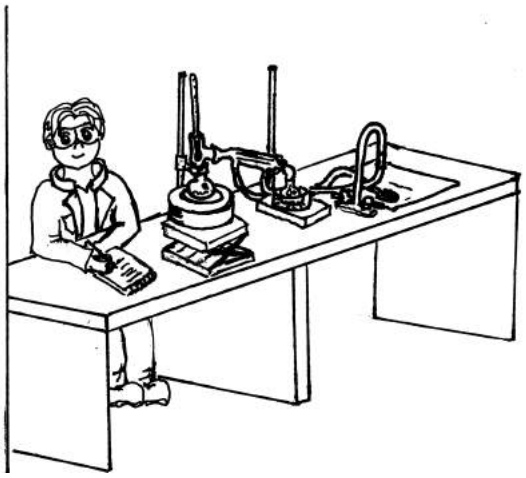
Chemistry 360

Organic Chemistry II

Report Book 2019-21



Athabasca University

Course team

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CHEM 360 Report Book Contents

CHEM 360 Report Book Contents (cont.)

Welcome to Organic Chemistry 360's Laboratory Report Workbook

This Report Book, along with the 'Chemistry 360 Lab Manual', will help you prepare for four-five days straight of supervised lab instruction. All preparatory work in this report book (~12 h to finish, see list on page 3), may be completed and shown to the Chemistry Lab Co-ordinator / Lab Instructor prior to beginning the labs.

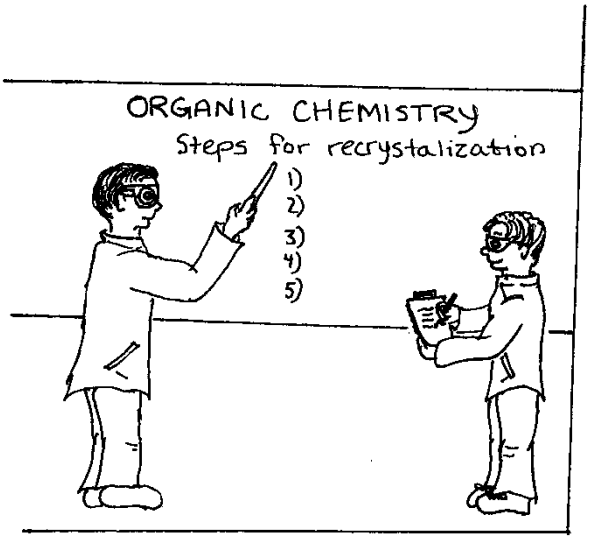
In order to successfully complete the laboratory component, please be aware of the following 4 step process of instruction. It is the intention of this Chem360 Report Workbook to provide you with the means of completing all four steps.

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 the usual 32 hours.)

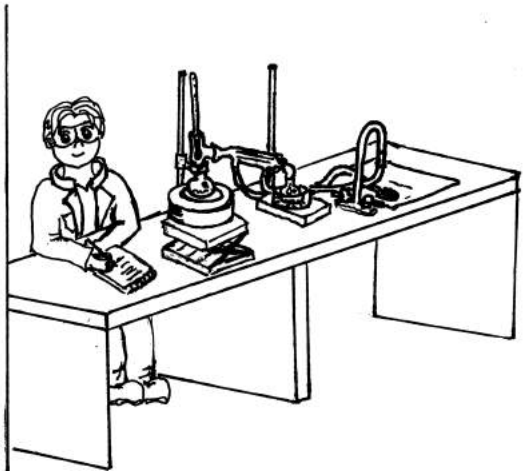
Step 2: Next we show you how to do it.

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.



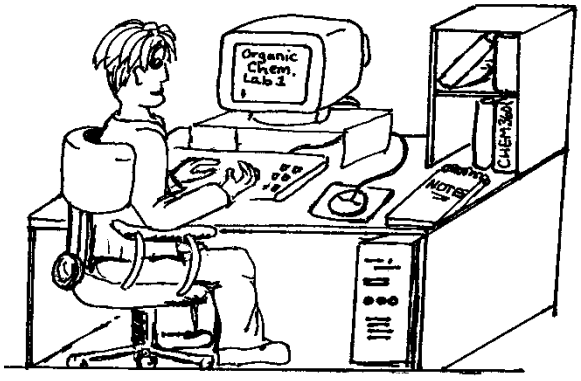
Step 3: Lab Time: Now you do what you've read, been told, and shown.

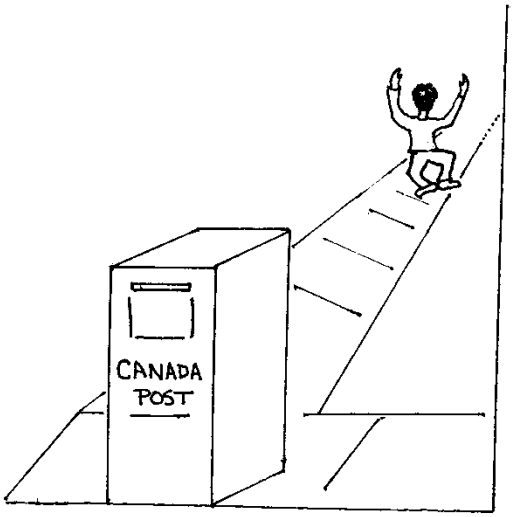
This is the time you spend in the lab performing your experiments, making your products, and recording your results in this report workbook



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 CHEM360 Report Book is to be used in conjunction with the CHEM360 Organic Chemistry II Lab Manual. It consists of an Introduction, Experiment Report Forms, Table of Reagents and Unknown Spectra. 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.
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 CHEM360 Lab Manual, and to read the 'Introduction to Concept', and 'Background Information' sections of this manual. To get feedback, **you may submit** them to the lab coordinator before the lab session begins.*
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 or procedural step table to record your work and observations. No need to do it both ways.
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 the Course Evaluation.

*CHEM360 Prelab questions will soon be available online at: http://science.pc.athabascau.ca/chem360.nsf
**CHEM360 Reagents is available online at: http://science.pc.athabascau.ca/chem360.nsf

Acknowledgements:

The 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 their permission to use the IR and 1H-NMR Spectra used in our Lab Manual and Report Book (pp. 15, 35, 46, 60, and 73). They were obtained from the SDBS web site: http://www.aist.go.jp/RIODB/SDBS/.

Each experiment in CHEM360 has been modified and rewritten from other sources, keeping the particular needs of Athabasca University students in mind. The format of this Report Book have been checked in our Athabasca laboratories by Dr. Lawton Shaw, Klaus Thomson, Nyron Jaleel, and Robert Carmichael. Special thanks to Ms. Aimee Caouette for her help on the Infrared Tutorial (Summer 1999). Also thanks to Mr. Douglas Woudstra and Mr. James Taylor (CHEM360 students 2004-05) for their helpful suggestions to improve the report book. The comments and suggestions received from all the individuals mentioned above were greatly appreciated by the Course Co-ordinator.

The following sources are also hereby acknowledged:

L.M. Browne, 2005. *Laboratory Report Book, Chemistry 161*, University of Alberta.
L.M. Browne, 2005. *Laboratory Report Book, Chemistry 163*, University of Alberta.

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.

CHEM360 Experiment 10 Report Date:_____

Student Name:_____ ID Number:_____

Experiment 10 Prelab Questions:

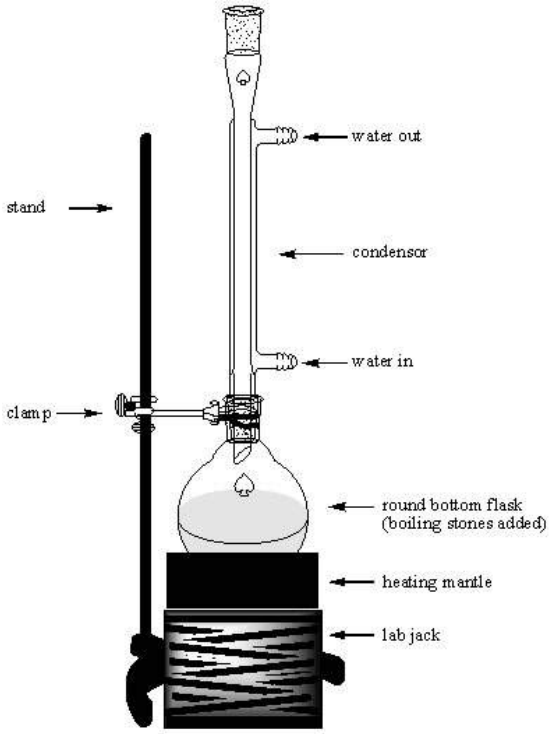
Lab Safety

1. What are the hazards of working with concentrated acids like glacial acetic acid and sulfuric acid?
   a) They are both extremely flammable
   b) They are only mildly corrosive and no significant precautions are needed
   c) Boiling these highly corrosive acids increases the danger to the experimenter, especially if the reaction flask should crack and break during heating

Equipment Preparation

2. Why must the condenser be 'clean and dry' prior to use?
   a) Clean and dry glassware automatically guarantees a higher yield
   b) Water is a by-product of the reaction and having 'wet glassware' will slow the reaction down
   c) Chemists are just neat

3. The following diagram for the 'reflux apparatus' used in Experiment 10 is correctly labelled.
   a) True
   b) false



Reagent Preparation

4. What are the two starting reagents used in a Fisher Esterification?
   a) carboxylic acid and a ketone
   b) alcohol and an ester
   c) alcohol and a carboxylic acid
   d) carboxylic acid and an ester

Reaction

5. Is the Fisher esterification reaction reversible?
   a) yes
   b) no

6. How long must you 'reflux the reaction' in order to maximize the amount of product formed?
   a) 20 min.
   b) 20-40 min.
   c) 60 min. or more (The longer the better!)

7. What acts as the nucleophile (Nu), and what acts as the electrophile (E) in this reaction?
   a) Nu = sulphuric acid, E = acetic acid
   b) Nu = isoamyl alcohol, E = acetic acid (protonated form)
   c) Nu = isoamyl alcohol, E = acetic acid
   d) Nu = acetic acid, E = isoamyl acetate

Reaction Workup

8. What gas is evolved during the reaction workup phase, when you wash the crude ester with 5% Na_2CO_3 (Procedure step C-5)?
   a) $H_{2\,(g)}$
   b) $N_{2\,(g)}$
   c) $O_{2\,(g)}$
   d) $CO_{2\,(g)}$

Product Characterization

9. How is the ester product purified and characterized?
   a) Yield, refractive index, % yield, infrared spectral analysis
   b) Boiling point, refractive index, and infrared spectral analysis
   c) Yield, boiling point, refractive index, % yield, and infrared spectral analysis

10. What major differences in absorption bands would you expect to see in the infrared spectra of isoamyl alcohol, and isoamyl acetate, the ester product?
    a) Broad 3300 cm^{-1} absorption for alcohol, sharp ~1740 cm^{-1} C=O of carbonyl in the ester
    b) Sharp ~2900 cm^{-1} absorption(s) for sp^3 C-H, no ~2900 cm^{-1} absorption for sp^3 C-H in ester.
    c) Sharp 3300 cm^{-1} absorption for alcohol, broad ~1740 cm^{-1} C=O of carbonyl in the ester.
    d) Sharp ~1200 cm^{-1} absorption for C-O of alcohol, none for the ester

CHEM360 Experiment 10 Report

Date:_____

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

Title:

Objective(s):

Equation(s): (General and specific reaction, draw structures, and provide names)

Introduction:

Procedure: (Reference: use proper format. Any Changes/Modifications?)

Procedure for the Fisher esterification of acetic acid with isoamyl alcohol.

Procedural Step	Observations/Comments/Inferences
Record amounts of reagents used.	

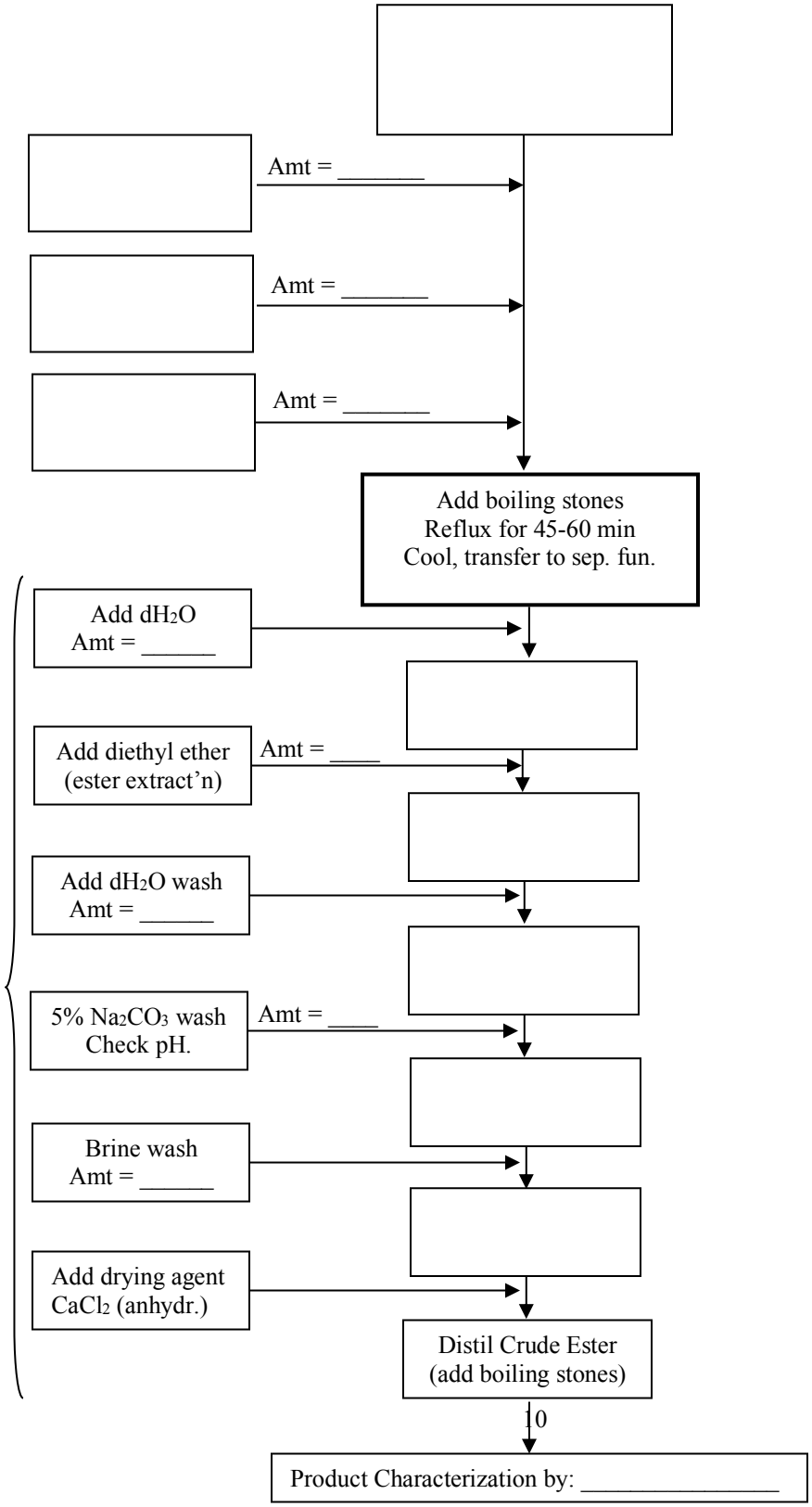
Table 10.1 Table of Reagents for Experiment 10 Fisher Esterification.

Reagent	Formula	Mwt. (g/mol)	d (g/ml)	Mp* (°C)	Bp* (°C)	Haz. Properties
Acetic acid, glacial (conc.) 17.4 M						
Isoamyl alcohol (3-methyl -1-butanol)	$(CH_3)_2CH(CH_2)_2OH$	88.15			130	Irritant
Sulfuric acid, conc. 18 M	H_2SO_4		1.840			
Distilled water	H_2O	18.02	1.000	0	100	none
Diethyl ether						
5% sodium carbonate (aq)	$Na_2CO_{3\,(aq)}$					
Sat. sodium chloride (aq) (brine)	$NaCl_{\,(aq)}$					
Calcium chloride (anhydr)	$CaCl_2$					
acetone, wash						
Isoamyl acetate	$CH_3CO_2C_5H_{11}$	130.19			142	

*find and record either the melting point if the reagent is a solid at room temperature, or the boiling point if the reagent is a liquid at room temperature.

EXPERIMENT 10 FLOW CHART

REAGENT **PROCEDURE / STEP** **OBSERVATION**



Experiment 10 Results:

Table 10.2. Summary Table of Observations (this table is optional. Use only to tidy up observations if necessary from the previous pages. Otherwise just say "see previous pages 9-10.")**:**

Procedural Step	Comment or Observation and Inference

Table 10.3. Table of Product Data for Isoamyl acetate, Fisher Esterification Product.

Table 10.3. 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.

	Yield Mass (g)	Appearance of Liquid	Boiling Pt.* (°C)	Refractive Index n_D obs.	Refractive Index^ (n_D^{20})	Theoretical Yield (g)	% Yield
Isoamyl acetate							

*Corrected for barometric pressure effects using the formula…
^Corrected to 20°C using the formula:

Limiting Reagent and Theoretical Yield Calculation:

Moles of acetic acid used in the reaction =

Moles of isoamyl alcohol used in the reaction =

Theoretical Yield of iosamyl acetate =

% Yield Calculation:

Table 10.4. Tabulation of Characteristic Infrared Absorptions for Starting regents and Product.

Table 10.4 contains the results of the Infrared Spectral Analyses for the isoamyl alcohol, acetic acid, and isoamyl acetate as obtained by thin film in a FTIR. See also attached spectra for peak numbering and identification.

Isoamyl alcohol	Peak Code#	Wavenumber (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strg,, med., or weak)	Functional Group Indicated
>3000 cm^{-1} Region					
3000-2000 cm^{-1} Region					
2000-1400 cm^{-1} Region					
Fingerprint Region					

Functional Groups Absent:

Acetic acid	Peak Code#	Wavenumber (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strg,, med., or weak)	Functional Group Indicated
>3000 cm^{-1} Region					
3000-2000 cm^{-1} Region					
2000-1400 cm^{-1} Region					
Fingerprint Region					

Functional Groups Absent:

Isoamyl acetate	Peak Code#	Wavenumber (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strg,, med., or weak)	Functional Group Indicated
>3000 cm^{-1} Region					
3000-2000 cm^{-1} Region					
2000-1400 cm^{-1} Region					
Fingerprint Region					

Functional Groups Absent:

Discussion:

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

Conclusion:

Structure of Product

Experiment 10 Post Lab Questions:

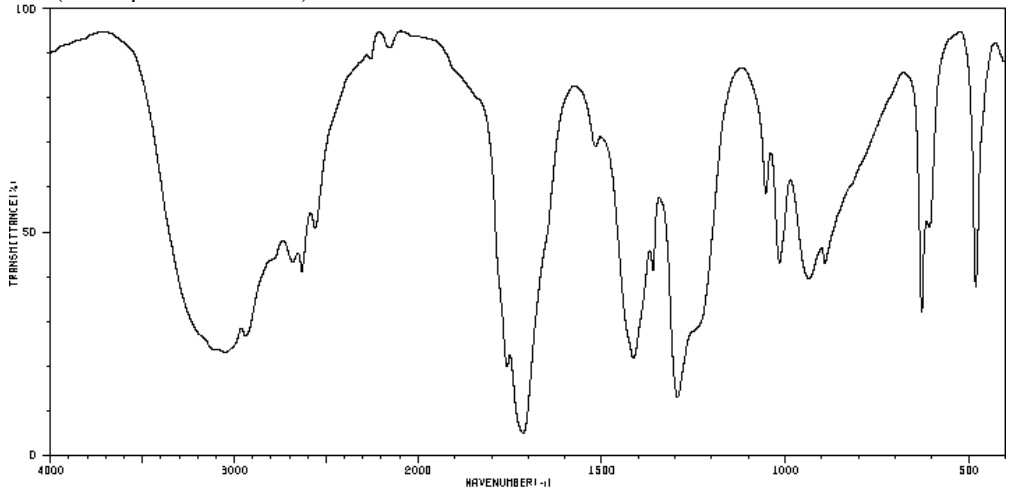
1. If the cost of **both** the alcohol and carboxylic acid were prohibitive, how would you maximize the yield of your Fischer esterification product while keeping costs down?

2. Why did you wash your product with water (3X), before washing it with the solution of sodium hydrogen carbonate? What was the purpose of washing with sodium hydrogen carbonate?

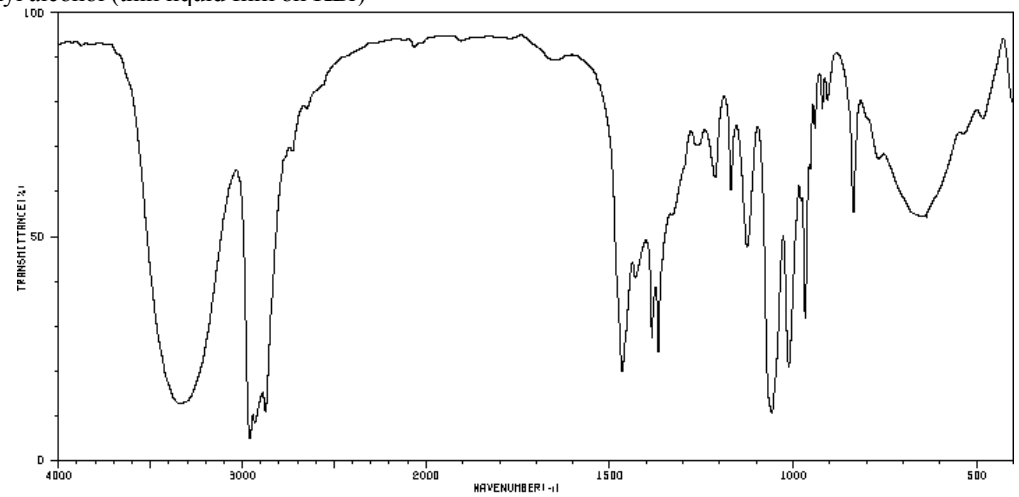
3. Explain the function of the acid catalyst in a Fisher esterification reaction.

4. What would the reactants be to produce isoamyl valerate via Fischer esterification reaction?

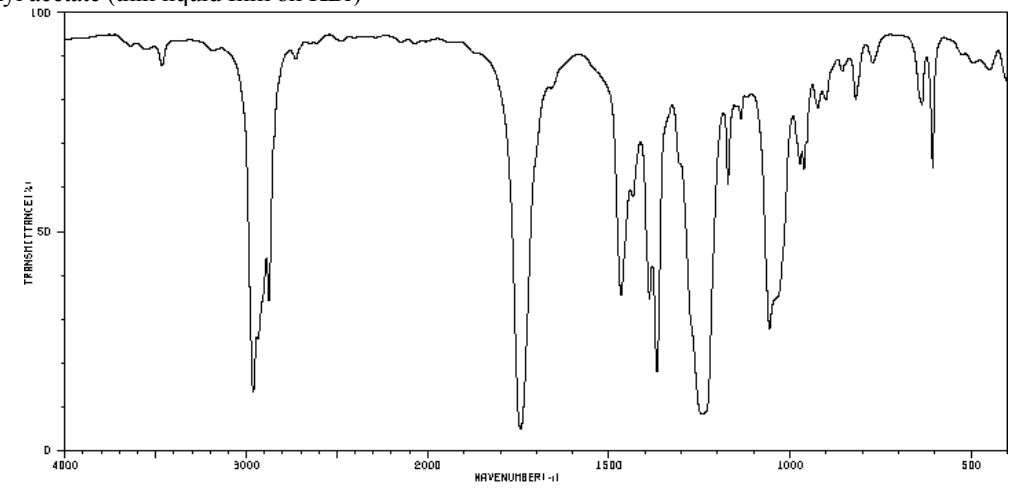
Acetic acid (thin liquid film on KBr)



Isoamyl alcohol (thin liquid film on KBr)



Isoamyl acetate (thin liquid film on KBr)



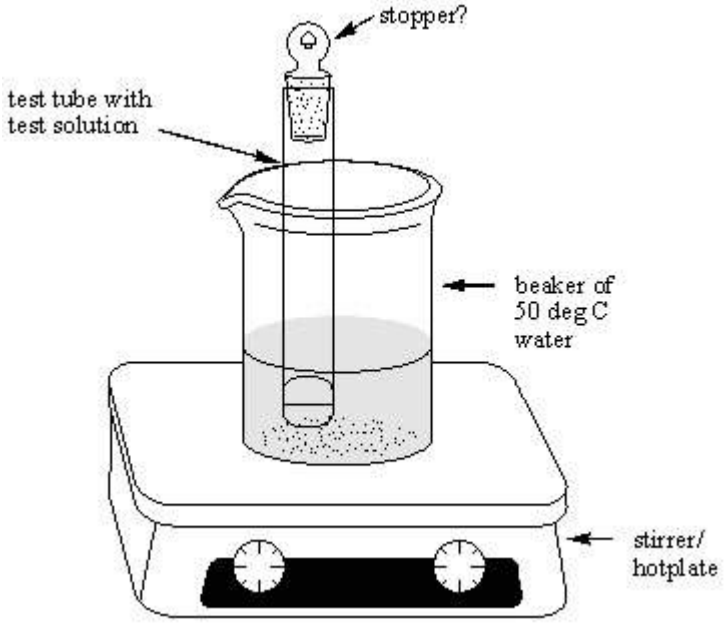
CHEM360 Experiment 11 Report Date:_____

Student Name:_____ ID Number:_____

Experiment 11 Prelab Questions:

Lab Safety

1. Should you stopper the test tubes prior to heating the tubes in the Ethanolic Silver Nitrate and Sodium Iodide/Acetone Tests?
   a) Yes. You must stopper the test tubes to prevent evaporation of the test compound
   b) No. You should never heat closed vessel as it may explode!



