Chemistry 360
Organic Chemistry II

Pre-lab questions

Athabasca University
Faculty of Science and Technology

Experiment 10 Prelab Questions

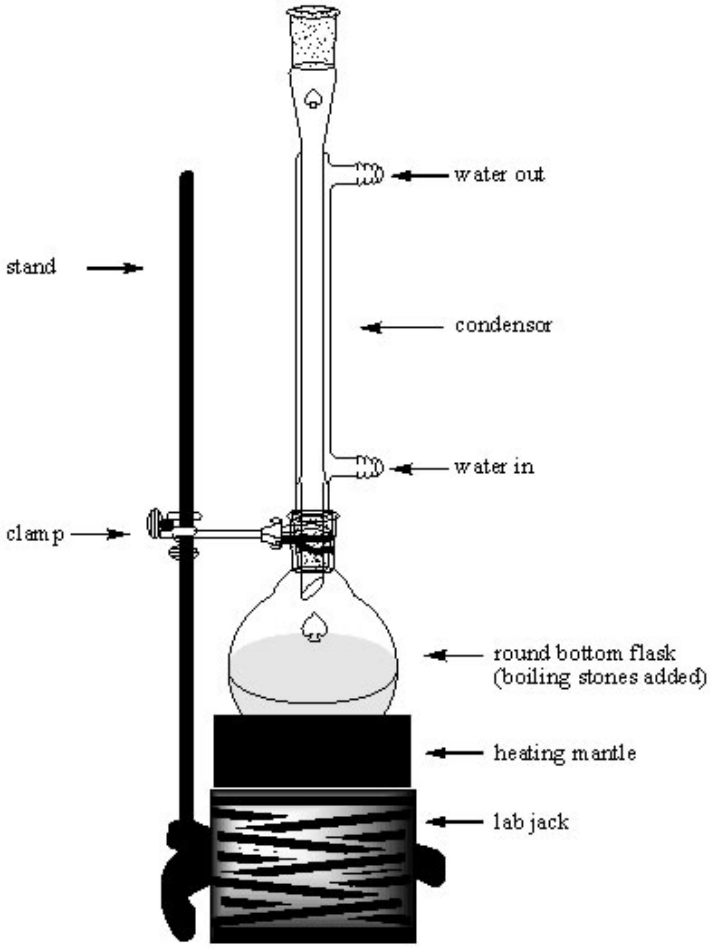
Lab Safety

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

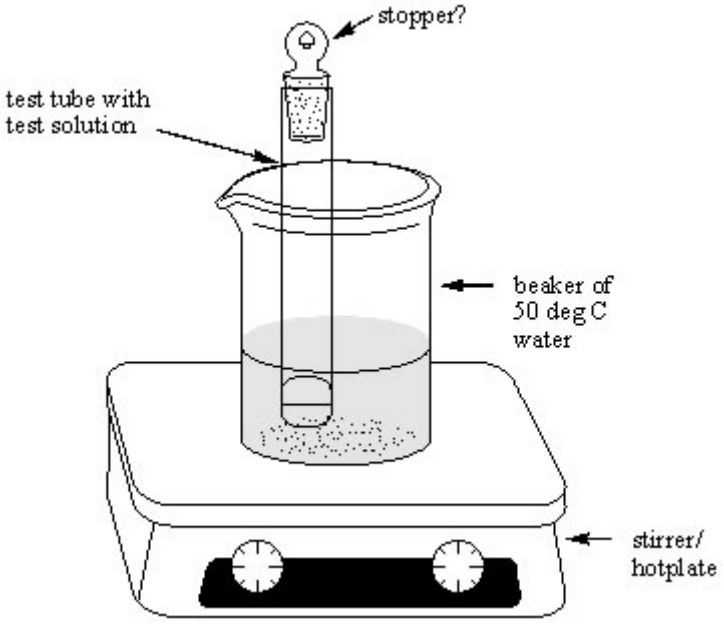
9. How is the ester product purified and characterized?
   a) Yield, refractive index, % yield, infrared spectral analysis
   b) Boiling point, refractive index, and infrared spectral analysis
   c) Yield, boiling point, refractive index, % yield, and infrared spectral analysis

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

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

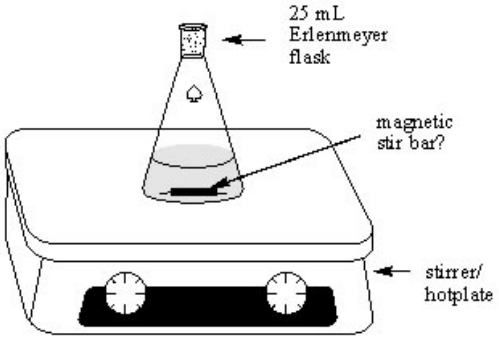
Experiment 12 Prelab Questions:

