

MELTING POINTS

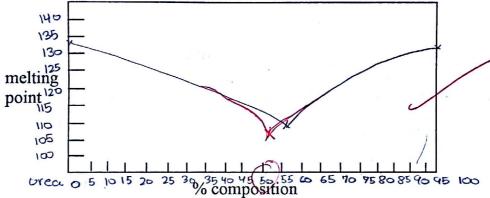
Section | 3 | لين بكر أبوزر

الاثنين (١-٥)

Determination of Melting Points.

<u> </u>	Τ,	T2.		
Compound	Start	End	m.p Range	Midpoint
Pure Compounds:			10	
1. urea	130%	1322	2	131°c
2. chnomic acid	132°c	133 ⁸	1 1 23	132.5 %
Mixtures: 50:50 1:1 20:80 80:20	98°	1228	24	110°c

Plot the midpoints of the melting points of the two pure compounds and their mixtures.



Cinnamic 100 95 90 85 80 75 70 66 60 55 50 45 40 35 30 25 20 15 10 0 Identification of an Unknown.

Unknown number: 43

Melting point of unknown: 130%

Possible Compounds:

Compound	Melting Point
1. urea	132°c
-2. cinnamic acid	133°C
3.	7

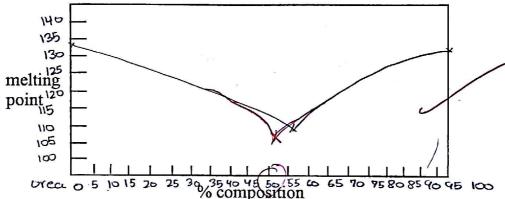


الاثنين (۱-5) لين بكر أبوز (Section) كالمناب المحتمد المحتم

Determination of Melting Points.

	٦,	T2.	re		1.
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Mixtures: 50:50 \\ 20:80	982	1225	24	110%	
80 : 20					

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Compound	Melting Point
1. urea	132°c
-2. cinnamic acid	133°C
3.	1

BOILING POINTS AND DISTILLATION

Name Leen Abuzic الين أبوزر Section

Simple Distillation of Pure Acetone.

Boiling point of pure acetone found: 50°c report

Account for any difference (if any) between the reported by

reported: 56°c

Account for any difference (if any) between the reported boiling point and the value obtained: external pressure differs from

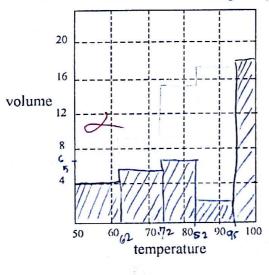
the atmosphere at sea level which is

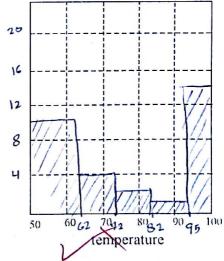
60 ml ??!

Separation of a Mixture of Acetone and Water.

Fraction	Boiling Range	Volume of Simple	of Distillate Fractional	Composition of distilla
I	50 - 62	4 mL	10 mL	acetone
II	62 - 72	5 mL	(4. m)	mixture
III	72 - 82	6 mL	2 mL	mixture
IV	82 - 95	2 mL	1 ml	mixture
V	residue	18 mL	14 ml	water

2. Plot the boiling point versus the volume of distillate for the acetone-water mixtures using simple and fractional distillation.





Which procedure was more efficient in separating the mixture into its components? Fractional distilution

QUESTIONS

- 1) A pure liquid has a constant boiling point, but a liquid with a constant boiling point is not necessarily pure. Explain.

 Because a liquid mixture of components of the same boiling point will have constant boiling point; not necessarily pure
- 2) What is the effect of each of the following on the observed bp?

 a) The thermometer is not kept moist with condensate.

 Super hearting

 b) The presence of a non-volatile impurity.
 - 13.P increases if it was soluble if not soluble b.p doesn't change
- 3) What effect does a reduction of the external pressure have on the boiling point?

