sup> )	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or weak)	Functional Group Indicated

Functional Group(s) absent:

<b>benzaldehyde</b>	Absorption Band#	Wavenumber (cm <sup>-1</sup> )	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or weak)	Functional Group Indicated

Functional Group(s) absent:

<b>ethyl benzoate</b>	Absorption Band#	Wavenumber (cm <sup>-1</sup> )	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or weak)	Functional Group Indicated

Functional Group(s) absent:

<b>benzoic acid</b>	Absorption Band#	Wavenumber (cm <sup>-1</sup> )	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.

<b>phenylacetylene</b>	Absorption Band#	Wavenumber (cm <sup>-1</sup> )	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or weak)	Functional Group Indicated

Functional Group(s) absent:

<b>benzonitrile</b>	Absorption Band#	Wavenumber (cm <sup>-1</sup> )	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or weak)	Functional Group Indicated

Functional Group(s) absent:

<b>styrene</b>	Absorption Band#	Wavenumber (cm <sup>-1</sup> )	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or weak)	Functional Group Indicated

Functional Group(s) absent:

<b>diethyl ether</b>	Absorption Band#	Wavenumber (cm <sup>-1</sup> )	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 ( $\text{cm}^{-1}$ )	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or weak)	Functional Group Indicated

Functional Group absent:

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

Functional Group absent:

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

Functional Group absent:

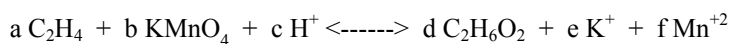
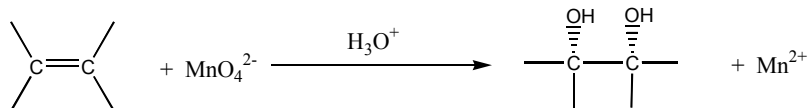
Code: Name:	Absorption Band#	Wavenumber ( $\text{cm}^{-1}$ )	Peak Shape (sharp, broad)	Peak Intensity (strong, medium or weak)	Functional Group Indicated

Functional Group absent:

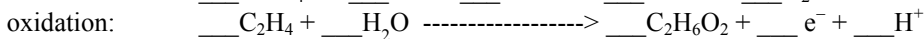
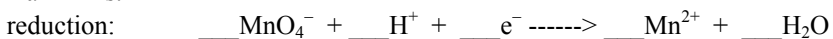
**Experiment 6 Questions:**

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.



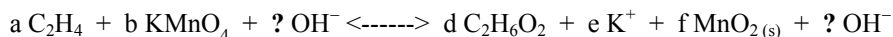
Half Rxns.



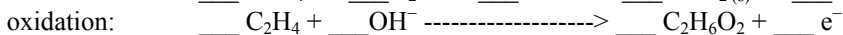
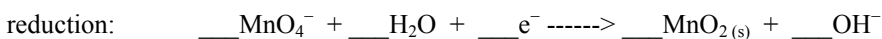
Bal. Equation:

---

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.



Half Rxns.



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**

- Which of the following compounds is optically active:
  - ultra-pure water.
  - acetone.
  - tetrahydrofuran.
  - dichloromethane.
  - none of the above.
- The measured optical activity of a solid compound is affected by three major factors. They are:
  - concentration and temperature of the solution, and length of sample tube.
  - size of the molecule, natural source of chemical, and solubility.
  - density and temperature of compound, and length of sample tube.
- 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  $\alpha$  (observed rotation) of  $+3.2^\circ$ , what would the specific rotation be?
  - $+50^\circ$
  - $+100^\circ$
  - $+200^\circ$
  - $-1000^\circ$
- During the solid-liquid extraction of the lichen with acetone, the lab manual (p.83, Part A of lab manual) suggests we extract for 30 minutes. What would happen if the extraction went longer than 30 minutes?
- Why is all the extraction solvent removed prior to beginning the recrystallization part of the procedure?

**Chem350 Experiment 7 Report**

**Date:** \_\_\_\_\_

**Student Name:** \_\_\_\_\_

**ID Number:** \_\_\_\_\_

**Title:**

**Objective(s):**

**Introduction:**



**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 ( $\alpha$ )*	Corrected Observed Rotation ( $\alpha$ -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



**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.
2. 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.
3. 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
4. How do you separate the aqueous and the cyclohexene organic layer?
5. Suggest 5 ways to characterize your final product and thereby prove that you have converted cyclohexanol to cyclohexene.
  - i)
  - ii)
  - iii)
  - iv)
  - v)
6. What is the first step called in the mechanism for an acid catalyzed dehydration.
7. State Alexander Zaitzev's rule for elimination reactions.

Exp.8

CHEM350 Report Book 2004-05

**Chem350 Experiment 8 Report**

**Date:** \_\_\_\_\_

**Student Name:** \_\_\_\_\_

**ID Number:** \_\_\_\_\_

**Title:**

**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
cyclohexanol					
phosphoric acid					
cyclohexene					
sodium chloride	NaCl				
sodium carbonate					
calcium chloride	CaCl <sub>2</sub>				
acetone (wash)	CH <sub>3</sub> COCH <sub>3</sub>			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.

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

<b>cyclohexene</b>	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 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).