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

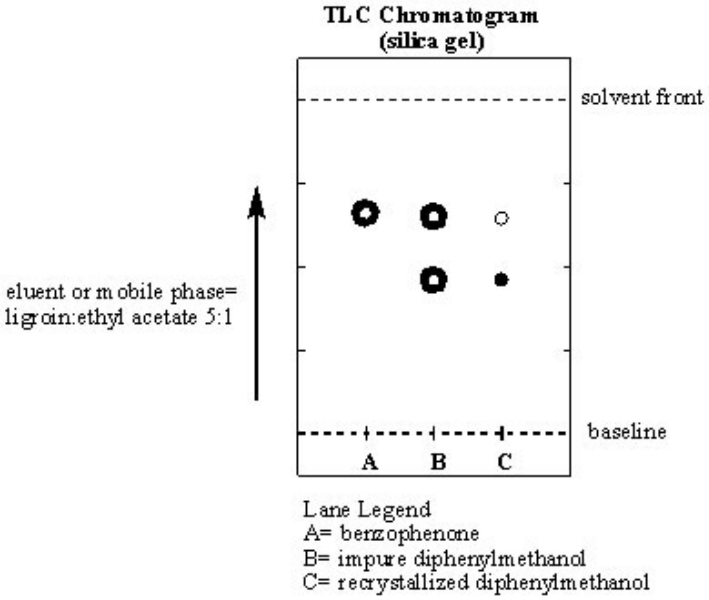
b) Erlenmeyer flask. The narrower neck of the Erlenmeyer flask acts like a condenser and prevents the solvent from evaporating too quickly. Also a flask can eventually be sealed with a stopper while crystal growth is occurring.
c) Beaker. A beaker is easier to add solvent to and the recrystallized product 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



10. 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

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

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

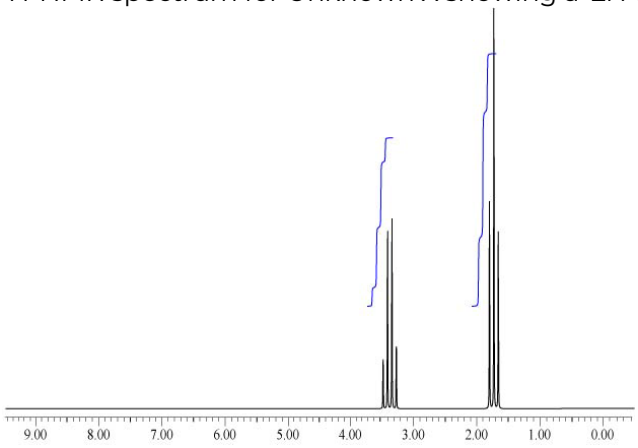
5. The N=n+1 rule is used to determine the number of neighbouring H
   a) true
   b) false

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

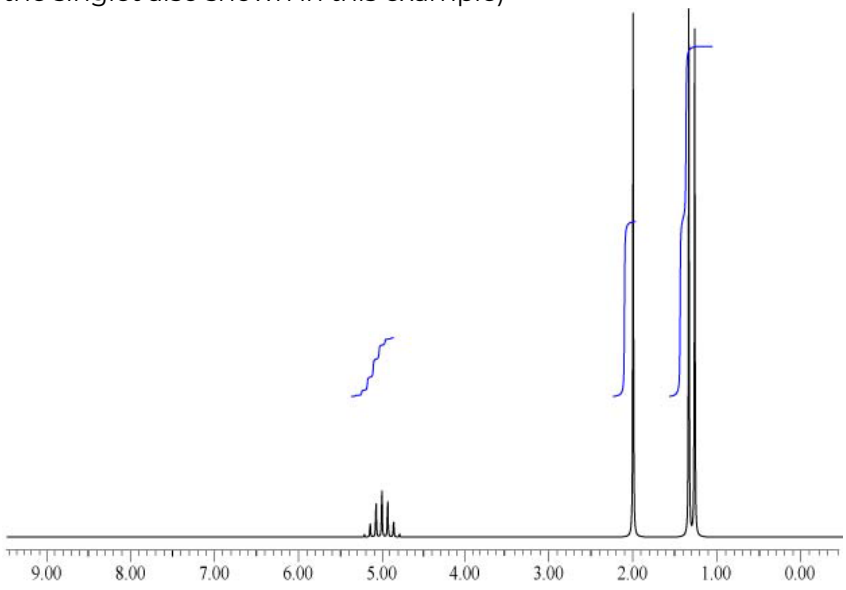
^{1}H-NMR spectrum for Unknown X showing a '2H quartet coupled to a 3H triplet'



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?