 B.P. decreases
- 4) Why is it important that cooling water enters at the lower end and exits at the upper end of the condenser jacket, and not vice versa? to ensure that condensed jacket is always full of water; because if it entered from the upper end i the water is so cold the
- 5) During a distillation why should the distilling flask be filled to two thirds of its capacity only?

 To prevent bumping when heated

To allow enough space for boiling.

9.50

RECRYSTALLIZATION

Name	a de la companya de l	Section	soluble	+
			insom	

Selection of Recrystallizing Solvent

Compound	111	rater hot		ibility cohol hot	cold	ligroin hot	Suitable Solvent
salicylic acid	_	+	+	+1		-	water
anthracene	-	+,		***	.—	+	ligroin
sodium benzoate	#	+ 1	-	+		-	alcovel

60.8

Recrystallization of an Unknown

Unknown No.: 37

		P	1
Solvent	water cold hot	alcohol cold hot	
Solubility	- +	(

A

Suitable recrystallizing solvent:

ا مراح المادة فيل المادة الما

Mass of the purified unknown:

% yield: Actualat +100%

Melting point of crude unknown:

Melting point of pure unknown:

0.337

QUESTIONS

instead of chilling immediately in an ice-bath?

Because immediately cooling decrease the amount of crystal

2) Mention three properties a solvent should have to be suitable for

recrystallizing a particular organic compound?

2) Mention three properties a solvent should have to be suitable for recrystallizing a particular organic compound?

17 wastreact with the substance 12) Dissolve alorge amount of the solid to be purfified at high temperatury [3] Evaporat readily from the crystals

3) For what purpose is charcoal used in recrystallization? remove the colored impurities

4) How are insoluble impurities removed during recrystallization?

filtroadion of the host solution is necessary to remove insoluble impurities, Assuled Filter paper and a short-stem funnel allow rapid Filtrandian and avoid premature crystallization hot solution 5) Why must the flask and funnel be heated before the hot solution is

avoid premature crystallizadian inside the steam and on the filter paper and Stem of furmel

6) Why is it important to minimize evaporation during the filtration of the hot solution?

A slight excess of the solvent is usually added to compensate for any losses during not Filtration

to minimize Loss of substance through crystalization on filter paper

Scanned by CamScanner

EXTRACTION

Name	THE BULL	Section	3
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mass of beaker: 93.8119

Isolation of Caffeine from Tea Leaves

mass of beaker

×93.9314

Mass of tea leaves: 15 cx

+ carfeine

Mass of extracted caffeine: 0.12

212 × 100 = 0.8%

Percentage of caffeine in tea leaves:

Separation of a Two-Component Mixture

Mass of recovered benzoic acid:

Mass of recovered p-dichlorobenzene:

QUESTIONS

- 1) Why should the stopper be removed from the separatory funnel before liquid can be withdrawn through the stopcock? In order for the pressure in the separatory Funnel to be the same everywhere.
- 2) What are the properties of a suitable solvent for the extraction of an organic solute from an aqueous solution?

9- immiscible with H20

B- 1000 b.p

non-Flammable

15- readily dissolves your substance more than in

3) What is the role of sodium carbonate in the extraction of caffeine layer. from tea leaves? It is used to remove acidic

tannins by converting them to water-

soluble salts

4) What effect does partial miscibility of the two solvents have on the efficiency of the extraction?

That will reduce the efficiency of the extraction because some of the desired solvent will be dissolved in the other solvent and as a result the amount of the desired solvent will be less

5) The distribution coefficient, K_D (C_{ether}/C_{water}), for an organic substance X at room temperature is 10. What relative volumes of ether to water should be used for the extraction of 90 percent of X from a water solution in a single extraction?

$$K_D = 10 = \frac{C \text{ ether}}{C \text{ water}}$$

$$10 = \frac{0.9 \text{ From } 90.}{V.\text{ org}}$$

$$\frac{0.1}{V.\text{ H}_20} \approx \frac{10}{100}$$

$$\frac{V \text{ org}}{V \text{ H}_20} = 0.9$$

STEAM DISTILIATION

1,0

(10
Section

Steam Distillation of Bromobenzene.

