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BIRZEIT UNIVERSITY
Chemistry Department
Chemistry 221

Mid-Term Exam
Time:75 minutes

2nd Sem.2012/2013
April.23, 2013.

Student Name: Donia Sheikh ..... Student No: 1111270 .....

Laboratory Instructor:

☐ Dr.Imad Qamhieh Monday at 2:00 ☑ Dr. Simon Kuttab, Tuesday at 11:00

☐ Dr.Adel Hidmi Thursday at 11:00 ☐ Dr. Sami Sayrafi , Thursday at 2:00

I-(60%) Circle the correct answer

1. Compounds A and B both have melting points of 140°C. If A is mixed with B at a ratio which leads to melting at the eutectic point, the melting point of the mixture

a) will remain 140°C (b) will be less that 140°C
c). will be greater than 140°C d) will contain two separate melting points

2- A compound can be separated by steam distillation if it satisfies the following conditions:

a) polar, viscous and water insoluble b) high melting point, water miscible and polar
(c) thermally stable, has some vapor pressure and water insoluble
d) polar, has very low vapor pressure and high melting point

3. An ideal recrystallization solvent will

a) dissolve lots of the compound at room temperature and little at high temperatures
b) dissolve lots of the compound at room temperature and lots at high temperatures
c. dissolve very little of the compound at any temperature
(d) dissolve little of the compound at room temperature and lots at high temperatures
e) react with the compound being recrystallized

4-In the preparation of cyclohexene, you performed a potassium permanganate test on the product. Hopefully, you witnessed the immediate formation of a brown precipitate. What was the precipitate?

a) Mn_2O_3 (b) MnO_2 c. cyclohexanol d) 1,2-cyclohexanediol

5. If you take the melting point of an impure substance, the observed melting point range will be _____ relative to the melting point range observed for the pure substance.

a) higher and broader b) higher and narrower (c) lower and broader
d) lower and narrower e) neither higher nor lower, but broader

6. When a solution is cooled very rapidly in recrystallization:

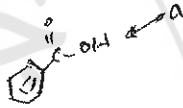
a) no crystals will form b) ideal recrystallization takes place
c). the impurities don't have time to stick to the crystals
d) small crystals form that have a large surface area on which impurities will be absorbed

7- Why do you add boiling chips to a solution that will be heated?

a) to keep the solution homogeneous b) to increase the rate of reflux
c) to prevent bumping d) to lower the boiling point

8- When performing a distillation of any type, it is necessary for the system to have an opening to the outside, otherwise excess pressure can build up inside the apparatus and:

a) lower the boiling point. b) keep the sample from distilling.
c) decompose the sample. d) cause the apparatus to explode.



9- Which extraction procedure below would you use to isolate benzoic acid from a mixture of neutral organic compounds?

a) dissolve in dichloromethane, extract with base, acidify solution, filter precipitate
b) dissolve in dichloromethane, extract with base, extract aqueous with dichloromethane
c) dissolve in dichloromethane, extract with acid, extract aqueous with dichloromethane
d) dissolve in dichloromethane, extract with acid, neutralize solution, extract aqueous with dichloromethane
e. dissolve in dichloromethane, extract with base, acidify solution, extract aqueous with dichloromethane

10- A common measure of the efficiency of a fractionating column is

a) the number of components in the solution b) the number of theoretical plates in the column
c) the number of basic types of distillation methods
d) the variation in temperature at the top of the column

11- The primary reason for the addition of anhydrous magnesium sulfate to the organic solvent layer resulting from the extraction of an organic compound from water is to

a) remove residual water b) remove residual NaCl
c) remove residual acid catalyst d) neutralize the residual NaOH

12- What device is used in the chemical laboratory to separate two immiscible liquids of differing density

a)Buret b) Erlenmeyer Flask c) Dropping funnel d) Separatory funnel

13- Which statement is correct regarding the **boiling point** of a pure liquid:

a) It is the temperature at which the total vapor pressure of the liquid is equal to the external (atmospheric) pressure.

b) Boiling points can be determined using the technique of steam distillation.

c) An alternative method to determine the boiling point is by gas chromatography .

d) Variation in the external pressure has little or no effect on the boiling point.

14- Recrystallized product is most efficiently collected by:

a) vacuum filtration b) gravity filtration c) solvent evaporation d) extraction

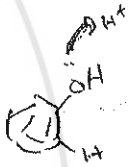
15- In the preparation of cyclohexene from cyclohexanol, what reaction conditions would not favor the formation of cyclohexene?

a) the acid used has a conjugate base which is a poor nucleophile

b) high reaction temperature

c) remove product from the reaction mixture as rapidly as it is formed

d) use a strong base to form cyclohexene



16- Sulfuric acid serves the following function in the dehydration of cyclohexanol:

a) neutralizes the basic cyclohexanol b) protonates the hydroxyl group

c) neutralizes the sodium bicarbonate d) converts hydrogen to a better leaving group

17- Compounds **A**, **B** and **C** are three unknown compounds. Compound **A** melts at 130-133°C, Compound **B** melts at 132-134°C and Compound **C** melts at 140-143°C. Compound **D** is a known compound which melts at 131-134°C. The mixed mp **A**+**B** is 131-133°C. The mixed mp **A**+**C** is 138-142°C. The mixed mp **B**+**C** is 139-144°C. The mixed mp **A**+**D** is 130-134°C. The mixed mp **C**+**D** is 134-140°C. What can you conclude about your unknowns?

a. A=B=D b. B=C=D c. A=B=C d. B=C e. C=D

18- Which of the following statements is correct regarding steam distillation:

a) the two components (water and organic) behave as distinct entities (independent).

b) It follows Raoult's Law: $P_{total} = P°_A \times N_A + P°_B \times N_B$, (observed $P_A = P°_A N_A$)

c) It works only for separation of liquids d) distillation proceeds at around 150 °C.

II 2. Continued:-

1 moles of β-naphthol ⟶ 1 mole of Nerolin.

0.045 moles of β-naphthol ⟶ 0.045 moles Nerolin

⇒ 0.045 moles = x (g) / 159 (g/mol) so x = 7.16 g ? it will is the theo. yield. Check this

M.wt: 159 g/mol.

7.765

3. % yield = Exp: yield / Theo. yield × 100%.

= 6.0 / 7.16 × 100% = 83.9%.

77.27%

Procedure is correct but calculations are off!

19- When you do a recrystallization experiment, the impurities should either be very insoluble or highly soluble in the recrystallization solvent. If they are insoluble they may be removed by _____ at _____ temperatures.

a) filtration, high b) recrystallization, high c) filtration, low
d) extraction, high e) extraction, low

20- Which compound can be classified as a fixative?

a) 2-ethoxynaphthalene b) β-naphthol c) cyclohexene d) naphthalene

II.(5%) Five grams of Compound A is dissolved in 90 mL of water. The distribution coefficient for Compound A between hexanes and water is 5 (in other words for compound A, $K_{(hexanes/water)} = 5$.) How much of Compound A will be in the hexanes if you extract it from the water one time with 90 mL of hexanes?

5 gm(s) of A, 90 mL water, 90 mL hexane, $K_D = 5$.

$K_D = \frac{\text{Solubility hexane}}{\text{Solubility water}} \Rightarrow 5 = \frac{\frac{y}{90}}{\frac{5-y}{90}} \Rightarrow$ y: # of gm(s) in hexane.

$\frac{y}{90} \times \frac{90}{5-y} = 5 \Rightarrow \frac{y}{5-y} = 5 \Rightarrow 25 - 5y = y \Rightarrow 25 = 6y$

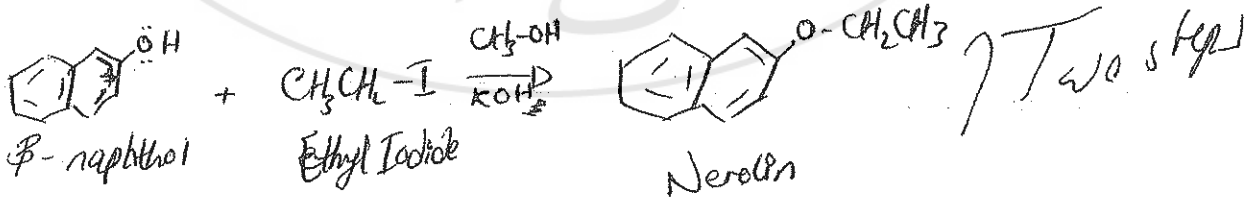
$+5y \quad +5y$

$\therefore y = 4.17$ gm.

III.(10%) In the preparation of nerolin, a student used 40 g of methanol(MW: 32), 6.5 g of β-Naphthol (MW:144.17) and 3.3 g of potassium hydroxide(MW: 56.11) and then added 7.8 g of ethyl iodide (MW: 156). At the end of the reaction, the student obtained 6.0 g of pure nerolin,

1) Write the **equation** of this reaction.
2) What is the **theoretical yield** of this reaction?
3) What is the **percent yield** of this reaction?

1.



β-naphthol Ethyl Iodide Nerolin Two steps

2. * β-naphthol:

$\#\text{ of mols} = \frac{6.5\text{ g}}{144.17\text{ (g/mol)}} = 0.045$ moles.

* ethyl iodide:-

$\#\text{ of moles} = \frac{7.8\text{ g}}{156\text{ (g/mol)}} = 0.05$ moles.

∴ β-naphthol is the limiting reagent.

(continue on the opposite s. please)

(IV) Continued/3-

(D) ~~Pure compou~~

~~Her~~ Maximum amount of A recovered =

10 - 0.2 - 0.43 = 9.37 g of A

(E) It'll be pure, since the 0.2 g of B is still soluble in the solvent after ~~co~~ cooling.

(V) 1. (hermometer ~~Su~~ pulb should be located just below the sidearm.

2. The condenser is not fixed with a clamp & ~~ring~~ on a stand.

3. The apparatus is not fixed with plastic clamps.

4. The level of liquid inside the round bottom flask should be higher than the half (water is needed to be added).

5. The condenser is located in away that lets the gas without the proper cooling. It should be tightened from the right side.

جامعة بيرزيت
BIRZEIT UNIVERSITY

Chemistry dept.

Chem. 221

Midterm Exam
Time: One Hour

2nd sem. 2006/2007
Date: 12/5/2007

Instructor: Prof. Dr. Simon Kuttab

Student Name:--

Student No.------

GOOD LUCK

Please provide clear answers to the following questions:

1- You have 100 ml of water and you add 4 ml of ether to it. You notice only one layer. Then you add 6 ml more of ether and see a small layer of a clear liquid on top of the water. Explain what is happening (4 pts).

When we add 4 ml to 100 ml it's very small ratio of ether to water so no layer appeared. but when we add more the layer will appear more easily.

2- One gram of a compound requires the following quantities of solvent to dissolve: 47 ml of water of 8.1 ml of chloroform. Estimate the partition coefficient of the compound between chloroform and water (8 pts).

$$P = \frac{K_u}{K_u + 1}, \quad u = \frac{v_1}{v_2}\ (\text{organic}) / \text{water} = \frac{8.1}{47} = 0.17$$

$$P = \frac{0.17}{1.17} = 0.147 \quad K_u = 0.588$$

$$q = 1 - P$$

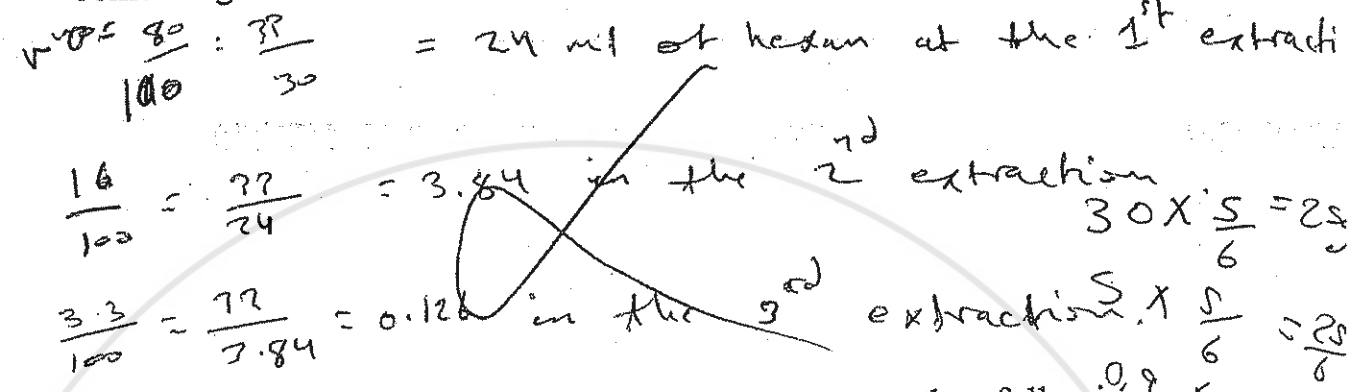
$$q = 1 - 0.588 = 0.412$$

3- Five grams of compound A is dissolved in 90 ml of water. The distribution coefficient for compound A between hexane and water is equal to 5.