**Chem350 Experiment 8 (Optional) Report Date:** \_\_\_\_\_**Student Name:** \_\_\_\_\_ **ID Number:** \_\_\_\_\_**Experiment 8 (Optional) 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) 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 'salt out' the water from the organic layer.
  - c) to preserve the product
  - d) to add water to the organic layer
5. Suggest 5 ways to characterize your final product and thereby prove that you have converted 4-methyl-2-pentanol to methylpentenes.
  - i)
  - ii)
  - iii)
  - iv)
  - v)
6. What is the first step called in the mechanism for an acid catalyzed dehydration.
7. State Alexander Zaitzev's rule for elimination reactions.

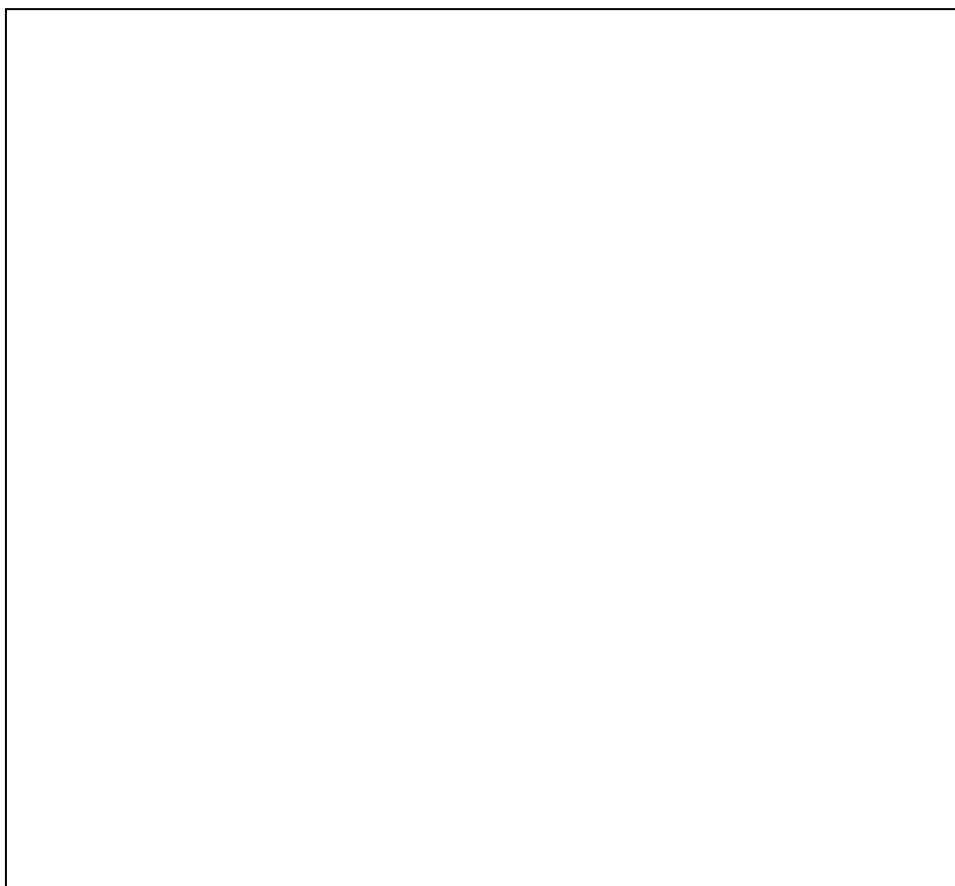
**Chem350 Experiment 8 (Optional) Report** Date: \_\_\_\_\_

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 <sub>6</sub> H <sub>14</sub> O	102.18		132	
sulfuric acid	H <sub>2</sub> SO <sub>4</sub>				
sodium hydroxide (10%)	NaOH				
sodium chloride	NaCl				
calcium chloride	CaCl <sub>2</sub>				
acetone (wash)	CH <sub>3</sub> COCH <sub>3</sub>			56.5	Flammable liquid, irritant
1-pentene, 2-methyl	C <sub>6</sub> H <sub>12</sub>	84.16		62	Flammable liquid, irritant
1-pentene, 4-methyl	C <sub>6</sub> H <sub>12</sub>	84.16		53-54	Flammable liquid, irritant
2-pentene, 2-methyl	C <sub>6</sub> H <sub>12</sub>	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.

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

<b>methylpentenes</b>	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**

<b>Component</b>	<b>%(v/v)</b>
4-methyl-1-pentene	
<i>cis</i> and <i>trans</i> -4-methyl-2-pentene	
2-methyl-1-pentene	
2-methyl-2-pentene	
<i>cis</i> and <i>trans</i> -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 (Optional) 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.



**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?
2. What happens when you mix  $\text{H}_2\text{SO}_4$  with nitric acid?
3. What is the name of the electrophile used in this experiment?
4. What acts as the nucleophile?
5. Why do you wash the product several times (Procedure Steps 8-10) with water.
6. How is the product characterized?
7. What major differences in absorption bands would you expect to see in the infrared spectra of acetanilide and *p*-nitroacetanilide?

Exp.9

CHEM350 Report Book 2004-05

**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.

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

<b><i>p</i>-nitroacetanilide</b>	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 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?