Fraction Number	Boiling Range	Volume of Water	Volume of Bromobenzene
1	93	5.4	4.2
2	43	5.9	4.9
3	93	5.5	4.5

Isolation of Essential Oils.

Name of spice used:

Mass of ground spice: 10 %

Mass of essential oil: 0⋅32 4

Percentage of essential oil in the spice: 3.2 ×

QUESTIONS

1) Discuss the results of part 1, concerning the boiling point, and composition of the distillate. Compare these results with those obtained from the water-acetone mixture in the distillation experiment (p. 30).

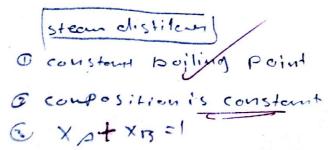
Simple distilation)

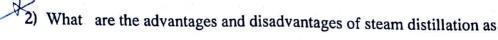
() water of acetone have boiling point close to other

(i) composition different

(i) Variable

(ii) XA = XB = 1





d's adventeuge;

Osteam-volatile

Thert toward steam and steable under the conditions

Immiscible with water

Relvantages) = pwification of high-boiling compounds 3) Suggest another possible method that might be used to obtain distillation

essential oils from the spices.

- Solid - liquid extraction . vacoum distillation

4) At 95.5 °C, the vapor pressure of water is 641 mm, and that of bromobenzene is 119 mm. Calculate the molar ratio and the weight ratio of bromobenzene to water when a mixture of the two is distilled at 760 mm. Compare the answers with your experimental results. (density of bromobenzene = 1.5 g/mL).

$$\frac{1.62}{1.62}$$
 *100% = $\frac{1.621}{100}$

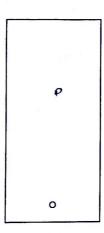
CHROMATOGRAPHY

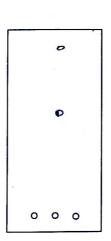
		_
Name	Section	

TLC Examination of Isomeric Nitroanilines.









ortho-isomer

para-isomer

mixture

$$R_f = \frac{y \cdot 6}{5 \cdot y} = 0.95 R_f = \frac{3.1}{5 \cdot y} = 0.57 R_f = \frac{y \cdot 6}{5 \cdot y}$$
 (orthorized) (para

Analysis of Analgesic Drugs.

Name of the analgesic drug:

The components of the analgesic drug are:

- 1)
- 2)
- 3)

Paper Chromatographic Analysis of a Dye.

 R_f -value for the yellow dye: $\frac{1.2}{2} = 0.6$

 R_f -value for the blue dye: $\frac{1.6}{2} = 0.8$

QUESTIONS

- 1) How will the following affect the TLC separation?
 - a) too much sample applied.

and over laping of class spot of (RF)
will not clear prot accurate

B) Stationary phase is too active.

RE will decrease because the molucul of the component with be bounded to the adsorbant and will move ashort distance

Forgetting to remove the plate when the solvent has reached the top of the plate.

the slower moving spots will cotch up with the Faster moving spots at the top of the Plate of we can't find (RF)

d) Having too much solvent in the developing chamber so that its surface extends above the origin.

and show the differ between the spot

e) Polarity of the solvent being too high.

RFP - poore seperation.

The mix will be Eluided to Fact

2) Which dye is more soluble in *n*-propyl alcohol, the blue or the yellow? How can you tell?

Blue it travelled more than yellow

3) Which compound is more strongly adsorbed on silica, ortho- or para-nitroaniline? Correlate the R_f values with the structures.