Equipment Preparation

2. Should the test tubes used in this experiment be clean and dry prior to use?
   a) yes
   b) no

Reagent Preparation

3. What oxidizing agent is used to detect primary and secondary alcohols, but not tertiary alcohols?
   a) sulphuric acid
   b) sodium dichromate
   c) mixture of sulphuric acid and sodium dichromate
   d) Lucas reagent

Reaction

4. What is the organic product of the reaction of Lucas reagent with an alcohol?
   a) ketone
   b) silver halide
   c) water
   d) alkyl halide

5. What type of alcohol would be positive in both the Dichromate and Lucas Reagent tests?
   a. phenol
   b) primary
   c) secondary
   d) tertiary

6. S_N1 stands for?
   a) substitution nucleophilic unimolecular
   b) substitution nucleophilic bimolecular
   c) substitution nucleophilic first

7. S_N2 stands for?
   a) substitution nucleophilic unimolecular
   b) substitution nucleophilic bimolecular
   c) substitution nucleophilic second

8. Why is the silver nitrate test a good one to observe S_N1 reaction mechanism behaviour?
   a) A positive reaction shows a change of color and is easily distinguished from a negative reaction
   b) precipitates that form are easily seen in positive tests
   c) the gas produced is easily seen in positive tests

9. Why is the sodium iodide in acetone test a good one to observe S_N2 reaction mechanism behaviour?
   a) A positive reaction shows a change of color and is easily distinguished from a negative reaction
   b) precipitates that form are easily seen in positive tests
   c) the gas produced is easily seen in positive tests

Cleanup

10. What should be done with the completed test solutions?
    a) they should be rinsed into the Halogentated organic waste container
    b) they can be rinsed down the drain
    c) store them in the fumehood

CHEM360 Experiment 11 Report

Date:_____

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

Title:

Objective(s):

General Equation(s): (structures and names)

Procedure: (Ref:)

Changes/Modifications?

Table 11.1. Table of Reagents for Experiment 11.

Reagent	Formula	Mwt.	Vol/Mass	d	mp	bp	Haz. Properties
1-butanol	C_4H_9OH	74.12	4 drops	0.810	-89.5	117-118	Flamm., Irritant
2-butanol	C_4H_9OH	74.12	4 drops	0.807		100	Flamm., Irritant
2-methyl-2-propanol	C_4H_9OH	74.12	4 drops	0.7887	25.5	82.3	Flamm., Irritant
Cyclohexanol	$C_6H_{11}OH$	100.16	4 drops	0.9624	25.1	161.1	Irritant, Hygroscopic
1-chlorobutane	C_4H_9Cl	92.57	8 drops	0.8862	-123	78.4	Flammable
2-chlorobutane	C_4H_9Cl	92.57	8 drops	0.8732	-131	68.2	Flammable
2-chloro-2-methylpropane	C_4H_9Cl	92.57	8 drops	0.8420	-25.4	52	Flammable
1-bromobutane	C_4H_9Br	136.9	8 drops	1.2758	-112	101.6	Flammable, Irritant
2-bromobutane	C_4H_9Br	136.9	8 drops	1.2585	-112	91.2	Flammable, Irritant
Chlorobenzene	C_6H_5Cl	122.4	8 drops	1.1058	-45.6	132	Flammable, Irritant
benzyl chloride	$C_6H_5CH_2Cl$	126.59	8 drops	1.1002	-39	179.3	Toxic, Cancer susp.agent
3-chloro-1-butene	C_4H_7Cl	90.55	8 drops	0.8978		64-65	Flamm., Lachrymator
Bromocylclohexane	$C_6H_{11}Br$	163.06	8 drops	1.3359	-56.5	166.2	Flammable, Irritant
Bromocyclopentane	C_5H_9Br	149.04	8 drops	1.3873		136.7	Flammable, Irritant
β-bromostyrene	$C_6H_5CHCHBr$	183.05	8 drops	1.4269	7	219	Irritant
sodium dichromate	$Na_2Cr_2O_7$	261.6	12 mL				Toxic, Cancer susp.agent
sulfuric acid	H_2SO_4	98.08					Corrosive, Toxic, Oxidizer
Lucas reagent	Solution of $ZnCl_2$ and HCl						Toxic, Irritant
zinc chloride, anhydrous	$ZnCl_2$	136.28		2.91	283	732	Corrosive, Toxic
hydrochloric acid, conc.	HCl	36.46		1.20		(110)	Corrosive, Highly toxic
sodium iodide	NaI	149.9					Mosit. Sens., Irritant
silver nitrate	$AgNO_3$	169.8					Highly toxic, Oxidizer
nitric acid	HNO_3	63.01		1.400			Corrosive, Oxidizer
Acetone	CH_3COCH_3	58.09		0.818		56.5	Flammable, Irritant
ethanol	CH_3CH_2OH	46.07		0.785		78.5	Flammable, Poison

Experiment 11 Part A Results: Reactions of Alcohols (1°, 2°, and 3°)

1. Alcohol Oxidation by Sodium Dichromate			
Test Substance	**Observation**	**Inference**	**Equation**
1-butanol			
2-butanol			
cyclohexanol			
2-methyl-2-propanol			

2. Lucas Reagent Test			
Test Substance	**Observation**	**Inference**	**Equation**
1-butanol			
2-butanol			
cyclohexanol			
2-methyl-2-propanol			

Experiment 11 Part B Results: Reactions of Alkyl Halides

Silver Nitrate Test (S_N1 Mechanism)			
Test Substance	**Observation**	**Inference**	**Equation**
1-chlorobutane			
2-chlorobutane			
2-chloro-2-methylpropane			
1-bromobutane			
2-bromobutane			
Chlorobenzene			
Benzyl chloride			
3-chloro-1-butene			
Bromocyclohexane			
Bromocyclopentane			
β-bromostyrene			

Experiment 11 Part B Results: (cont.)

Sodium Iodide/Acetone Test (S_N2 Mechanism)			
Test Substance	**Observation**	**Inference**	**Equation**
1-chlorobutane			
2-chlorobutane			
2-chloro-2-methylpropane			
1-bromobutane			
2-bromobutane			
Chlorobenzene			
Benzyl chloride			
3-chloro-1-butene			
Bromocyclohexane			
Bromocyclopentane			
β-bromostyrene			

Conclusion:

Experiment 11 Post Lab Questions:

1. There are four isomeric alcohols having the formula $C_4H_{10}O$, and in this experiment you investigated the properties of three of them. How would you expect the fourth isomer to behave when treated with (i) acidic sodium dichromate, and (ii) Lucas reagent?

2. On the basis of your results, arrange the eleven halogen-containing compounds in order of *decreasing* reactivity in (i) S_N1 reactions and (ii) S_N2 reactions.

3. a. What results would you expect to observe when benzyl alcohol, $C_6H_5CH_2OH$, is treated with (i) acidic sodium dichromate, and (ii) Lucas reagent?

   b. What results would you expect to obtain when 1-chloro-2,2-dimethylpropane is treated with (i) ethanolic silver nitrate, and (ii) sodium iodide in acetone?

CHEM360 Experiment 12 Report Date:_____

Student Name:_____ ID Number:_____

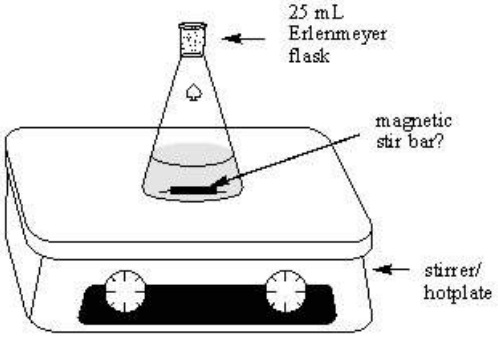
Experiment 12 Prelab Questions:

Lab Safety

1. Sodium borohydride ($NaBH_4$) is much safer to use than lithium aluminium hydride ($LiAlH_4$)?
   a) yes
   b) no
   c) This statement is false. They are both safe
   d) This statement is false. They are both highly dangerous reagents!

Equipment Preparation

2. Why must you place a magnetic stir bar into the reaction vessel and use a stir plate for this reaction?
   a) for the reaction to occur to its fullest extent, the reagents need to be continuously mixed
   b) the magnetic stir bar serves as a site for crystal nucleation
   c) to keep the crystalline product from settling to the bottom of the flask



Reagent Preparation

3. Why must you prepare the two main reagents, sodium borohydride and benzophenone, separately and then mix them together?
   a) as a safety precaution so as to avoid an uncontrolled premature reaction
   b) the two reagents are not miscible
   c) you can only do one thing at a time in a chemistry lab

Reaction

4. Why do you add the sodium borohydride slowly to the benzophenone?
   a) to avoid spilling the reagent
   b) sodium borohydride is very difficult to handle
   c) as a safety precaution; to control the rate of the exothermic reaction

Reaction Workup

5. What is the purpose of adding the hydrochloric acid/ice in the procedure step 5?
   a) To dilute and lower the pH of the mixture and thereby prevent product precipitation
   b) To decompose the excess sodium borohydride, and protonate the alcohol moiety of the final product
   c) To prevent the decomposition of the sodium borohydride

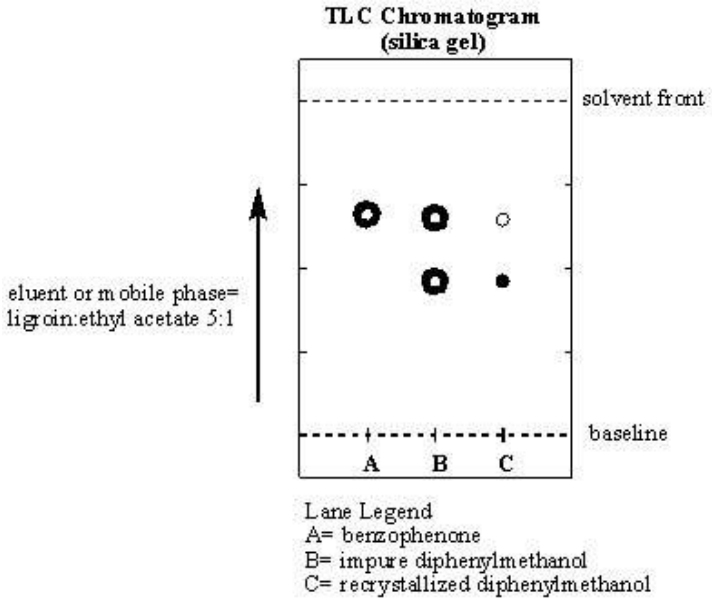
6. Should an Erelnmeyer flask be used as the vessel to recrystallize the final product, or a beaker?
   a) There is no advantage. Either one could be used
   b) Erlenmeyer flask. The narrower neck of the Erlenmeyer flask acts like a condenser and prevents the solvent from evaporating too quickly. Also a flask can eventually be sealed with a stopper while crystal growth is occurring.
   c) Beaker. A beaker is easier to add solvent to and the recrystallized product is easier to recover from a beaker.

7. Why is hexane used as the recrystallization solvent?
   a) **because** diphenylmethanol is soluble in hot hexane and insoluble in cold hexane
   b) because diphenylmethanol is insoluble in hot hexane and isoluble in cold hexane
   c) because it is the only solvent available

Product Characterization

8. What is the purpose of performing TLC on the crude and recrystallized product, and benzophenone?
   a) To compare and determine the purity of the final product
   b) To measure the yield of the final product
   c) To purify the final product

9. Does the TLC sketch shown here indicate the product diphenylmethanol to be pure or impure after recrystallization?
   a) pure
   b) impure



11. What major differences in absorption bands would you expect to see in the infrared spectra of benzophenone, and diphenylmethanol, the alcohol product?
   a) sharp ~1710 cm^{-1} C=O of carbonyl in the ketone, broad 3300 cm^{-1} absorption for alcohol
   b) broad ~1740 cm^{-1} C=O of carbonyl in the ketone, sharp 3300 cm^{-1} absorption for alcohol
   c) Sharp ~1200 cm^{-1} absorption for C-O for the ketone, not present for the alcohol

CHEM360 Experiment 12 Report

Date:_____

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

Title:

Objective(s):

Reaction Equation: (structures and names)

Introduction:

Procedure: (Reference format: author surname, initials, date. Title of text, publisher, pages)

Any Changes/Modifications?

Part A: Reduction of Benzophenone

Procedural Step	Observations/Comments/Inferences
Preparation of organic reagent Preparation of reducing agent	

Part B: Thin Layer Chromatography of Reagent and Product.

Procedural Step	Observations/Comments/Inferences
1. Preparation of eluent 2. Preparation of spotting solutions 3. Number of spots per lane: 4. Appearance of developed TLC	

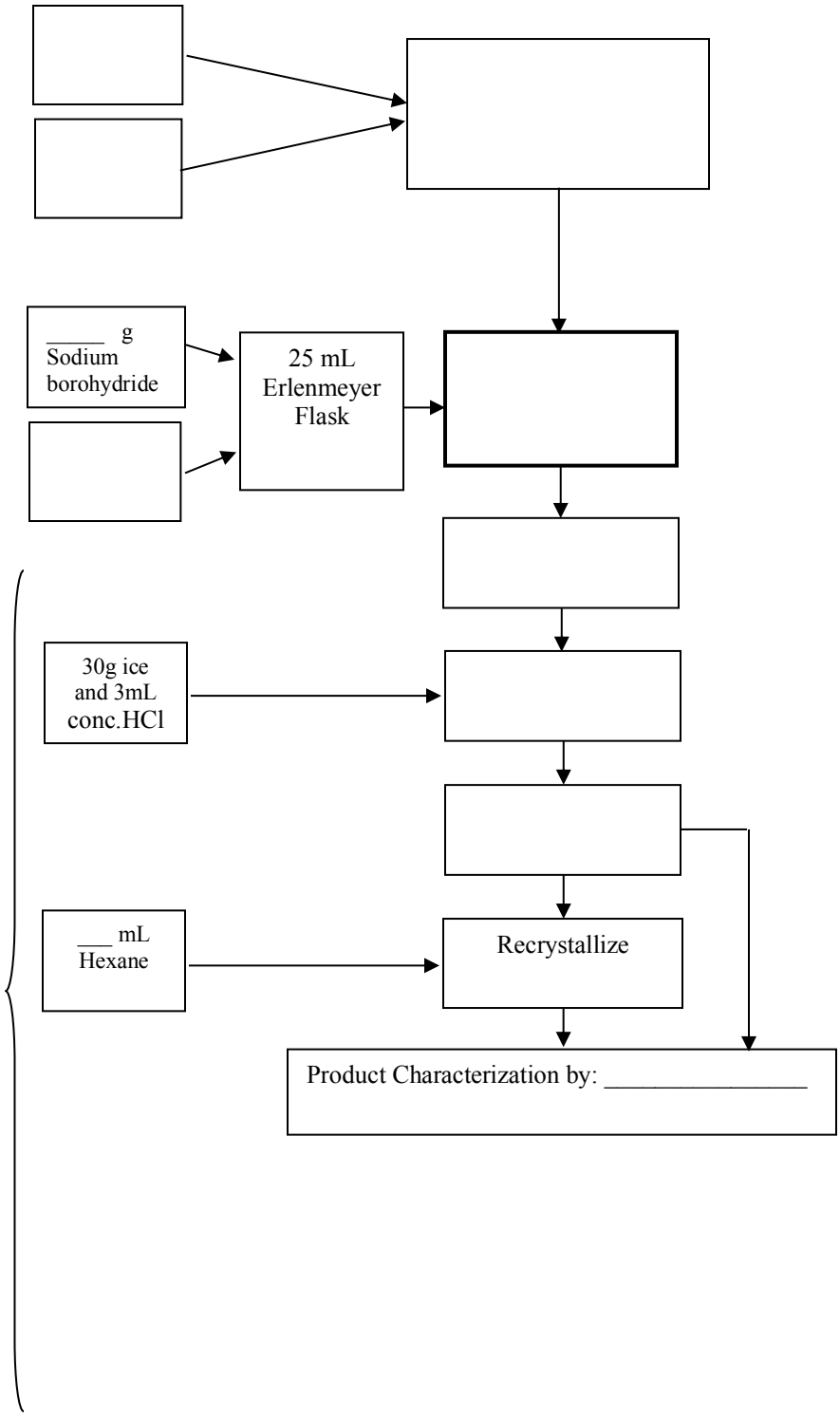
Table 12.1. Table of Reagents for Exp. 12

Reagent	Formula	Mwt.	Vol/Mass	d	moles	mp	bp	Haz. Properties
Benzophenone (or diphenylketone)	$C_6H_5COC_6H_5$	182.22		NA			NA	
Sodium borohydride	$NaBH_4$	37.83		NA		400	NA	
Methanol	CH_3OH	32.04		1.3288		-93.9	65	
Hydrochloric acid (conc. = 12M)	HCl	36.46		1.20		NA		
Hexane	C_6H_{14}	86.18		0.659		NA	69	
Ethyl acetate	$C_2H_5CO_2\ C_2H_5$	88.11	2 mL	0.902		NA	77	
Ligroin	Mineral spiritis		10 mL	0.656		NA	60-80	
Chloroform	$CHCl_3$	119.39	3 mL	1.500		NA	61.3	
Iodine	I_2	253.81	trace	NA		133	NA	
Diphenylmethanol (or benzhydrol)			?				NA	

NA = not applicable

SAMPLE EXPERIMENT 12 FLOW CHART

REAGENT **PROCEDURE / STEP** **OBSERVATION**



Experiment 12 Results:

Table 12.2. Summary Table of Observations (this table is optional. Use only to tidy up your observations from the previous page if necessary.):

Procedural Step	Comment or Observation

Table 12.3. Table of Product Data for Diphenylmethanol, the Carbonyl Reduction Product.

Table 12.3. 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.

Product Name	Yield (Mass in g)	Appearance of Solid	Observed Melting Pt.* (°C)	Lit. Melting Pt. (°C)	Theoretical Yield (g)	% Yield

*uncalibrated thermometer used.

Limiting Reagent and Theoretical Yield Calculation:

Moles of benzophenone used in the reaction =

Moles of sodium borohydride used in the reaction =

Theoretical Yield of diphenylmethanol =

% Yield Calculation:

Table 12.4. Infrared Spectrum Data Analysis (see attached spectra)

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

Functional Group absent:

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

Functional Group absent:

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

Functional Group absent:

Fig 12.1. TLC Analysis of Benzophenone, Crude and Recrystallized Diphenylmethanol

Discussion:

Comments on and reasons for yield (high/med/low), purity (high/med/low), sources of error (uncalibrated thermometer?, side reactions?):

Conclusion:

Structure of Product

Experiment 12 Post Lab Questions:

1. Aldehydes and ketones can be reduced to alcohols using hydrogen gas and a metal catalyst. Suggest two reasons why the use of sodium borohydride is preferred over the catalytic hydrogenation in order to prepare diphenylmethanol from benzophenone.

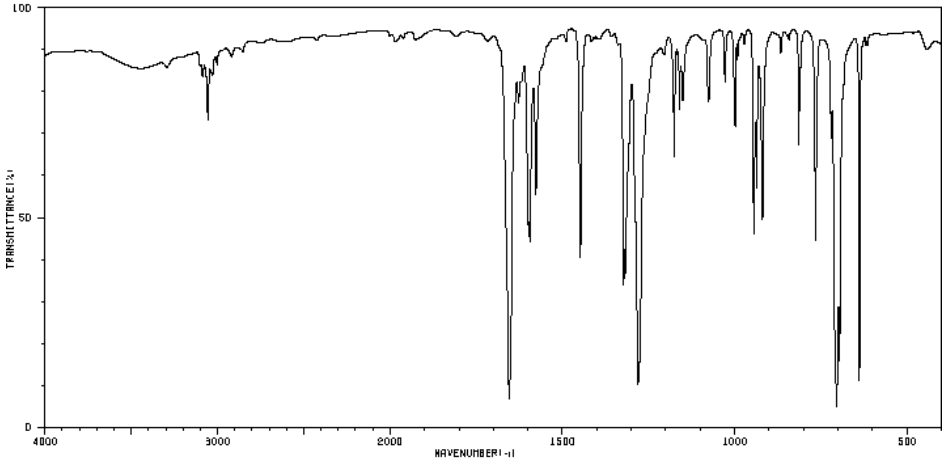
2. In this experiment, you destroyed the excess sodium borohydride by reacting it with hydrochloric acid (Part A, Step 5). What gas was evolved during the process? Write a balanced equation for the reaction that occurred. (**Hint:** one of the products was boric acid).

Literature Infrared Spectrum of Benzophenone (cast thin film on KBr)

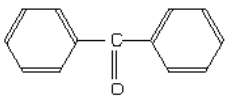
HIT-NO=1393	SCORE= ( )	SDBS-NO=1294	IR-NIDA-03202 : KBR DISC

BENZOPHENONE

$C_{13}H_{10}O$



3291	81	1656	6	1205	86	992	84	720	56
3088	81	1627	74	1176	82	945	44	705	4
3067	79	1595	42	1162	72	936	55	697	15
3057	70	1577	69	1151	74	919	47	639	10
3030	81	1449	38	1076	74	867	86		
3003	84	1323	52	1028	79	814	64		
2919	84	1281	10	999	68	766	42		

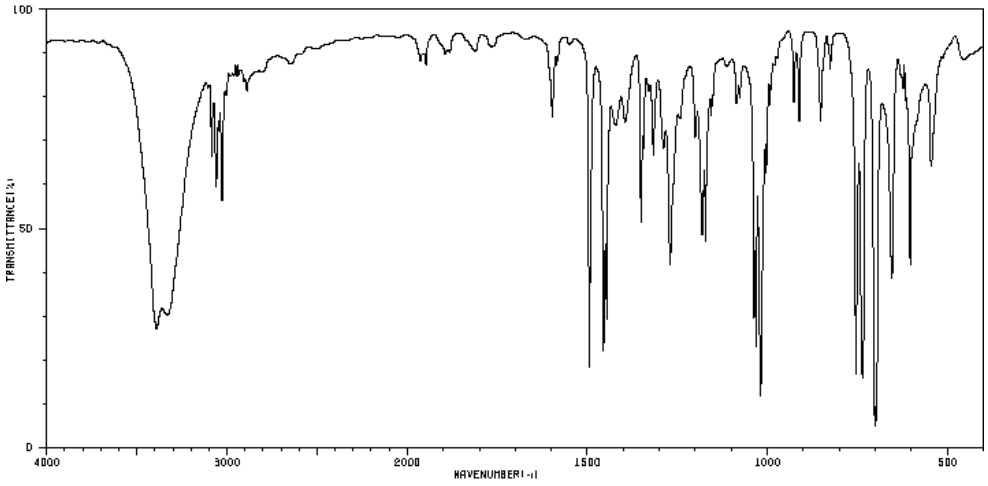


Literature Infrared Spectrum of Diphenylmethanol (cast thin film on KBr)

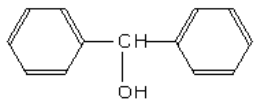
HIT-NO=1144	SCORE= ( )	SDBS-NO=869	IR-NIDA-47684 : KBR DISC

BENZHYDROL

$C_{13}H_{12}O$



3392	26	1598	72	1345	66	1172	44	863	72
3359	30	1495	17	1317	64	1156	72	754	16
3331	29	1458	21	1289	86	1037	28	735	15
3085	64	1446	27	1271	39	1032	22	702	4
3059	57	1422	70	1244	72	1019	11	654	37
3049	70	1595	72	1201	88	1003	60	604	39
3027	63	1361	49	1182	46	912	72	645	62



CHEM360 Experiment 13 Report Date:_____

Student Name:_____ ID Number:_____

Experiment 13 Prelab Questions

Lab Safety

1. What is the major danger of using 95% ethanol?
   a) It is corrosive
   b) It is flammable
   c) It is an oxidizer

Equipment Preparation

2. A stirrer/hot plate is used in this experiment in order to:
   d) continuously mix the reagents and allow the reaction to occur to its fullest extent
   e) the magnetic stir bar serves as a site for crystal nucleation
   f) to keep the crystalline product from settling to the bottom of the flask

Reagent Preparation

3. How do you use the molar mass (MM, g/mol) and density (d, g/mL) of your starting aldehyde or ketone to determine the volume amount of reagent to add (mL) to the reaction, from knowing only the number of moles to use?
   a) Moles reagent to use is divided by the MM = grams reagent, divided by the density = mL of reagent to use, i.e., (mol / MM)/d = mL
   b) Moles reagent to use multiplied by the MM = grams reagent, divided by the density = mL of reagent to use, i.e., (mol × MM)/d = mL
   c) Moles reagent to use divided by the MM = grams of reagent, multiplied by the density = mL of reagent to use, i.e., (mol /MM) × d = mL

4. What is the purpose of adding the reagent 95% ethanol (in water) to the reaction mixture of the ketone and aldehyde?
   a) 95% ethanol is the solvent for the reaction
   b) 95% ethanol prevents unwanted side reactions
   c) 95% ethanol reacts with sodium hydroxide to form sodium ethoxide, which is the base catalyst for the reaction

Reaction

5. The aldol condensation is used by synthetic chemists:
   a) because it is a reversible reaction
   b) because it is non-reversible reaction
   c) to form a new carbon-carbon bond

Reaction Workup

6. Will your product be essentially pure after cooling the flask in ice in Step 2 of the procedure?
   a) yes
   b) no

7. Will your product be essentially pure after washing your product with ice cold 95% ethanol, ice cold 95% ethanol + 4% acetic acid and again with ice cold 95% ethanol in Step 3 of the procedure?
   a) yes
   b) no

8. Why must the washing solutions be ice-cold in Step 3 of the procedure?
   a) to prevent dissolving the product, which is more soluble in warm solvent than cold
   b) to keep the reaction from warming up
   c) to prevent unwanted side reactions
9. How will you know which solvent (95% ethanol or toluene) is more suitable for the recrystallization of your product?
   a) the chosen solvent will dissolve less crystals when hot, and form more crystals when cold
   b) the chosen solvent will dissolve more crystals when hot, and form more crystals when cold
   c) the chosen solvent will dissolve less crystals when hot, and form less crystals when cold

Product Characterization

10. What reason(s) may help to explain a low percentage yield for your aldol condensation reaction
    a) the reagents where incorrectly measured
    b) all the washing steps resulted in the loss of some product, even when ice cold solvents were used
    c) the chosen recrystallization solvent still kept some of the product dissolved
    d) all of the above

CHEM360 Experiment 13 Report Date:_____

Student Name:_____ ID Number:_____

Title:

Objective(s):

Reaction Equation: (structures and names)

Introduction:

Introduction: (cont.)