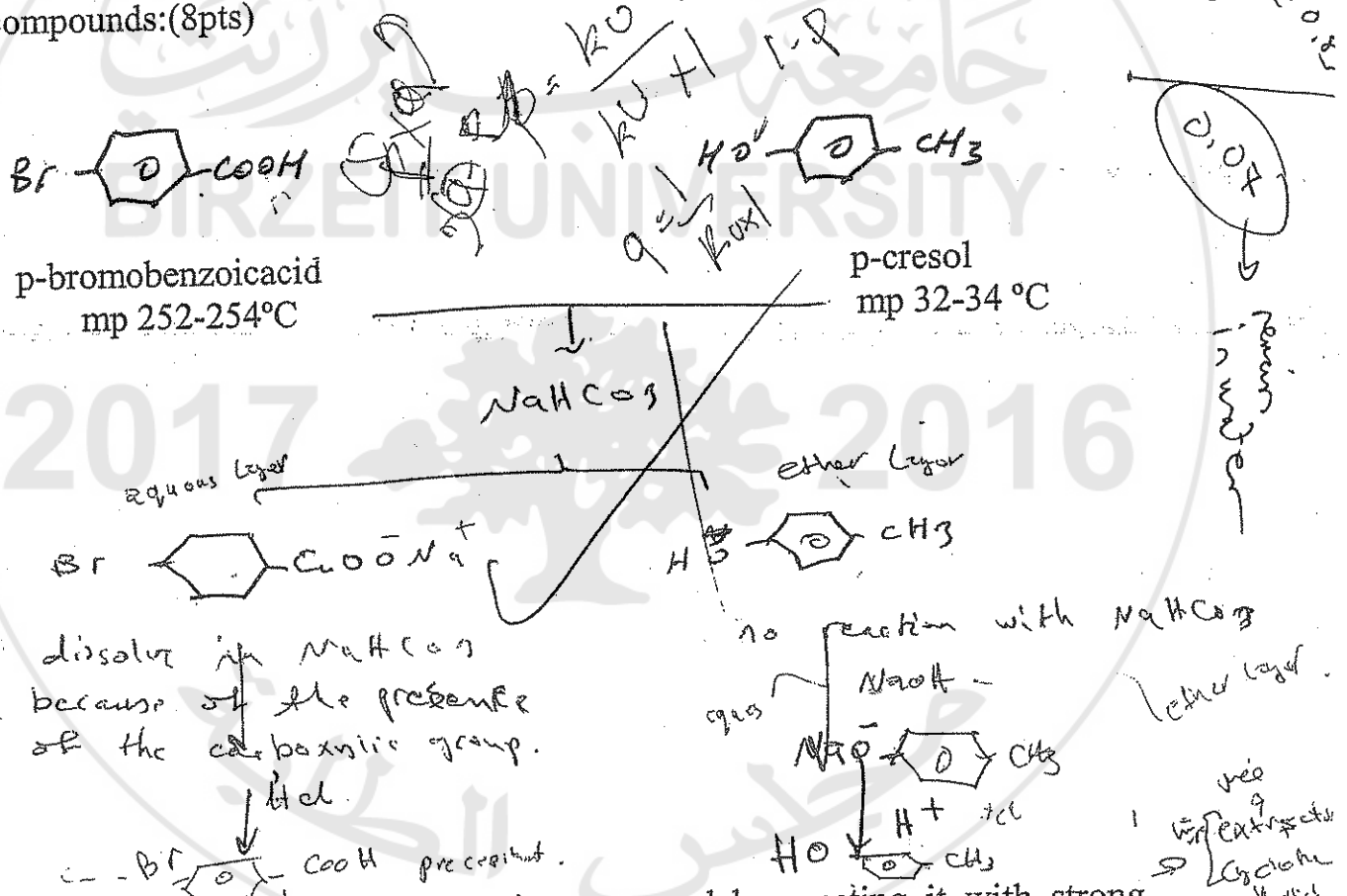
a) How much of compound A will be in the hexane if you extract it from water one time with 90 ml hexane (6 pts).

$$q = 1 - P$$

b) How much of A will be in the hexane, if you extract it from water with three extractions using 30 ml of hexane each time and then combining the hexane extracts (6 pts).



4- Outline in a flow chart how would you separate the following compounds:(8pts)

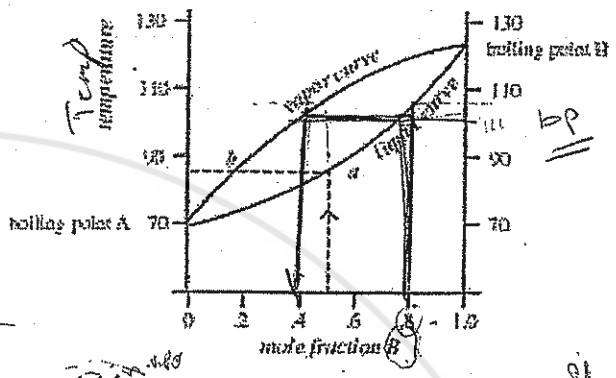


5-You have prepared an organic compound by reacting it with strong aqueous acid. The procedure calls for extraction into methylene chloride, then wash with 5 % Na HCO_3. What is the purpose of the Na HCO_3 wash (4pts).

to remove any impurities by reacting of the NaHCO3 with the organic compound that dissolve it and to remove acid.

Methylene chloride is soluble in sodium bicarbonate solution. which converts the molecule to its Water Soluble 2 Sodium salt by

6- Refer to the figure below. If the mole fraction of B in the liquid is 0.8. What is the composition of B in the vapor above the liquid ? (8 pts)



What will be the reading on the Thermometer at this point?

7- You prepare a fractionating column by filling it with glass beads. The glassware in the laboratory is not completely the same. You notice that when your neighbor pack (fills) his column; he was able to fit a lot fewer beads than you. Whose column will be more efficient as a fractionating column and why?(6pts).

8- When distilling 50 ml of aniline (b.p. 184 °C), prepared by a procedure involving an aqueous reaction, you record a boiling point of 92 °C. The temperature remains at this value through most of the distillation. However the distillate drops into the receiving flask as cloudy drops and separates in the flask into two layers. Explain (6 pts).

azeotropic mixture

This because of the fixed boiling point: the composition of the liquid is equal to the composition of the vapor. azeotrop mixture

→ has a certain fixed composition in the liquid & the same / the liquid have a fixe is boiling as

9- In doing a distillation, what error would result form placing the thermometer bulb too high in the distilling head (4pts)

The bulb will not be in the vapor stream. Therefore the reading is lower than the thermometer

M.P

stopper holding the thermo

- permit seeing the mercury level
- to avoid heating a closed system.

- immersed in the vapor
- equilibrium b/w liquid & vapor.
- wrong reading →

10- When determining a melting point on an unknown compound, it is important to raise the temperature slowly. What would be the error introduced by raising the temperature too quickly. (5pts).

The unknow compound will melt quickly on the wrongs range of temperature, so we can't determine what the unknown compound was.

difficult to give accurate melting point has been approached

~~to not avoid~~ (the sample melting immediately or incorrect T°)

11- If you obtain a crystalline compound during a research project. How could you use its observed melting point as a criterion of purity (4pts).

If there was a wide rang in melting point this tell us that the compound was not pure lit: (127 – 133).
If there was no rang lit: 155.5 – 156 this tell us that the compound was pure

12- If you begin a recrystallization with 15.0 g of solid and recovered 3.0g after the operation. What would your percentage yield be: (5 pts).

percentage yield = $\frac{\text{Experimental yield}}{\text{theoretical yield}} \times 100\%$

$= \frac{3}{15} \times 100\%$

$= 20\%$

13- You and your laboratory partner take melting point of the same sample. You observe a m. p of 101-107 °C while your partner observe a value of 110-112 °C. Explain how you can get two different values with exactly the same sample (4pts).

the observed m.p of 101-107 → tell us that the sample was not pure. but in the second value of 110-112°c the sample was more pure.

Pure organic compound have sharp melting

14- Sometimes recrystallization does not take place spontaneously from a supersaturated solution. What are two techniques commonly used to induce crystallization (6 pts).

1- colorizing 1) stirring
2- washing 2) scratching

1) Suction filtration
2) decolorizing carbon

1) Seed
2) Suction

15- In no more than three lines, explain how a recrystallization procedure is carried out (6pts).

4

prepare a solvent → dissolve the solid by adding the sol
with a small amount → heating → cooling (in ice
washing to remove any impurities → collect it → dr
it → calculate the weight & mp.
Dissolve in minum hot solvent → filter → cool → wash crystal → collect

16- You have 2 g of phenol and you have been asked to recrystallize it from hexanes. How much hexane you will use. (4pts).

0

at 75°c.

hexane

17- A student was recrystallizing a compound. As the hot solution cooled to room temperature, no crystals appeared. The flask was then placed in an ice-water both. Suddenly a large amount of solid appeared in the flask. The student isolated a good yield of product however the product was impure. Explain (6pts)

3

I d), impure because of the slow recrystallization at first so large crystals will form the impurities in it which will impde)

Birzeit University

Chemistry Department

Chemistry 221- Lab

Instructor: Adel Hidmi

Midterm Exam — Second Semester. 2010/2011

Time: 60 min.

Key

- **Student Name:** _____
- **Student No:** _____

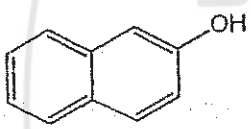
GOOD Luck

Q1 (2/15) A mixture solution of 1-propanol (B.P = 97.1 °C, 50ml) and toluene (BP 110.6 °C, 50ml) was distilled by fractional distillation and collected in 10 ml fractions. Predict the boiling range of each fraction.

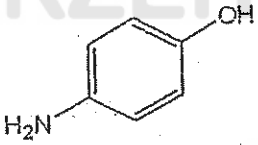
fractions	vol. collected(ml)	temp. °C (predicted)
1	10	97-98
2	20	97-98
3	30	98-99
4	40	99-100
5	50	

Q2 (2/15)

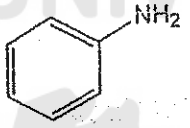
Draw a flow chart to show how to separate the following four compounds from each other. Give enough information so that someone could do it –include stuff like "organic layer", "aqueous layer", "anhydrous sodium sulfate" and the names of solvents used, as well as techniques like "extraction" or "crystallization".



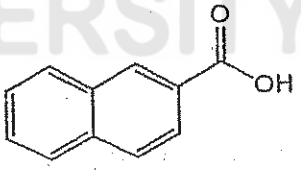
Naphthalen-2-ol



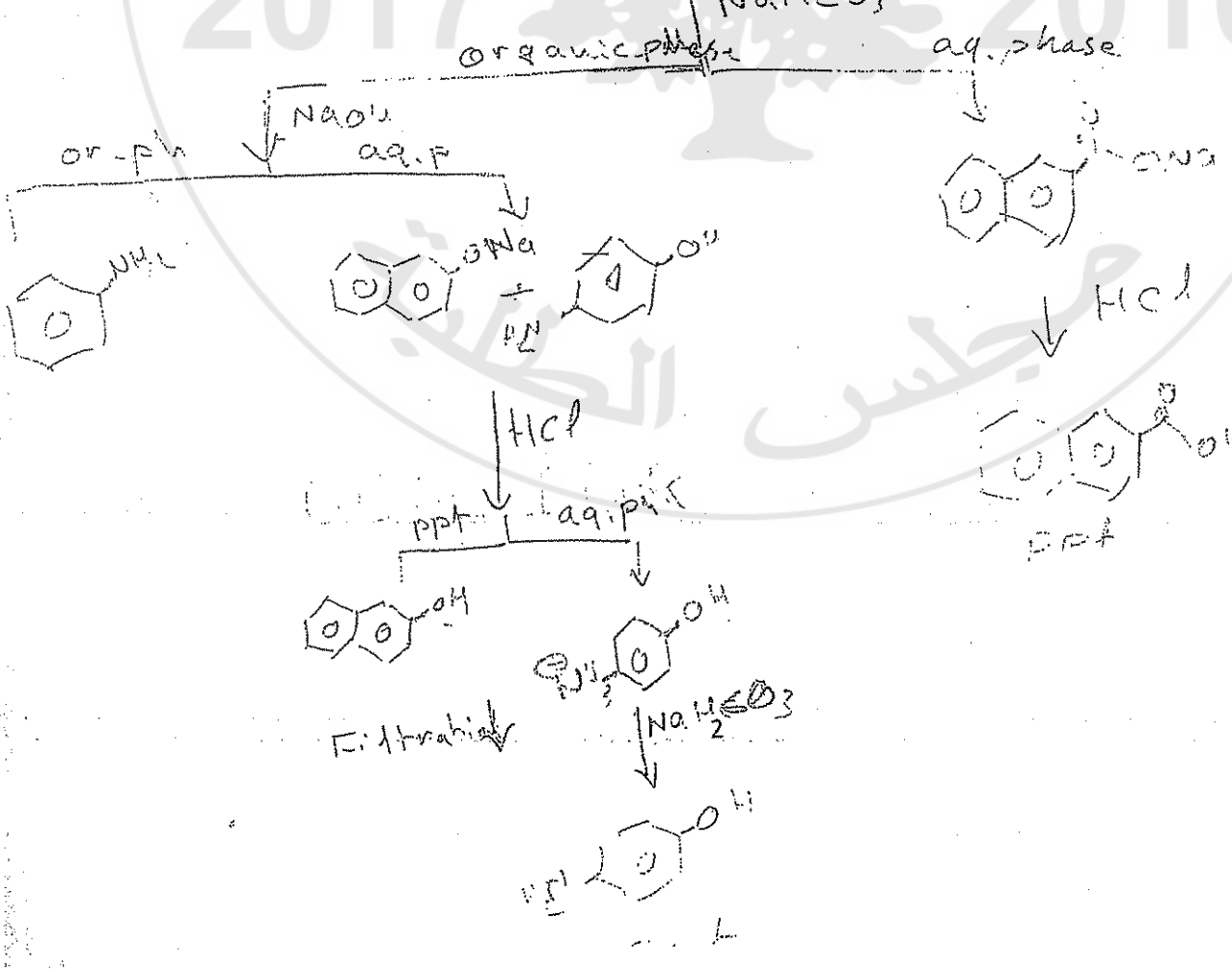
4-Amino-phenol



Phenylamine



Naphthalene-2-carboxylic acid



Q3 (2/15)

a. The normal boiling point of cyclohexanol is 160.5 °C. What is the vapor pressure of cyclohexanol at 160.5 °C?

b. Given the following mole fraction and vapor pressures for miscible liquids A and B, calculate the composition (in mole percentage) of the vapor from a distilling an ideal binary solution at 110 °C and 760 mmHg for the solution

XA = 0.50 PAo = 1216 mmHg
XB = 0.05 PBo = 3040 mmHg

Where: Ptotal = PA + PB = XA. PAo + XB. PBo

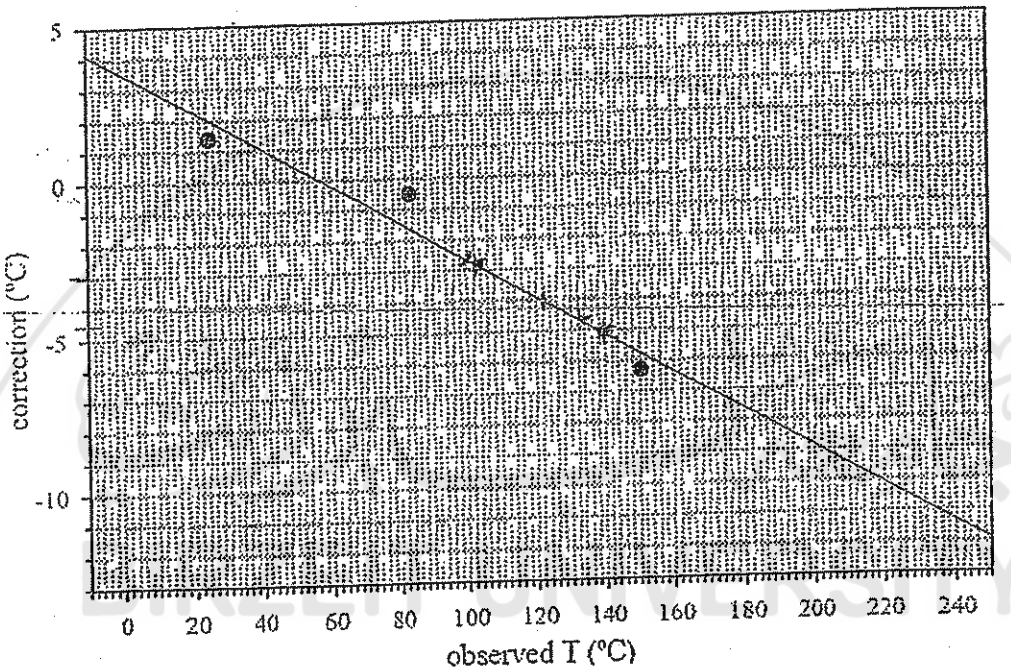
Q4 (2/15)

a. The solubility of compound A in ethanol is 0.5 g per 100 ml at 0 °C and 5.0 g per 100 ml at 75 °C. What is the minimum amount of solvent needed to recover 9.0g of compound A, after recrystallization processes? How much would be lost in the recrystallization?

b. The solubility of acetic acid in hot water (15 g/100 ml at 100 °C), and its solubility in cold water (2.0 g/ 100 ml at 0 °C). What would be the maximum theoretical yield recovery from the crystallization of 10.0 g of ~~acetanilide~~ acetic acid from 200 ml water at 0 °C?