Para - nitroduitine RF-parais less than RE-ortho

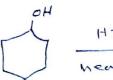
DEHYDRATION OF ALCOHOLS

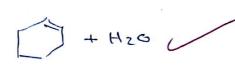
Section Name

Excelle

I (or II). Preparation of Alkenes

Write a balanced equation for the preparation of the alkene.





Alcohe	ol	All	kene	
molecular weight	100	molecular weight	82	
grams used	9.6 a	moles expected	0:1	
moles used	0.1	grams expected (theoretical yield)	8.2	
		grams obtained (actual yield)	1.6	
percentage	yield = $\frac{\text{actuall}}{\text{theoretic}}$	yield × 100	1.6 × 100%.	Ē

III. Tests for Unsaturation

a) Bromine test: (equations and observations)

-C=C-+13r2 (colorless)

b) Baeyer test: (equations and observations)

Alkene:

c-c- + >KMNUM + 4H20 - net react
purple

QUESTIONS:

1) Dehydration reactions are acid-catalyzed. What is the function of the acid in such reactions? alcohole having B-Hydrogen can be converted in to alkenar by dehydration (elmination of amoluces of water convert of-band leaving group to other young group.

2) Could hydrochloric acid be an acceptable substitute for the acid used in this experiment? Explain. No —

instead of to alkenes

3) Why was it necessary to wash the crude alkene with a solution of aqueous base? to remove trucks of acid in crude alkene to secuent the ingelration of Alkenec back to alchele



beaker + alkyl = 34, 457 g

NUCLEOPHILIC SUBSTITUTION

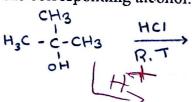


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I (or II). Preparation of Alkyl Halide

Write an overall equation for the preparation of the alkyl halide from

the corresponding alcohol.



CH3 - C - CH3

Theoretical yield of alkyl halide: moles of alkyl halide

moles of molar mass
alkyl halide of alkyl halide
0.045 * 92.5 = 6.93459

Actual yield: 2.38 9

Percentage yield: 2.389 x 100% = 34.34%

III. Relative Reactivities of Alkyl Halides

Alkyl Halide	Reaction Time	
1-chlorobutane	AgNO3 Na	il
2-chlorobutane	intermediate ppt	
2-chloro-2-methylpropane	Fast ppt	
allyl chloride (x)		
chlorobenzene. (no rxn	

Order of reactivity towards AgNO3: Ly NaI

2.chloro -2-methyl propone λ 2-chloro butane λ 1-chlorobutane λ 3° λ 2° λ 1°

Order of reactivity towards NaI:

1-chlorobutane 2 2-chlorobutane 22-chloro-2-methyl propune

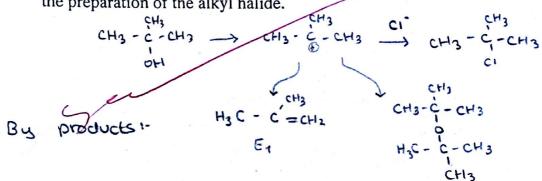
10 2 20 230

QUESTIONS

3-decant Into a beaker and weigh the beaker with contents 1) Write all the steps in the purification of the alkyl halide mentioning the purpose of each.

1- After seperating the aqueous layer from the organic layer => wash with cold water to get rid of excess unreacted alcohol and to avoid evaporation

- 2- Seperate once again and pour the organic layer Pn a Florice Containing anhydrous calcium chlorice
- 2) Write the structure of all possible by-products that may form during the preparation of the alkyl halide.



3) Account for the low reactivity of chlorobenzene towards silver Su₁ Su₂ nitrate and sodium iodide.

the carbocation it doesn't undergo

ELECTROPHILIC AROMATIC SUBSTITUTION

	·	
Name	Section	0.
	Section	5

I. Bromination of Acetanilide

Write an equation for the preparation of p-bromoacetanilide

Theoretical yield of p-bromoacetanilide:

Actual yield:

Percentage yield:

QUESTIONS

- 1) Write an equation for the formation of the bromonium ion in this experiment
- 2) How can you remove excess bromine?