Compound Name	Chemical Formula	Solid (S) or Liquid (L)	Formula Weight	MP or BP (°C)	Density (g/mL)	Refract. Index	Hazardous Properties*
acetanilide	CH <sub>3</sub> CONHC <sub>6</sub> H <sub>5</sub>	S	135.17	113-115			Toxic, irritant
acetanilide, 4-methyl	CH <sub>3</sub> CONHC <sub>6</sub> H <sub>4</sub> CH <sub>3</sub>	S	149.19	149-151			Irritant
acetanilide, <i>p</i> -nitro	CH <sub>3</sub> CONHC <sub>6</sub> H <sub>4</sub> NO <sub>2</sub>	S	180.16	216			Irritant
acetanilide, <i>o</i> -nitro	CH <sub>3</sub> CONHC <sub>6</sub> H <sub>4</sub> NO <sub>2</sub>	S	180.16	94			Irritant
acetanilide, <i>m</i> -nitro	CH <sub>3</sub> CONHC <sub>6</sub> H <sub>4</sub> NO <sub>2</sub>	S	180.16	154-156			Irritant
acetic acid, glacial (17.4 M)	CH <sub>3</sub> CO <sub>2</sub> H	L	60.05	118.1	1.049		Corrosive, hygroscopic
acetic acid, <i>p</i> -ethoxyphenyl	C <sub>2</sub> H <sub>5</sub> OC <sub>6</sub> H <sub>4</sub> CH <sub>2</sub> CO <sub>2</sub> H	S	180.2	87-90			Irritant
acetic anhydride	(CH <sub>3</sub> CO) <sub>2</sub> O	L	102.09	140	1.082	1.3900	Corrosive, lachrymator
acetone	CH <sub>3</sub> COCH <sub>3</sub>	L	58.08	56.5	0.7899	1.3590	Flammable, irritant
acetone, diethylamino	(C <sub>2</sub> H <sub>5</sub> ) <sub>2</sub> NCH <sub>2</sub> COCH <sub>3</sub>	L	129.2	64/16mm	0.832	1.4250	Irritant
acetophenone	C <sub>6</sub> H <sub>5</sub> COCH <sub>3</sub>	L	120.15	202	1.030	1.5325	Irritant
activated carbon		S					(see charcoal)
allyl alcohol (2-propen-1-ol)	CH <sub>2</sub> =CHCH <sub>2</sub> OH	L	58.08	96-98	0.854	1.4120	Highly Toxic, flammable
ammonia (14.8 M)	NH <sub>3</sub>	L	17.03		0.90		Corrosive, lachrymator
ammonium hydroxide (14.8 M)	NH <sub>4</sub> OH	L	35.05		0.90		Corrosive, lachrymator
aniline	C <sub>6</sub> H <sub>5</sub> NH <sub>2</sub>	L	93.13	184	1.022	1.5860	Highly toxic, irritant
aniline, 4-bromo	BrC <sub>6</sub> H <sub>4</sub> NH <sub>2</sub>	S	172.03	62-64			Toxic, irritant
aniline, 4-chloro	ClC <sub>6</sub> H <sub>4</sub> NH <sub>2</sub>	S	127.57	72.5			Highly toxic, irritant
aniline, <i>o</i> -ethyl	CH <sub>3</sub> CH <sub>2</sub> C <sub>6</sub> H <sub>4</sub> NH <sub>2</sub>	L	121.18	210		1.5590	Toxic, irritant
aniline, 2-ethoxy	CH <sub>3</sub> CH <sub>2</sub> OC <sub>6</sub> H <sub>4</sub> NH <sub>2</sub>	L	137.18	231-233	1.051	1.5550	Irritant, light sensitive
aniline, 4-methyl	CH <sub>3</sub> C <sub>6</sub> H <sub>4</sub> NH <sub>2</sub>	L	107.16	196	0.989	1.5700	Toxic, irritant
aniline, 3-nitro	NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> NH <sub>2</sub>	S	138.13	114			Highly toxic, irritant
aspirin (see salicylic acid, acetate)	CH <sub>3</sub> CO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> CO <sub>2</sub> H	S	180.16	138-140			Irritant, toxic
<b>benzaldehyde</b>	C <sub>6</sub> H <sub>5</sub> CHO	L	106.12	179.5	1.044	1.5450	Hi.toxic, cancer susp.agent
benzaldehyde, 4-methyl	CH <sub>3</sub> C <sub>6</sub> H <sub>4</sub> CHO	L	120.15	204-205	1.019	1.5454	Irritant ( <i>p</i> -tolualdehyde)
benzaldehyde, 4-methoxy	CH <sub>3</sub> OC <sub>6</sub> H <sub>4</sub> CHO	L	136.15	248	1.119	1.5730	Irritant, (anisaldehyde)
benzaldehyde, 4-nitro	NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> CHO	S	151.12	106			Irritant
benzene	C <sub>6</sub> H <sub>6</sub>	L	81.14	80.1	0.908	1.4990	Flamm., cancer susp.agent
benzene, bromo	C <sub>6</sub> H <sub>5</sub> Br	L	157.02	155-156	1.491	1.5590	Irritant
benzene, chloro	C <sub>6</sub> H <sub>5</sub> Cl	L	112.56	132	1.107	1.5240	Flammable, irritant
benzoate, ethyl	C <sub>6</sub> H <sub>5</sub> CO <sub>2</sub> C <sub>2</sub> H <sub>5</sub>	L	150.18	212.6	1.051	1.5050	Irritant
benzoate, methyl	C <sub>6</sub> H <sub>5</sub> CO <sub>2</sub> CH <sub>3</sub>	L	136.15	198-199	1.094	1.5170	Irritant
benzocaine, 4-aminobenzoic acid, ethyl ester,	H <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> CO <sub>2</sub> C <sub>2</sub> H <sub>5</sub>	S	165.19	88-92			Irritant
benzoic acid	C <sub>6</sub> H <sub>5</sub> CO <sub>2</sub> H	S	122.12	122.4			Irritant
benzoic acid, 4-acetamido	CH <sub>3</sub> CONHC <sub>6</sub> H <sub>4</sub> CO <sub>2</sub> H	S	179.18	256.5			Irritant
benzoic acid, 4-amino	H <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> CO <sub>2</sub> H	S	137.14	188-189	1.374		Irritant
benzoic acid, 3-chloro	ClC <sub>6</sub> H <sub>4</sub> CO <sub>2</sub> H	S	156.57	158			Irritant
benzoic acid, 4-chloro	ClC <sub>6</sub> H <sub>4</sub> CO <sub>2</sub> H	S	156.57	243			Irritant
benzoic acid, 3-hydroxy	HOC <sub>6</sub> H <sub>4</sub> CO <sub>2</sub> H	S	138.12	210-203			Irritant
benzoic acid, 4-hydroxy	HOC <sub>6</sub> H <sub>4</sub> CO <sub>2</sub> H	S	138.12	215-217			Irritant
benzoic acid, 2-methyl	CH <sub>3</sub> C <sub>6</sub> H <sub>4</sub> CO <sub>2</sub> H	S	136.15	103-105			See also <i>o</i> -toluic acid
benzoic acid, 4-methyl	CH <sub>3</sub> C <sub>6</sub> H <sub>4</sub> CO <sub>2</sub> H	S	136.15	180-182			See also <i>p</i> -toluic acid
benzoic acid, 4-nitro	O <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> CO <sub>2</sub> H	S	167.12	239-241			Irritant
benzonitrile	C <sub>6</sub> H <sub>5</sub> CN	L	103.12	191	1.010	1.5280	Irritant
benzophenone	(C <sub>6</sub> H <sub>5</sub> ) <sub>2</sub> CO	S	182.22	49-51			Irritant
benzoyl chloride	C <sub>6</sub> H <sub>5</sub> COCl	L	140.57	198	1.211	1.5530	Corrosive, toxic
benzyl alcohol	C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub> OH	L	108.14	205	1.045	1.5400	Irritant, hygroscopic
benzyl amine	C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub> NH <sub>2</sub>	L	107.16	184-185	0.981	1.5430	Corrosive, lachrymator
benzyl chloride	C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub> Cl	L	126.59	179	1.1002		Hi.toxic, cancer susp.agent
biphenyl	C <sub>6</sub> H <sub>5</sub> C <sub>6</sub> H <sub>5</sub>	S	154.21	69-71	0.992		Irritant
boric acid	H <sub>3</sub> BO <sub>3</sub>	S	61.83		1.435		Irritant, hygroscopic
Brady's Reagent	(NO <sub>2</sub> ) <sub>2</sub> C <sub>6</sub> H <sub>3</sub> NHNH <sub>2</sub>	L		See hydrazine, 2,4-dinitrophenyl			
bromine	Br <sub>2</sub>	L	159.82	58.8	3.102		Highly toxic, oxidizer
butanal	CH <sub>3</sub> CH <sub>2</sub> CH <sub>2</sub> CHO	L	72.11	75			Flammable, corrosive
1,3-butadiene, E,E-1,4-diphenyl	C <sub>6</sub> H <sub>5</sub> C <sub>4</sub> H <sub>4</sub> C <sub>6</sub> H <sub>5</sub>	S	206.29	153			Irritant
butane, 1-bromo	CH <sub>3</sub> CH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> Br	L	137.03	101.3	1.276	1.4390	Flammable, irritant
butane, 2-bromo	CH <sub>3</sub> CH <sub>2</sub> CHBrCH <sub>3</sub>	L	137.03	91.3	1.255	1.4369	Flammable, irritant