Q5 (2/15)

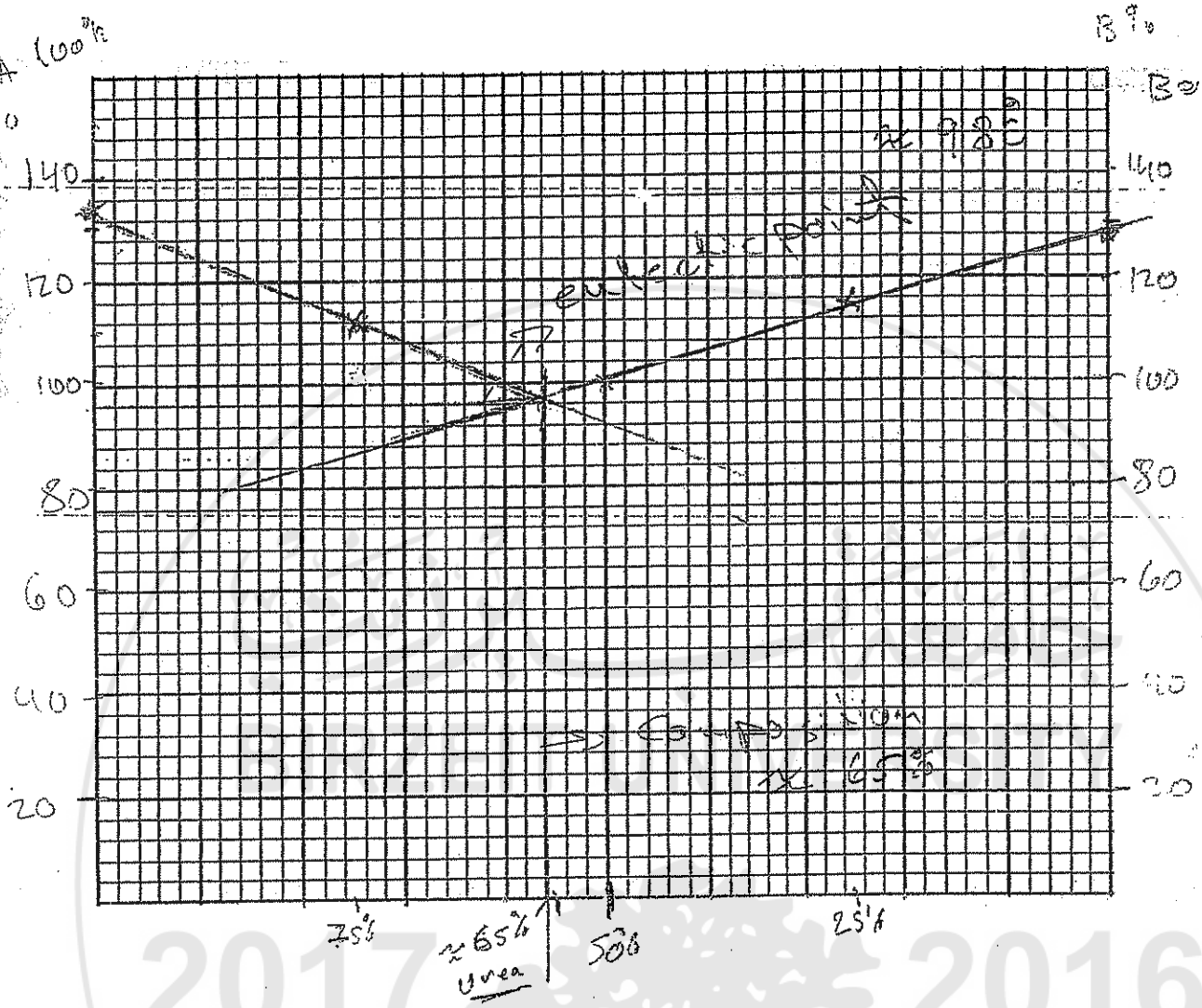
From the following data obtained, estimate what composition of urea and benzamide form a eutectic mixture.



Thermometer calibration curve.

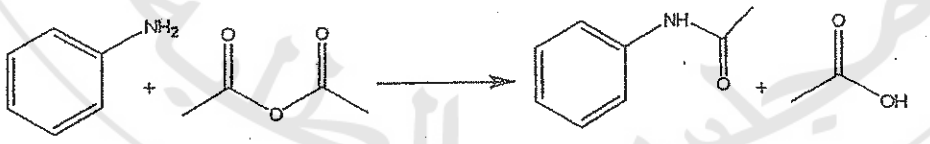
Corrected T = observed T + correction

Sample	Observed m.p (°C)	Corrected m.p (°C)
1) Urea	140°C	140+(-5) = 135
2) Benzamide	132°C	132+(-4.5) = 127.5
3) 75% Urea, 25% Benzamide	115°C	115+(-3) = 112
4) 50% Urea, 50% Benzamide	103°C	103+(-2.5) = 100.5
5) 25% Urea, 75% Benzamide	122°C	122+(-4) = 118



Q6 (2/15)

Assume that 3.0 ml of aniline and 4.5 ml of acetic anhydride were used in the preparation of acetanilide according to the following reaction.



	aniline	acetic anhydride	acetanilide	acetic acid
M.Wt g/mol	93.13	102.1	135.2	60.0
Density g/ml	1.0217	1.082	1.219	1.05
Wt(g) =	3.07	4.87		

a. What is the limiting reagent? What is the theoretical yield of acetanilide? What is the percentage yields if 3.3 g of acetanilide is obtained?

# of moles of Aniline = $\frac{wt}{MW} = \frac{3.07}{93.13} = 0.033$ mol L.R

# of moles of Acetic anhydride = $\frac{wt}{Mt} = \frac{4.87}{102.1} = 0.0477$ mol

⇒ Theoretical yield of acetanilide =

$0.033 \times 135.12 = 4.462$ g ⇒ % yield = $\frac{3.3}{4.462} \times 100 = 74\%$

b. If your experiment yield of acetanilide is greater than 100%, how could this occur?

* may it contained impurities
* may it's not dry.

c. Describe how you would separate a mixture of acetanilide and sand.

By dissolving acetanilide with organic solvent and then do filtration

Q7 (2/15)

Give at 20 °C only 0.24 g of an organic acid "A" dissolves in 100 ml of water, but 2.70 g of the same acid dissolves in 100 ml of ether.

Where: Distribution coefficient ,

$$KD = \frac{[solute]o}{[solute]aq} = \frac{Co}{Caq} = \frac{Wo/Vo}{Waq/Vaq}$$

a. Calculate the value Kd.

$$Kd = \frac{2.7g/100ml}{0.24g/100ml} = 11.25$$

b. Calculate the percentage of extraction if 0.12 g of acid extracted in 100 ml of aqueous solution.

$$11.25 = \frac{Wo/100ml}{0.12/100ml} \Rightarrow Wo = 1.35\,g \text{ extracted in } 100ml \text{ B.S.}$$

total grams = $1.35 + 0.12 = 1.47g \Rightarrow \%\ extraction = \frac{1.35}{1.47} = 91.8\%$

c. Calculate the volume of ether required to extract 85% of a 3.00 g of acid "A" in 100 ml solution.

grams extracted in ether = 2.55 g

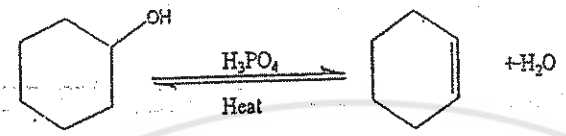
" " " water = 0.45 g

$$Kd = \frac{Wo/Vo}{Waq/Vaq} \Rightarrow 11.25 = \frac{2.55/Vo}{0.45g/100ml}$$

$$Vo = \frac{2.55g/100ml}{11.25 \times 0.45g} = 50.6\ ml$$

Q8 (2/15)

a. Cyclohexene, obtained from cyclohexanol by the following reaction (equilibrium reaction).



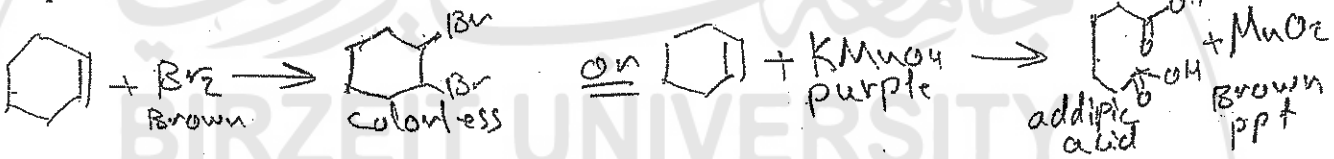
Cyclohexanol B.P 161°C — Cyclohexene B.P 83°C

1. How we can shift the reaction toward the products in order to achieve the maximum product? Give at least two techniques. 1- Distillation remove the product. 2- By drying agent. 3- Increase the conc. of the reactant

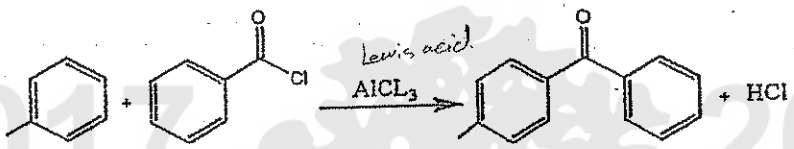
2. The major impurity of above reaction is water, how could we remove it?

Extraction

3. Write equation reaction for one test that would be done to indicate the product.



b. P-methyl benzophenone prepared according to the following reaction:



Toluene — benzoyl Chloride

a. Write the name of the reaction. Friedel-Crafts reaction (Acylation Reaction)

b. Write the name of all the reactants.

c. This reaction should be done under very dry conditions. Why? (Explain by reaction equation).

To prevent the hydrolysis of $AlCl_3$

$AlCl_3 + 3H_2O \rightarrow Al(OH)_3 + 3HCl \uparrow$

Good Luck

These are true and false questions. Circle T for the true statement and F for the false ones. (you will be given +1 point for the correct answer and –1/2 for the wrong one and zero for the blank).

T – F knowing that K_D values for acetone equals 1/13 and of chloroform is 1/19 in extracting citric acid from water. So chloroform is better extracting solvent than acetone, if we use 20ml of organic solvent to extract 30mg of citric acid dissolved in 20ml of water.

T – F The ease of dehydration of the following alcohols is:-
1 – phenylethanol 〉 tert – butyl alcohol 〉1 – propanol

T – F When you are collecting a solid by suction filtration, we always turn off the water pump before disconnecting the suction flask.

T – F Sodium is a good drying agent for cyclohexanol.

T – F A certain substance melting at 152 – 159° is suspected to be salicylic acid. To confirm this we do mixed melting point one time.

T – F Knowing that the distribution coefficient (K_D = C ether / C water), between ether and water for 2 – naphthol at room temperature is 3.5. The weight of 2–naphthol which would be removed by ____ extraction with 20ml portions of water is less than 0.67g. knowing that we have 5g of 2 – naphthol in 100ml ether

T – F Naphtalene is very soluble in benzene and slightly soluble in water, in contrast sodium butyrate is water soluble and in benzene is slightly soluble. So benzene is the best crystallizing solvent for naphthalene and water for sodium butyrate.

T – F In determination of melting point, using two much sample has no effect on melting point value but increase the melting point range.

T – F If two miscible liquids are each found to boil at exactly the same temperature, we conclude with no doubt that they are identical.

T – F Presence of sand as impurities in benzoic acid lowers its melting point.

T – F Ethanol is not a good solvent to extract aspirin from water because this solvent is flammable.

T – F The order reactivity addition of Br_2 in CCl_4 to following compounds is Benzene 〉 1,3 – cyclohexadiene 〉 cyclohexene.

T – F "Hold up" is a term used to show much an organic compound is absorbed to charcoal during crystallization.

T – F Acetanilicle is prepared from benzoic acid and methyl amine.

T – F If the thermometer bulb is not kept moist with condensate during distillation, the boiling point reading tends to be higher than what it should be.

٩

جامعة بيرزيت

BirZeit University

Chemistry Department

Chemistry 221

Mid Term Exam **Summer 2005**

Time: 50 min.

By: Dr. A. Laila

- **Student Name:_____**
- **Student No:_____**

GOOD LUCK

9/10

Date: 14.02.2013 **Org-Chem 221-Lab**
Student Name: Shatha Mari
Student Number: 1120716

Experiment #2

Distillation-Fractional Distillation
Azeotropic mixtures

Q.1

a. What effect would a decrease or increase in barometric pressure have on the boiling point?

the decrease of pressure will decrease the boiling point

2

b. The efficiency of the distillation increase or decrease with a decreasing of theoretical plates.

The efficiency decrease with a decreasing of theoritical plates of distillation

2

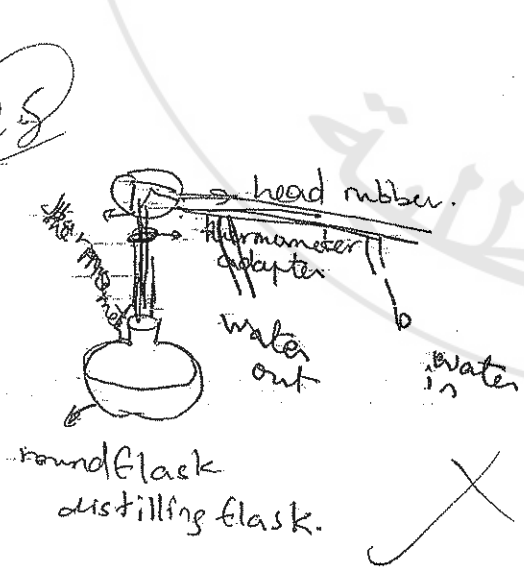
Q.2

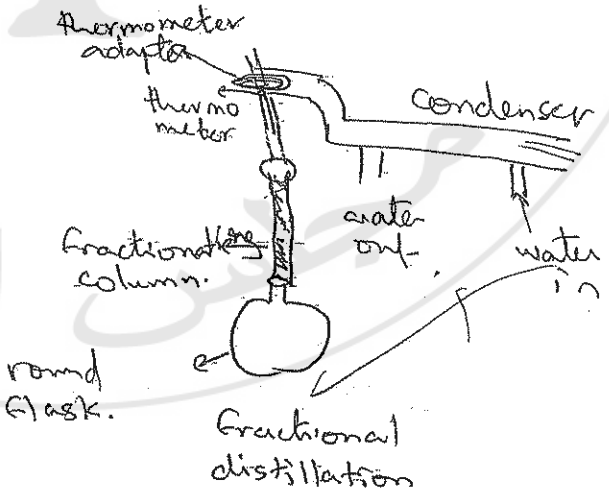
If two miscible liquids are each found to boil at exactly the same temperature, could you conclude that they are identical? Why?.