Procedure: (Reference: author surname, initials, date. Title of text, publisher, pages)
Any Changes/Modifications?

Mixed Aldol Condensation Reaction Observations

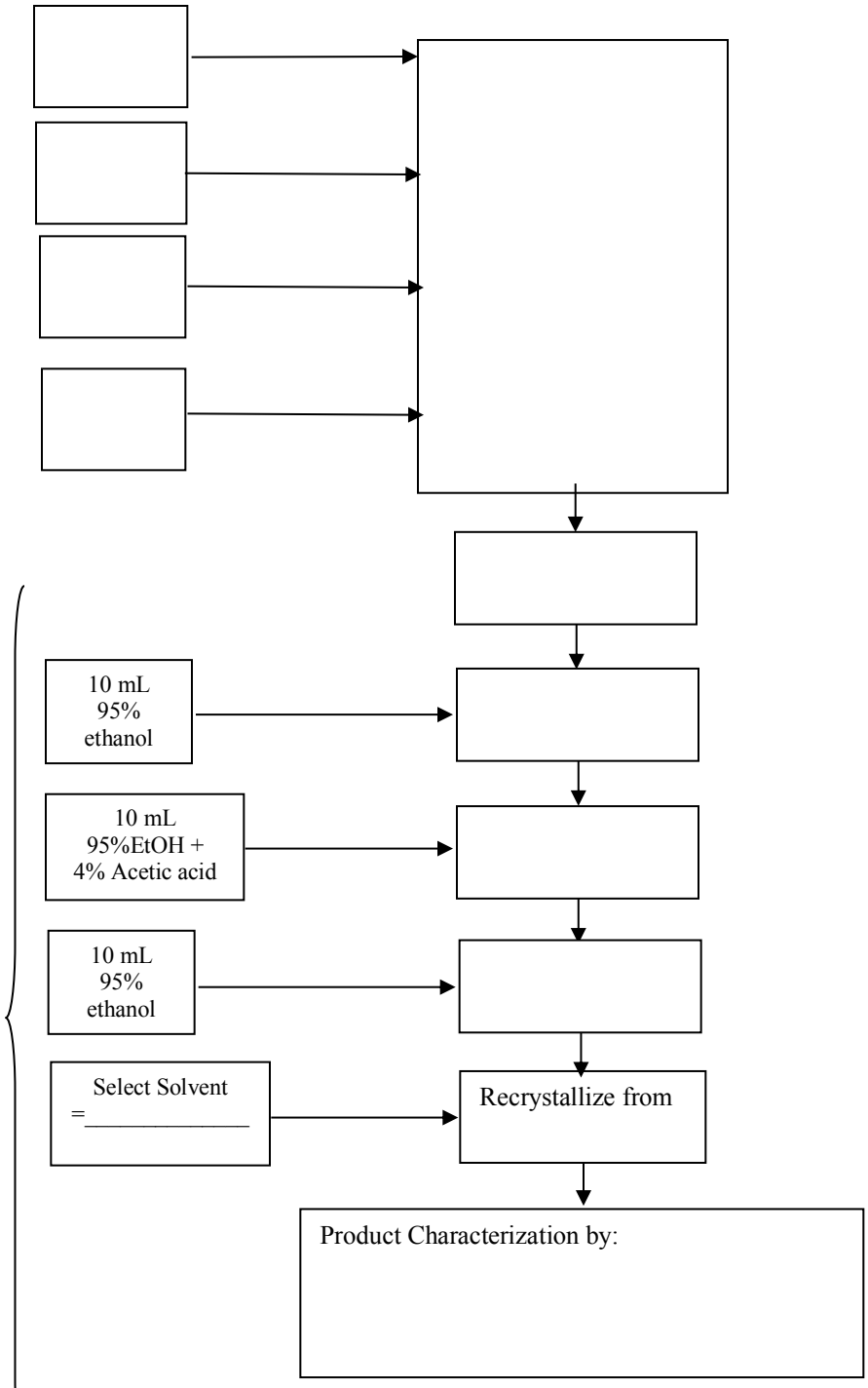
Procedural Step	Observations/Comments/Inferences

Table 13.1. Table of Reagents for Exp. 13

Reagent	Formula	Mwt.	d	mp	bp	Haz. Properties
Benzaldehyde			1.044		179.5	
4-methylbenzaldehyde	$CH_3C_6H_4CHO$		1.019		204-205	
4-methoxybenzaldehyde	$CH_3OC_6H_4CHO$		1.119		248	
Cinnamaldehyde	$C_6H_5CH{=}CHCHO$		1.048		248	
Acetone	CH_3COCH_3		0.818		56.5	
Cyclopentanone	C_5H_8O		0.951		130.6	
Cyclohexanone	$C_6H_{10}O$		0.947		155	
4-methylcyclohexanone	$CH_3C_6H_9O$		0.914		169-171	
Ethanol	CH_3CH_2OH		0.785		78.5	
Sodium hydroxide	NaOH (1M)		~1.00			
Acetic acid	CH_3COOH		1.049		118.1	
Toluene	$C_6H_5CH_3$		0.865		110.6	
Benzalacetone	$C_6H_5CHCHCOCH_3$					
Dibenzalacetone						
Dibenzalcyclohexanone						
Dibenzalcyclopentanone						
Chloroform	$CHCl_3$					
Carbon tetrachloride	CCl_4					

SAMPLE EXPERIMENT 13 FLOW CHART

REAGENT **PROCEDURE / STEP** **OBSERVATION**



Experiment 13 Results:

Table 13.2. Summary Table of Observations (this table is optional. Use only to tidy up your observations from the previous page if necessary.):

Procedural Step	Comment/Observation/Inferences

Table 13.3. Table of Data for Aldol Condensation Product.

Table 13.3 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: Both reagents are added in equal stoichiometric amounts. The _____ was used as the limiting reagent as it is a 1:1 ratio with the product.

Product Name	Yield Mass (g)	Appearance of Solid	Melting Pt.* (°C)	Literature Melting Pt. (°C)	Theoretical Yield (g)	% Yield

*Uncorrected for temperature calibration

Limiting Reagent and Theoretical Yield Calculation:

Moles of ketone actually used in the reaction =

Moles of aldehyde actually used in the reaction =

Theoretical Yield of Aldol product =

% Yield Calculation:

Table 4. Infrared Spectrum Data Analysis (see attached spectra)

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

Functional Group absent:

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

Functional Group absent:

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

Functional Group absent:

Discussion:

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

Conclusion:

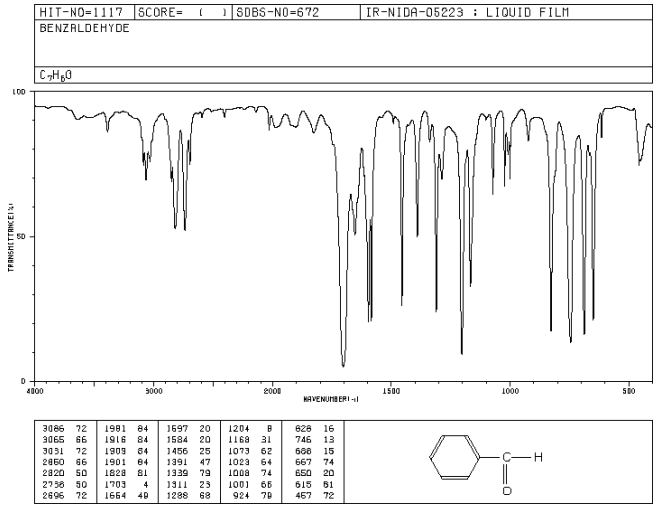
Structure of Product

Experiment 13 Post Lab Questions:

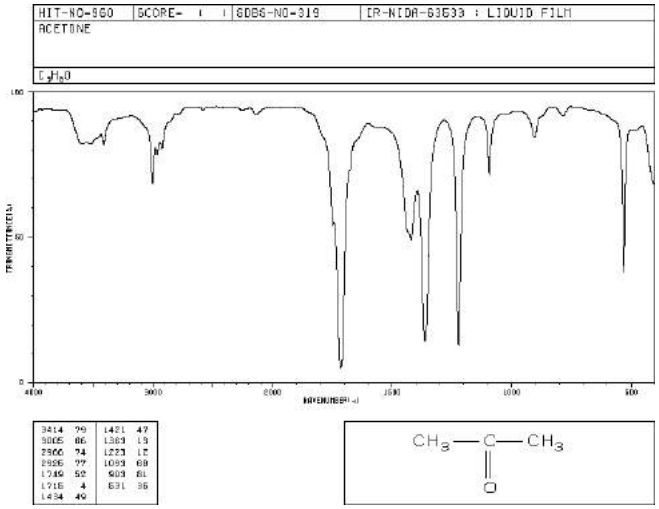
1. The product obtained in this experiment results from a crossed condensation between an aldehyde and a ketone. Identify two other base-initiated reactions that could conceivably occur involving either or both of these reactants. Suggest reasons why these reactions do not result in the formation of large quantities of by-products.

2. What is the purpose of adding sodium hydroxide to the reaction mixture? (use equations if necessary to explain your answer).

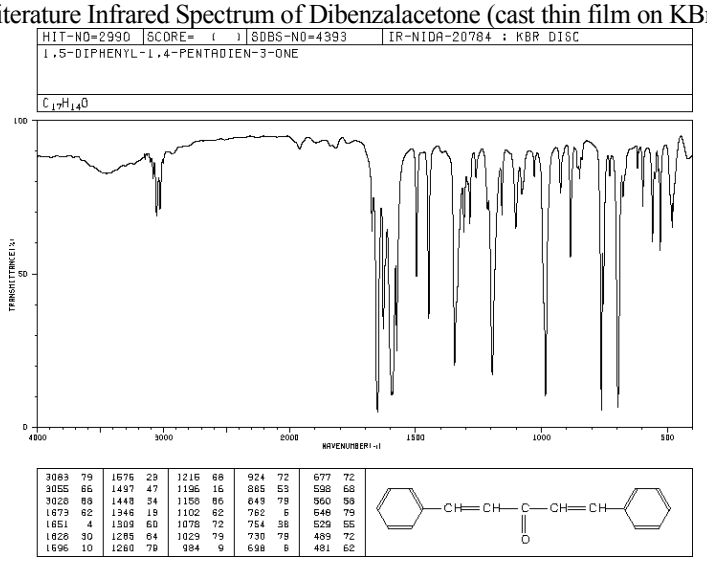
Literature Infrared Spectrum of Benzaldehyde



Literature Infrared Spectrum of Acetone



Literature Infrared Spectrum of Dibenzalacetone (cast thin film on KBr)



CHEM360 Experiment 14 Report Date:_____

Student Name:_____ ID #:_____

Experiment 14 Prelab Questions:

1. ^{1}H-NMR is an abbreviation meaning:
   a) nuclear magnetic resonance spectroscopy
   b) proton nuclear magnetic resonance spectroscopy
   c) carbon 13 nuclear magnetic resonance spectroscopy

2. What types of information are to be found in a ^{1}H-NMR spectrum?
   a) chemical shift and multiplicity (splitting patterns)
   b) chemical shift, number of equivalent hydrogens, and the multiplicity
   c) chemical shift, number of equivalent hydrogens, multiplicity, and the number of neighbouring hydrogens
   d) chemical shift, number of equivalent hydrogens, multiplicity, the number of neighbouring hydrogens, and the potential signal assignment (after consulting the Shifts for Various Functional Groups table.

3. If you know the chemical formula of your unknown, the 'degrees of unsaturation' calculation (deg Unsaturation . = nC + 1 +1/2N -1/2nH -1/2nX) helps you to:
   a) determine the number of 'equivalent double bonds' present in your unknown (e.g., alkene = 1, alkyne = 2, cylcoalkane = 1, benzene ring = 4)
   b) determine the functional groups present in the unknown
   c) determine the structure of your unknown

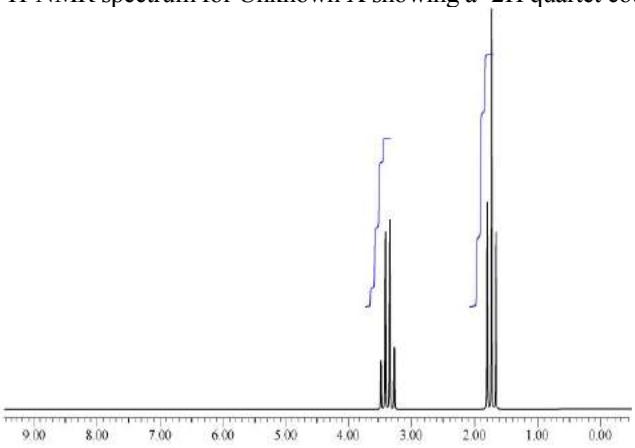
4. Chemical shifts are the result of 'shielding' and 'deshielding' in the environment the proton finds around it.
   a) true
   b) false

5. The N=n+1 rule is used to determine the number of neighbouring H
   a) true
   b) false

6. What is the purpose of adding tetramethylsilane (TMS) to a ^{1}H-NMR sample prior to determining its spectrum?
   a) to give an example of a highly shielded hydrogen environment
   b) to serve as a reference standard
   c) to serve as a blank

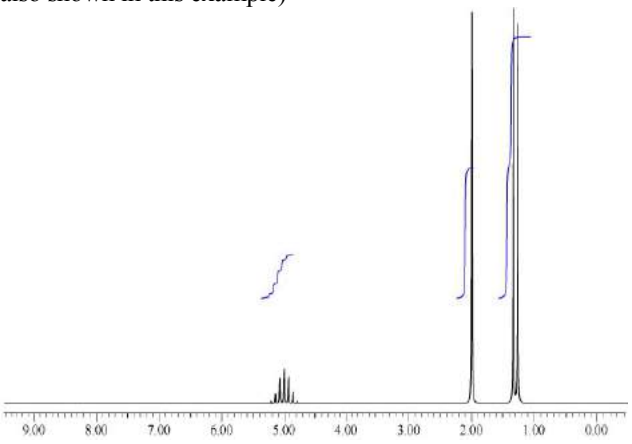
7. A very common splitting pattern seen in a ^{1}H-NMR spectrum (see Unknown X spectrum below) is the '2H quartet coupled to a 3H triplet'. Which of the following molecular fragments does this represent?
   a. a methyl group
   b. an ethyl group
   c. an isopropyl group
   d. a *tert*-butyl group

   ^{1}H-NMR spectrum for Unknown X showing a '2H quartet coupled to a 3H triplet'



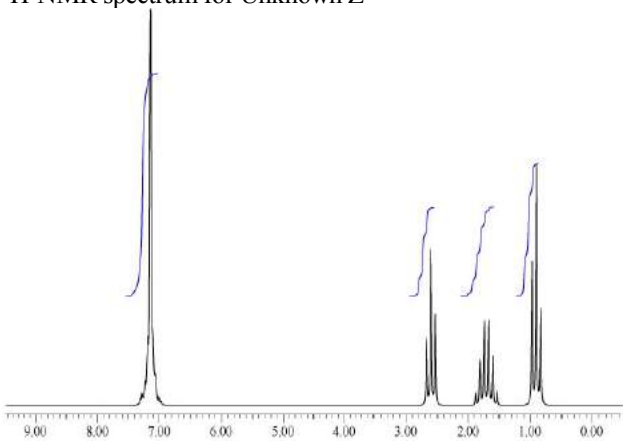
8. Another very common splitting pattern seen in a ^{1}H-NMR spectrum (see Unknown Y spectrum below) is a '1H septet coupled to a 6H doublet'. Which of the following molecular fragments does this usually represent?
   a) a methyl group
   b) an ethyl group
   c) an isopropyl group
   d) a *tert*-butyl group

   ^{1}H-NMR spectrum for Unknown Y showing a '1H septet coupled to a 6H doublet' (ignore the singlet also shown in this example)



9. The following ^{1}H-NMR spectrum for Unknown Z shows Unknown Z to have an aromatic ring.
   a) true
   b) false

^{1}H-NMR spectrum for Unknown Z



10. The hardest part in determining the structure of an unknown from your NMR data, is usually the final assembly of all the fragments.
   a) true
   b) false

CHEM360 Experiment 14 Report Date:_____

Student Name:_____ ID Number:_____

Title:

Objective(s):

Procedure:

(Ref:)
Changes/Modification:

ATTACH YOUR 4 UNKNOWN IR/NMR SPECTRA PROBLEMS TO THIS TITLE PAGE.
The unknown spectra can be found either at the end of this Report Book or online at:
http://science.athabascau.ca/Labs/resources/360Unkns/index.php
username = auchem360 password = synthesis

CHEM360 Experiment 15 Report Date:_____

Student Name:_____ ID Number:_____

Experiment 15 Prelab Questions

Lab Safety

1. What danger exists with the Tollen's reagent?
   a) The silver mirror that forms is high reflective to light
   b) ammonia used is very corrosive
   c) Tollen's reagent decomposes on standing to an explosive substance

Equipment Preparation

2. Test tube size is very important in performing functional group tests?
   a) true. The test tube must not become so filled with reagent and test substance, that it becomes difficult to mix
   b) true. The test tube must be completely filled with the reagent and test substance, so that it can only be mixed by stoppering and inverting the test tube
   c) false

Reagent Preparation

3. What should the Tollen's reagent appear like after the addition of the 1.0 M ammonium hydroxide?
   a) a brownish colored precipitate in solution
   b) a silver colored solution
   c) a clear and colorless solution

Reaction(s)

4. What does the Brady's Test detect?
   a) methyl ketone groups in aldehydes and ketones
   b) carbonyl groups of aldehydes and ketones
   c) aldehydes only
   d) ketones only

5. What does the Tollens' Test detect?
   a) methyl ketone groups of aldehydes and ketones
   b) carbonyl groups of aldehydes and ketones
   c) aldehydes only
   d) ketones only

6. What does the Schiff's Test detect?
   a) methyl ketone groups of aldehydes and ketones
   b) carbonyl groups of aldehydes and ketones
   c) aldehydes and aldehyde impurities mostly
   d) ketones only

7. What does the Iodoform Test Detect?
   a) methyl ketone groups of aldehydes and ketones
   b) carbonyl groups of aldehydes and ketones
   c) aldehydes only
   d) ketones only

8. Which of the following compounds gives a positive reaction to Brady's, Tollens', and Schiff's Tests above?
   a) benzaldehyde
   b) cylcopentanone
   c) acetone

9. Of the following compounds, which does not react in any of the four above tests (Brady's, Tollens', Schiff's, and Iodoform Tests):
   a) an ethyl ketone
   b) cinnamaldehyde
   c) ethyl benzoate
   d) cyclohexanone

10. What must be done to the unused Tollens' reagent and any unreacted Tollens' test samples:
   a) rinse the reagent into the General Organic waste container
   b) dilute with water
   c) add concentrated nitric acid to decompose the Tollens' reagent
   d) leave reagent in the hood for someone else to find

CHEM360 Experiment 15 Report

Date:_____

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

Title:

Objective(s):

Reaction Equations:

Introduction:

Procedure:

(Ref:)
Changes/Modification:

Table 15.1. Table of Reagents for Experiment 15.

Reagent	Formula	Mwt.	d	mp	bp	Haz. Properties
Benzaldehyde	C_6H_5CHO	106.12	1.044	-26	179.5	
4-methylbenzaldehyde	$CH_3C_6H_4CHO$	120.15	1.019		204-205	
4-methoxybenzaldehyde	$CH_3OC_6H_4CHO$	136.15	1.119	-1	248	
trans-cinnamaldehyde	$C_6H_5CHCHCHO$	132.16	1.048		248	
Acetone	CH_3COCH_3	58.08	0.791	-94	56	
Cyclopentanone	C_5H_8 (=O)	84.12	0.951	-51	130-131	
Cyclohexanone	C_6H_{10} (=O)	98.15	0.947	-47	155	
4-methylcyclohexanone	$CH_3C_6H_9$ (=O)	112.17	0.914		169-171	
Formaldehyde	HCHO	30.03	1.083			
Acetophenone	$C_6H_5COCH_3$	120.15	1.030	19-20	202	
1-butanol	$CH_3(CH_2)_3OH$	74.12	0.810	-90	117.7	
2-butanol	$C_2H_5CH(OH)CH_3$	74.12	0.807		99-100	
Methanol	CH_3OH	32.04	0.791	-98	64.7	
Brady's Reagent	Solution			See hydrazine, 2,4-dinitrophenyl		
2,4-dinitrophenyl hydrazine	$(O_2N)_2C_6H_3NHNH_2$	198.14				
Sulfuric acid, conc. (18 M)	H_2SO_4	98.08	1.840			
Ethanol, 95%	CH_3CH_2OH	46.07	0.785		78.5	
Tollen's Reagent	Solution		See ammonia + silver nitrate			
Schiff's Reagent	Solution		mixture of roseaniline hydrochloride and sulfur dioxide, Toxic			
Ammonium hydroxide	NH_4OH	35.05	0.90			
Silver nitrate	$AgNO_3$, 0.3 M	169.87	4.352	212		
Nitric acid	HNO_3	63.01				
Sodium hydroxide	NaOH, 3 M	40.00	~1.00			
Iodine in potassium iodide	I_2 in KI					

Experiment 15 Part A Results:

Brady's Test 2,4-dinitrophenylhydrazine			
Test Substance	**Observation**	**Inference**	**Equation**
Aldehyde =			
Ketone =			
Positive control			
Negative control			

Part B Results:

Tollen's Test - Silver Mirror			
Test Substance	**Observation**	**Inference**	**Equation**
Aldehyde =			
Ketone =			
Positive control			
Negative control			

Part C Results:

Schiff's Test			
Test Substance	**Observation**	**Inference**	**Equation**
Formaldehyde			
Aldehyde =			
Ketone =			

Iodoform Test			
Test Substance	**Observation**	**Inference**	**Equation**
Acetone			
Cyclohexanone			
Acetophenone			
1 butanol			
2-butanol			

Conclusion:

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

Experiment 15 Post Lab Questions:

1. Write a balanced equation for the reaction of acetaldehyde (i.e. ethanal) with ammoniacal silver nitrate. Remember that this is a redox reaction.

2. Outline a systematic functional group test procedure that would enable you to distinguish among hexanal, 2-hexanone, 3-hexanone, 2-hexanol, and cyclohexanol.

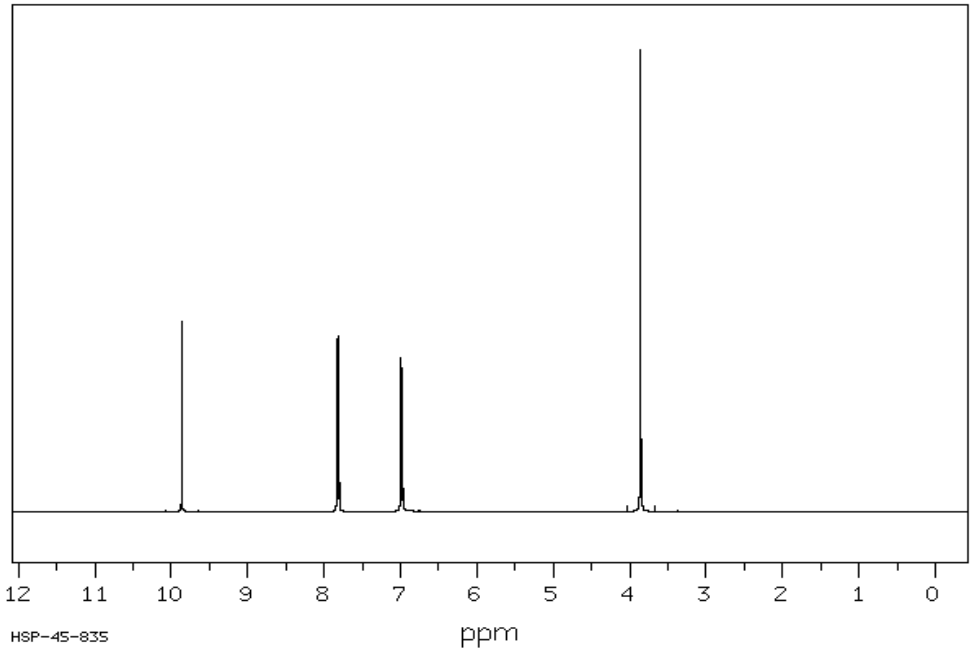
3. Aldehydes and ketones can also be easily distinguished by their infrared spectra and their identity deduced from their ^{1}H-NMR spectra. Explain why this is.