Table of Reagents

CHEM350 Report Book 2004-05

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 <sub>3</sub> CH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> Cl	L	92.57	78.4	0.886	1.4024	Flammable liquid	
butane, 2-chloro	CH <sub>3</sub> CH <sub>2</sub> CHClCH <sub>3</sub>	L	92.57	68.2	0.873	1.3960	Flammable liquid	
1-butanol	CH <sub>3</sub> CH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> OH	L	74.12	117-118	0.810	1.3990	Flammable, irritant	
2-butanol	CH <sub>3</sub> CH <sub>2</sub> CHOHCH <sub>3</sub>	L	74.12	99.5-100	0.807	1.3970	Flammable, irritant	
2-butanone	CH <sub>3</sub> CH <sub>2</sub> COCH <sub>3</sub>	L	72.11	80	0.805	1.3790	Flammable, irritant	
2-butanone, 3-hydroxy-3-methyl	(CH <sub>3</sub> ) <sub>2</sub> C(OH)COCH <sub>3</sub>	L	102.13	140-141	0.971	1.4150	Irritant	
1-butene, 3-chloro-	CH <sub>3</sub> CH(Cl)CH=CH <sub>2</sub>	L	90.55	62-65	0.900	1.4155	Flammable, lachrymator	
3-buten-2-ol	CH <sub>2</sub> =CHCH(OH)CH <sub>3</sub>	L	72.11	96-97	0.832	1.4150	Flammable, irritant	
<i>n</i> -butyl butyrate	C <sub>3</sub> H <sub>7</sub> CO <sub>2</sub> C <sub>4</sub> H <sub>9</sub>	L	144.21	164-165	0.871	1.4060	Irritant	
3-buten-2-ol, 2-methyl	CH=CC(CH <sub>3</sub> ) <sub>2</sub> OH	L	84.12	104	0.868	1.4200	Flammable, toxic	
<b>calcium carbonate</b>	CaCO <sub>3</sub>	S	100.99		2.930		Irritant, hygroscopic	
calcium chloride, anhydr.	CaCl <sub>2</sub>	S	110.99		2.150		Irritant, hygroscopic	
camphor (1R, +)	C <sub>10</sub> H <sub>16</sub> O	S	152.24	179-181	0.990	1.5462	Flamm., irritant	
carbon dioxide, solid	CO <sub>2</sub>	S	44.01	-78.5(subl.)			Frost bite burns	
carbon tetrachloride	CCl <sub>4</sub>	L	153.82	76	1.594		Susp. Cancer agent	
charcoal (Norit)		S	Decolourizing agent, used in recrystallizations					Irritant
chloroform	CHCl <sub>3</sub>	L	119.38	61.3	1.500		Highly toxic	
cinnamaldehyde, <i>trans</i>	C <sub>6</sub> H <sub>5</sub> CH=CHCHO	L	132.16	246(decomp)	1.048	1.6220	Irritant	
cinnamic acid, <i>trans</i>	C <sub>6</sub> H <sub>5</sub> CH=CHCO <sub>2</sub> H	S	148.16	135-136			Irritant	
crotonaldehyde	CH <sub>3</sub> CH=CHCHO	L	70.09	102.4	0.846	1.4365	Highly toxic, flammable.	
Cyclohexane	C <sub>6</sub> H <sub>12</sub>	L	84.16	80.7	0.779	1.4260	Flammable, irritant	
cyclohexane, bromo	C <sub>6</sub> H <sub>11</sub> Br	L	163.06	166.2	1.324	1.4950	Flammable, irritant	
cyclohexane, methyl	C <sub>6</sub> H <sub>11</sub> CH <sub>3</sub>	L	98.19	101	0.770	1.4220	Flammable, irritant	
cyclohexene	C <sub>6</sub> H <sub>10</sub>	L	82.15	83	0.811	1.4460	Flammable, irritant	
cyclohexanol	C <sub>6</sub> H <sub>11</sub> OH	L	100.16	161.1	0.963	1.4650	Irritant, hygroscopic	
cyclohexanone	C <sub>6</sub> H <sub>10</sub> (=O)	L	98.15	155.6	0.947	1.4500	Corrosive, toxic	
cyclohexanone, 4-methyl	CH <sub>3</sub> C <sub>6</sub> H <sub>9</sub> (=O)	L	112.17	170	0.914	1.4460	Corrosive, toxic	
cyclopentane	C <sub>5</sub> H <sub>10</sub>	L	70.14	49.5	0.751	1.4000	Flammable, irritant	
cyclopentane, bromo	C <sub>5</sub> H <sub>9</sub> Br	L	149.04	137-138	1.390	1.4881	Flammable	
cyclopentanone	C <sub>5</sub> H <sub>8</sub> (=O)	L	84.12	130.6	0.951	1.4370	Flammable, irritant	
<b>dichloromethane</b>	CH <sub>2</sub> Cl <sub>2</sub>	L	84.93	40.1	1.325	1.4240	Toxic, irritant	
diethyl ether (see ethyl ether)	C <sub>2</sub> H <sub>5</sub> OC <sub>2</sub> H <sub>5</sub>	L	74.12	34.6	0.708	1.3530	Flammable, toxic	
1,4-dioxane	C <sub>4</sub> H <sub>8</sub> O <sub>2</sub>	L	88.11	100-102	1.034	1.4220	Flamm., cancer susp.agent	
diphenylmethanol	(C <sub>6</sub> H <sub>5</sub> ) <sub>2</sub> CH(OH)	S	184.24	65-67			Irritant	