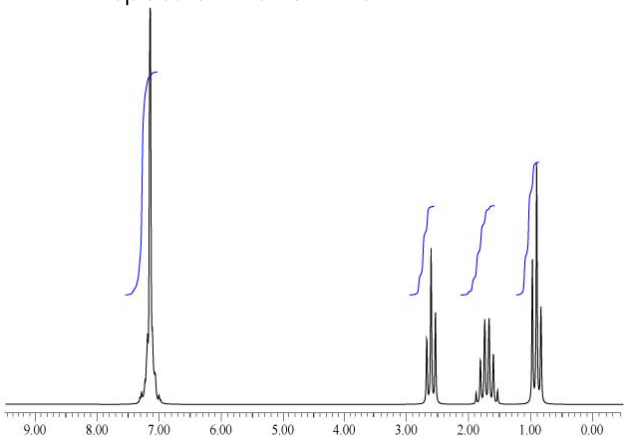
a) a methyl group
b) an ethyl group
c) an isopropyl group
d) a *tert*-butyl group

^{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

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

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

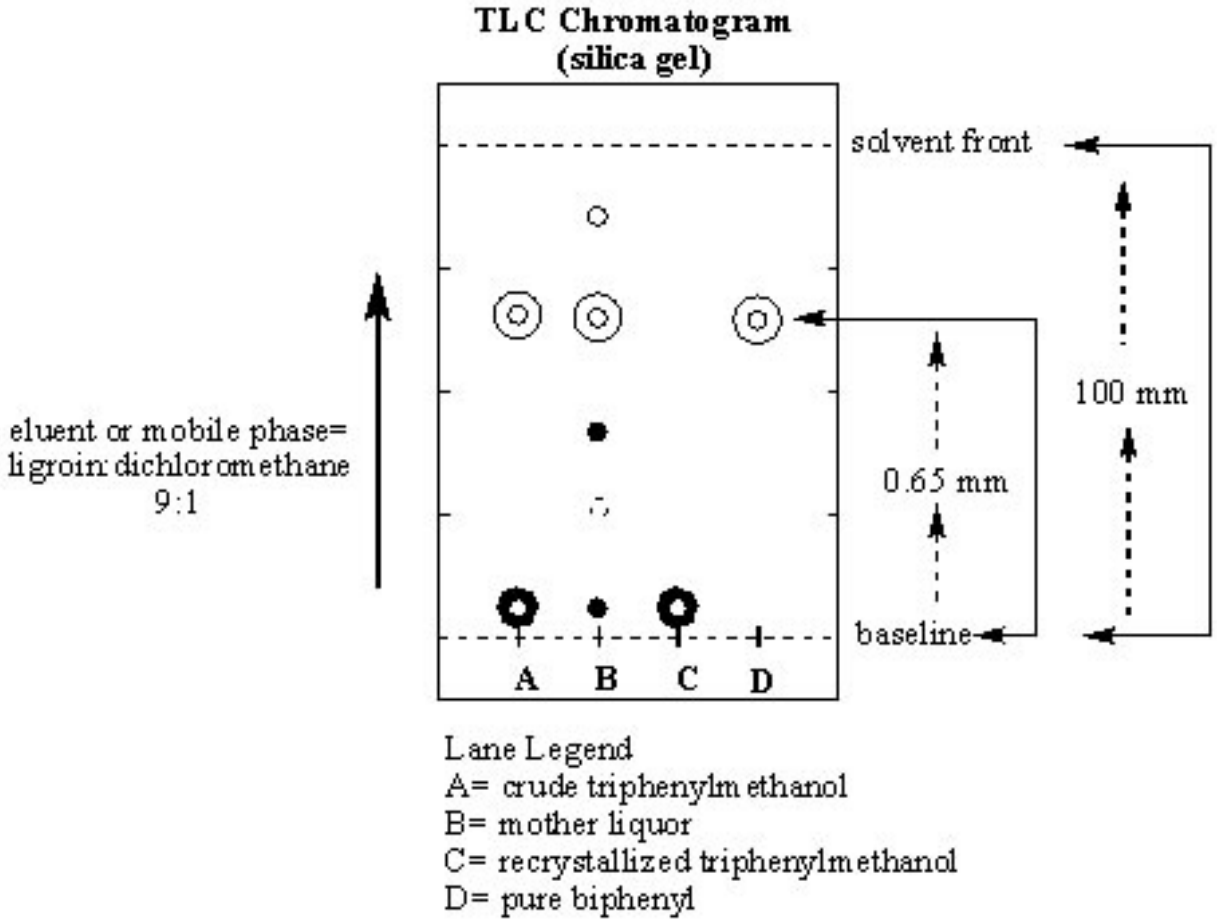
7. What layer will your product be in after addition of the diethyl ether in Part C Step 3?
   a) the aqueous layer
   b) the ether layer

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



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%

Athabasca University

CHEM360 Organic Chemistry II

PRELAB ANSWERS

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