4. From the following results, identify the unknown compounds.

   a) Compound A: 2,4-DNPH positive, Tollens Test positive, Schiff's test positive, Iodoform negative (see Spectrum (A) next page).

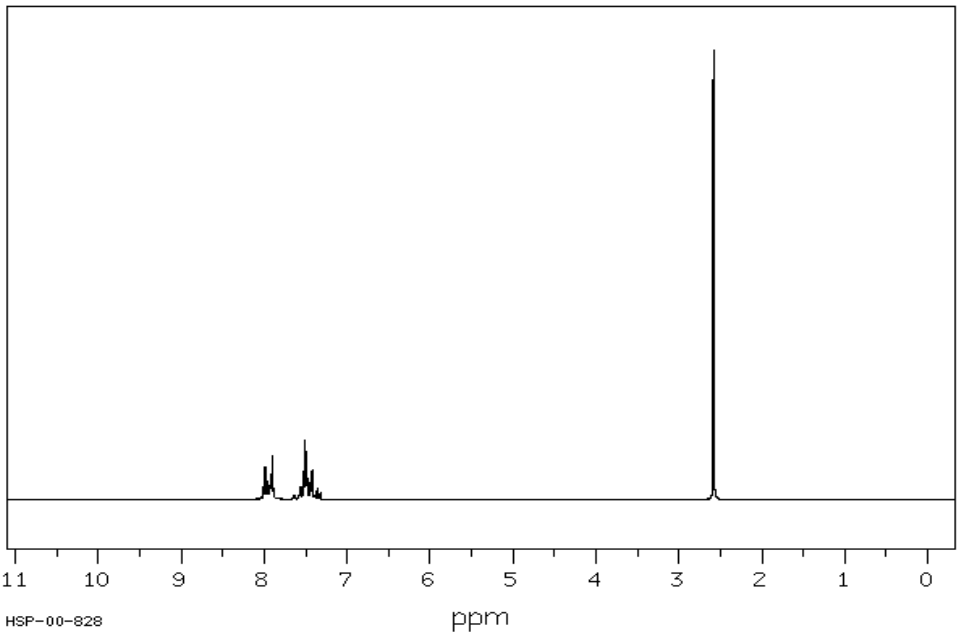
   b) Compound B: 2,4-DNPH positive, Tollens Test negative, Schiff's test negative, Iodoform positive (see Spectrum (B) next page).

Spectrum (A): ^{1}H-NMR, 400 MHz in $CDCl_3$



Molecular Formula $C_8H_8O_2$, δ 9.9 = 1H, δ 7.8 = 2H, δ 7.0 = 2H, δ 3.9 = 3H.

Spectrum (B): ^{1}H-NMR, 90 MHz in $CDCl_3$



Molecular Formula C_8H_8O , δ 7.9 = 2H, δ 7.3-7.7 = 3H, δ 2.6 = 3H.

CHEM360 Experiment 16 Report Date:_____

Student Name:_____ ID Number:_____

Experiment 16 Prelab Questions

Lab Safety

1. A procedureal flowchart is **highly recommended** for performing this experiment.
   a) true
   b) false

2. No source of flame is allowed in this experiment because of the use of diethyl ether.
   a) true
   b) false

Equipment Preparation

3. Dry glassware is essential for the Grignard reaction to work because:
   a) the Grignard reaction is easily contaminated with water
   b) the Grignard reagent is very moisture sensitive
   c) the Grignard reagent is very stable

Reagent Preparation

4. What is the purpose of the magnesium and diethyl ether used in the Grignard Reaction?
   a) magnesium reacts with bromobenzene to form the alcohol product, triphenylmethanol, and diethyl ether is the solvent which keeps the product in solution
   b) magnesium reacts with ethyl benzoate to form the Grignard reagent, and diethyl ether is the solvent for the reaction, which also helps to stabilize the Grignard reagent
   c) magnesium reacts with bromobenzene to form the Grignard reagent, and diethyl ether is the solvent for the reaction, which also helps to stabilize the Grignard reagent

Reaction

5. Which of the following are the limitations of the Grignard Reaction?
   a) the Grignard reagent is only useful in preparing tertiary alcohols, and it is moisture sensitive
   b) the Grignard reagent can only be formed from certain organohalides, and it is moisture, and oxygen sensitive
   c) the Grignard reagent can only be formed from certain organohalides, and it is oxygen sensitive

Reaction Workup

6. What is the purpose of using 50mL 2M H_2SO_4 in Part C Step 1?
   a) To dilute and lower the pH of the mixture and thereby prevent product precipitation
   b) To protonate the alcohol moiety of the final product
   c) To prevent the decomposition of the Grignard reagent

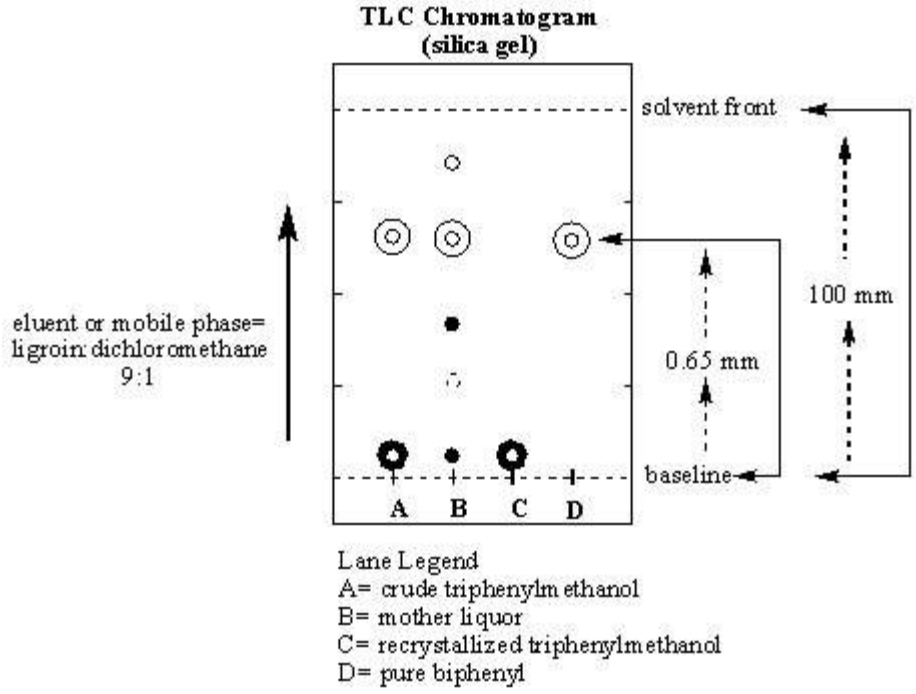
7. What layer will your product be in after addition of the diethyl ether in Part C Step 3?
   a) the aqueous layer
   b) the ether layer

8. What is the purpose of washing the organic layer with water in Part C Step 6?
   a) to remove water soluble impurities
   b) to remove organic impurities
   c) to extract the product into the aqueous layer
   d) to 'pre-dry' the organic layer, thereby removing the bulk of the water from the ether

9. What is the purpose of washing the organic layer with brine in the same Part C Step 6?
   a) To remove water soluble impurities
   b) To remove organic impurities
   c) To 'pre-dry' the organic layer, thereby removing the bulk of the water from the ether
   d) To remove all water from the organic layer

Product Characterization

10. What is the Retention Factor or R_f of pure biphenyl in Lane D in the TLC sketch shown below:
   a) 1.54
   b) 0.65
   c) 0.154
   d) 0.065



CHEM360 Experiment 16 Report Date:_____

Student Name:_____ ID Number:_____

Title:

Objective(s):

Reaction Equation(s):

Introduction:

Procedure:

(Ref:)
Changes/Modification:

A. Procedure for formation of Grignard Reagent.

Procedural Step	Observations/Comments/Inferences

B. Procedure for the reaction of the Grignard Reagent with ethyl benzoate to form Tiphenylmethanol.

Procedural Step	Observations/Comments/Inferences

C. Procedure for isolation of Triphenylmethanol.

Procedural Step	Observations/Comments/Inferences

D. Procedure for the observation of the carbocation.

Procedural Step	Observations/Comments/Inferences

E. Procedure for assessment of product purity by Thin Layer Chromatography.

Procedural Step	Observations/Comments/Inferences

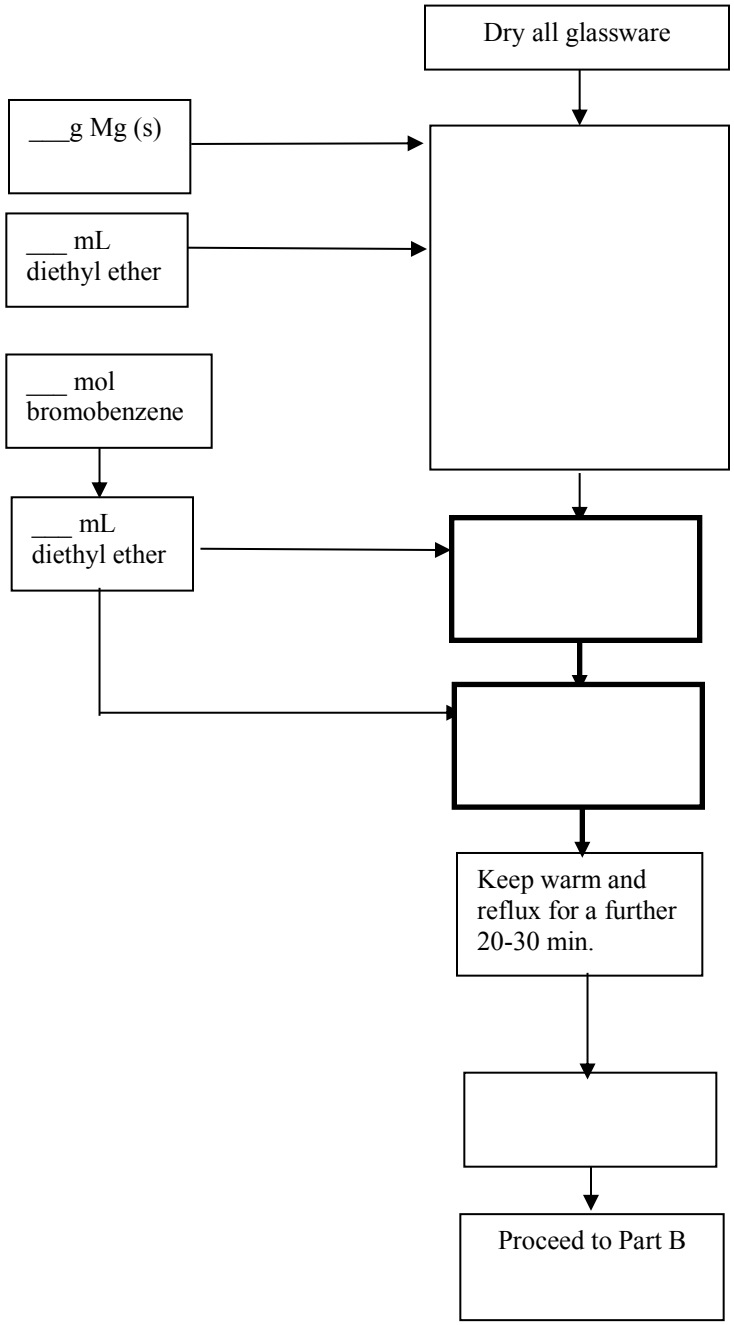
Procedure: (cont.)

Table 16.1. Table of Reagents for Experiment 16.

Reagent	Formula	Mwt.	d	mp	bp	Haz. Properties
Bromobenzene	C_6H_5Br					
Magnesium		24.31	1.75	649	1090	
Ethyl benzoate	$C_6H_5CO_2C_2H_5$					
Diethyl ether	$C_2H_5OC_2H_5$	74.12	0.7138	-116	34.5	
Sulfuric acid, 2M		98.07				
Sodium H carbonate		84.01	2.159	270		
Sodium sulfate, anhyd		142.04	2.68			
Dichloromethane		84.93	1.325	-95.1	40.1	
Biphenyl	$(C_6H_5)_2$	154.21	0.992	71	255.9	
Ligroin	(high bp pet. ether) alkane mixt.		0.656		60-80	
Iodine		253.8	4.930	133		
Methanol	CH_3OH	32.04	0.7914	-94	65	
Ethanol	C_2H_5OH	46.07	0.7893	-117	78.5	
Triphenylmethanol	$(C_6H_5)_3COH$					

SAMPLE EXPERIMENT 16 FLOW CHART Part A

REAGENT **PROCEDURE / STEP** **OBSERVATION**



SAMPLE EXPERIMENT 16 FLOW CHART Part B and C

REAGENT	**PROCEDURE / STEP**	**OBSERVATION**

Experiment 16 Results:

Table 16.2. Summary Table of Observations (this table is optional. Use only to tidy up your observations from the previous page if necessary.):

Procedural Step	Comment or Observation

Table 16.3. Table of Product Data for Triphenylmethanol, the Grignard Reaction Product.

Table 16.3. 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.

Product Name	Yield (Mass in g)	Appearance of Solid	Observed Melting Pt.* (°C)	Lit. Melting Pt. (°C)	Theoretical Yield (g)	% Yield

*uncalibrated thermometer used.

Limiting Reagent and Theoretical Yield Calculation:

Moles of magnesium used in the reaction =

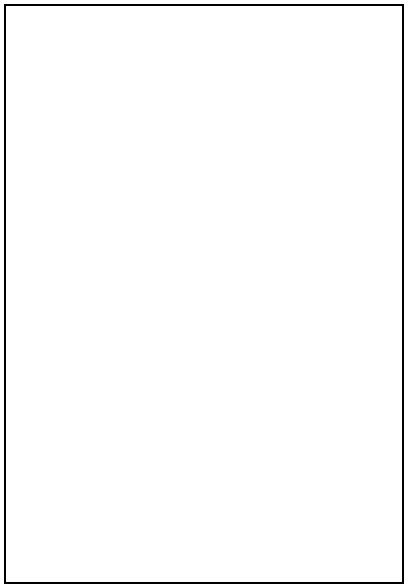
Moles of bromobenzene used in the reaction =

Moles of ethyl benzoate used in the reaction =

Theoretical Yield of triphenylmethanol =

% Yield Calculation:

Fig 16.1. TLC Analysis of Biphenyl, Mother Liquor, Crude and Recrystallized Triphenylmethanol



Lane#	Dist to Center Spot ( )	Dist to Solvent Front ( )	Rf

Discussion:

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

Conclusion:

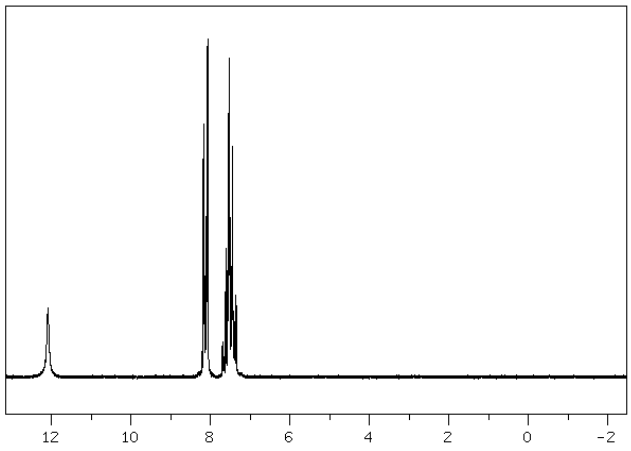
Structure of Product

Experiment 16 Post Lab Questions:

1. How do you account for the fact that biphenyl is formed as a by-product in this reaction?

2. Why do you think that reactions involving Grignard reagents are sometimes carried out in an atmosphere of nitrogen or argon?

3. A Grignard reaction was performed and the following ^{1}H-NMR (90 MHz in $CDCl_3$) was obtained of the purified product. Deduce the product's structure (Molecular Formula = $C_7H_6O_2$). Also write the overall reaction for its formation from any organohalide and carbonyl compound.



^{1}H-NMR Spectral Data:

Signal #	Shift	Integrat'n	Splitting	Comment	#Neighbour H	Signal Assignment
1	δ 7.5	3H				
2	δ 8.1	2H				
3	δ 12.1	1H		Xchngs with D_2O		

CHEM360 Experiment 17 Report Date:_____

Student Name:_____ ID Number:_____

Experiment 17 Prelab Questions

Lab Safety

1. In Part A, the reduction of 4-nitrotoluene, a dangerous gas, HCl, must be trapped using a:
   a) vacuum take off adaptor
   b) sodium hydroxide acid-vapour gas trap
   c) calcium chloride

Equipment Preparation

2. In Part C, Step 8, the reaction mixture must be cooled in an ice bath prior to suction filtration because:
   a) the product is least soluble in ice cold water
   b) it will prevent dangerous side reactions from occuring
   c) it will stop the reaction between sodium hydrogen sulfite and manganese dioxide

Reagent Preparation

3. In Part C, Step 3, the reason for splitting the potassium permanganate into 10 equal portions is:
   a) the potassium permanganate is not very soluble in hot water and therefore cannot be all added at once
   b) the potassium permanganate is to be carefully added in portions so as to control the rate of the oxidation reaction
   c) to test the student's ability to follow procedures

Reaction

4. In Part C, the oxidation of 4'-methylacetanilide, the oxidizing agent is:
   a) magnesium sulphate heptahydrate
   b) 4'methylacetanilide
   c) potassium permanganate
   d) 4-acetamidobenzoic acid

5. In Part E, what is the purpose of the 100% ethanol used in Step 2.
   a) it is the solvent for the Fisher esterification reaction
   b) it is both the solvent and co-substrate for the Fisher esterification reaction
   c) since benzocaine is highly soluble in ethanol, it is used so that the final product will not precipitate from the reaction mixture

Reaction Workup

6. In Part D, the 4-acetamidobenzoic acid is hydrolyzed by the addition of _____?
   a) dilute hydrochloric acid
   b) water
   c) water and 6.0 M hydrochloric acid

7. In Part D, what is hydrolyzed, i.e., what is removed from the starting reagent to form the 4-aminobenzoic acid product?
   a) acetic acid
   b) ethanol
   c) water

8. During a reflux, what constant states are maintained while the reaction proceeds? (there are 3)
   a) homogeneity, temperature, and volume of the solvent
   b) homogeneity, temperature, and volume of the starting reagent
   c) moles of reagent, moles of product, and temperature of the reaction

Product Characterization

9. Why should you not bother to perform a melting point on 4-acetamidobenzoic acid?
   a) the melting point of this compound will be to low to record accurately
   b) the compound is very unstable and cannot be heated
   c) the melting point of this compound will be to high to read for the thermometers provided in this course to read
   d) the melting point of this compound is unknown

10. If you obtained % yields of 85%, 65% and 90% in a three-step synthesis, what is the overall % yield?
   a) 240%
   b) 50%
   c) 145%
   d) 18%

CHEM360 Experiment 17 Report Date:_____

Student Name:_____ ID Number:_____

Title:

Objective(s):

Reaction equation(s):

Introduction:

Introduction (cont.):

Procedure:

(Ref:)
Changes/Modification:

Part C. Procedure for the synthesis of 4-acetamidobenzoic acid.

Procedural Step	Observations/Comments/Inferences
Record amount/appearance of pure 4-methylacetanilide used Reaction/Equipment Setup Reaction Reaction Work-up	

Part D. Procedure for the synthesis of 4-aminobenzoic acid.

Procedural Step	Observations/Comments/Inferences
Record amount of pure 4-acetamidobenzoic acid used Reaction/Equipment Setup Reaction Reaction Work-up	

Part E. Procedure for the synthesis of benzocaine.

Procedural Step	Observations/Comments/Inferences
Record amount/appearance of pure *p*-aminobenzoic acid used Reaction/Equipment Setup Reaction Reaction Work-up	

Table 17.1. Table of Reagents for Experiment 17

Reagent	Formula	Mwt.	d	mp	bp	Haz. Properties
4-methylacetanilide						
magnesium sulfate -7H_2O	$MgSO_4$ -7H_2O					
potassium permanganate	$KMnO_4$					
sodium hydrogen sulfite	$NaHSO_3$					
4-acetamidobenzoic acid						
hydrochloric acid	HCl					
Ammonia (conc.)	NH_3					
acetic acid, glacial						
4-aminobenzoic acid						
Ethanol						
sulfuric acid	H_2SO_4					
sodium carbonate	Na_2CO_3					
ethyl-4-aminobenzoate						

SAMPLE EXPERIMENT 17 FLOW CHART Part C

REAGENT	**PROCEDURE / STEP**	**OBSERVATION**

SAMPLE EXPERIMENT 17 FLOW CHART Part D

REAGENT	**PROCEDURE / STEP**	**OBSERVATION**

SAMPLE EXPERIMENT 17 FLOW CHART Part E

REAGENT	**PROCEDURE / STEP**	**OBSERVATION**

Experiment 17 Results:

Table 17.2. Table Summarizing Observations (This table is optional. Use only to tidy up your observations from the previous page if necessary.**:**

Procedural Step	Observations/Comments/Inferences

Table 17.3.1 Properties of the 4-methylacetanilide Oxidation Product, **4-acetamidobenzoic acid**

Table 17.3.1 shows a summary of the results of the 4-acetamidobenzoic acid synthesis. The calculations for theoretical yield and percent yield are shown below the table. Note: _____ was the limiting reagent, since the only other reagent involved in the reaction, potassium permanganate was added in excess.

Name of Product	Mass (g)	Appearance of Solid	Melting Pt. (°C)	Theoretical Yield (g)	% Yield

Theoretical Yield Calculation:

% Yield Calculation:

Table 17.3.2 Properties of the 4-methylacetanilide Oxidation Product, **4-aminobenzoic acid**

Table 17.3.2 shows a summary of the results of the 4-aminobenzoic acid synthesis. The calculations for theoretical yield and percent yield are shown below the table. Note: _____ was the limiting reagent, since the only other reagent involved in the reaction, phosphoric acid, served as a catalyst.

Name of Product	Mass (g)	Appearance of Solid	Melting Pt. (°C)	Theoretical Yield (g)	% Yield

Theoretical Yield Calculation:

% Yield Calculation:

Table 17.3.3 Properties of Fisher Esterification Product, **benzocaine**

Table 17.3.3 shows a summary of the results of the benzocaine synthesis. The calculations for theoretical yield and percent yield are 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 Solid	Melting Pt. (°C)	Theoretical Yield (g)	% Yield

Theoretical Yield Calculation:

% Yield Calculation:

(Theoretical) Overall % Yield Calculation

Table 4.1 Tabulation of Characteristic Infrared and ^{1}H-NMR Absorptions for 4-acetamidobenzoic acid.

Infrared Data:

	Absorption Band#	Wavenumber (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, med. or weak)	Functional Group Indicated
> 3000 cm^{-1}					
Between 3000 and 2000 cm^{-1}					
Between 2000 and 1400 cm^{-1}					
< 1400 cm^{-1}					

Functional Group(s) absent:

^{1}H-NMR Data:

Signal #	Shift	Integrat'n	Splitting	Comment	#Neighbour H	Signal Assignment

Table 4.2 Tabulation of Characteristic Infrared and ^{1}H -NMR Absorptions for 4-aminobenzoic acid.

Infrared Data:

	Absorption Band#	Wavenumber (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, med. or weak)	Functional Group Indicated
> 3000 cm^{-1}					
Between 3000 and 2000 cm^{-1}					
Between 2000 and 1400 cm^{-1}					
< 1400 cm^{-1}					

Functional Group(s) absent:

^{1}H-NMR Data:

Signal #	Shift	Integrat'n	Splitting	Comment	#Neighbour H	Signal Assignment

Table 4.3 Tabulation of Characteristic Infrared and ^{1}H -NMR Absorptions for benzocaine.

Infrared Data:

	Absorption Band#	Wavenumber (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, med. or weak)	Functional Group Indicated
> 3000 cm^{-1}					
Between 3000 and 2000 cm^{-1}					
Between 2000 and 1400 cm^{-1}					
< 1400 cm^{-1}					

Functional Group(s) absent:

^{1}H-NMR Data:

Signal #	Shift	Integrat'n	Splitting	Comment	#Neighbour H	Signal Assignment

Discussion:

Comments on and reasons for yield (high or low), purity (high/med/low), sources of error, overall synthesis effectiveness, etc.