<b>ethyl acetate</b>	CH <sub>3</sub> CO <sub>2</sub> C <sub>2</sub> H <sub>5</sub>	L	88.11	76-77	0.902	1.3720	Flammable, irritant	
ethyl alcohol, anhydrous	CH <sub>3</sub> CH <sub>2</sub> OH	L	46.07	78.5	0.785	1.3600	Flammable, poison	
ethyl ether, absolute	CH <sub>3</sub> CH <sub>2</sub> OCH <sub>2</sub> CH <sub>3</sub>	L	74.12	34.6	0.708	1.3530	Flammable, irritant	
<b>fluorene</b>	C <sub>13</sub> H <sub>10</sub>	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 <sub>3</sub> ) <sub>2</sub>	L	73.10	149-156	0.9487	1.4310	suspect. Cancer agent	
furfuryl amine	(C <sub>4</sub> H <sub>3</sub> O)CH <sub>2</sub> NH <sub>2</sub>	L	97.12	145-146	1.099	1.4900	Irritant	
<b>gold</b>	Au	S	196.97	1064	19.28		Expensive/valuable	
<b><i>n</i>-hexane</b>	CH <sub>3</sub> (CH <sub>2</sub> ) <sub>4</sub> CH <sub>3</sub>	L	86.18	69	0.659	1.3750	Flammable, irritant	
hydrazine, 2,4-dinitrophenyl	(NO <sub>2</sub> ) <sub>2</sub> C <sub>6</sub> H <sub>3</sub> NHNH <sub>2</sub>	70% soln	198.14				Flammable, irritant	
hexanes	C <sub>6</sub> H <sub>14</sub>	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	
<b>iodine</b>	I <sub>2</sub>	S	253.81	133	4.930		Corrosive, highly toxic	
lichen		S					Allergen	
ligroin (high bp petrol. Ether)	C <sub>6</sub> -C <sub>7</sub> (light naphtha)	L		60-80	0.656	1.3760	Flammable, irritant	
Lucas Reagent		Solution	of hydrochloric acid/zinc chloride (from zinc dust)					Toxic, irritant
<b>magnesium (metal)</b>	Mg	S	24.31	651	1.75		Flammable	
magnesium oxide	MgO	S	40.31		3.58		Moist. Sens., irritant	
magnesium sulfate, anhydrous	MgSO <sub>4</sub>	S	120.37		2.660		Hygroscopic	
magnesium sulfate, 7-hydrate	MgSO <sub>4</sub> ·7H <sub>2</sub> O	S	246.48		1.670		(□psom salt)	
manganese dioxide	MnO <sub>2</sub>	S	86.94	535 (dec.)	5.026		Oxidizer, irritant	
methanol, anhyd.	CH <sub>3</sub> OH	L	32.04	64.5	0.791	1.3290	High. Toxic, flammable	
methanol, diphenyl	(C <sub>6</sub> H <sub>5</sub> ) <sub>2</sub> CH(OH)	S	184.24	69			Irritant	
methanol, triphenyl	(C <sub>6</sub> H <sub>5</sub> ) <sub>3</sub> C(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 <sub>2</sub> Cl <sub>2</sub>	L	84.93	40.1	1.325	1.4230	See dichlormethane
mineral spirits (light kerosene)	C <sub>12</sub> -C <sub>14</sub>	L		179-210	0.752	1.4240	Flammable, irritant
<b>naphthalene</b>	C <sub>10</sub> H <sub>8</sub>	S	128.17	80.5			Flamm., susp.cancer agent
nitric acid (conc. 15.4 M)	HNO <sub>3</sub>	L	63.01		1.400		Corrosive, oxidizer
<b>2-octanone</b>	CH <sub>3</sub> (CH <sub>2</sub> ) <sub>5</sub> COCH <sub>3</sub>	L	128.22	173	0.819	1.4150	Irritant
<b>pentane</b>	C <sub>5</sub> H <sub>12</sub>	L	72.15	36.1	0.626	1.3580	Flammable, irritant
2-pentanol, 4-methyl	C <sub>6</sub> H <sub>14</sub> O	L	102.18	132	0.802	1.4110	Irritant
3-pentanol	C <sub>2</sub> H <sub>5</sub> CH(OH)C <sub>2</sub> H <sub>5</sub>	L	88.15	115/749mm	0.815	1.4100	Flammable, irritant
3-penten-2-one, 4-methyl	(CH <sub>3</sub> ) <sub>2</sub> C=CHCOCH <sub>3</sub>	L	98.15	129	0.858	1.4450	Flammable, lachrymator
1-pentene, 2-methyl	C <sub>6</sub> H <sub>12</sub>	L	84.16	62	0.682	1.3920	Flammable, irritant
1-pentene, 4-methyl	C <sub>6</sub> H <sub>12</sub>	L	84.16	53-54	0.665	1.3820	Flammable, irritant
2-pentene, 2-methyl	C <sub>6</sub> H <sub>12</sub>	L	84.16	67	0.690	1.400	Flammable, irritant
2-pentene, 3-methyl	C <sub>6</sub> H <sub>12</sub>	L	84.16	69	0.698	1.4040	Flammable, irritant
2-pentene, 4-methyl	C <sub>6</sub> H <sub>12</sub>	L	84.16	57-58	0.671	1.3880	Flammable, irritant
petroleum ether, (Skelly B)	Mixt. of C <sub>5</sub> -C <sub>6</sub>	L		35-60	0.640		Flammable, toxic
petroleum ether, hi bp (ligroin)	Mixt. of C <sub>6</sub> -C <sub>7</sub>	L		60-80	0.656	1.3760	Flammable, toxic