2.5 no, because there are some compounds that are found in petroleum for example that have nearly the same boiling point, therefore we need fraction distillation.

Q.3 Draw a simple distillation apparatus and fractional distillation apparatus and mention the difference between them.

2.5





Good Luck

simple distillation

Date: 21.02.2013
Student Name: Shatha Hani
Student Number: 1120716

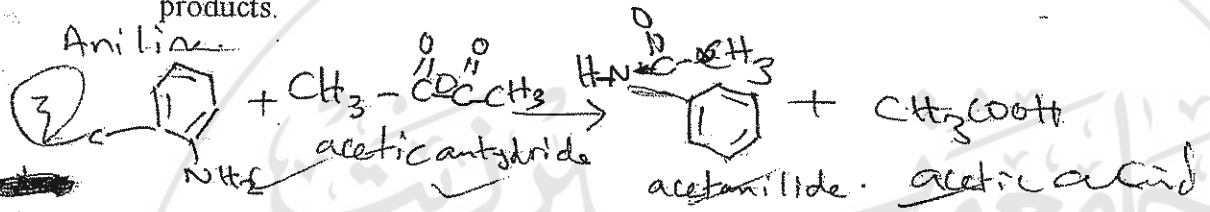
Org-Chem 221-Lab

9/10

Experiment #3

Recrystallization of Acetanilide Prepared from Aniline

Q1. Write a reaction equation of acetanilide preparation, mention all of the reactants and the products.



Q2. What properties are necessary for a solvent in order that it is well suited for recrystallizing a particular organic compound?.

1. It dissolves at high temp. ✓
2. It doesn't dissolve at room temp
3. Safe to use, not toxic

2

Q3. In our experiment, what is the best solvent will be used for the purification of acetanilide?

acetic hydride. ?

Q.4 a. Mention two types of filtration used in this experiment.

1. Suction filteration ✓
2. gravity filteration ✓

b. Which phase (saved) collected after each type (the filtrate or the solid)?

1- crystals (solid) ✓
2- filtrate. ✓

Good Luck

5/10

Date: 07.03.2013 **Org-Chem 221-Lab**

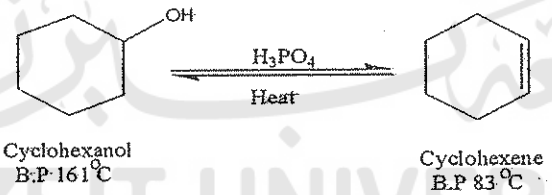
Student Name: Shatha Mari

Student Number: 1120716

Experiment # 5

Preparation of Cyclohexene from Cyclohexanol

Q-1. In this experiment, cyclohexene, obtained from cyclohexanol by the following reaction (equilibrium reaction). How we can shift the reaction toward the products in order to achieve the maximum product.

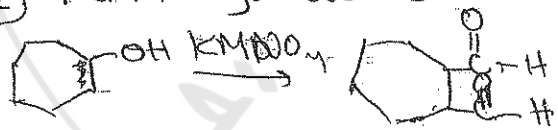


by adding H+ to the reactants, and then making another reaction forem water which produce carbonylcation and at last removing H ion from cyclohexanol to produce cyclohexene which will produce protonated cyclohexane.

Q-2. In order to check that our product is cyclohexene, we should do two tests, write the reaction equation for these tests.

(1) Bromine test

(2) Permanganate test



Q-3. What is the function of each of the following reagents in this experiment:

1. Phosphoric acid PO_4^- we used it so that we'd have cyclohexanol but slowly.
2. Anhydrous sodium sulfate Na_2SO_4?
3. Sodium carbonate solution 10% $NaCO_3$?
4. Saturated sodium chloride solution. → it's used as a drying agent

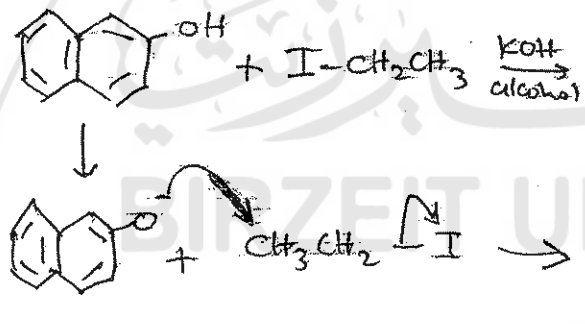
Good Luck

Date: 14.03.2013 Org-Chem 221-Lab
Student Name: Shatha Mari (5)
Student Number: 1120716

Experiment # 6

PERFUME-The Synthesis of Nerolin

Q-1 Describe how the order of reagent addition avoided a possible side reaction in this synthesis. Show a mechanism for this side reaction below.



We have to add KOH and dissolve it so that we'll get the salt and the nucleophile potasium β-naphthoxide and then we'll get add I-CH2CH3 to get the Nerolin

2.5

Q-2 In the experiment "The synthesis of Nerolin", by mistake one student react excess of KOH relative to the reactants. If this mistake affect on the reaction and the products. Explain

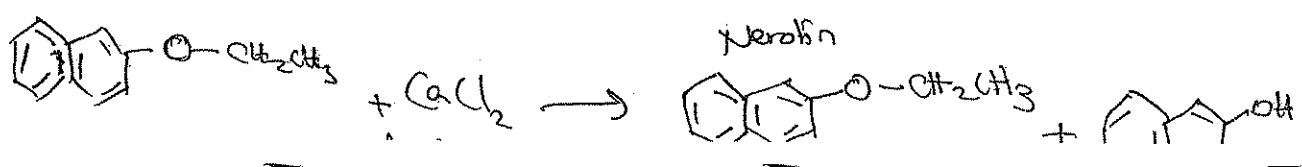
It will result in more and more salt (potasium β-naphthoxide) nucleophile and will cause an increase in the amount of Nerolin.

Q-3 If you suspect that the product nerolin contained unreacted β-naphthol, describe method (purification technique & reaction equation) to purify the product and remove the contaminant.

(we'll use the seperatory funnel) in which when we put and shake the product their will be two layers and we'll get rid of the β-naphthol layer. Good Luck

2

Nerolin



9+2 ⇒ 11/10

Date: 28.03.2013 Org-Chem-221-Lab

Student Name: Shatha Mari

Student Number: 1120716

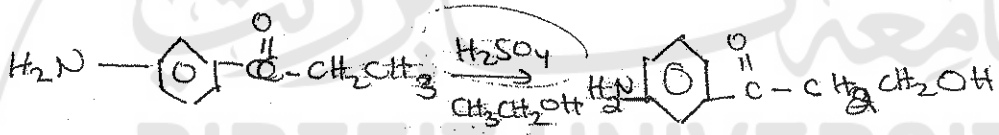
+2

Experiments # 8

The Synthesis of Benzocaine

Q-1 In this experiment (**The synthesis of benzocaine**).

a. Explain by using balanced chemical equations, The synthesis of benzocaine from P-aminobenzoic acid.



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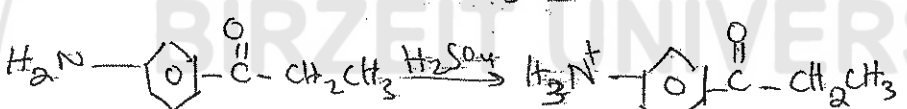


When we add H_2SO_4 it will be changed into an acid (ion) and when we add Na_2CO_3 it will precipitate

b. The crude product should be washed with aqueous sodium bicarbonate. Why?

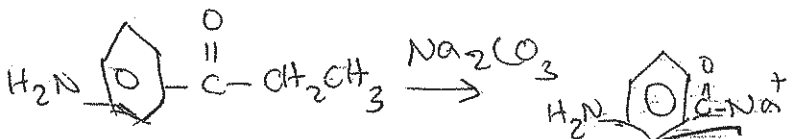
So that the crude product will not turn into acid again (to stop the job of H^+)

1

Q-2 If you suspect that the product (Benzocaine) contained un reacted P-aminobenzoic acid, describe method purification technique to purify the product and remove the contaminant, write reaction equation.

We will use the extraction technique in which

5



Good Luck

Date: 25.04.2013
Student Name: Shatha Mari
Student Number: 1120716

6/10

Org-Chem 221-Lab

Experiment # 11

Chromatography

Q-1 In column chromatography why does one begin with a non-polar eluting solvent and then progress to solvent of higher and higher polarity?

Because if we started with the higher polarity it will dissolve quickly and react quickly so we won't be able to recognize the rate.

3/4

Q-2 An unknown compound, X, was analyzed by TLC using two different eluting solvent mixtures. Three standard compounds, G, H, and I, were used. The Rf values for the analyses are given below. Which of the three standards is most likely to be compound X? Explain your reasoning in two short sentences.

5:1 Hexane: Acetone		3:1 Hexane: Ethyl acetate	
Cpd.	Rr	Cpd.	Rf
G	0.35	G	0.55
H	0.65	H	0.95
I	0.65	I	0.85
X	0.65	X	0.95

α

$Rf = \frac{\text{distance of the compound}}{\text{distance of the solvent}} =$

$0.95 = \frac{x}{0.65}$

$x = H$

Q-2 What would happen if your solvent level is above the level of the initial spots?

we can't see the rate of the solvents correctly. because it shows that we started with higher polar than the ones.

3/4

Good Luck

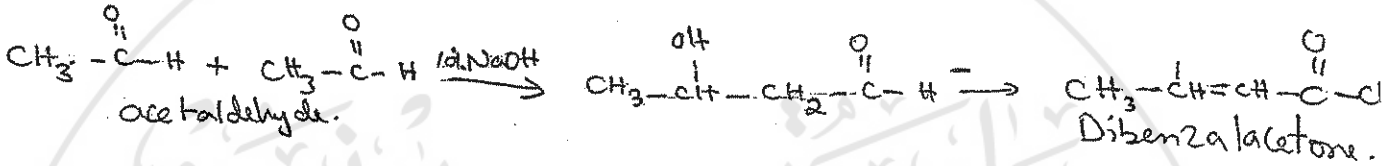
Date: 04.04 .2013
Student Name: Shatha Hani
Student Number: 1120316

Org-Chem 221-Lab

Experiment # 10

Aldol condensation: Preparation of Dibenzalacetone

Q-1 a. In this experiment (**Aldol condensation**). Explain by using balanced chemical equations, the preparation of Dibenzalacetone.



b. write in brief step-by-step the procedure and mention the purpose of all the that will be used.

(1)

Good Luck

Birzeit University

Chemistry Department

Chemistry 221- Lab

Instructor: Adel Hidmi

Second Semester. 2010/2011

Midterm Exam

Time: 60 min.

Key

- **Student Name:** _____
- **Student No:** _____

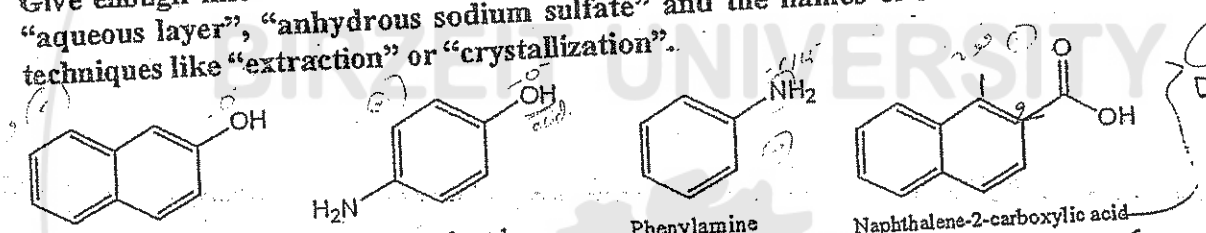
GOOD Luck

Q1 (2/15) A mixture solution of 1-propanol (B.P = 97.1 °C, 50ml) and toluene (BP 110.6 °C, 50ml) was distilled by fractional distillation and collected in 10 ml fractions. Predict the boiling range of each fraction.

fractions	vol. collected(ml)	temp. °C (predicted)
1	10	~ 97 - 98
2	20	~ 97 - 98
3	30	~ 98 - 99
4	40	~ 99 - 100
5	50	~ 100 - 105

Q2 (2/15)

Draw a flow chart to show how to separate the following four compounds from each other. Give enough information so that someone could do it –include stuff like "organic layer", "aqueous layer", "anhydrous sodium sulfate" and the names of solvents used, as well as techniques like "extraction" or "crystallization".



Disolve in ether

NaHCO3

organic phase

aq. phase

separate

NaOH

aq.p

ppt

crystalization

HCl

base

increasing E

decreasing basicity

HCl

ppt

evaporate ether

& dry it!

ppt

aq.ph

Filtration

benzocaine

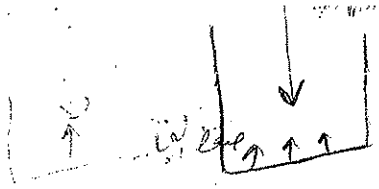
pKa = 10.0

NaHCO3

+ Na2CO3

+ HCO3⁻ conj. acid

pKa = 10.2



↓ pressure ↓ B.P.
external pressure
apply heat
Liquid → gas
Liquid ← gas
Vapor pressure = ...
... initially ... equilibrium

Q3 (2/15)

a. The normal boiling point of cyclohexanol is 160.5 °C. What is the vapor pressure of cyclohexanol at 160.5 °C?

B.P.

1 atm = 760 mmHg

b. Given the following mole fraction and vapor pressures for miscible liquids A and B, calculate the composition (in mole percentage) of the vapor from a distilling an ideal binary solution at 110 °C and 760 mmHg for the solution

X P

XA = 0.50 Liquid mix. PAo = 1216 mmHg

XB = 0.05 PBo = 3040 mmHg ⇒ 3040 mmHg

4 equations

Raoult's law.

Where: Ptotal = PA + PB = XA. PAo + XB. PBo

$P_{total} = 0.5 \times 1216\ mmHg + 0.05 \times 3040\ mmHg = 760\ mmHg$

$\%A = \frac{0.5 \times 1216\ mmHg}{760\ mmHg} \times 100\% = 80\%$ In vapor.

$\%B = \frac{0.05 \times 3040}{760} \times 100\% = 20\%$

Q4 (2/15)

a. The solubility of compound A in ethanol is 0.5 g per 100 ml at 0 °C and 5.0 g per 100 ml at 75 °C. What is the minimum amount of solvent needed to recover 9.0g of compound A, after recrystallization processes? How much would be lost in the recrystallization?

(4.5) ppt. recovered

recrystallization

in 100 ml → 5.0 − 0.5 = 4.5 g recovered ⇒ grams lost

4.5 g → 200 ml

9 g → ?