Conclusion:

Structure of Product

Experiment 17 Post Lab Questions:

1. In Part C, what is the purpose of adding magnesium sulfate to the reaction mixture?

2. In the discussion pertaining to the hydrolysis of 4-acetamidobenzoic acid, it was argued that the presence of the electron-withdrawing carboxyl group in the para position could result in the occurrence of some nucleophilic displacement if the hydrolysis was carried out under acidic conditions and an elevated temperature. What would the product of such a nucleophilic displacement reaction?

3. Write the balanced equation for the oxidation of 4'-methylacetanilide to 4-acetamidobenzoic acid as carried out in Part C of the synthesis.

4. In Part E, what is the purpose of adding 100% ethanol to the reaction mixture? (use equations if necessary to answer this question)

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	1.419		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 sens.
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

Compound Name	Chemical Formula	Solid (S) or Liquid (L)	Formula Weight	MP or BP (°C)	Density (g/mL)	Refract. Index	Hazardous Properties*
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
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
1-butanol, 3-methyl	$(CH_3)_2CH(CH_2)_2OH$	L	88.15	130	0.8092	1.4053	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
butyric acid	$CH_3(CH_2)_2CO_2H$	L	88.11	165.5	0.9577	1.3980	Corrosive, toxic
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	100.09		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, flammab.
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

Compound Name	Chemical Formula	Solid (S) or Liquid (L)	Formula Weight	MP or BP (°C)	Density (g/mL)	Refract. Index	Hazardous Properties*
iodine	I_2	S	253.81	133	4.930		Corrosive, highly toxic
isoamyl acetate (isopentyl acetate)	$CH_3CO_2C_5H_{11}$	L	130.19	142	0.8670	1.4000	Flammable, irritant
isoamyl alcohol	$(CH_3)_2CH(CH_2)_2OH$	L					(see 1-butanol, 3-methyl-)
isopentyl alcohol	$(CH_3)_2CH(CH_2)_2OH$	L					(see 1-butanol, 3-methyl-)
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		(92psom 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	164.3			Irritant
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
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
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.
propanoic acid (or propionic acid)	$CH_3CH_2CO_2H$	L	74.08	141	0.9930	1.3869	Corrosive, toxic
1-propanol	$CH_3CH_2CH_2OH$	L	60.11	97.4	0.8035	1.3850	Flammable, irritant
1-propanol, 2-methyl-	$(CH_3)_2CHCH_2OH$	L	74.12	108.1	0.8018	1.3955	Flammable, irritant
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 acetate, trihydrate	$CH_3CO_2Na\ 3H_2O$	S	136.08	58	1.45		Hygroscopic

Compound Name	Chemical Formula	Solid (S) or Liquid (L)	Formula Weight	MP or BP (°C)	Density (g/mL)	Refract. Index	Hazardous Properties*
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	1.5805		Tooth Decay!
sulfur dioxide	SO_2	Gas	64.06	-10 bp			Nonflamm, corrosive
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	164.3			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.
zinc chloride, anhydrous	$ZnCl_2$	S	136.28	283	2.91		Corrosive, toxic

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

Athabasca University

CHEM360/Labc360-Organic Chemistry II

PRELAB ANSWERS (2019-21)

http://science.pc.athabascau.ca/chem360.nsf

(Click on 'CHEM360 Prelab Questions' in the side menu)

Question	Exp.10	Exp. 11	Exp. 12	Exp. 13	Exp.14	Exp. 15	Exp. 16	Exp. 17
1	c	b	a	b	b	c	a	b
2	b	a	a	a	d	a	a	a
3	a	c	a	c	a	c	b	b
4	c	d	c	c	a	b	c	c
5	a	c	b	c	a	c	b	b
6	c	a	b	b	b	c	b	c
7	b	b	a	b	b	a	b	a
8	d	b	a	a	c	a	a	a
9	c	b	b	b	a	c	c	c
10	a	a	a	d	a	c	b	b

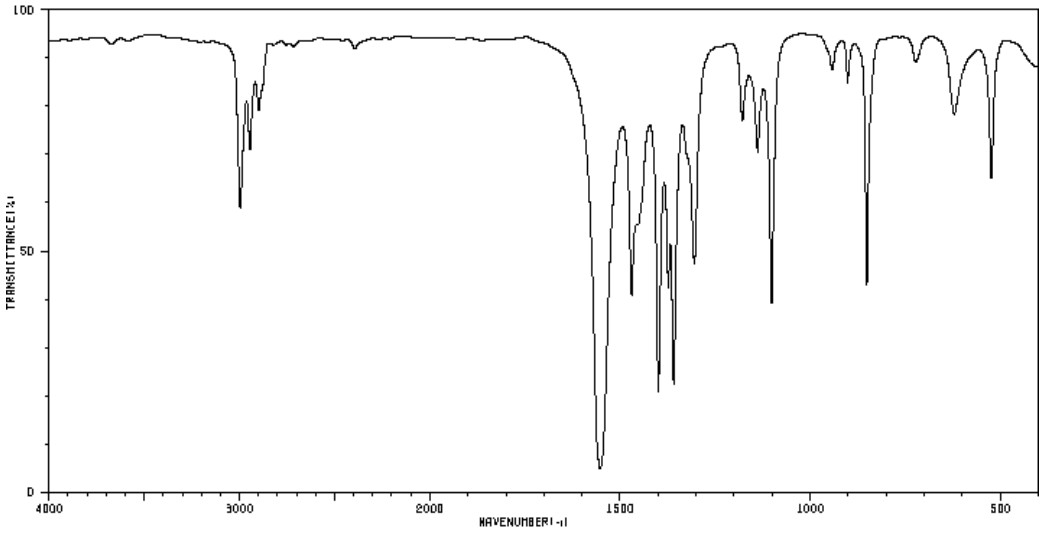
CHEM360 2019-21 **Exp.14**

Unknown # 360-14-1

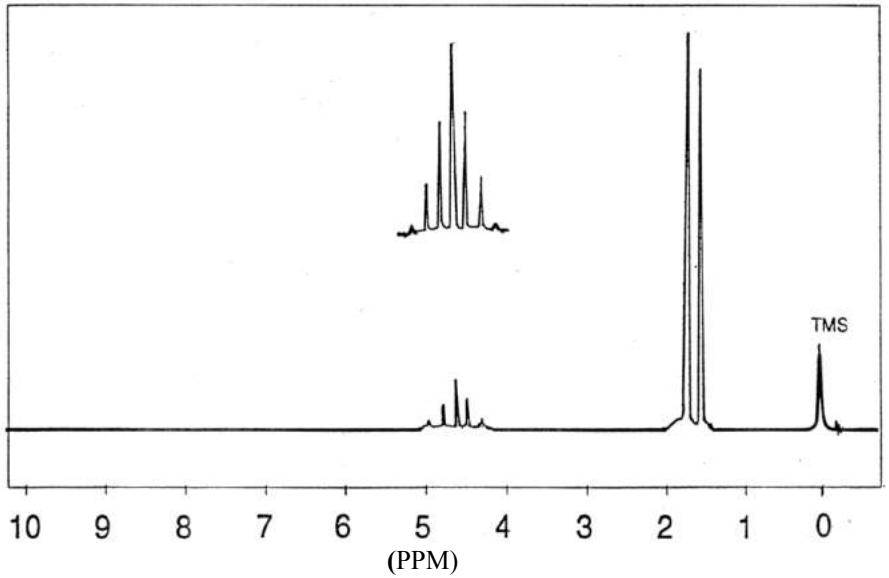
Mol. Wt. = 89.09 g/mol Chemical Formula = $C_3H_7O_2N$ Degrees of Unsaturation = _____

b.p. = 120°C

Infrared Spectrum (liquid film (neat) on KBr Disc)



^{1}H-NMR Spectra (at 60 MHz in $CDCl_3$)



Infrared Spectrum (liquid film (neat) on KBr disc):

Spectrum Region	Absorp'n Band #	Frequency (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, med. or weak)	Functional Group Indicated

^{1}H-NMR Integration Data: (at 60 MHz in $CDCl_3$)

Signal #	Shift	Integrat'n	Splitting	Comment	#Neigh-bouring H	Signal Assignment
1	δ 1.6	6H				
2	δ 4.65	1H				

Structure Assigned to Unknown 360-14-1

Compound Name:

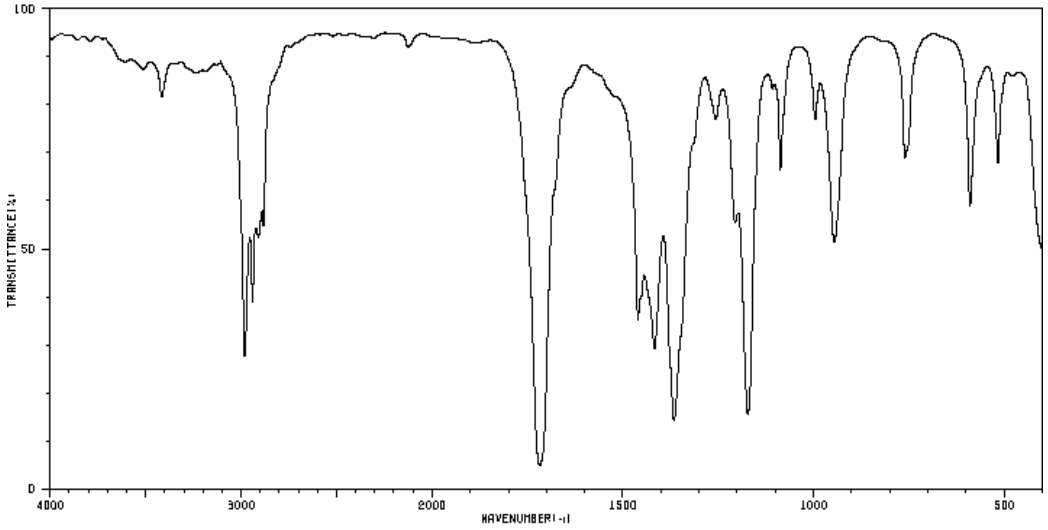
CHEM360 2019-21 **Exp.14**

Unknown # 360-14-2

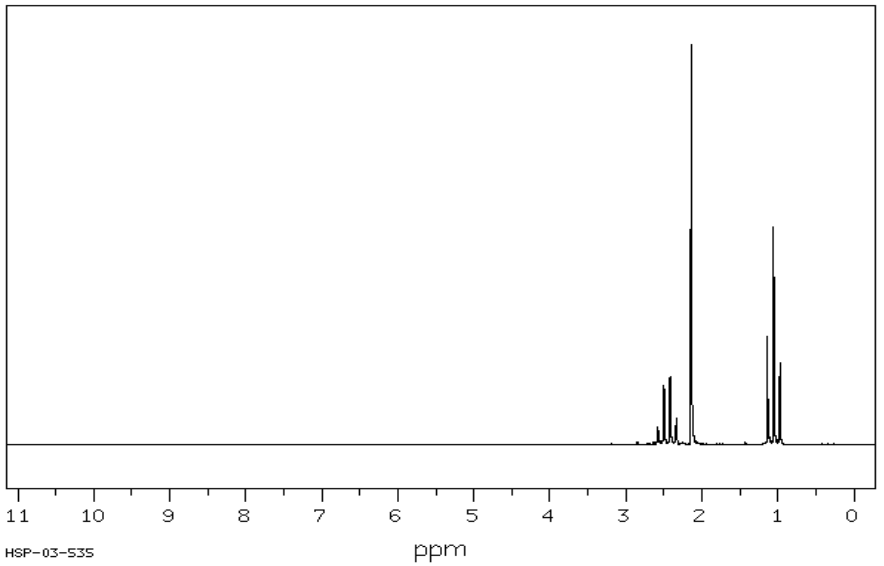
Mol. Wt. = 72.112 g/mol Chemical Formula = C_4H_8O Degrees of Unsaturation = _____

b.p. = 80°C

IR Spectra (liquid film (neat) on KBr Disc)



^{1}H-NMR Spectra (at 90 MHz in $CDCl_3$)



Infrared Spectra (liquid film (neat) on KBr disc):

Spectrum Region	Absorp'n Band #	Frequency (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, med. or weak)	Functional Group Indicated

^{1}H-NMR Integration Data: (at 90 MHz in $CDCl_3$)

Signal #	Shift	Integrat'n	Splitting	Comment	#Neigh-bouring H	Signal Assignment
1	δ 1.1	3H				
2	δ 2.1	3H				
3	δ 2.45	2H				

Structure Assigned to Unknown 360-14-2

Compound Name:

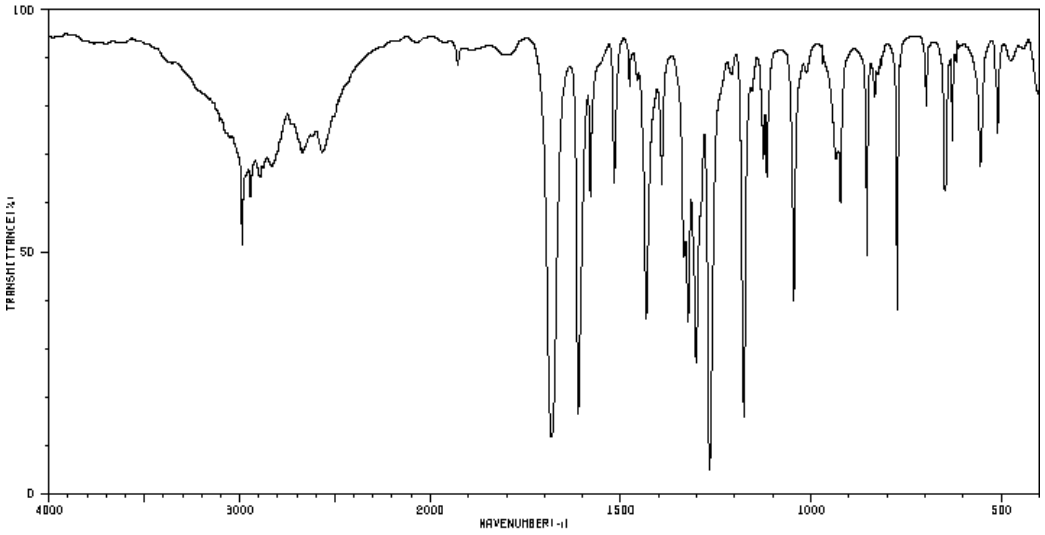
CHEM360 2019-21 **Exp.14**

Unknown # 360-14-3

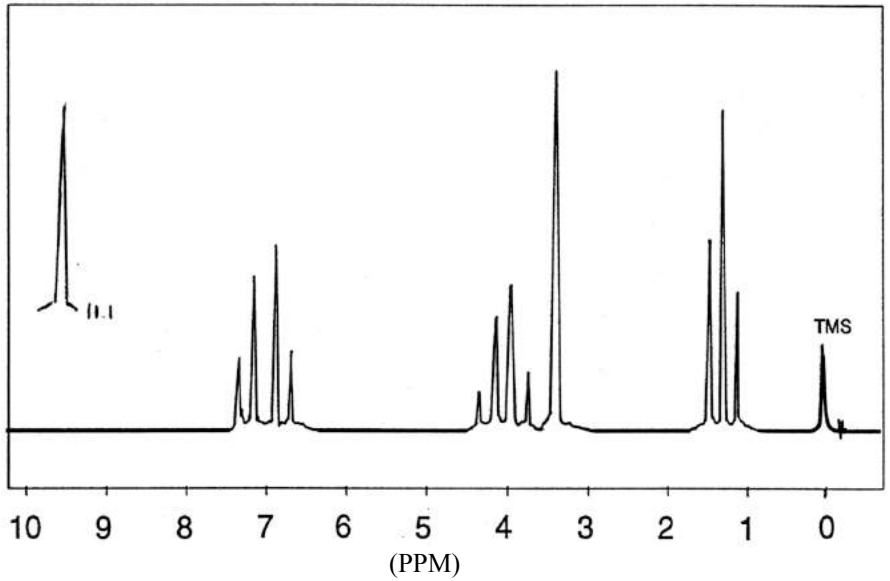
Mol. Wt. = 180.2 g/mol Chemical Formula = $C_{10}H_{12}O_3$ Degrees of Unsaturation = _____

m.p. = 90°C

Infrared Spectrum (liquid film (neat) on KBr Disc)



^{1}H-NMR Spectra (at 60 MHz in $CDCl_3$)



Infrared Spectrum (liquid film (neat) on KBr disc):

Spectrum Region	Absorp'n Band #	Frequency (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, med. or weak)	Functional Group Indicated

^{1}H-NMR Integration Data: (at 60 MHz in $CDCl_3$)

Signal #	Shift	Integrat'n	Splitting	Comment	#Neigh-bouring H	Signal Assignment
1	δ 1.3	3H				
2	δ 3.6	2H				
3	δ 4.0	2H				
4	7.0	4H				
5	δ 11.1	1H				

Structure Assigned to Unknown 360-14-3

Compound Name:

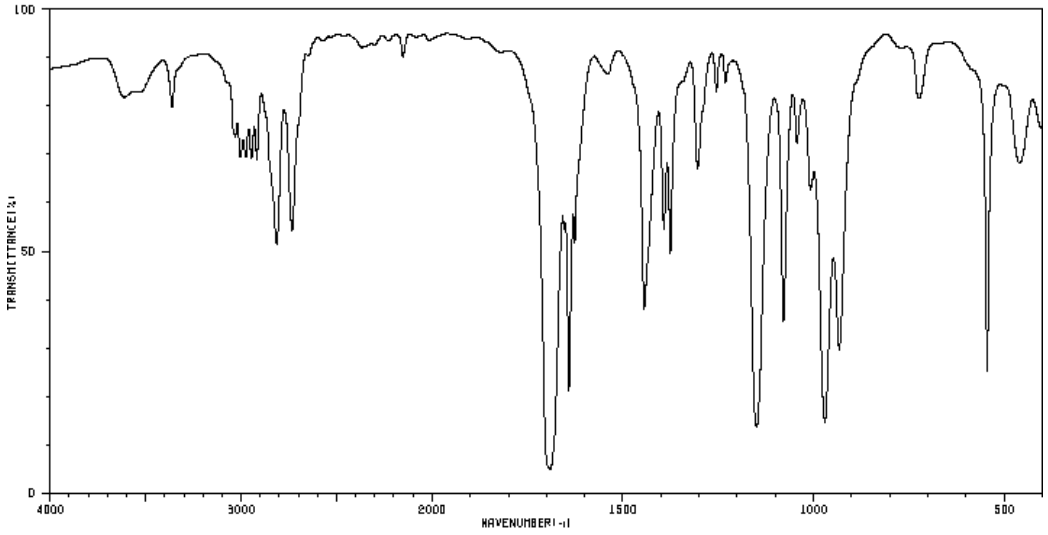
CHEM360 2019-21 **Exp.14**

Unknown # 360-14-4

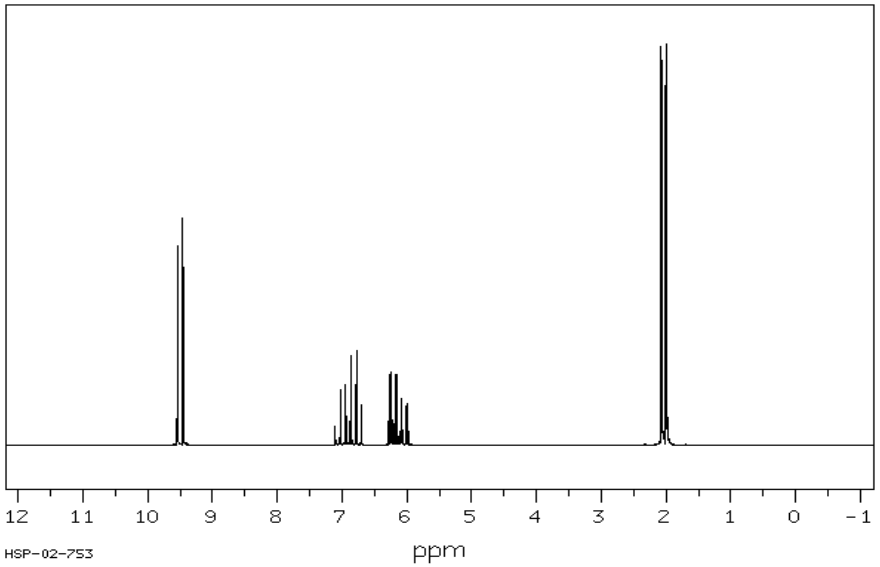
Mol. Wt. = 70.09 g/mol Chemical Formula = C_4H_6O Degrees of Unsaturation = _____

b.p. = 104°C

IR Spectra (liquid film (neat) on KBr Disc)



^{1}H-NMR Spectra (at 90 MHz in $CDCl_3$)



Infrared Spectra (liquid film (neat) on KBr disc):

Spectrum Region	Absorp'n Band #	Frequency (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, med. or weak)	Functional Group Indicated

^{1}H-NMR Integration Data: (at 90 MHz in $CDCl_3$)

Signal #	Shift	Integrat'n	Splitting	Comment	#Neigh-bouring H	Signal Assignment
1	δ 2.0	3H				
2	δ 6.15	1H				
3	δ 6.9	1H				
4	δ 9.5	1H				
Hint: J(2,4)=7.8Hz, J(2,3)=15.5Hz, J(1,3)=6.8Hz, J(1,2)=1.5Hz						

Structure Assigned to Unknown 360-14-4

Compound Name:

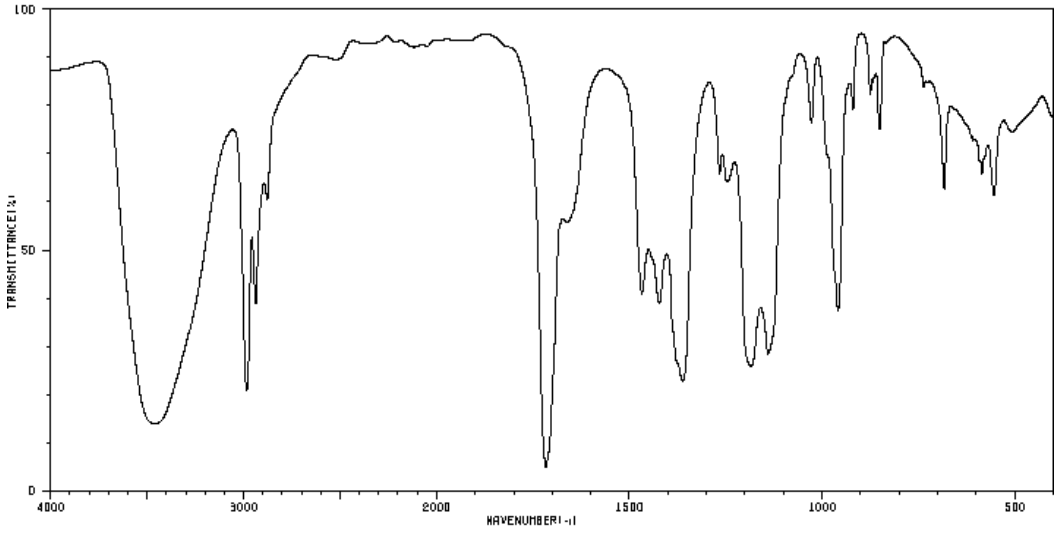
CHEM360 2019-21 **Exp.14**

Unknown # 360-14-5

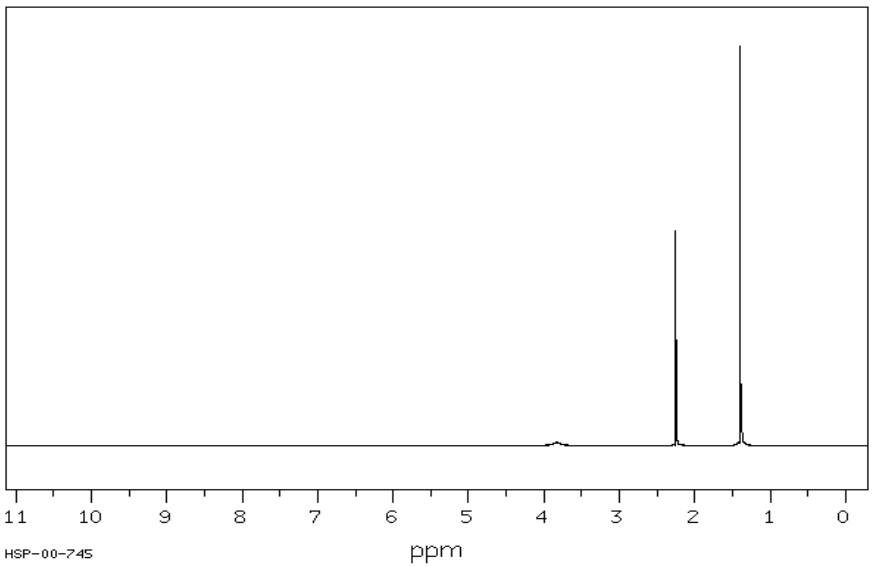
Mol. Wt. = 102.13 g/mol Chemical Formula = $C_5H_{10}O_2$ Degrees of Unsaturation = _____

b.p. = 141°C

IR Spectra (liquid film (neat) on KBr Disc)



^{1}H-NMR Spectra (at 90 MHz in $CDCl_3$)



Infrared Spectra (liquid film (neat) on KBr disc):

Spectrum Region	Absorp'n Band #	Frequency (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, med. or weak)	Functional Group Indicated