phenethyl alcohol	C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub> CH <sub>2</sub> OH	L	122.17	221/750mm	1.023	1.5320	Toxic, irritant
phenol	C <sub>6</sub> H <sub>5</sub> OH	S	94.11	40-42	1.071		Highly toxic, corrosive
phenol, 2,4-dimethyl	(CH <sub>3</sub> ) <sub>2</sub> C <sub>6</sub> H <sub>3</sub> OH	S	122.17	22-23	1.011	1.5380	Corrosive, toxic
phenol, 2,5-dimethyl	(CH <sub>3</sub> ) <sub>2</sub> C <sub>6</sub> H <sub>3</sub> OH	S	122.17	75-77	0.971		Corrosive, toxic
phenylacetylene	C <sub>6</sub> H <sub>5</sub> C≡CH	L	102.14	142-144	0.930	1.5490	Flamm., cancer susp.agent
phenylmagnesium bromide	C <sub>6</sub> H <sub>5</sub> MgBr	L	181.33		1.134		Flammable, moist.sensit.
phosphoric acid (85%, 14.7 M)	H <sub>3</sub> PO <sub>4</sub>	L	98.00		1.685		Corrosive
potassium chromate	K <sub>2</sub> CrO <sub>4</sub>	S	194.20	968	2.732		Canc.susp.agent, oxidizer
potassium dichromate	K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub>	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 <sub>4</sub>	S	158.04	d<240	2.703		Oxidizer, corrosive
propane, 2-chloro, 2-methyl	(CH <sub>3</sub> ) <sub>2</sub> CCl	L	92.57	50	0.851	1.3848	Flammable
propane, 2-nitro	(CH <sub>3</sub> ) <sub>2</sub> CHNO <sub>2</sub>	L	89.09	120	0.992	1.3940	Canc.susp.agent, flamm.
2-propanol, 2-methyl-	(CH <sub>3</sub> ) <sub>2</sub> COH	L	74.12	82.3	0.7887		Flammable, irritant
propionate, ethyl	C <sub>2</sub> H <sub>5</sub> CO <sub>2</sub> C <sub>2</sub> H <sub>5</sub>	L	102.13	99	0.891	1.3840	Flammable, irritant
propionic acid	C <sub>2</sub> H <sub>5</sub> CO <sub>2</sub> H	L	74.08	141	0.993	1.3860	Corrosive, toxic
<b>rosaniline hydrochloride</b>	C <sub>20</sub> H <sub>14</sub> (NH <sub>2</sub> ) <sub>3</sub> Cl	Solution	337.86	250 (dec)			Susp. cancer agent
<b>salicylic acid</b>	HOC <sub>6</sub> H <sub>4</sub> CO <sub>2</sub> H	S	138.12	158-160			Toxic, irritant
salicylic acid, acetate ester	CH <sub>3</sub> CO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> CO <sub>2</sub> H	S	180.16	138-140			Irritant, toxic
Schiff's Reagent		Solution	of roseaniline hydrochloride & sulfur dioxide				Toxic
silane, tetramethyl	Si(CH <sub>3</sub> ) <sub>4</sub>	L	88.23	26-28	0.648	1.3580	Flammable, hygroscopic
silica, sand	SiO <sub>2</sub>	S	60.09	NA			abrasive
silver nitrate	AgNO <sub>3</sub>	S	169.88	212	4.352		Highly toxic, oxidizer
sodium acetate	CH <sub>3</sub> CO <sub>2</sub> Na	S	82.03				hygroscopic
sodium bisulfite	NaHSO <sub>3</sub>	S			1.480		Severe irritant
sodium borohydride	NaBH <sub>4</sub>	S	37.38	400			Flam. solid, corrosive
sodium bicarbonate	NaHCO <sub>3</sub>	S	84.01		2.159		Moist. sensitive
sodium carbonate	Na <sub>2</sub> CO <sub>3</sub>	S	105.99	851	2.532		Irritant, hygroscopic
sodium chloride	NaCl	S	58.44	801	2.165		Irritant, hygroscopic
sodium dichromate, dihydrate	Na <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> ·2H <sub>2</sub> O	S	298.00		2.350		Hi.toxic, cancer susp.agent
sodium hydrogen carbonate	NaHCO <sub>3</sub>	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 <sub>2</sub> S <sub>2</sub> O <sub>5</sub>	S	190.10		1.480		Moist.sens., toxic
sodium methoxide	NaOCH <sub>3</sub>	S	54.02				Flam. solid, corrosive
sodium sulfate	Na <sub>2</sub> SO <sub>4</sub>	S	142.04	884	2.680		Irritant, hygroscopic
styrene	C <sub>6</sub> H <sub>5</sub> CH=CH <sub>2</sub>	L	104.15	146	0.909		Flammable
styrene, β-bromo	C <sub>6</sub> H <sub>5</sub> CH=CHBr	L	183.05	112/20mm	1.427	1.6070	Irritant
sucrose	C <sub>12</sub> H <sub>22</sub> O <sub>11</sub>	S	342.30	185-187			Tooth Decay!
sulfur dioxide	SO <sub>2</sub>	Gas	64.06	-10 bp			Nonflamm, corrosive