$\frac{9.0 \times 100\ ml}{4.5} = 200\ ml$

0.5 g → 100 ml

? → 200 ml

$\frac{0.5 \times 200\ ml}{100\ ml} = 1.0\ g$

b. The solubility of acetic acid in hot water (15 g/100 ml at 100 °C), and its solubility in cold water (2.0 g/ 100 ml at 0 °C). What would be the maximum theoretical yield recovery from the crystallization of 10.0 g of ~~acetanilide~~ acetic acid from 200 ml water at 0 °C?

cooling

grams lost ⇒ 2.0 g — 100 ml

? — 200 ml ⇒ $\frac{2.0\ g \times 200\ ml}{100\ ml} = 4.0\ g$

used at high temp.

theoretical yield ⇒

⇒ grams recovery = 10.0 g − 4.0 g = 6.0 g

15 g → 100 ml at 100 °C.

66.7 ml

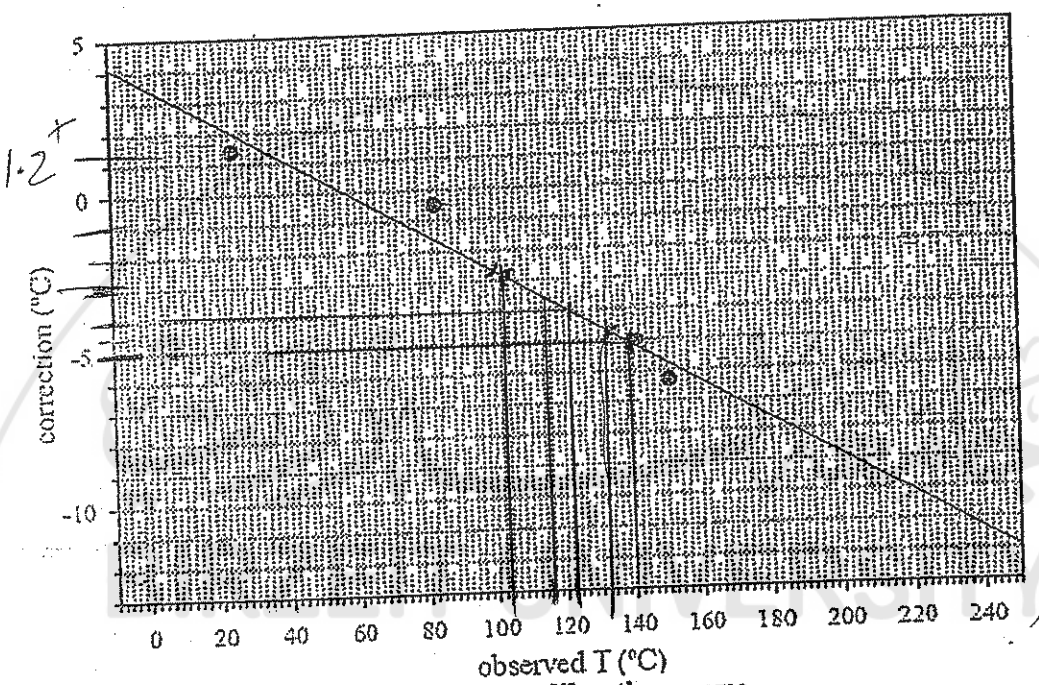
2.0 g → 100 ml at 0 °C.

66.7 ml

1.3 g soluble

Q5 (2/15)

From the following data obtained, estimate what composition of urea and benzamide form a eutectic mixture. ≡ low point



Thermometer calibration curve.

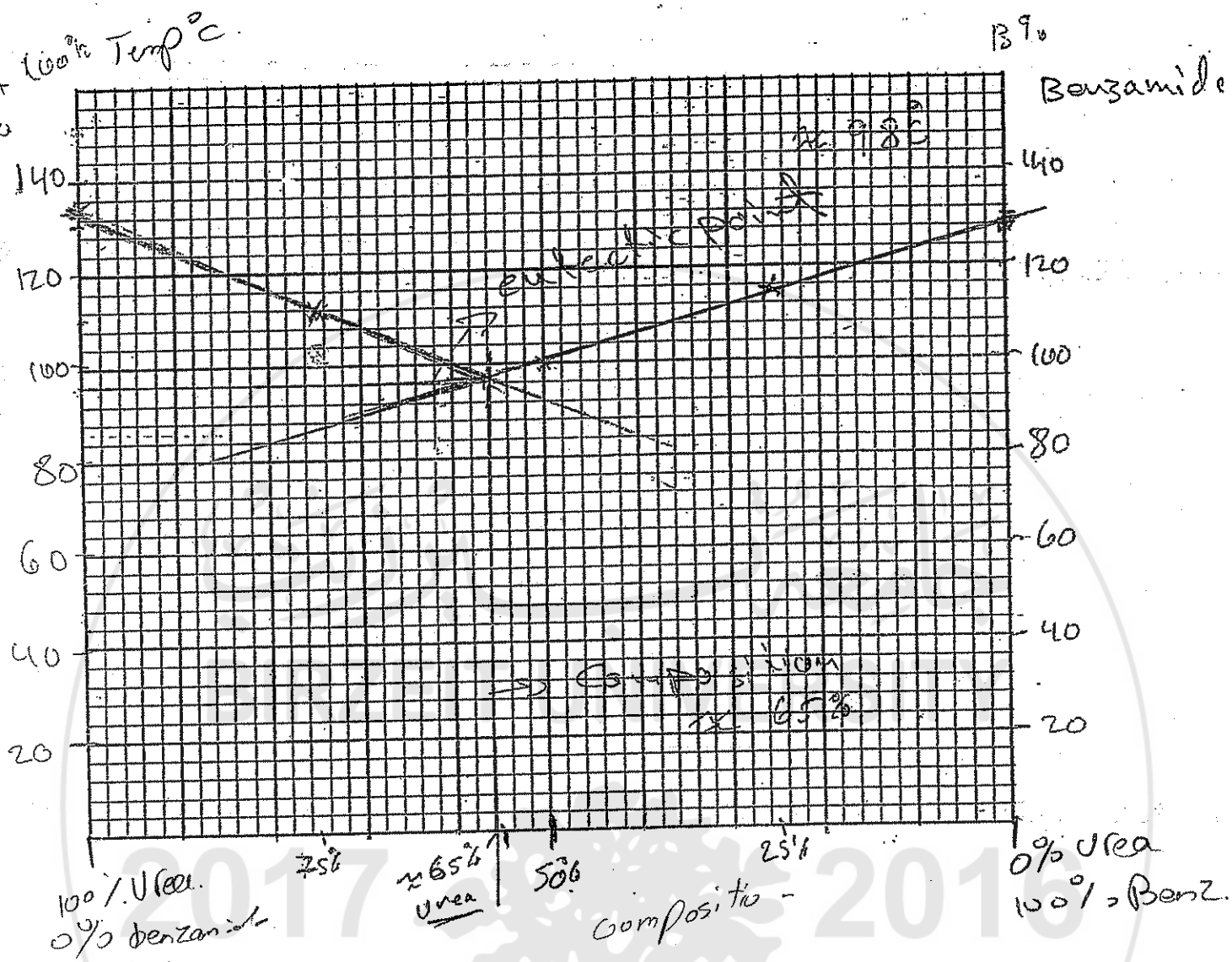
Corrected T = observed T + correction

140 + -5

Sample	Observed m.p (°C)	Corrected m.p (°C)
1) Urea	140°C	140 + (-5) = 135
2) Benzamide	132°C	132 + (-4.5) = 127.5
3) 75% Urea, 25% Benzamide	115°C	115 + (-3) = 112
4) 50% Urea, 50% Benzamide	103°C	103 + (-2.5) = 100.5
5) 25% Urea, 75% Benzamide	122°C	122 + (-4) = 118

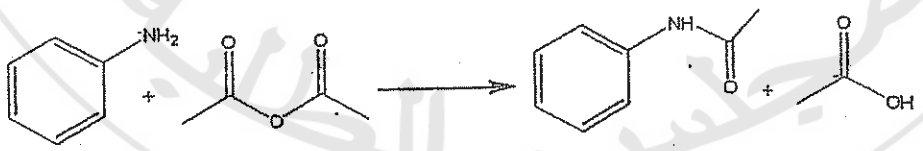
observed 140°C — 135°C Literature — correction

correction −5 = b



Q6 (2/15)

Assume that 3.0 ml of aniline and 4.5 ml of acetic anhydride were used in the preparation of acetanilide according to the following reaction.



	aniline	acetic anhydride	acetanilide	acetic acid
M.Wt g/mol	93.13	102.1	135.2	60.0
Density g/ml	1.0217	1.082	1.219	1.05
Wt(g) =	3.07	4.87		

a. What is the limiting reagent? What is the theoretical yield of acetanilide? What is the percentage yields if 3.3 g of acetanilide is obtained?

# of moles of Aniline $= \frac{wt}{MW} = \frac{3.07}{93.13} =$ 0.033 mol L.R

# of moles of Acetic anhydride $= \frac{wt}{MW} = \frac{4.87}{102.1} =$ 0.0477 mol

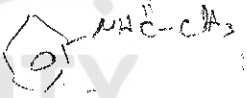
$\Rightarrow$ Theoretical yield of acetanilide = $0.033 \times 135.2 =$ 4.462 g $\Rightarrow$ % yield $= \frac{3.3}{4.462} \times 100 = 74\%$

b. If your experiment yield of acetanilide is greater than 100%, how could this occur? Recrystallization

* may it contained impurities
* may it is not dry.

c. Describe how you would separate a mixture of acetanilide and sand.

By dissolving acetanilide with organic solvent and then do filtration

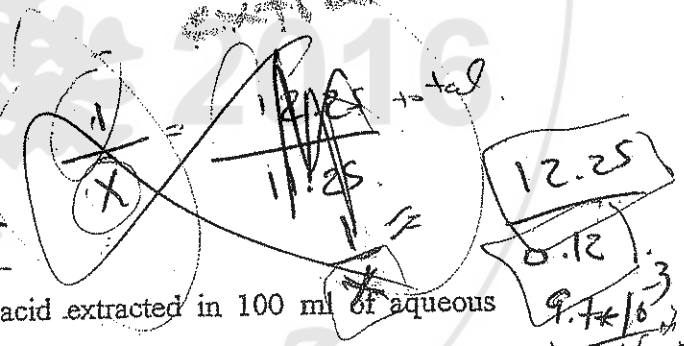


Q7 (2/15)

Give at 20 °C only 0.24 g of an organic acid "A" dissolves in 100 ml of water, but 2.70 g of the same acid dissolves in 100 ml of ether.

Where: Distribution coefficient,

$$KD = \frac{[solute]o}{[solute]aq} = \frac{Co}{Caq} = \frac{Wo/Vo}{Waq/Vaq}$$



a. Calculate the value Kd.

$$Kd = \frac{2.7\,g/100ml}{0.24\,g/100ml} = 11.25$$

b. Calculate the percentage of extraction if 0.12 g of acid extracted in 100 ml of aqueous solution.

$$11.25 = \frac{Wo/100ml}{0.12/100ml} \Rightarrow Wo = 1.35\,g \text{ extracted in } 100ml$$

total grams: $1.35 + 0.12 = 1.47\,g \Rightarrow$ % extraction $= \frac{1.35}{1.47} =$ 91.8%

c. Calculate the volume of ether required to extract 85% of a 3.00 g of acid "A" in 100 ml solution.

grams extracted in ether = 2.55 g
grams in water = 0.45 g

$$Kd = \frac{Wo/Vo}{Waq/Va} \Rightarrow 11.25 = \frac{2.55\,g \times Vo}{0.45\,g/100ml}$$

$$Vo = \frac{2.55\,g \times 100ml}{11.25 \times 0.45\,g} = 50.6\,ml$$

$$\frac{\text{total } 0.12}{X} = \frac{12.25}{1} \quad \text{total water}$$

9.7×10^{-3} remain in water

97

BIRZEIT UNIVERSITY
Chemistry Department
Chemistry 221

Mid-Term Exam
Time:75 minutes

2nd Sem.2012/2013
April.23, 2013.

Student Name:....Hanieen Nakhleh..... Student No:....1110954.
Laboratory Instructor:

☐ Dr.Imad Qamhieh Monday at 2:00 ☑ Dr. Simon Kuttab, Tuesday at 11:00

☐ Dr.Adel Hidmi Thursday at 11:00 ☐ Dr. Sami Sayrafi , Thursday at 2:00

I-(60%) Circle the correct answer

1. Compounds A and B both have melting points of 140°C. If A is mixed with B at a ratio which leads to melting at the eutectic point, the melting point of the mixture

a) will remain 140°C (b) will be less that 140°C
c). will be greater than 140°C d) will contain two separate melting points

2- A compound can be separated by steam distillation if it satisfies the following conditions:

a) polar, viscous and water insoluble b) high melting point, water miscible and polar
(c) thermally stable, has some vapor pressure and water insoluble
d) polar, has very low vapor pressure and high melting point

3. An ideal recrystallization solvent will

a) dissolve lots of the compound at room temperature and little at high temperatures
b) dissolve lots of the compound at room temperature and lots at high temperatures
c. dissolve very little of the compound at any temperature
(d) dissolve little of the compound at room temperature and lots at high temperatures
e) react with the compound being recrystallized

4-In the preparation of cyclohexene, you performed a potassium permanganate test on the product. Hopefully, you witnessed the immediate formation of a brown precipitate. What was the precipitate?

a) Mn_2O_3 (b) MnO_2 c. cyclohexanol d) 1,2-cyclohexanediol

5. If you take the melting point of an impure substance, the observed melting point range will be _____ relative to the melting point range observed for the pure substance.

a) higher and broader b) higher and narrower (c) lower and broader
d) lower and narrower e) neither higher nor lower, but broader

6. When a solution is cooled very rapidly in recrystallization:

a) no crystals will form b) ideal recrystallization takes place
c) the impurities don't have time to stick to the crystals
d) small crystals form that have a large surface area on which impurities will be absorbed

7- Why do you add boiling chips to a solution that will be heated?

a) to keep the solution homogeneous b) to increase the rate of reflux
c) to prevent bumping d) to lower the boiling point

8- When performing a distillation of any type, it is necessary for the system to have an opening to the outside, otherwise excess pressure can build up inside the apparatus and:
a) lower the boiling point. b) keep the sample from distilling.
c) decompose the sample. d) cause the apparatus to explode.

9- Which extraction procedure below would you use to isolate benzoic acid from a mixture of neutral organic compounds?

a) dissolve in dichloromethane, extract with base, acidify solution, filter precipitate
b) dissolve in dichloromethane, extract with base, extract aqueous with dichloromethane
c) dissolve in dichloromethane, extract with acid, extract aqueous with dichloromethane
d) dissolve in dichloromethane, extract with acid, neutralize solution, extract aqueous with dichloromethane
e) dissolve in dichloromethane, extract with base, acidify solution, extract aqueous with dichloromethane

10- A common measure of the efficiency of a fractionating column is

a) the number of components in the solution b) the number of theoretical plates in the column
c) the number of basic types of distillation methods
d) the variation in temperature at the top of the column

11- The primary reason for the addition of anhydrous magnesium sulfate to the organic solvent layer resulting from the extraction of an organic compound from water is to

a) remove residual water b) remove residual NaCl
c) remove residual acid catalyst d) neutralize the residual NaOH

12- What device is used in the chemical laboratory to separate two immiscible liquids of differing density

a) Buret b) Erlenmeyer Flask c) Dropping funnel d) Separatory funnel

13- Which statement is correct regarding the **boiling point** of a pure liquid:

a) It is the temperature at which the total vapor pressure of the liquid is equal to the external (atmospheric) pressure.

b) Boiling points can be determined using the technique of steam distillation.

c) An alternative method to determine the boiling point is by gas chromatography

d) Variation in the external pressure has little or no effect on the boiling point.