^{1}H-NMR Integration Data: (at 90 MHz in $CDCl_3$)

Signal #	Shift	Integrat'n	Splitting	Comment	#Neigh-bouring H	Signal Assignment
1	δ 1.4	6H				
2	δ 2.2	3H				
3	δ 3.8	1H		xchanges with D_2O		

Structure Assigned to Unknown 360-14-5

Compound Name:

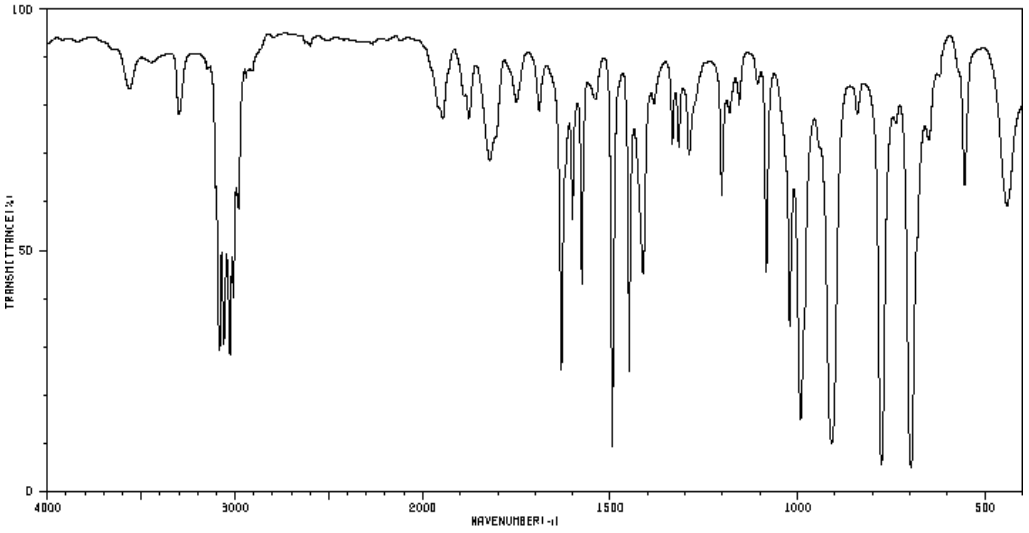
CHEM360 2019-21 **Exp.14**

Unknown # 360-14-6

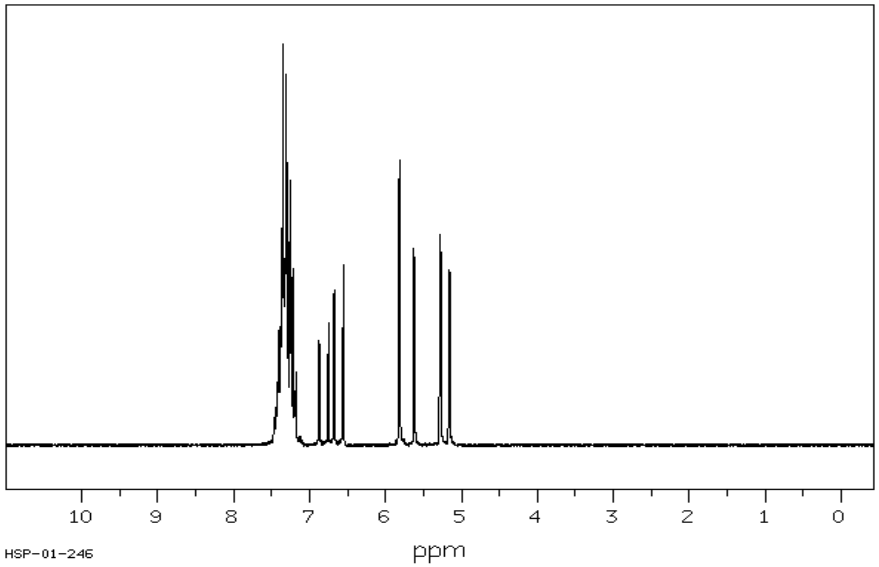
Mol. Wt. = 104.15 g/mol Chemical Formula = C_8H_8 Degrees of Unsaturation = _____

b.p. = 146°C

IR Spectra (liquid film (neat) on KBr Disc)



^{1}H-NMR Spectra (at 90 MHz in $CDCl_3$)



Infrared Spectra (liquid film (neat) on KBr disc):

Spectrum Region	Absorp'n Band #	Frequency (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, med. or weak)	Functional Group Indicated

^{1}H-NMR Integration Data: (at 90 MHz in $CDCl_3$)

Signal #	Shift	Integrat'n	Splitting	Comment	#Neigh-bouring H	Signal Assignment
1	δ 5.2	1H				
2	δ 5.7	1H				
3	δ 6.7	1H				
4	δ 7.3	5H				

Structure Assigned to Unknown 360-14-6

Compound Name:

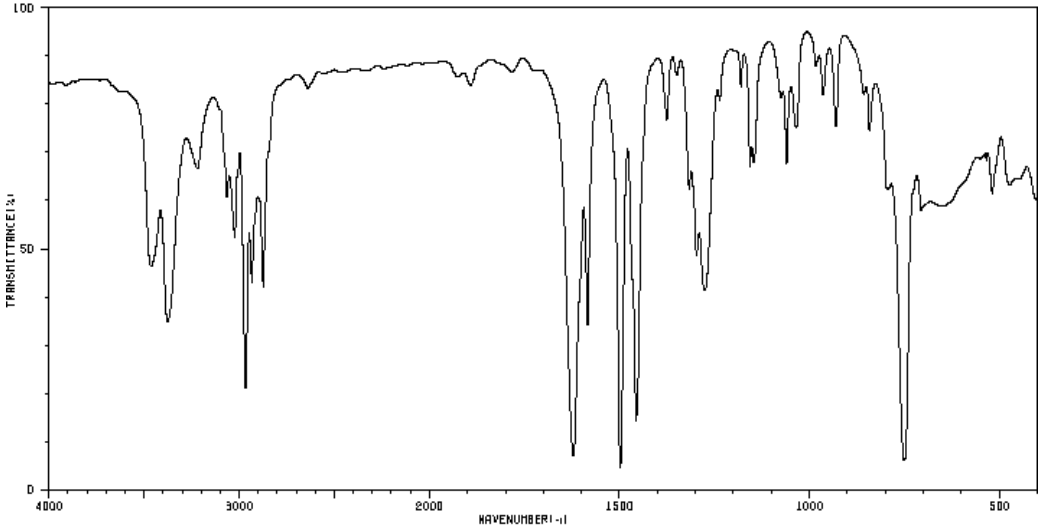
CHEM360 2019-21 **Exp.14**

Unknown # 360-14-7

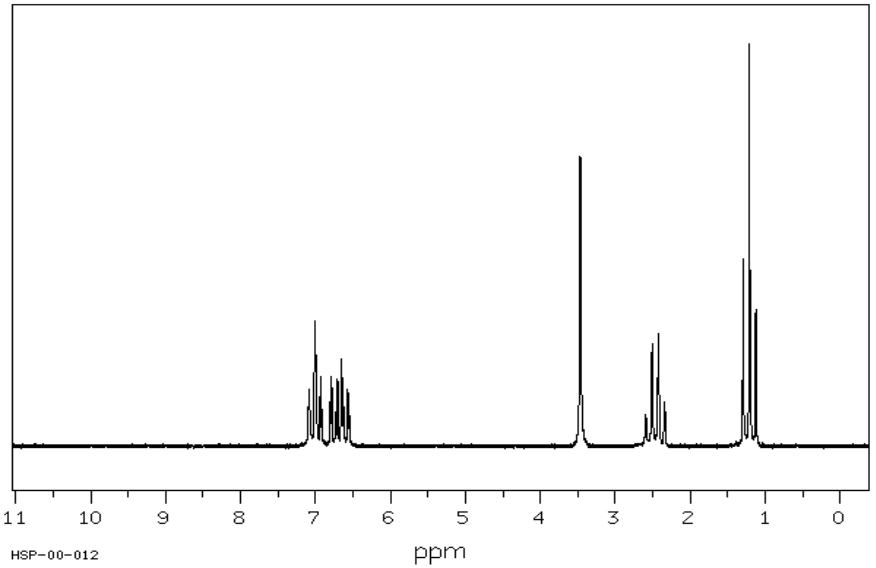
Mol. Wt. = 121.18 g/mol Chemical Formula = $C_8H_{11}N$ Degrees of Unsaturation = _____

b.p. = 210°C

IR Spectra (liquid film (neat) on KBr Disc)



^{1}H-NMR Spectra (at 90 MHz 10.5 mol% in $CDCl_3$)



Infrared Spectra (liquid film (neat) on KBr disc):

Spectrum Region	Absorp'n Band #	Frequency (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, med. or weak)	Functional Group Indicated

^{1}H-NMR Integration Data: (at 90 MHz 10.5 mol% in $CDCl_3$)

Signal #	Shift	Integrat'n	Splitting	Comment	#Neigh-bouring H	Signal Assignment
1	δ 1.2	3H				
2	δ 2.46	2H				
3	δ 3.47	2H		xchanges with D_2O		
4	δ 6.65	2H				
5	δ 7.0	2H				

Structure Assigned to Unknown 360-14-7

Compound Name:

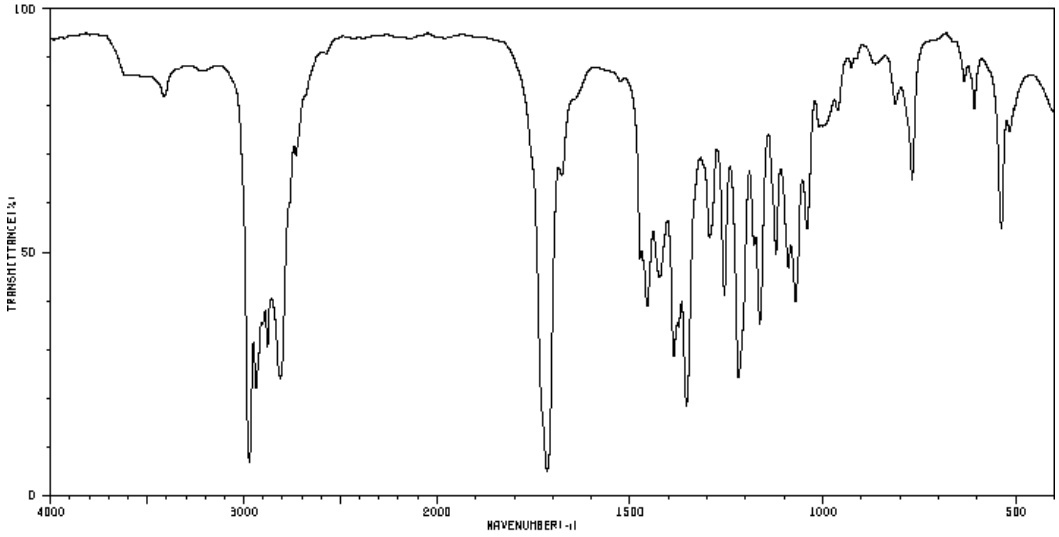
CHEM360 2019-21 **Exp.14**

Unknown # 360-14-8

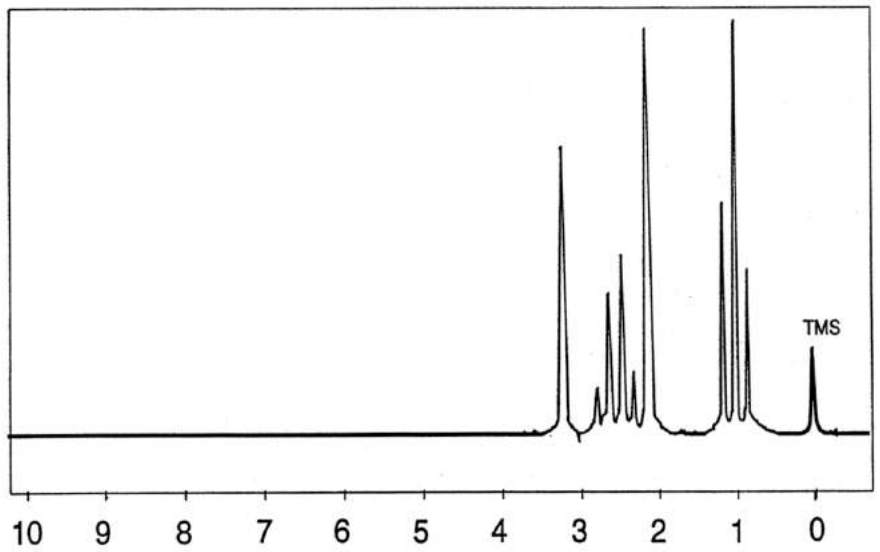
Mol. Wt. = 129.2 g/mol Chemical Formula = $C_7H_{15}NO$ Degrees of Unsaturation = _____

m.p. = 64°C (16mm)

Infrared Spectrum (liquid film (neat) on KBr Disc)



^{1}H-NMR Spectra (at 60 MHz in $CDCl_3$)



Infrared Spectrum (liquid film (neat) on KBr disc):

Spectrum Region	Absorp'n Band #	Frequency (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, med. or weak)	Functional Group Indicated

^{1}H-NMR Integration Data: (at 60 MHz in $CDCl_3$)

Signal #	Shift	Integrat'n	Splitting	Comment	#Neigh-bouring H	Signal Assignment
1	δ 1.0	6H				
2	δ 2.10	3H				
3	δ 2.55	4H				
4	δ 3.25	2H				

Structure Assigned to Unknown 360-14-8

Compound Name:

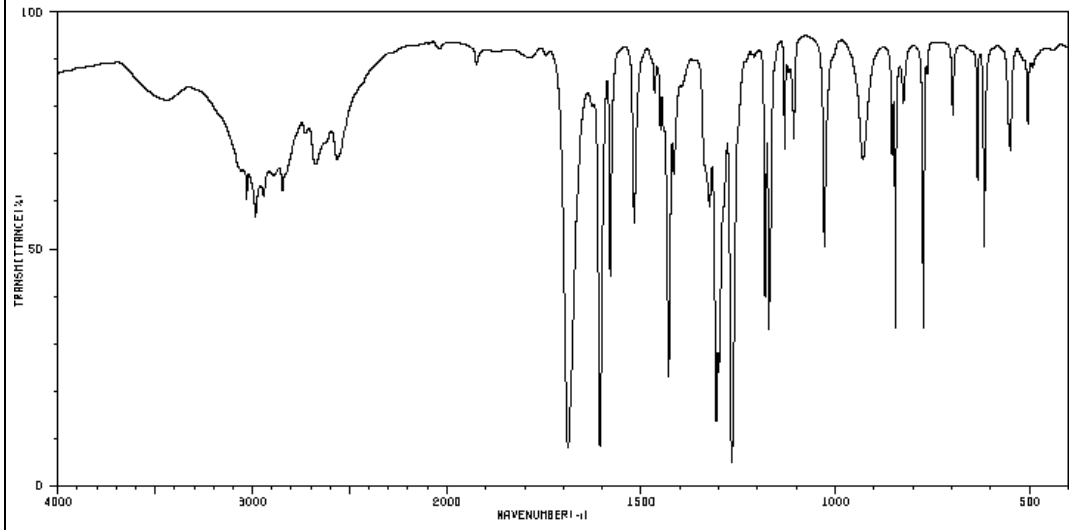
CHEM360 2019-21 **Exp.14**

Unknown # 360-14-9

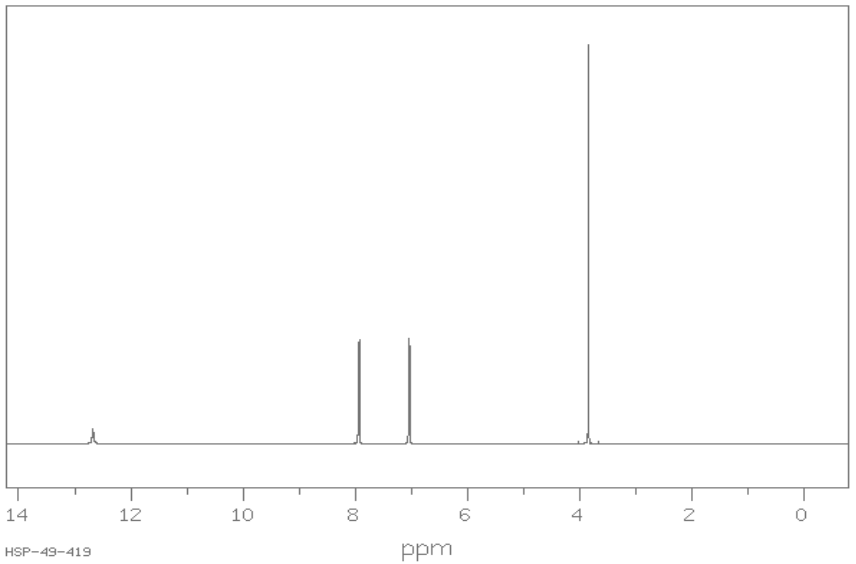
Mol. Wt. = 152.15 g/mol Chemical Formula = $C_8H_8O_3$ Degrees of Unsaturation = _____

m.p. = 182-185°C

IR Spectra (on KBr Disc)



^{1}H-NMR Spectra (at 400 MHz in DMSO-d_6)



Infrared Spectra (on KBr disc):

Spectrum Region	Absorp'n Band #	Frequency (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, med. or weak)	Functional Group Indicated

^{1}H-NMR Integration Data: (at 400 MHz in DMSO-d_6)

Signal #	Shift	Integrat'n	Splitting	Comment	#Neigh-bouring H	Signal Assignment
1	δ 3.8	3H				
2	δ 7.0	2H				
3	δ 7.9	2H				
4	δ 12.7	1H				

Structure Assigned to Unknown 360-14-9

Compound Name:

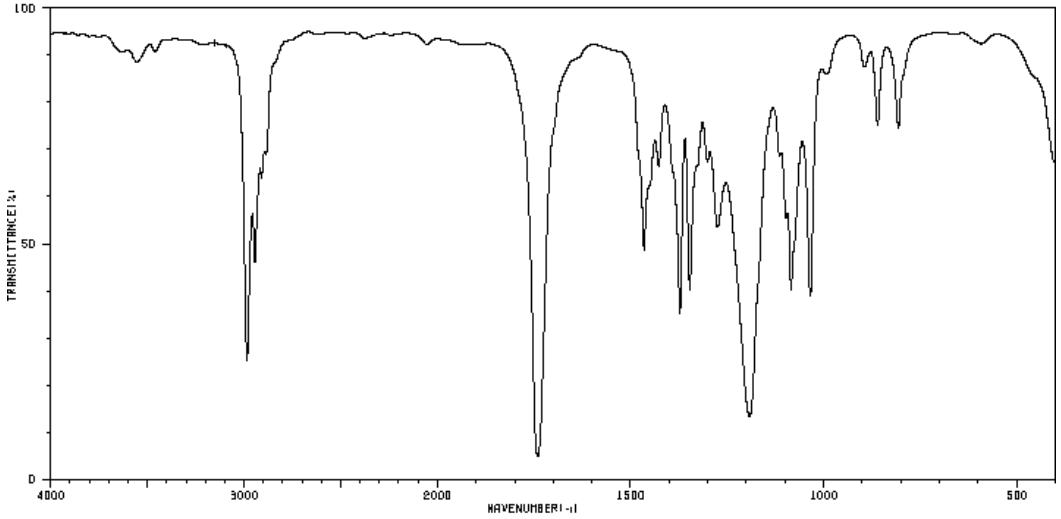
CHEM360 2019-21 **Exp.14**

Unknown # 360-14-10

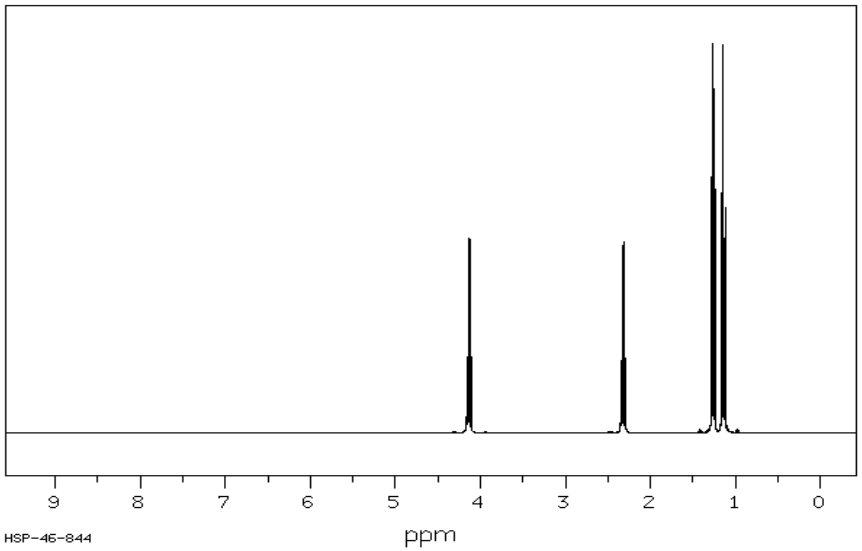
Mol. Wt. = 102.13 g/mol Chemical Formula = $C_5H_{10}O_2$ Degrees of Unsaturation = _____

b.p. = 99°C

IR Spectra (thin film (neat) on KBr Disc)



^{1}H-NMR Spectra (at 400 MHz in $CDCl_3$)



Infrared Spectra (thin film (neat) on KBr disc):

Spectrum Region	Absorp'n Band #	Frequency (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, med. or weak)	Functional Group Indicated

^{1}H-NMR Integration Data: (at 400 MHz in $CDCl_3$)

Signal #	Shift	Integrat'n	Splitting	Comment	#Neigh-bouring H	Signal Assignment
1	δ 1.1	3H				
2	δ 1.3	3H				
3	δ 2.3	2H				
4	δ 4.2	2H				

Structure Assigned to Unknown 360-14-10

Compound Name:

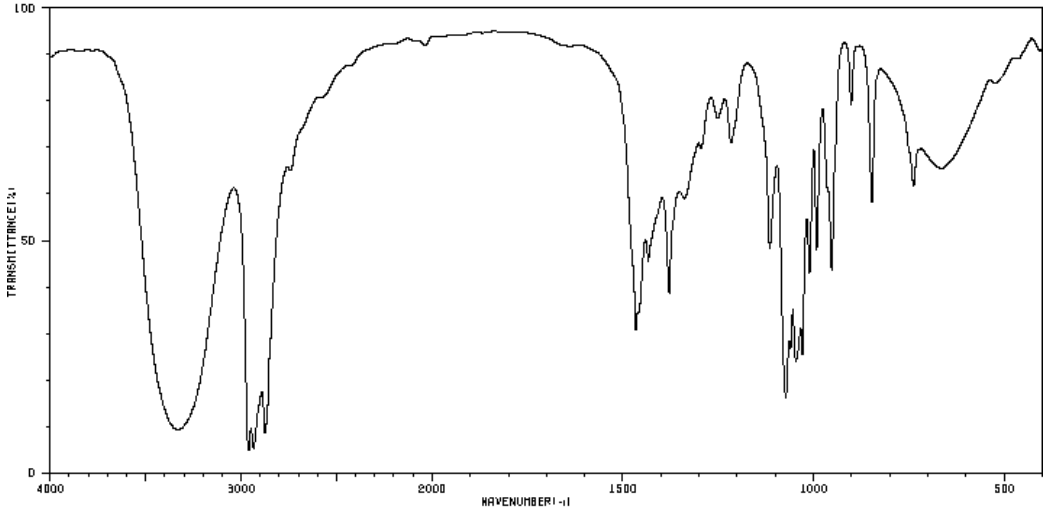
CHEM360 2019-21 **Exp.14**

Unknown # 360-14-11

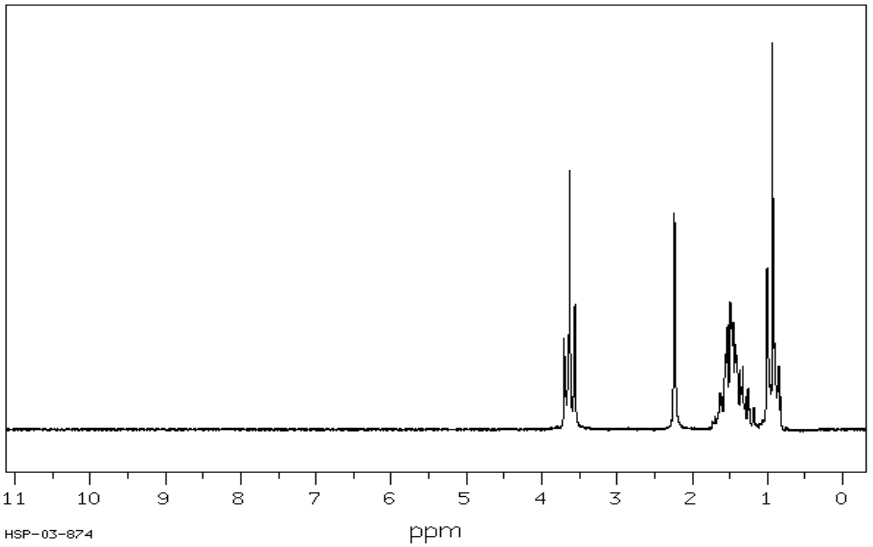
Mol. Wt. = 74.1 g/mol Chemical Formula = $C_4H_{10}O$ Degrees of Unsaturation = _____

b.p. = 117.7°C

IR Spectra (thin film (neat) on KBr Disc)