## Table of Reagents

## CHEM350 Report Book 2004-05

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 <sub>2</sub> SO <sub>4</sub>	L	98.08		1.840		Corrosive, oxidizer	
sulfurous acid	H <sub>2</sub> SO <sub>3</sub>	L	82.08		1.030		Corrosive, toxic	
<b>L-tartaric acid</b>	HO <sub>2</sub> CC <sub>2</sub> H <sub>2</sub> (OH) <sub>2</sub> CO <sub>2</sub> H	S	150.09	171-174			Irritant	
tetrahydrofuran	C <sub>4</sub> H <sub>8</sub> O	L	72.11	65-67	0.889	1.4070	Flammable, irritant	
tetramethylsilane	Si(CH <sub>3</sub> ) <sub>4</sub>	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 <sub>6</sub> H <sub>5</sub> CH <sub>3</sub>	L	92.14	110.6	0.867	1.4960	Flammable, toxic	
toluene, 4-nitro	NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> CH <sub>3</sub>	S	137.14	52-54	1.392		Hi.toxic, irritant	
<i>o</i> - or 2-toluic acid	CH <sub>3</sub> C <sub>6</sub> H <sub>4</sub> CO <sub>2</sub> H	S	136.15	103-105			Probable irritant	
<i>p</i> - or 4-toluic acid	CH <sub>3</sub> C <sub>6</sub> H <sub>4</sub> CO <sub>2</sub> H	S	136.15	180-182			Probable irritant	
triethylphosphite	(C <sub>2</sub> H <sub>5</sub> O) <sub>3</sub> P	L	166.16	156	0.969	1.4130	Moist. sens., irritant	
triphenylmethanol	(C <sub>6</sub> H <sub>5</sub> ) <sub>3</sub> C(OH)	S	260.34	160-163			Probable irritant	
<b>urea</b>	NH <sub>2</sub> CONH <sub>2</sub>	S	60.06	135	1.335		Irritant	
(-) usnic acid	C <sub>18</sub> H <sub>16</sub> O <sub>7</sub>	S	344.32	198			Toxic	
(+) usnic acid	C <sub>18</sub> H <sub>16</sub> O <sub>7</sub>	S	344.32	201-203			Toxic	
<b>water</b>	H <sub>2</sub> O	L	18.02	100		1.33	Will burn skin when hot	
water, ice	H <sub>2</sub> O	S/L	18.02	0	1.00		Frostbite, hypothermia	
xylenes	CH <sub>3</sub> C <sub>6</sub> H <sub>4</sub> CH <sub>3</sub>	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.