II. Nitration of Phenol

Write an equation for the preparation of o- and p-nitrophenol.

	o-Nitrophenol	p-Nitrophenol
Theoretical yield		
Actual yield	r a war ng	
Percentage yield		

QUESTIONS

1) Write equations that show the mechanism for the nitration of phenol.

- 2) Why should the temperature be kept between 45-50 during the experiment?
- 3) What makes it possible to separate *o*-nitrophenol from the *p*-isomer by steam distillation?

III. Nitration of Bromobenzene

Write an equation for the preparation of o- and p-nitrobromobenzene.

H250 H Ortha

Theoretical yield of p-nitrobromobenzene:

10.02 mol bromo x 1 mol P x 202

Actual yield: 4.016 of Percentage yield: 4.016 × 100%= 99.4 %

= 4.049

QUESTIONS

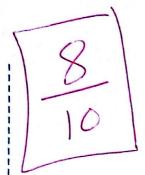
- 1) What has happened to the o-nitrobromobenzene that was formed in this experiment? How can it be recovered? It is dissolved in the ethanol solvent and is recovered through evaporation of ethanol
- 2) Why should the temperature be kept between 45-50 during the experiment? Because if it exceeds that temperature range 94 will lead to dinitration of bromobenzene

IV. Relative Bromination Rates.

<u>্</u>	no reaction
~ 0	110
NHC-CH3	medium
JOH	fastest

From your results, arrange the groups: NH-CO-CH₃, H and OH in decreasing order of ring activation towards bromination.

OH > NH-C-CH3 > H



ALCOHOLS AND PHENOLS

Name	Section	3
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I. ALCOHOLS

1. Solubility of Alcohols in Water

Alcohol	Structure	Solubility
1-butanol	CH3 CH2CH2 CH2OH	7
2-methyl-2-propanol	HSC -C-CH3	+
cyclohexanol	Con Chi	_
ethylene glycol	CH 2- CH 2	+

What general conclusions can you draw concerning the solubility of alcohols in water? Alcohols of low molecular weight are water soluble due to their obility to form hydrogen bonds with 420, solubility in water excrease with increasing (Mw) but increase with branching & with the number of (014)

2. Acid Properties of Alcohols

	Alcohol	Reaction Equation	Observations
0	l-butanol	CH3CH2CH2OH +2N9-9	y-Past.
@	2-butanol	CH 3 CH2 CH2 CH3 + 2Na - 2 CH3 CH3 CH3 CH3 CH3	Past
3	2-methyl-2-propanol	CH3 C-C13 +2Na- C143-C-CH	2 NO L+N

Arrange the three alcohols according to their rates of reaction with 10 > 20 > 30 sodium:

1-butanol > 2-butanol > 2-methyl-2-propoun

? Account for the color change observed upon addition of phenolphthalein to the solution of 1-butanol:

The color becomes pint due to the basic medium result From the formation of the sodium soil of alcohols

3. Oxidation of Alcohols with Chromic Acid

Alcohol	Reaction Equation	Observations	
1-butanol	CH3CH2 CH2CH2OH + K)CT207 -	cysean L	7
2-butanol	CHICHICHICHI + KICTZOT	slowGr	een ol
2-methyl-2-propanol	CH3CH2CHCH3 + Cr+3	Fort	

4: Lucas Test

	Alcohol	Structure of Product	Observations	>
0	1-butanol	CH3CH2CH2 C142017	No ryn	()
2	2-butanol	CH3CHCH2CH3	Slow	100
3	2-methyl-2-propanol	CH3 + 2 CM3	Fast (turbiolit	

Arrange the three alcohols according to their rates of reaction with the

Lucas reagent:

5. Iodoform Test

Alcohol	Structure of Product	Observations
1-butanol	. The state of the	
2-butanol		
2-methyl-2-propanol		