^{1}H-NMR Spectra (at 90 MHz in $CDCl_3$)



Infrared Spectra (thin film (neat) on KBr disc):

Spectrum Region	Absorp'n Band #	Frequency (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, med. or weak)	Functional Group Indicated

^{1}H-NMR Integration Data: (at 90 MHz in $CDCl_3$)

Signal #	Shift	Integrat'n	Splitting	Comment	#Neigh-bouring H	Signal Assignment
1	δ 0.9	3H				
2	δ 1.4	4H				
3	δ 2.2	1H		xchanges with D_2O		
4	δ 3.6	2H				

Structure Assigned to Unknown 360-14-11

Compound Name:

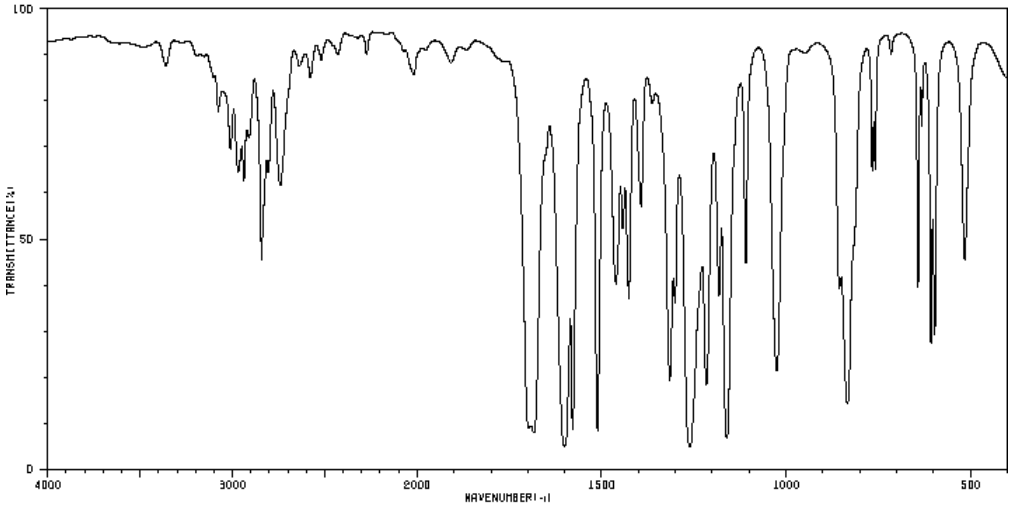
CHEM360 2019-21 **Exp.14**

Unknown # 360-14-12

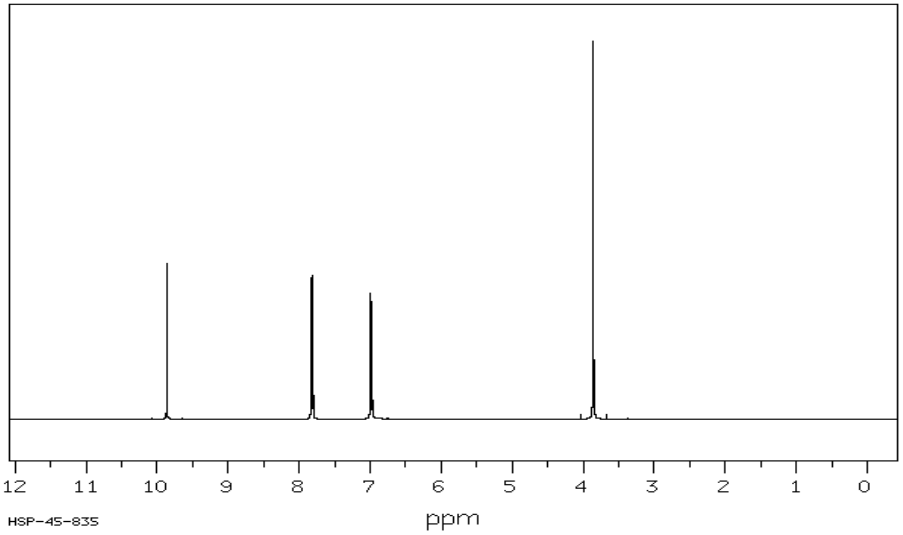
Mol. Wt. = 136.12 g/mol Chemical Formula = $C_8H_8O_2$ Degrees of Unsaturation = _____

b.p. = 230°C

IR Spectra (liquid film (neat) on KBr Disc)



^{1}H-NMR Spectra (at 400 MHz in $CDCl_3$)



Infrared Spectra (neat liquid film on KBr disc):

Spectrum Region	Absorp'n Band#	Frequency (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, med. or weak)	Functional Group Indicated

^{1}H-NMR Integration Data: (at 400 MHz in $CDCl_3$)

Signal #	Shift	Integrat'n	Splitting	Comment	#Neigh-bouring H	Signal Assignment
1	δ 3.86	3H				
2	δ 6.98	2H				
3	δ 7.82	2H				
4	δ 9.86	1H				

Structure Assigned to Unknown 360-14-12

Compound Name:

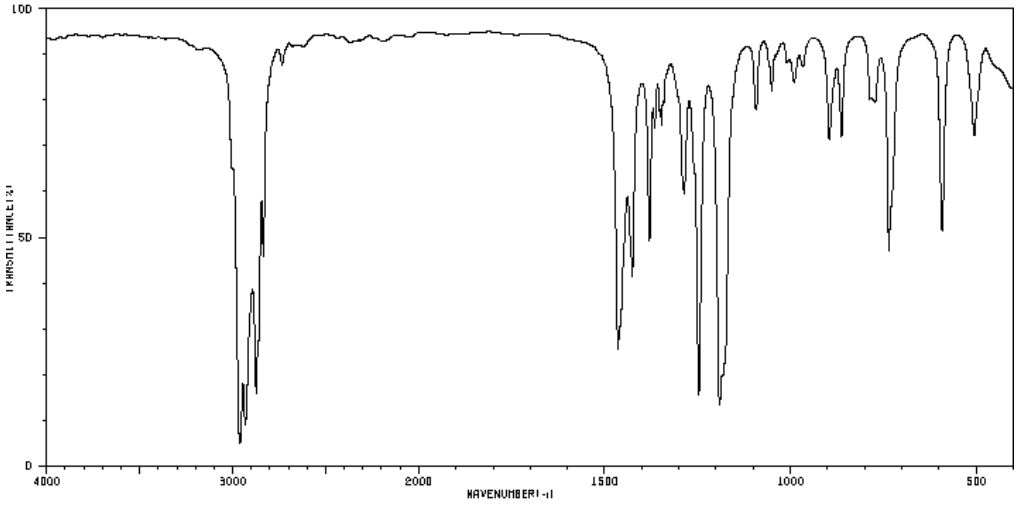
CHEM360 2019-21 **Exp.14**

Unknown # 360-14-13

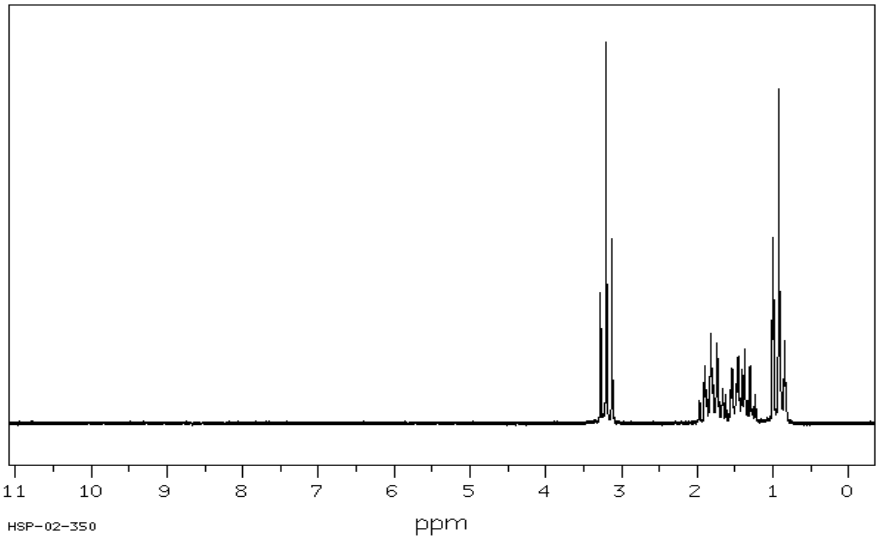
Mol. Wt. = 184.02 g/mol Chemical Formula = C_4H_9I Degrees of Unsaturation = _____

b.p. = 130.5°C

IR Spectra (liquid film (neat) on KBr Disc)



^{1}H-NMR Spectra (at 90 MHz in $CDCl_3$)



Infrared Spectra (neat liquid film on KBr disc):

Spectrum Region	Absorp'n Band#	Frequency (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, med. or weak)	Functional Group Indicated

^{1}H-NMR Integration Data: (at 90 MHz in $CDCl_3$)

Signal #	Shift	Integrat'n	Splitting	Comment	#Neigh-bouring H	Signal Assignment
1	δ 0.9	3H				
2	δ 1.4	2H				
3	δ 1.8	2H				
4	δ 3.2	2H				

Structure Assigned to Unknown 360-14-13

Compound Name:

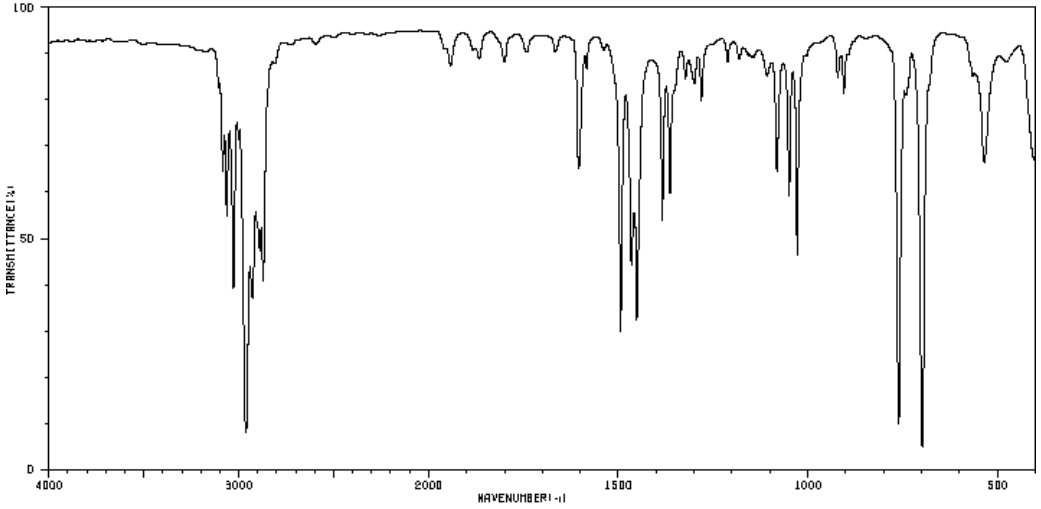
CHEM360 2019-21 **Exp.14**

Unknown # 360-14-14

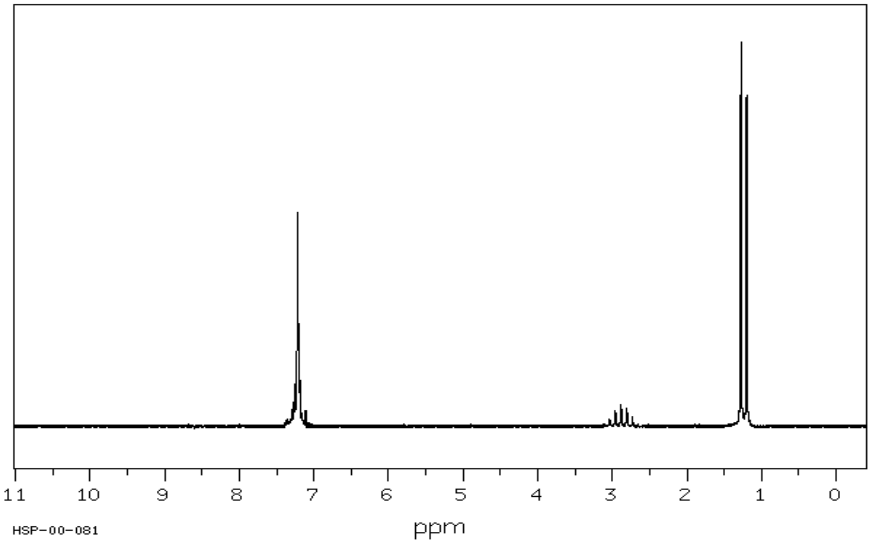
Mol. Wt. = 120.20 g/mol Chemical Formula = C_9H_{12} Degrees of Unsaturation = _____

b.p. = 152.4°C

IR Spectra (liquid film (neat) on KBr Disc)



^{1}H-NMR Spectra (at 90 MHz in $CDCl_3$)



Infrared Spectra (neat liquid film on KBr disc):

Spectrum Region	Absorp'n Band#	Frequency (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, med. or weak)	Functional Group Indicated

^{1}H-NMR Integration Data: (at 90 MHz in $CDCl_3$)

Signal #	Shift	Integrat'n	Splitting	Comment	#Neigh-bouring H	Signal Assignment
1	δ 1.2	6H				
2	δ 2.9	1H				
3	δ7-7.4	5H				

Structure Assigned to Unknown 360-14-14

Compound Name:

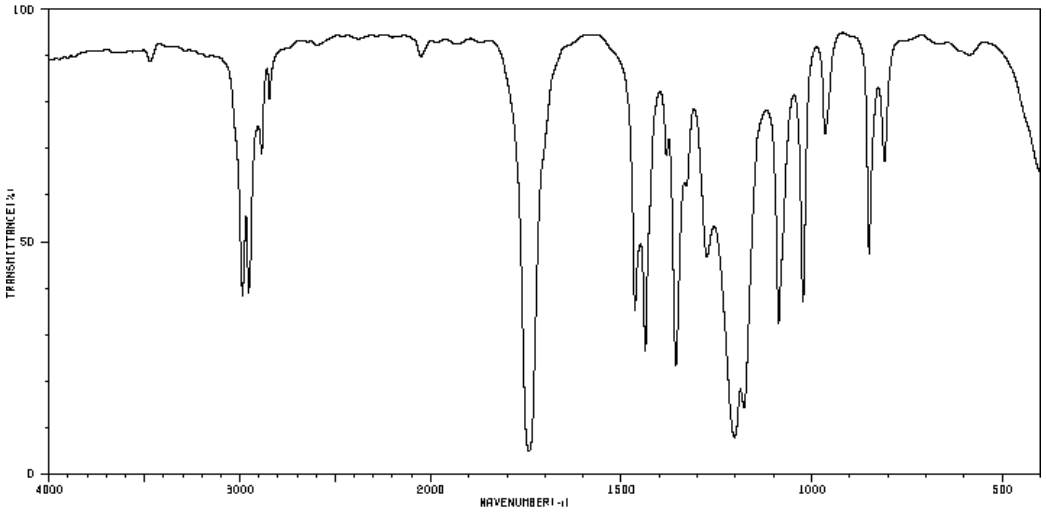
CHEM360 2019-21 **Exp.14**

Unknown # 360-14-15

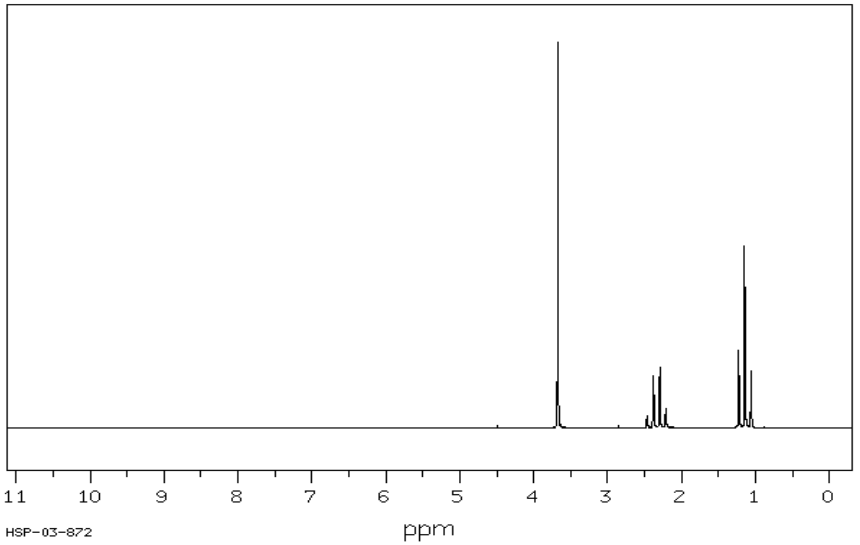
Mol. Wt. = 88.12 g/mol Chemical Formula = $C_4H_8O_2$ Degrees of Unsaturation = _____

b.p. = 79.9°C

IR Spectra (liquid film (neat) on KBr Disc)



^{1}H-NMR Spectra (at 90 MHz in $CDCl_3$)



Infrared Spectra (neat liquid film on KBr disc):

Spectrum Region	Absorp'n Band#	Frequency (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, med. or weak)	Functional Group Indicated

^{1}H-NMR Integration Data: (at 90 MHz in $CDCl_3$)

Signal #	Shift	Integrat'n	Splitting	Comment	#Neigh-bouring H	Signal Assignment
1	δ 1.15	3H				
2	δ 2.3	2H				
3	δ 3.7	3H				

Structure Assigned to Unknown 360-14-15

Compound Name:

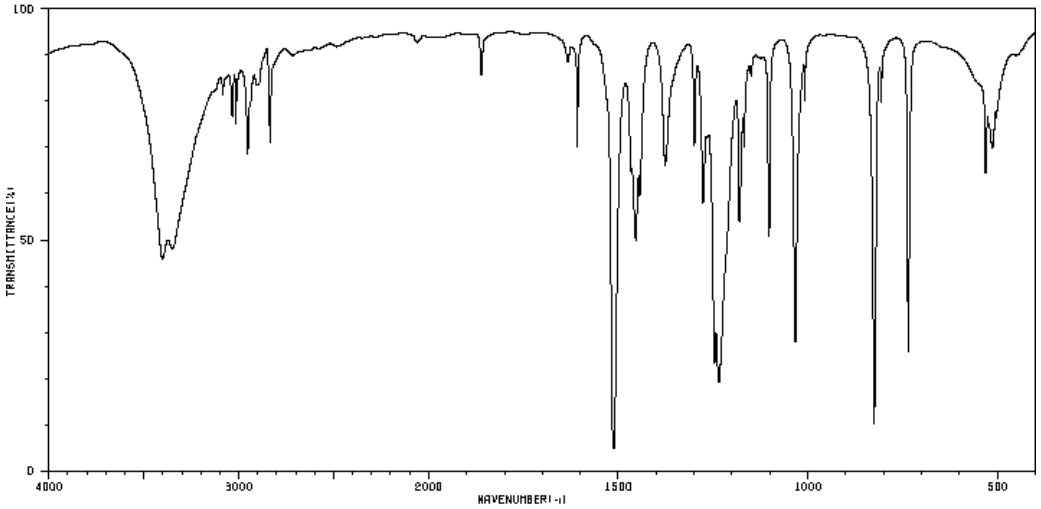
CHEM360 2019-21 **Exp.14**

Unknown # 360-14-16

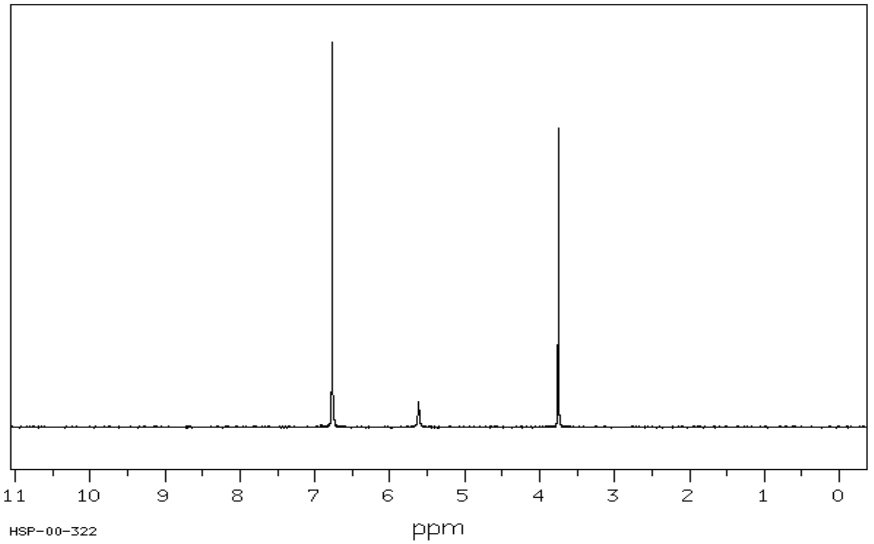
Mol. Wt. = 124.14 g/mol Chemical Formula = $C_7H_8O_2$ Degrees of Unsaturation = _____

m.p. = 55-57°C

IR Spectra (on KBr Disc)



^{1}H-NMR Spectra (at 90 MHz in $CDCl_3$)



Infrared Spectra (on KBr disc):

Spectrum Region	Absorp'n Band#	Frequency (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, med. or weak)	Functional Group Indicated

^{1}H-NMR Integration Data: (at 90 MHz in $CDCl_3$)

Signal #	Shift	Integrat'n	Splitting	Comment	#Neigh-bouring H	Signal Assignment
1	δ 3.75	3H				
2	δ 5.6	1H		xchanges With D_2O		
3	δ 6.8	4H				

Structure Assigned to Unknown 360-14-16

Compound Name:

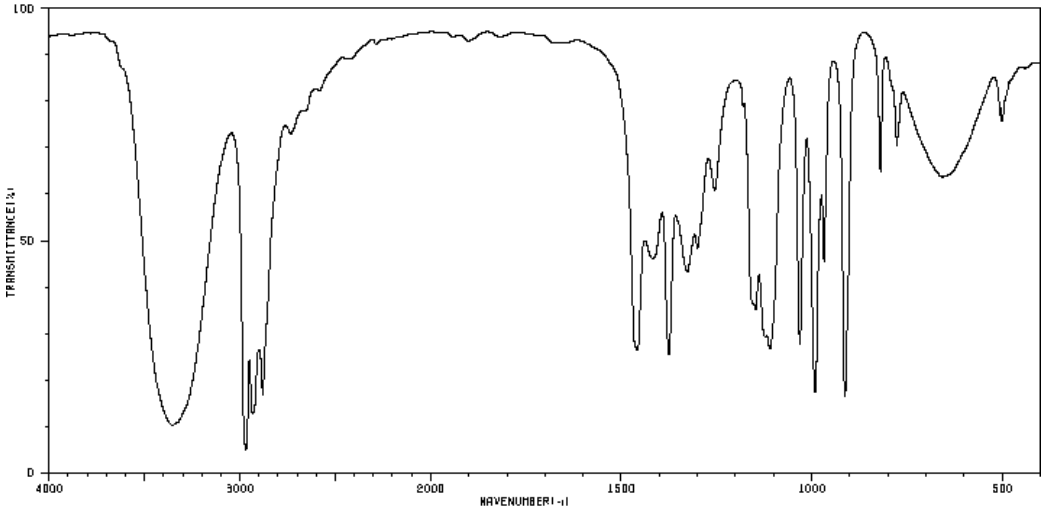
CHEM360 2019-21 **Exp.14**

Unknown # 360-14-17

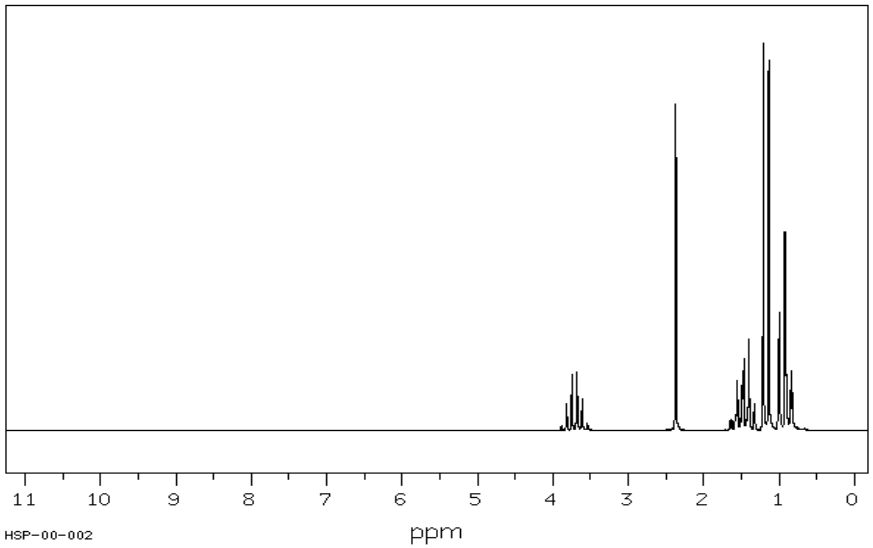
Mol. Wt. = 74.12 g/mol Chemical Formula = $C_4H_{10}O$ Degrees of Unsaturation = _____

b.p. = 99.5°C

IR Spectra (liquid film (neat) on KBr Disc)