**Organic Chemistry I, Chemistry 350 Laboratory Course Questionnaire**

**Lab Session Type (circle):**      **Weekend or Weeklong**

The Centre for Natural and Human Science would appreciate your confidential help in improving our laboratory program. Please complete the following questionnaire by circling one of the following responses on a scale of 1-5:

Strongly agree (5), Agree (4), Neutral (3), Disagree (2), Strongly Disagree (1)

Submit the completed questionnaire by mail to the Science Lab Coordinator at: Centre for Science, 1University Drive, Athabasca University, Athabasca, AB, T9S 3A3.

**Please rate the Lab Instructor(s) in the items specified below:**

1. The lab instructor was competent and made the experiments interesting and instructional.  
Strongly agree (5), Agree (4), Neutral (3), Disagree (2), Strongly Disagree (1)
2. The instructor presented an overview of the laboratory exercise in an interesting and helpful manner, pointing out likely problems in the lab procedure at the start of the experiment and stressing safety precautions.  
Strongly agree (5), Agree (4), Neutral (3), Disagree (2), Strongly Disagree (1)
3. The lab instructor speaks clearly and understandably.  
Strongly agree (5), Agree (4), Neutral (3), Disagree (2), Strongly Disagree (1)
4. The lab instructor circulates through the lab during the experiments and actively monitors our work.  
Strongly agree (5), Agree (4), Neutral (3), Disagree (2), Strongly Disagree (1)
5. The instructor treated me with respect.  
Strongly agree (5), Agree (4), Neutral (3), Disagree (2), Strongly Disagree (1)
6. Overall, my instructor was effective.  
Strongly agree (5), Agree (4), Neutral (3), Disagree (2), Strongly Disagree (1)

**Please rate the Lab Course in the items specified below by circling the appropriate number or letter:**

7. The Chem350 Laboratory Manual was easy to read and understandable.  
Strongly agree (5), Agree (4), Neutral (3), Disagree (2), Strongly Disagree (1)
8. The Chem350 Laboratory Report Book was easy to read and understandable.  
Strongly agree (5), Agree (4), Neutral (3), Disagree (2), Strongly Disagree (1)
9. Working on the procedure and table of reagents in the Chem350 Report Book before coming to the lab helped me understand what to do in the laboratory and what the dangers are in each experiment.  
Strongly agree (5), Agree (4), Neutral (3), Disagree (2), Strongly Disagree (1)
10. The information required to answer the pre-lab questions in the Chem350 Report Book was easy to find.  
Strongly agree (5), Agree (4), Neutral (3), Disagree (2), Strongly Disagree (1)
11. I found the whole Chem350 weekend lab session very rushed and poorly organized.  
Strongly agree (5), Agree (4), Neutral (3), Disagree (2), Strongly Disagree (1)
12. Recording my results was made easy by the prepared/formatted tables in the results section.  
Strongly agree (5), Agree (4), Neutral (3), Disagree (2), Strongly Disagree (1)

**Organic Chemistry I Chemistry 350 Laboratory Course Questionnaire (cont.)**

13. Which experiment did you enjoy the most?
- Melting Point Determination
  - Recrystallization
  - Distillation and Refractive Index
  - Organic Acid/Base Separations
  - Cyclohexene from cyclohexanol, or Methylpentenes
  - Extraction of Usnic Acid
  - Nitration of Acetanilide
14. Which experiment did you like the least?
- Melting Point Determination
  - Recrystallization
  - Distillation and Refractive Index
  - Organic Acid/Base Separations
  - Cyclohexene from cyclohexanol, or Methylpentenes
  - Extraction of Usnic Acid
  - Nitration of Acetanilide
15. From which experiment did you learn the most?
- Melting Point Determination
  - Recrystallization
  - Distillation and Refractive Index
  - Separations
  - Cyclohexene from cyclohexanol, or Methylpentenes
  - Usnic Acid
  - Nitration of Acetanilide
16. From which experiment did you learn the least?
- Melting Point Determination
  - Recrystallization
  - Distillation and Refractive Index
  - Separations
  - Cyclohexene from cyclohexanol, or Methylpentenes
  - Usnic Acid
  - Nitration of Acetanilide
17. Working on the experiment with a lab partner improved my understanding of the experiment and overall knowledge of organic chemistry.  
Strongly agree (5), Agree (4), Neutral (3), Disagree (2), Strongly Disagree (1)
18. I had many questions that went unanswered while I was in the lab.  
Strongly agree (1), Agree (2), Neutral (3), Disagree (4), Strongly Disagree (5)
19. I would recommend to a friend to take Athabasca University's organic chemistry course.  
Strongly agree (5), Agree (4), Neutral (3), Disagree (2), Strongly Disagree (1)
20. Overall, the laboratory component is a valuable part of AU's organic chemistry course.  
Strongly agree (5), Agree (4), Neutral (3), Disagree (2), Strongly Disagree (1)

Do you have any other comments to add about your instructor(s) or this Chem350 laboratory course?