14- Recrystallized product is most efficiently collected by:

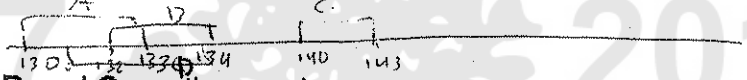
a) vacuum filtration b) gravity filtration c) solvent evaporation d) extraction

15- In the preparation of cyclohexene from cyclohexanol, what reaction conditions would not favor the formation of cyclohexene?

a) the acid used has a conjugate base which is a poor nucleophile
b) high reaction temperature
c) remove product from the reaction mixture as rapidly as it is formed
d) use a strong base to form cyclohexene

16- Sulfuric acid serves the following function in the dehydration of cyclohexanol:

a) neutralizes the basic cyclohexanol b) protonates the hydroxyl group
c) neutralizes the sodium bicarbonate d) converts hydrogen to a better leaving group



17- Compounds **A**, **B** and **C** are three unknown compounds. Compound **A** melts at 130-133°C, Compound **B** melts at 132-134°C and Compound **C** melts at 140-143°C. Compound **D** is a known compound which melts at 131-134°C. The mixed mp **A+B** is 131-133°C. The mixed mp **A+C** is 138-142°C. The mixed mp **B+C** is 139-144°C. The mixed mp **A+D** is 130-134°C. The mixed mp **C+D** is 134-140°C. What can you conclude about your unknowns?

a) A=B=D b. B=C=D c. A=B=C d. B=C e. C=D

18-Which of the following statements is correct regarding steam distillation:

a) the two components (water and organic) behave as distinct entities (independent).

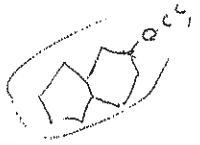
b) It follows Raoult's Law: $P_{total} = P°_A \times N_A + P°_B \times N_B$, (observed $P_A = P°_A N_A$)
c) It works only for separation of liquids d) distillation proceeds at around 150 °C.

19- When you do a recrystallization experiment, the impurities should either be very insoluble or highly soluble in the recrystallization solvent. If they are insoluble they may be removed by _____ at _____ temperatures.

a) filtration, high b) recrystallization, high c) filtration, low
d) extraction, high e) extraction, low

20- Which compound can be classified as a fixative?

a) 2-ethoxynaphthalene b) β-naphthol c) cyclohexene d) naphthalene



II.(5%) Five grams of Compound A is dissolved in 90 mL of water. The distribution coefficient for Compound A between hexanes and water is 5 (in other words for compound A, $K_{(hexanes/water)} = 5$.) How much of Compound A will be in the hexanes if you extract it from the water one time with 90 mL of hexanes?

$K_D = \frac{[C] \text{ in hexane}}{[C] \text{ in water}}$

$5 = \frac{wt_{hexane} / V_{hexane}}{wt_{water} / V_{water}}$

$5 = \frac{X/90}{5-X/90} \rightarrow 5 = \frac{X}{5-X} \rightarrow 25 - 5X = X$

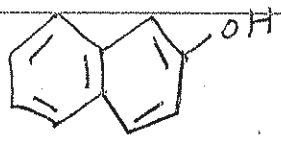
$25 = 6X$

$X = 4.166$ g of A in hexane

0.834 g of A in water

III.(10%) In the preparation of nerolin, a student used 40 g of methanol(MW: 32), 6.5 g of β-Naphthol (MW:144.17) and 3.3 g of potassium hydroxide(MW: 56.11) and then added 7.8 g of ethyl iodide (MW: 156). At the end of the reaction, the student obtained 6.0 g of pure nerolin. 172.17

1) Write the **equation** of this reaction.
2) What is the **theoretical yield** of this reaction?
3) What is the **percent yield** of this reaction?



$+ CH_3CH_2I \xrightarrow{KOH}$

1) β-naphthol → moles $= \frac{6.5}{144.17} = 0.045$ mol → β-naphthol is the limitting reagent

ethyl iodide → moles $= \frac{7.8}{156} = 0.05$ mol

theoretical yield = mwt of nerolin × 0.045 mol

$172 \times 0.045 = 7.74$ g

3) % yield $= \frac{\text{exp. wt}}{\text{theoretical wt}} \times 100\%$

$= \frac{6.0}{7.74} \times 100\% = 77.52\%$

IV. (15%)You want to purify 10 g of Compound A that has been contaminated with 0.2 g of Compound B. Solubilities in water of the two compounds are:
Compound A: 0.029 g/10mL at 20°C and 0.68g/10 mL at 100°C.
Compound B: 0.22 g/10mL at 20°C and 6.67g/10 mL at 100°C.

a) What volume of boiling water is needed to dissolve the 10 g of Compound A?
b) How much Compound A will crystallize after cooling to 20°C?
c) Will any Compound B crystals also form?
d) What is the maximum amount of Compound A that can be recovered in the first crop of this recrystallization?
e) Will the Compound A be pure?

(a) 0.68 g → 10 ml
10 g → ?
147 ml of water.

(b) solubility at 20 C°
0.029 → 10
s ← 147
0.4263 g

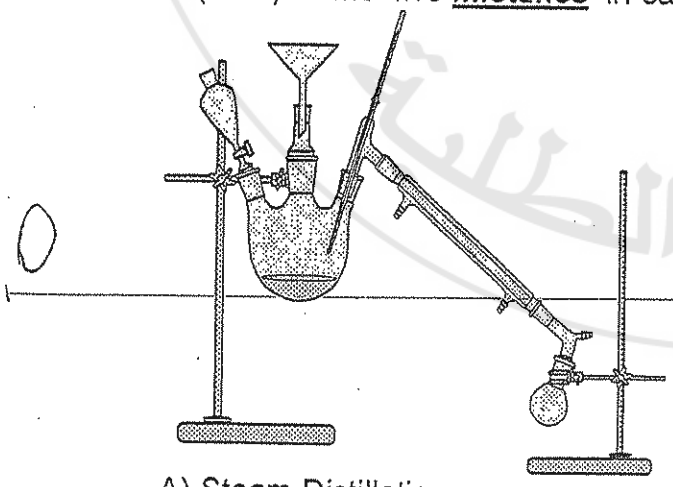
10 g - 0.4263 = 9.5737 g crystall.

(c) at 20 c°
0.22 g → 10 ml
s ← 147
3.234 g soluble of B in sln
so no crystals of B will be formed.

(d) 9.5737 g of A recovered.

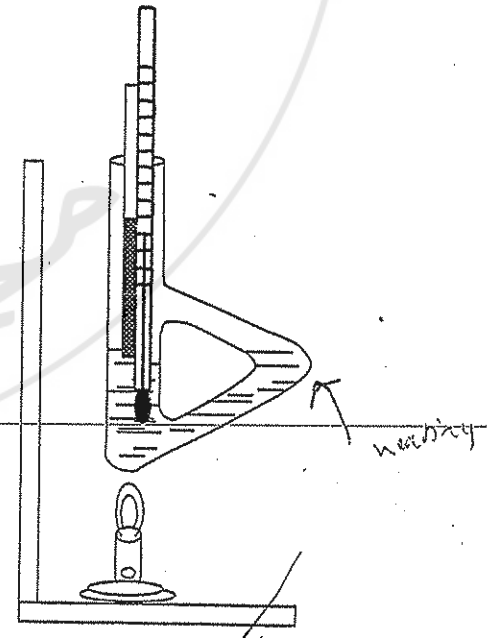
(e) compound A will be pure

V.(10%) Name five ***mistakes*** in each setup



A) Steam Distillation

→ closed system due receving flask must not be round bottom flask → it must be erlanmeyer flask.
(2) no clamps
(3) no thermometer adapter
(4) no boiling chips
(5) no bunsen burner and ring or wire goes



B) Melting point

→ (1) capillary not at the same level as thermometer bulb ending
→ (2) no ring and wire goes
→ (3) no clamp.
→ 4 heating must be from the other side
→ (5) capillary contains too much of substance

A : 15g → 100ml hot water

A : 2g → 100 ml 0°C "

I. (10pts) The solubility of compound A in hot water in 15g/100ml at 100°C and its solubility in cold water is 2.0g/100ml at 0°C. What would be the maximum theoretical yield recovery from the recrystallization of 10.0g of compound A from 200ml of water at 0°C.

2

2g → 100 ml
? ← 200 ml
? = 2×200/100 = 4g maximum theoritical yeild

10.0g dissolved
200 ml

Theor. yield: 10 − 4 = 6g

% 6/10 * 100 = 60%

2g → 100 ml
10g → ?
= 500 ml of water
and we have only 200 ml of H_2O, so it is the limiting agent.

II (5pts) What is a eutectic mixture and what is its main characteristic

2.5

eutectic mixture is the mixture of ~~two~~ compounds, that ~~are~~ have eutectic melting point; Lower possible melting point the we can get by combining different concentrations from the two component.

→

III. (5pts) When determining the melting point of an unknown compound, it is important to raise the temperature slowly. What would be the error if the temperature is raised too quickly.

3

If the temperature was raised too quickly ~~you~~ you will not be able to notice the exact ~~melting~~ temperature on the thermometer because it will rise quickly and you will ~~get~~ get a wide ~~range~~ and not accurat ~~ate~~ reading for the melting point.

lower or higher M.P.

There are are ... aldehydes and ketones which are ... reaction is the aldol condensation. this reaction ... to produce many compounds. The word aldol came ... aldehyde and the last two letters of the word Alcohol, and it is ... of these functional groups, and this reaction can be called an "aldol ac..."; the type of reaction gives it the name "aldol condensation", this reaction

IV. (6pts) In recrystallization, medium sized crystals are preferred. Explain.

6

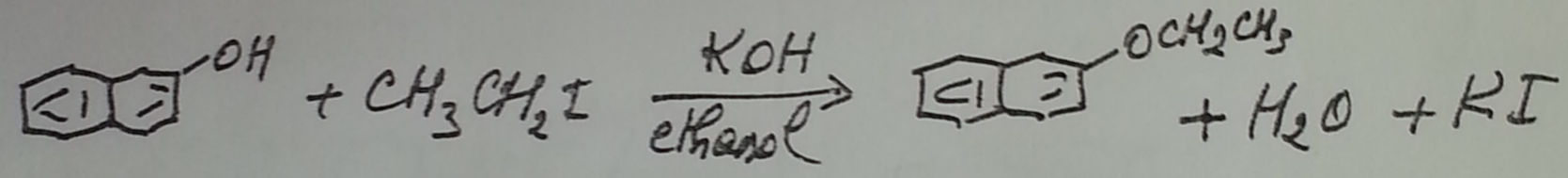
* Because fine crystals have very large surface area, ~~and this must be~~ in which the impurities will be ~~adsorbed~~ by those fine crystals (and won't be easy to remove)
* And the very large crystals will absorbe the impurities so the impurities will be ~~trapped~~ embeded inside the ~~s~~ crystals and can't be removed.
* that is why medium sized crystals are preferred, even if some ~~impurities~~ impurities had ~~been~~ been attached on the surface of the crystals it can be removed easily ~~by~~ washing the crystals with cold solvent.

V. (6pts) you prepare a fractionating column by filling it with glass beads. The glassware in the laboratory is not completely the same. You notice that when your neighbor packs (fills) his column, he was able to fit a lot fewer beads in the column, than you. Whose column will be more efficient and why?

6

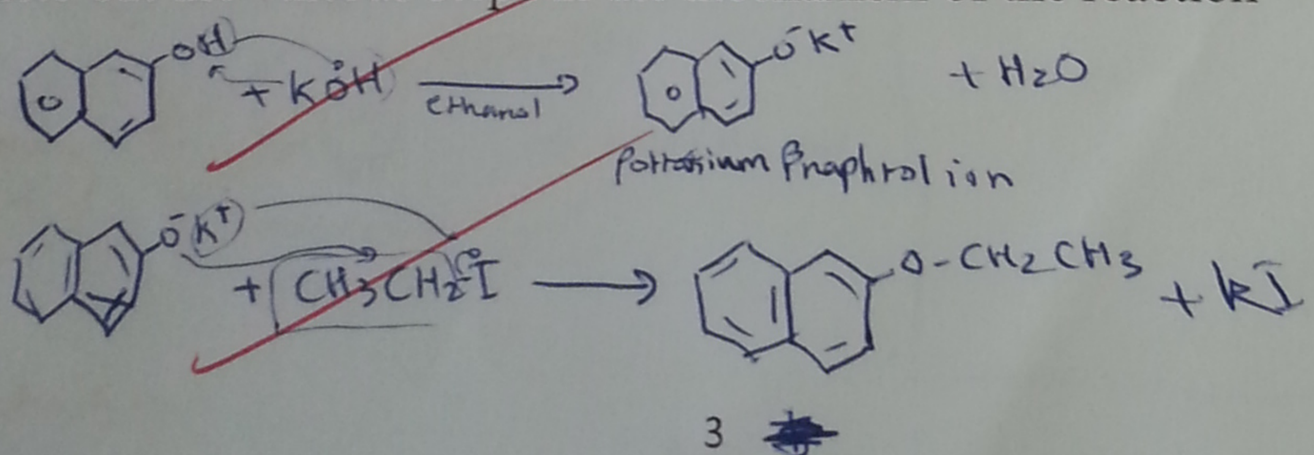
The column which have more beads inside it will be more efficient because it have more surfaces / area & larger no. of to work at the theoritical plates

VI. (13 pts) Nerolin can be synthesized by the following reaction which is an example of the Williamson synthesis of ether



4

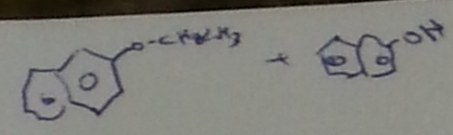
a. Write out the various steps in the mechanism of the reaction



3

Neroline

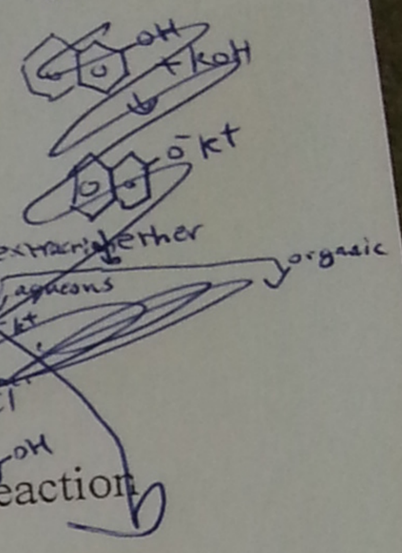
There are are re... aldehydes and ketones which are ... reaction is the aldol condensation. this reaction ... used to produce many compounds. The word aldol came ... and the last two letters of the word Alcohal, and it is ... ; The reaction ... functional groups, and this reaction can be called an "aldol addition" ... gives it the name "aldol condensation", this reaction



b. If you suspect that the product nerolin contained unreacted β-naphthol, describe method (purification technique and reaction equation) to purify the product and remove the β-naphthol.