^{1}H-NMR Spectra (at 90 MHz in $CDCl_3$)



Infrared Spectra (neat liquid film on KBr disc):

Spectrum Region	Absorp'n Band #	Frequency (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, med. or weak)	Functional Group Indicated

^{1}H-NMR Integration Data: (at 90 MHz in $CDCl_3$)

Signal #	Shift	Integrat'n	Splitting	Comment	#Neigh-bouring H	Signal Assignment
1	δ 0.9	3H				
2	δ 1.2	3H				
3	δ 1.5	2H				
4	δ 2.4	1H		xchanges With D_2O		
5	δ 3.7	1H				

Structure Assigned to Unknown 360-14-17

Compound Name:

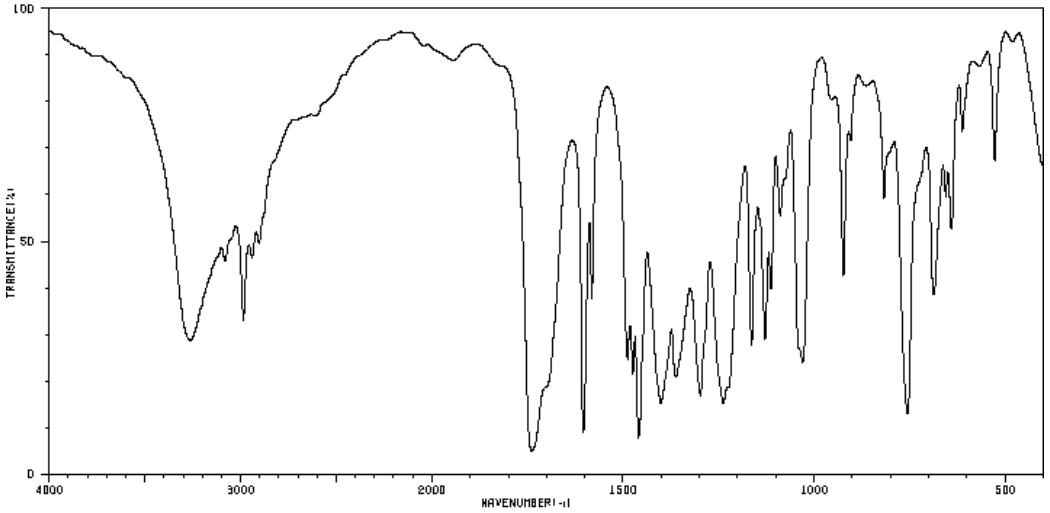
CHEM360 2019-21 **Exp.14**

Unknown # 360-14-18

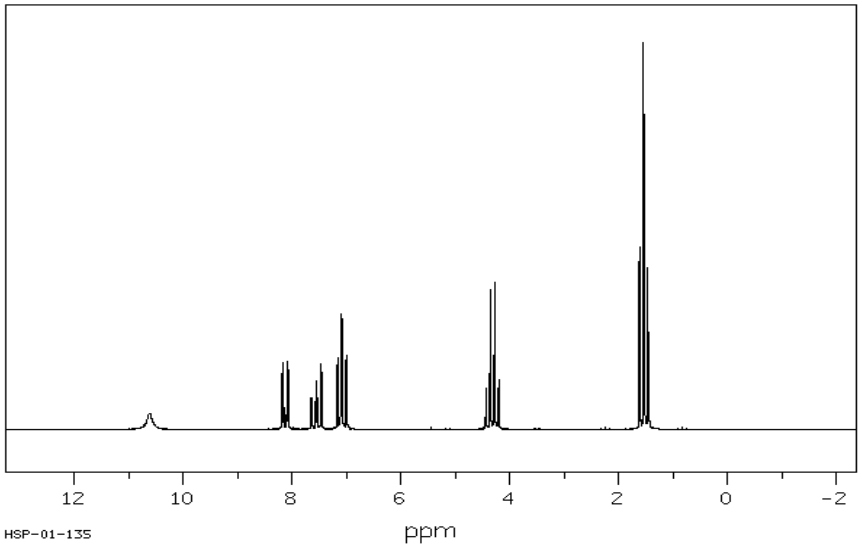
Mol. Wt. = 166.18 g/mol Chemical Formula = $C_9H_{10}O_3$ Degrees of Unsaturation = _____

m.p. = 20.7°C

IR Spectra (on KBr Disc)



^{1}H-NMR Spectra (at 90 MHz in $CDCl_3$)



Infrared Spectra (on KBr disc):

Spectrum Region	Absorp'n Band #	Frequency (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, med. or weak)	Functional Group Indicated

^{1}H-NMR Integration Data: (at 90 MHz in $CDCl_3$)

Signal #	Shift	Integrat'n	Splitting	Comment	#Neigh-bouring H	Signal Assignment
1	δ 1.54	3H				
2	δ 4.32	2H				
3	δ 7.05 &7.1	2H		Hint: ortho substituion pattern		
4	δ 7.5 &8.1	2H				
5	δ 10.6	1H		xchanges with D_2O		

Structure Assigned to Unknown 360-14-18

Compound Name:

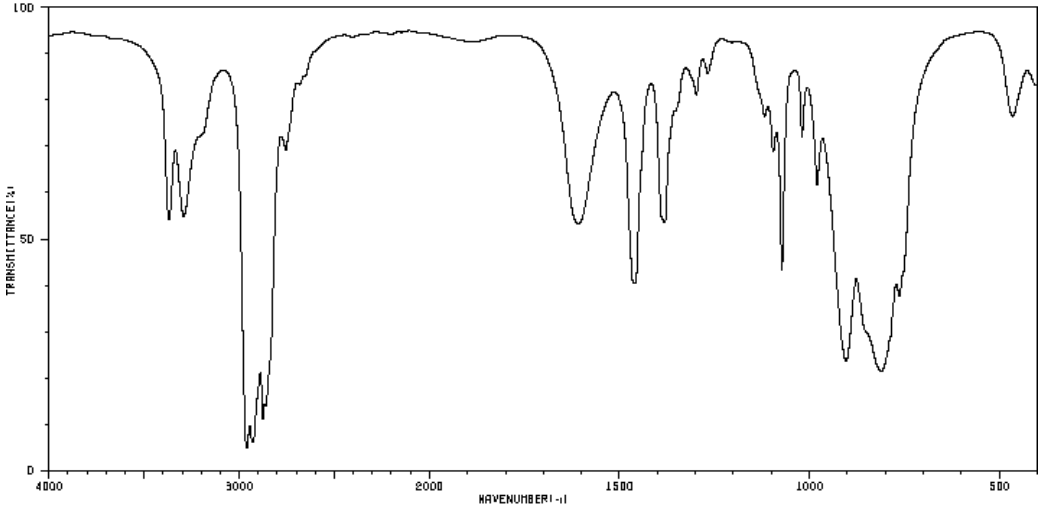
CHEM360 2019-21 **Exp.14**

Unknown # 360-14-19

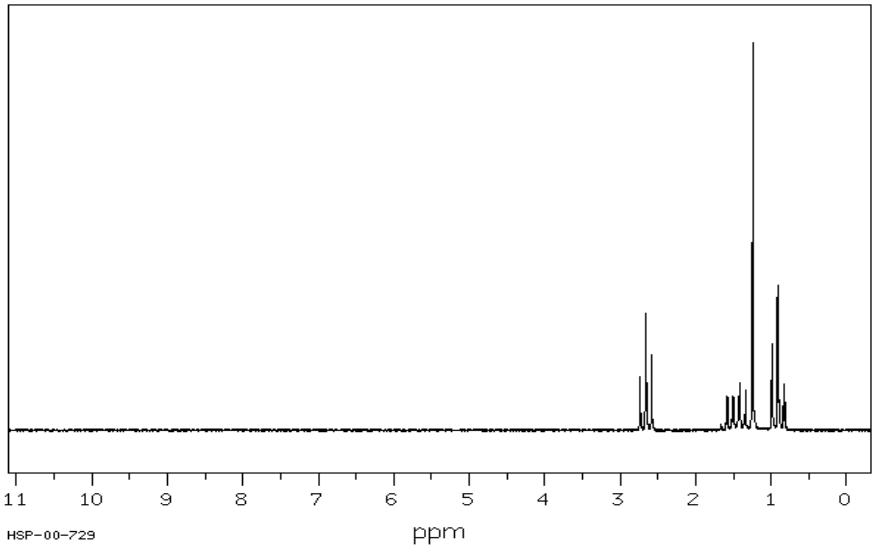
Mol. Wt. = 59.11 g/mol Chemical Formula = C_3H_9N Degrees of Unsaturation = _____

b.p. = 47.8°C

IR Spectra (liquid film on KBr Disc)



^{1}H-NMR Spectra (at 90 MHz in $CDCl_3$)



Infrared Spectra (liquid film on KBr disc):

Spectrum Region	Absorp'n Band #	Frequency (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, med. or weak)	Functional Group Indicated

^{1}H-NMR Integration Data: (at 90 MHz in $CDCl_3$)

Signal #	Shift	Integrat'n	Splitting	Comment	#Neigh-bouring H	Signal Assignment
1	δ 0.92	3H				
2	δ 1.24	2H		xchanges with D_2O		
3	δ 1.45	2H				
4	δ 2.65	2H				

Structure Assigned to Unknown 360-14-19

Compound Name:

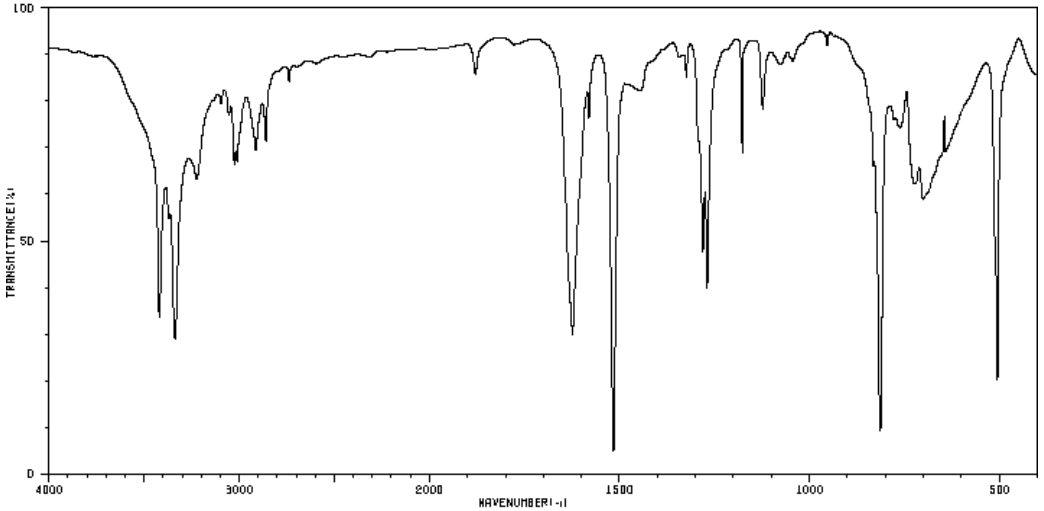
CHEM360 2019-21 **Exp.14**

Unknown # 360-14-20

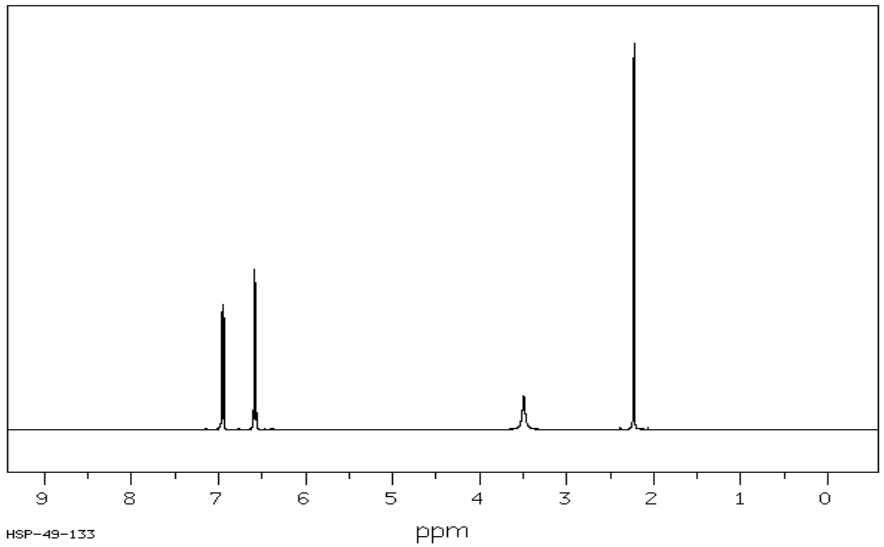
Mol. Wt. = 107.16 g/mol Chemical Formula = C_7H_9N Degrees of Unsaturation = _____

b.p. = 200.5°C

IR Spectra (on KBr Disc)



^{1}H-NMR Spectra (at 400 MHz in $CDCl_3$)



Infrared Spectra (on KBr disc):

Spectrum Region	Absorp'n Band #	Frequency (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, med. or weak)	Functional Group Indicated

^{1}H-NMR Integration Data: (at 400 MHz in $CDCl_3$)

Signal #	Shift	Integrat'n	Splitting	Comment	#Neigh-bouring H	Signal Assignment
1	δ 2.2	3H				
2	δ 3.5	2H		xchanges with D_2O		
3	δ 6.6	2H				
4	δ 6.95	2H				

Structure Assigned to Unknown 360-14-20

Compound Name:

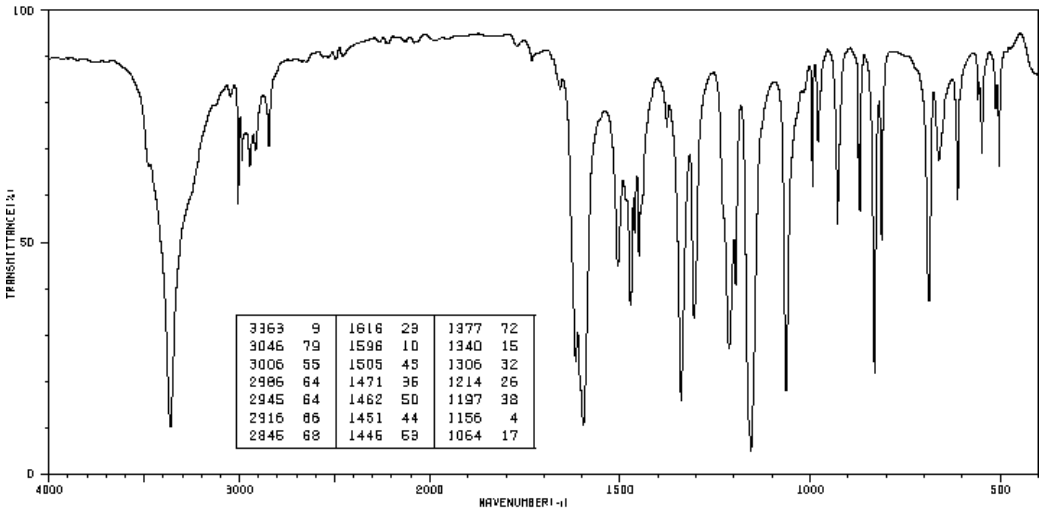
CHEM360 2019-21 **Exp.14**

Unknown # 360-14-21

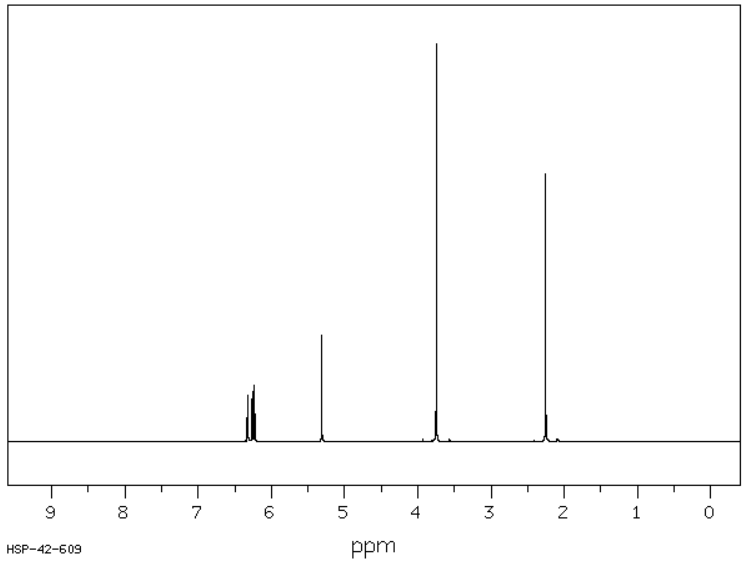
Mol. Wt. = 138.2 g/mol Chemical Formula = $C_8H_{10}O_2$ Degrees of Unsaturation = _____

b.p. = unknown °C

IR Spectra (thin film on KBr disc)



^{1}H-NMR Spectra (at 400 MHz in 0.029 g in 0.5 mLCDCl$_3$)



Infrared Spectra (on KBr disc):

Spectrum Region	Absorp'n Band #	Frequency (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, med. or weak)	Functional Group Indicated

^{1}H-NMR Integration Data: (at 400 MHz in CDCl$_3$)

Signal #	Shift	Integrat'n	Splitting	Comment	#Neigh-bouring H	Signal Assignment
1	δ 2.25	3H				
2	δ 3.75	3H				
3	δ 5.31	1H				
4	6.2	3H	multiplet			

Structure Assigned to Unknown 360-14-21

Compound Name:

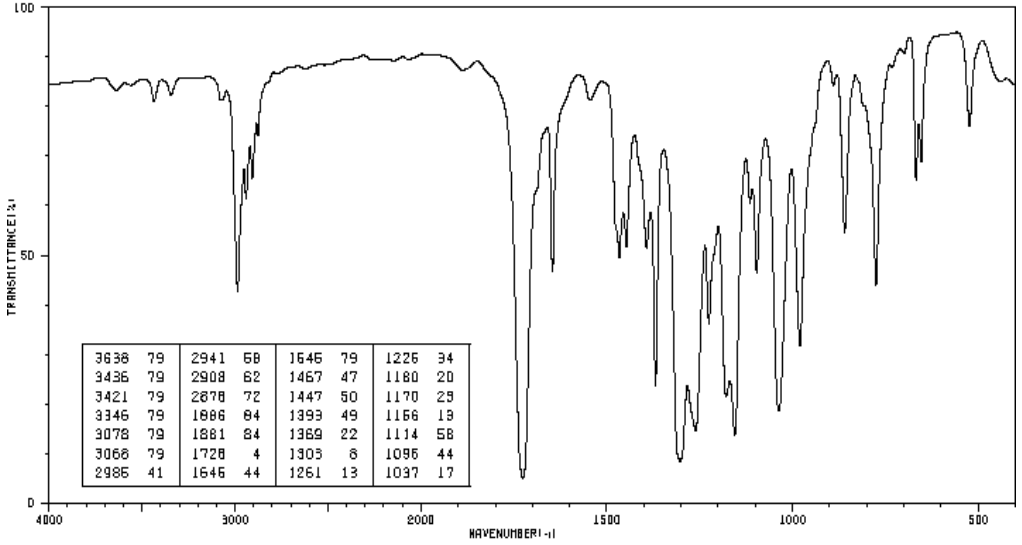
CHEM360 2019-21 **Exp.14**

Unknown # 360-14-22

Mol. Wt. = 172.2 g/mol Chemical Formula = $C_8H_{12}O_4$ Degrees of Unsaturation = _____

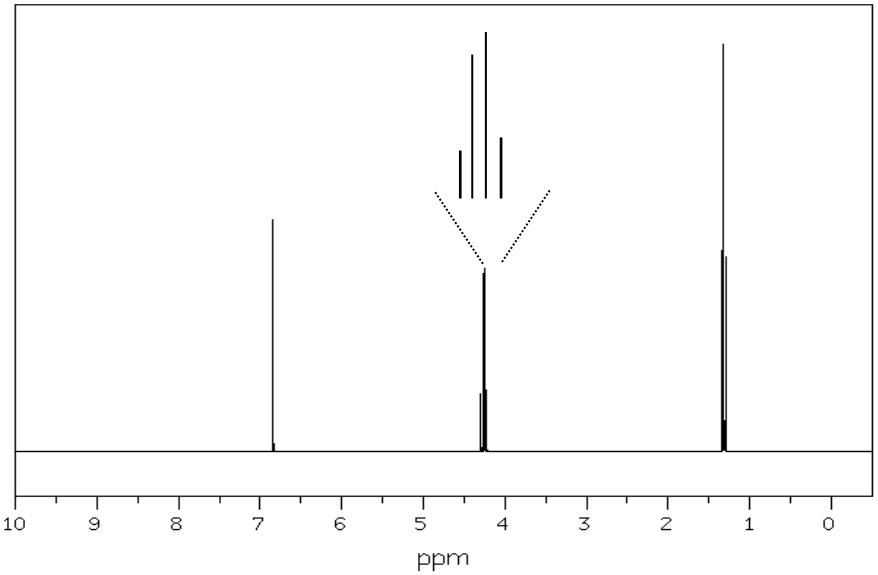
m.p. = 1-2 °C

IR Spectra (liquid)



3638	79	2941	58	1645	79	1225	34
3436	79	2908	62	1467	47	1180	20
3421	79	2878	72	1447	50	1170	25
3346	79	1886	84	1393	49	1156	13
3078	79	1881	84	1369	22	1114	58
3068	79	1728	4	1305	8	1095	44
2985	41	1646	44	1261	13	1037	17

^{1}H-NMR Spectra (at 300 MHz, 10.4 mol% $CDCl_3$)



Infrared Spectra (liquid film):

Spectrum Region	Absorp'n Band #	Frequency (cm^{-1})	Peak Shape (sharp, broad)	Peak Intensity (strong, med. or weak)	Functional Group Indicated

^{1}H-NMR Integration Data: (at 300 MHz in $CDCl_3$)

Signal #	Shift	Integrat'n	Splitting	Comment	#Neigh-bouring H	Signal Assignment
1	δ 1.32	6H		2 x 3H?		
2	δ 4.26	4H		2 x 2H?		
3	δ 6.9	2H				

Structure Assigned to Unknown 360-14-22

Compound Name:

CHEM360 ORGANIC CHEMISTRY II

Athabasca University

PREPARATION, PERFORMANCE, AND PRODUCT EVALUATION FORM*

STUDENT NAME: _____ **STUDENT ID#:** _____ **DATE:** _____

EXP.#	PRODUCT SUBMITTED	DATE SUBMITTED**	QUANTITY (units?)	PRODUCT CHARACTERISTICS						EXPERIMENT GRADE	INSTR. INIT.
				APPEARANCE	M.P./B.P.	IR (Y/N)	TLC	OTHER	B.PRESS.		
10	ESTER =						N/A				
	carboxylic acid=						N/A		N/A		
(5 marks)	alcohol=						N/A		N/A		
11	FUNCTIONAL GROUPS	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	
(0 marks)	ALCOHOLS AND ALKYL HALIDES								N/A		
12	DIPHENYLMETHANOL						Rf =		N/A		
	DIPHENYLMETHANOL (crude)						Rf =		N/A		
(5 marks)	BENZOPHENONE						Rf =	N/A	N/A		
13	ALDOL CONDENSATION*						N/A		N/A		
	aldehyde=						N/A		N/A		
(10 marks)	ketone=						N/A		N/A		
14	IR/NMR TUTORIAL	N/A	N/A	N/A	N/A	N/A	N/A	submit 4	N/A	N/A	
(0 marks)	Unknowns^^=										
15	FUNCTIONAL GROUPS	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	
(0 marks)	ALDEHYDES AND KETONES										
16	TRIPHENYLMETHANOL					N/A	Rf =				
	TRIPHENYLMETHANOL (crude)	N/A	N/A		N/A	N/A	Rf =	N/A	N/A		
	MOTHER LIQUOR	N/A	N/A		N/A	N/A	Rf =	N/A	N/A		
	BIPHENYL	N/A	N/A		N/A	N/A	Rf =	N/A	N/A		
(10 marks)	Amt.Bromobenzene = , Amt.Ethyl benzoate =				N/A	N/A	N/A	N/A	N/A		
17	4'-METHYLACETANILIDE	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A		
	4'-ACETOAMIDOBENZOIC ACID^				N/D	N/A	N/A		N/A		
(~7 ea.)	4-AMINOBENZOIC ACID^^					N/A	N/A		N/A		
(20 marks)	BENZOCAINE^^^					N/A	N/A				
	^Part C Amt. 4'-methylacetanilide used =			^^^Part E Amt. 4-aminobenzoic acid used =					**Total=**		
	^^Part D Amt. 4'-acetamidobenzoic acid used =								**(/50)**		

N/A = not applicable, N/D = not determined

* This form is the official results form for Chem360. To get credit for the lab it must be fully completed and then initialed by the instructor. Keep safe at all times.

** Lab reports are due 1 month after completion of each experiment. Late lab reports typically lose 10% per month late.

***This form will not be signed until the student has done a complete cleanup of their bench area.

^^CHEM360 H-NMR Unknowns are to be downloaded from: http://science.athabascau.ca/Labs/resources/organic_chemistry.php (username= auchem360, password = synthesis)

Last modified: 2019-06-21