4

we add KOH to the aqueous layer and this will convert the β-naphthol that is init to a potassium β-Naphthol ion and then we do extraction again using ether and water, the two layers appears the aqueous layer will have the potassium β-naphthol ion, we take this aqueous layer and acidify it using concentrated HCl, this will give back the β-Naphthol that we want.

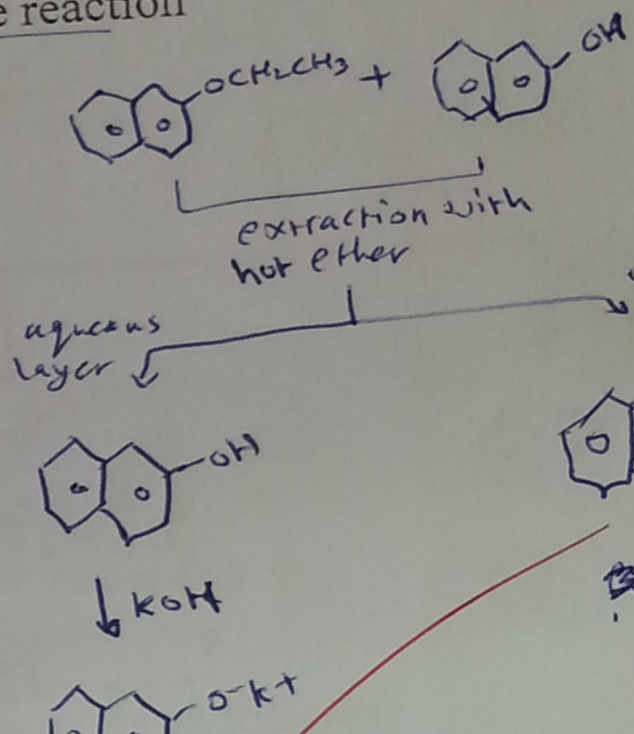


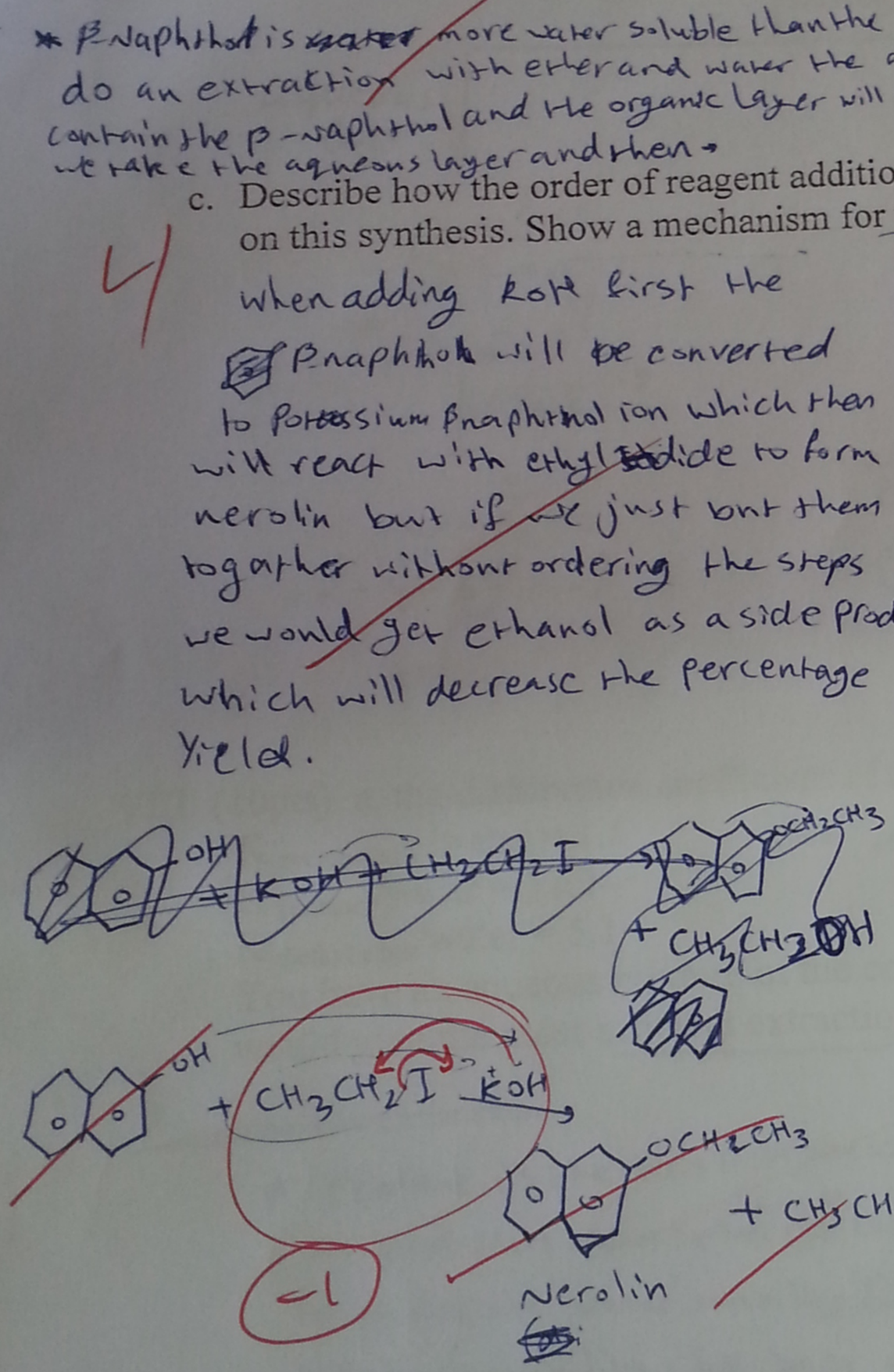
* β-Naphthol is more water soluble than the Nerolin, so we do an extraction with ether and water the aqueous layer will contain the β-naphthol and the organic layer will contain the Nerolin we take the aqueous layer and then →

c. Describe how the order of reagent addition avoided a possible side reaction on this synthesis. Show a mechanism for the side reaction

4

when adding KOH first the β-naphthol will be converted to Potassium β-naphthol ion which then will react with ethyl iodide to form nerolin but if we just dont them together without ordering the steps we would get ethanol as a side product which will decrease the percentage Yield.





Mechanism

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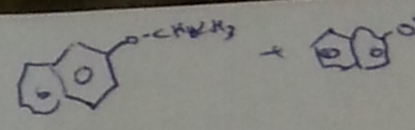

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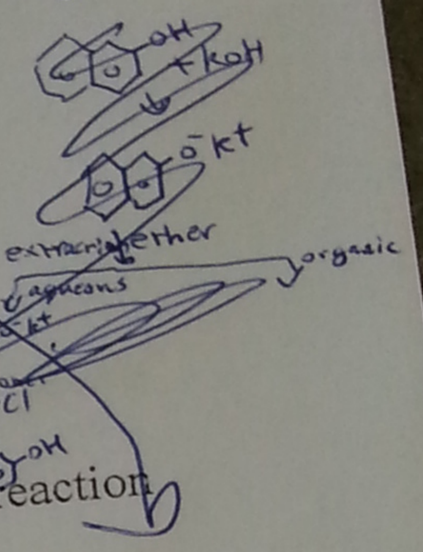

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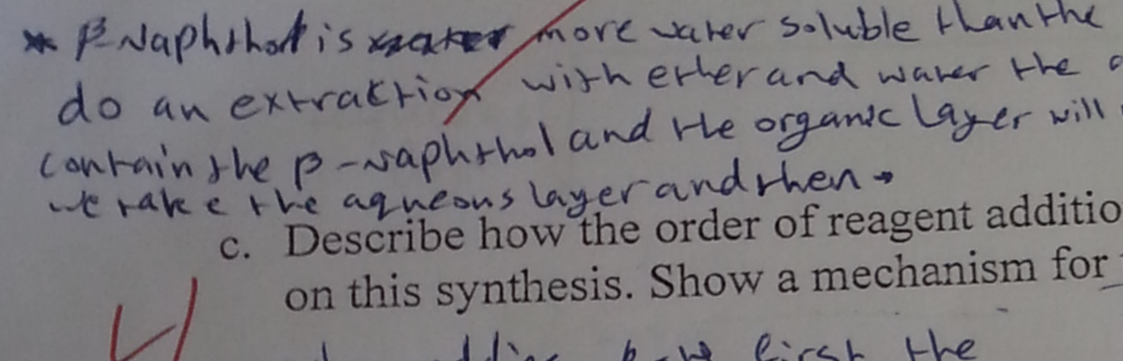

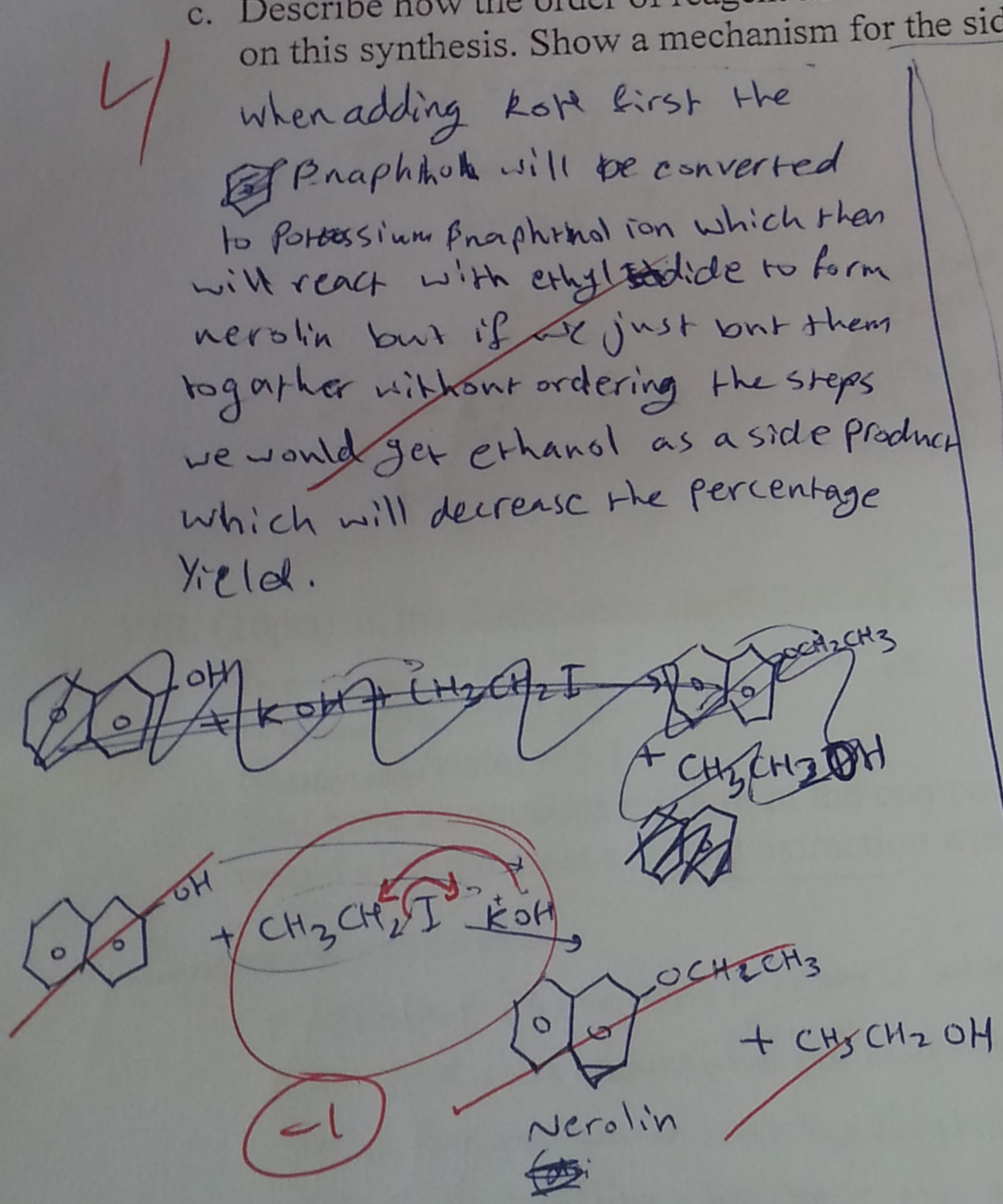

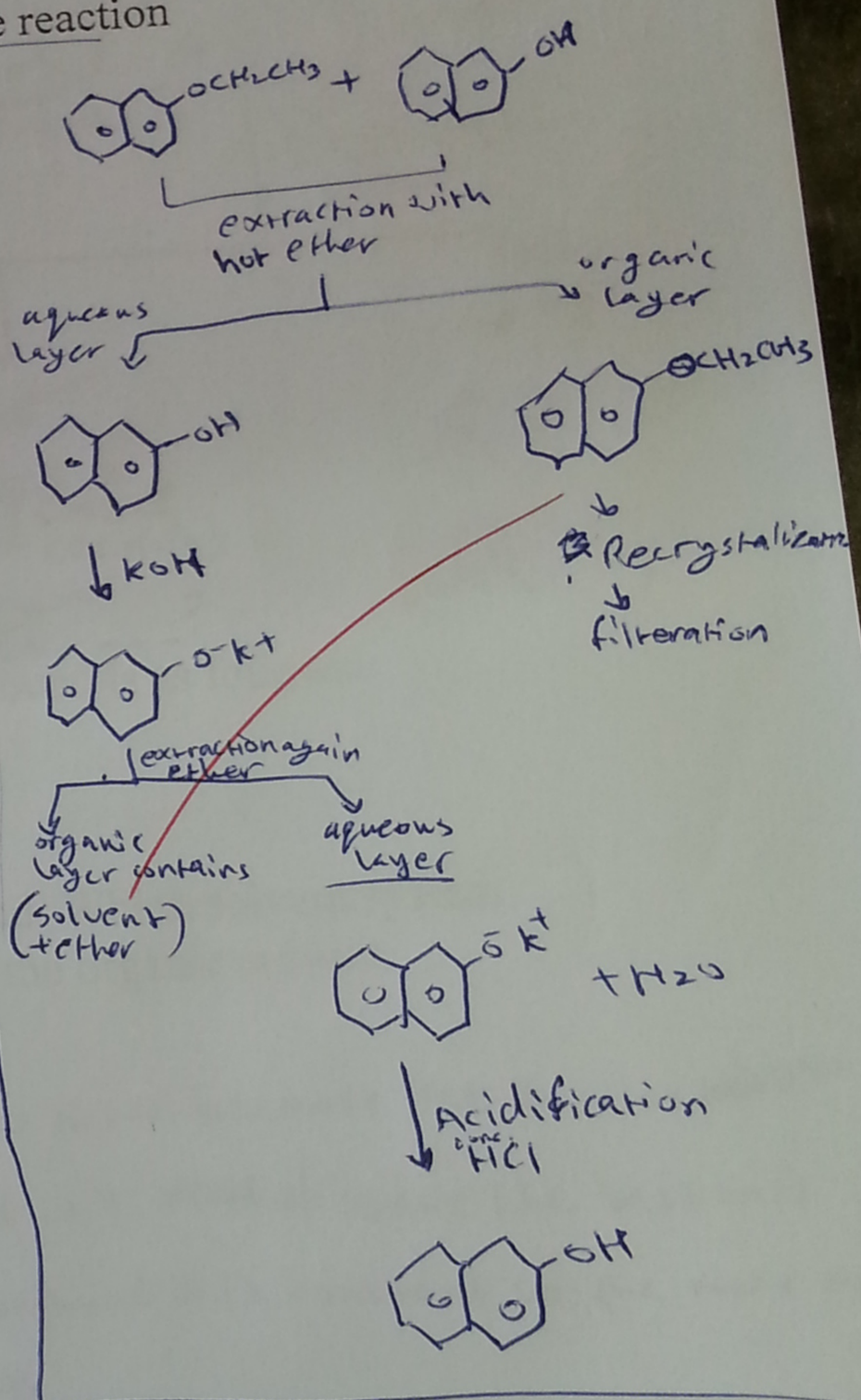

Mechanism

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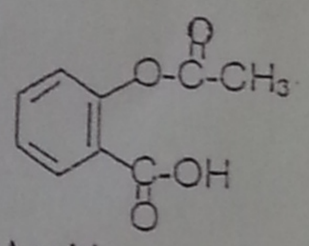
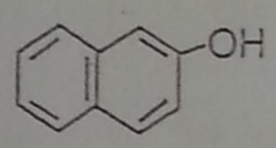
VII. (10pts) a mixture of aspirin, β-naphthol and naphthaline in ether is to be separated. Please complete the following chart

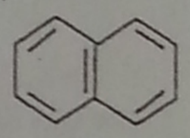
10



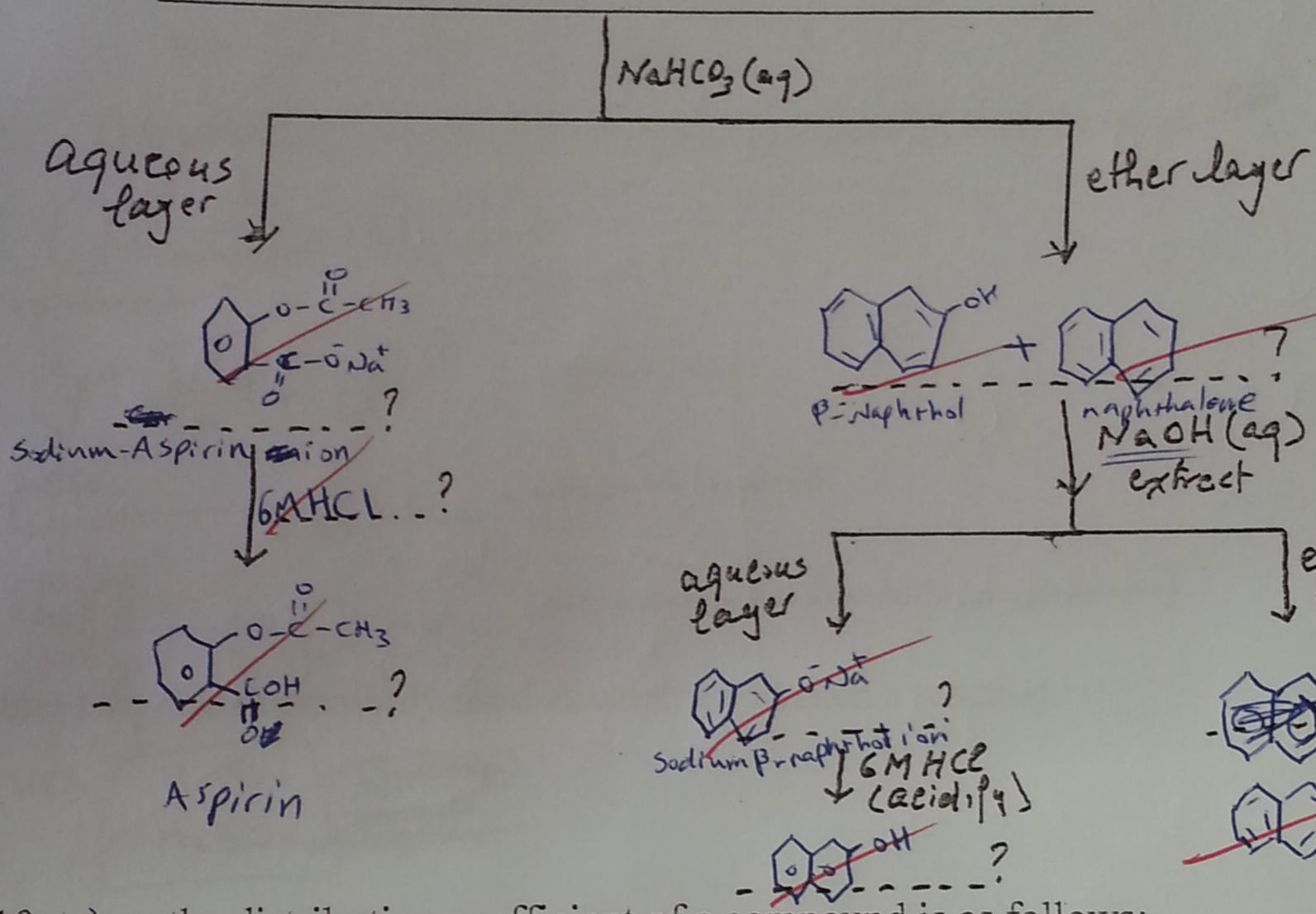
Aspirin



ß-naphthol



naphthalene



VIII. (10pts) a. the distribution coefficient of a compound is as follows:

$K_{cyclohexane}$ /water = 1.5

$K_{pentane}$ /water = 11.2

$K_{diethyl\ ether}$/water = 5.1

You have an aqueous mixture of the compound. Which solvent system would give the most efficient extraction with the organic solvent.

5

* Pentane is the best organic solvent to use because KD for it is ~~b~~ bigest on and this means that the compound will ~~dissolve~~ dissolve the best on it because the water solubility for the compound is constant so the more the organic solubility the biger the KD.

There are one reaction that combines the two reactions that can be undergone by aldehydes and ketones which are Nucleophilic addition and substitution reaction, and this one

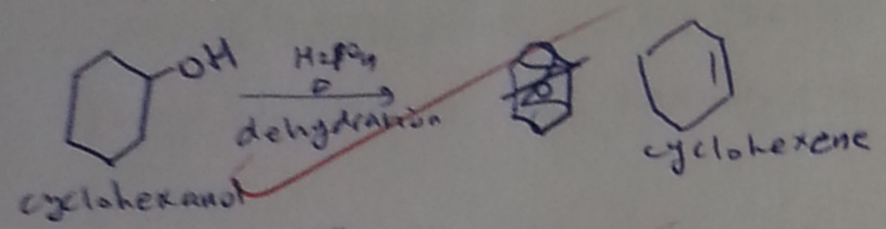
b. Butyric acid has a distribution coefficient of 6.5 in ether/water. What fraction of butyric acid will be extracted from 100 ml of water with 20 ml of ether one time. $K_D = 6.5 = \frac{\text{solubility organic}}{\text{solubility aqueous}}$

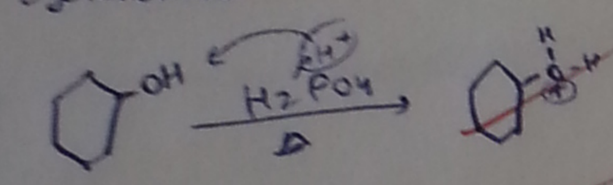
$u = \frac{20ml}{100ml} = 0.2$

$p = \frac{K_D \times u}{(K_D \times u)+1} = \frac{6.5 \times 0.2}{(6.5 \times 0.2)+1} = \frac{1.3}{2.3} = 0.565 = 56.5\%$

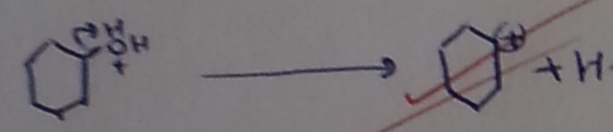
$q = 1 - 0.565 = 0.435 = 43.5\%$

IX. (15pts) write the mechanism for the dehydration of cyclohexanol.

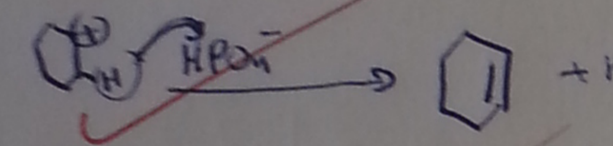


1) 

(Protonation)

2) 

(carbocation formation)

3) 

(deprotonation and formation of cyclohexene)

Name two acids commonly used as catalysts in such a reaction.

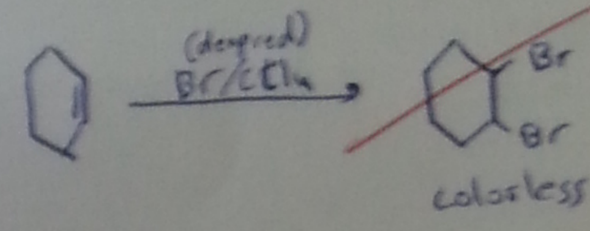
H_2PO_4
H_2SO_4

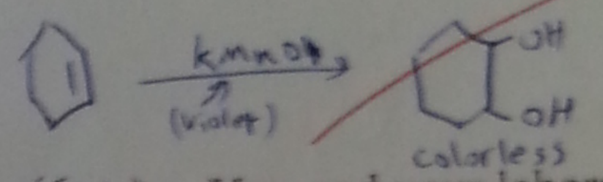
If the collected product was found turbid, how could you purify it.

- turbid means that there is water with the product, we can purify the product by removing the water by adding drying agent.

Write the reactions for two tests that would be done to indicate the product.

4

1) 

2) 

X. (6pts) a. You and your laboratory partner take melting point of the same sample. You observe a melting point of 101-107°C while your partner observes a value of 110-112°C. Give three possible reasons for getting two different readings for exactly the same sample.

2

1) may be one of was heating the sample quickly so he had a lower and wider range

2) ~~may be because one of them was slow in taking the reading so~~

3)

XI. (14pts) Assume that 15g of aniline and 15.0g of acetic anhydride were used in the preparation of acetanilide (mol. wt. aniline = 93.13), acetic anhydride = 102.1. acetanilide = 135.2)

5

1 mol aniline + 1 mol acetic anhydride → 1 mol acetanilide

What is the limiting reagent

*mol aniline = $\frac{15g}{93.13g/mol}$ = 0.16 mol

*mol acericathydride = $\frac{15}{102.1}$ = 0.147 mol → the limiting reagent

What is the theoretical yield of acetanilide

5

theoretical yield (acetanilide) = *mol limiting reagent = 0.147 mol

7

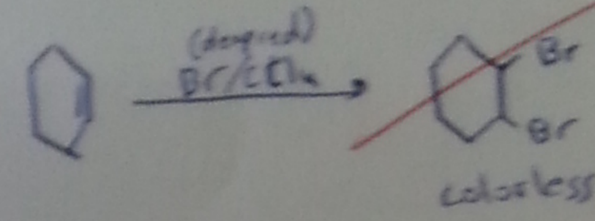
mass = MW x *mols = 0.147 x 135.2 = 19.87g of acetanilide

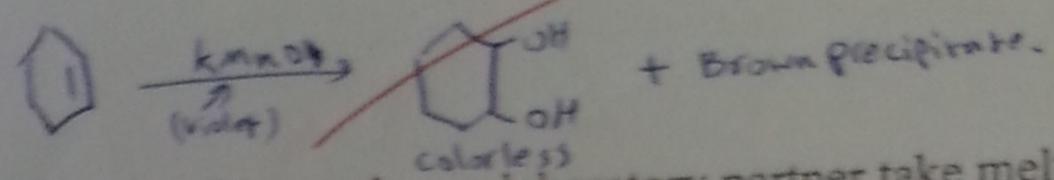
...tion and description.

There are one reaction that combines the two reactions that can be undergone by aldehydes and ketones which are Nucleophilic addition and

Write the reactions for two tests that would be done to indicate the product.

4

1) 

2) 

X. (6pts) a. You and your laboratory partner take melting point of the same sample. You observe a melting point of 101-107°C while your partner observes a value of 110-112°C. Give three possible reasons for getting two different readings for exactly the same sample.

2

1) may be one of was heating the sample quickly so he had a lower and wider range

~~2) may be because one of them was slow in taking the reading so~~

2)

3)

XI. (14pts) Assume that 15g of aniline and 15g of acetic anhydride were used in the preparation of acetanilide (mol. wt. aniline = 93.13), acetic anhydride = 102.1. acetanilide = 135.2)

5

1 mol aniline + 1 mol acetic anhydride → 1 mol acetanilide

What is the limiting reagent

*mol aniline = $\frac{15g}{93.13g/mol}$ = 0.16 mol

*mol acericathydride = $\frac{15}{102.1}$ = 0.147 mol → the limiting reagent

What is the theoretical yield of acetanilide

5

theoretical yield (acetanilide) = *mol limiting reagent = 0.147 mol

7

mass = MW x *mols = 0.147 x 135.2 = 19.87g of acetanilide

and description.

There are one reaction that combines the two reactions that can be undergone by aldehydes and ketones which are Nucleophilic addition and

What is the percentage yield if 13g of acetanilide are obtained

4. Percentage yield = $\frac{\text{experimental yield}}{\text{theoretical yield}} \times 100\%$.

$= \frac{13}{19.87} \times 100\%$.

$= 65.4\%$.

E

Object

BirZeit University
Department of Chemistry
Chemistry 221

Midterm Exam
Time: 1.5 hr

2nd sem. 2014/2015
April 16, 2015

Instructors: Simon Kuttab
Imad Qamhiyeh
Adi Qamhiyeh
Ghassan Daghra

Please Mark Your Laboratory Instructor:

- ☐ Imad Qamhiyeh
- ☐ Adi Qamhiyeh
- ☑ Ghassan Daghra

Student Name: [redacted]
Student Number: [redacted]

I 2
II 2.5
III 0
IV 6
V 6
VI 12
VII 10
VIII 10
IX 15
X 2
XI 14

79.5
+ 3
82.5

v. good

GOOD LUCK