II. PHENOLS

1. Acidity of Phenols

Compound	Structure	Solubility		
		Water	NaOH	
cyclohexanol	C→ OH	×	×	
phenol	(0) OH	X	1	
p-cresol	H3C (0)-OH	×	1	

seen

Write your conclusion about the solubility of alcohols and phenols in water and NaOH solutions:

acid,

2. Reaction of Phenols with Bromine Water

Write an equation for the reaction of phenol with bromine water giving your observations:

3. Ferric Chloride Test

cyclohexanol OH no reaction phenol OOH reacts -> violet	Alcohol	Structure	Observations	
phonol	cyclohexanol			
	phenol	(0)-OH		100

Write an equation for the reaction of phenol with FeCl3:

ALDEHYDES AND KETONES

as
01.0

Name	and the Property of	Section	
		~ CULLOII	

1. 2,4-Dinitrophenylhydrazine Test

Compound	Structure	Observations and Results
acetone	CH3CCH3	+ve wange Espit
benzaldehyde	() = H	the ?

2. Tollens' Test

Compound	g G :	
Compound	Structure Observations and Results	
formaldehyde	H-C-H Lorst to form mirror	سان
benzaldeliyde	Tol-c-H whiver form after heat	ing
acetone	-H3CCH3 Le NOTXI	7

-0.5

3. Fehling's or Benedict's Tests

Compound	Structure	Observations and	Results
formaldehyde	14 / C - H	red	4ve
benzaldehyde	<->-3-14	red	xue
acetone	CH3-C"-CH3	no rxn	Ne

4. Iodoform Test

Compound	Structure	Observations and R	esults
acetone	CH3 6 CH3	Yellow	PPF
2-propanol	c-c-c	yellow	bbt
2-pentanone	c-c-c-c	Mellon	PP
3-pentanone	c-c-c-c	/ he rxu	

Chemical equation:

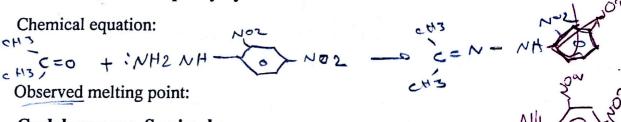
× 6. Cyclohexanone Phenylhydrazone

Chemical equation

Observed melting point:

7. Acetone 2,4-Dinitrophenylhydrazone

Chemical equation: Observed melting point:



✓ 8. Cyclohexanone Semicarbazone

Chemical equation Noz LO +NATURAL NOS NHSNHSNHS-

Observed melting point:

× 9. Identification of Unknown

Unknown No.:

Tollen's Test:

Iodoform Test:

Derivatives Prepared:	1)	mp:
	2)	. mp:
	2)	

Unknown is:

PREPARATION OF CARBOXYLIC ACIDS

Name Leen Abuzir Section 3

I. Benzoic Acid by Hydrolysis of Benzonitrile

Give the equation for the preparation of benzoic acid from benzonitrile.

Theoretical yield of benzoic acid:

Actual yield:

Percentage yield:

Melting point of benzoic acid:

QUESTIONS

1) Explain with the help of equations the function of the base in the hydrolysis of benzonitrile.

- 2) What impurity might be found in the benzoic acid prepared from benzonitrile? How does this impurity arise?
- 3) Would it be possible to use the nitrile method to achieve the following conversions? Illustrate with equations.

- a) Ph-Br ----- Ph-COOH
- b) Ph-CH₂Br → Ph-CH₂COOH
- c) t-Bu-Br ----- t-Bu-COOH

II. Benzoic Acid by the Haloform Reaction

Write an equation for the preparation of benzoic acid from acetophenone:

Theoretical yield of benzoic acid:

Actual yield:

Percentage yield:

Melting point of benzoic acid:

QUESTIONS

- 1) Which of the following compounds will give the haloform reaction?
 - a) CH₃COOH
 - b) C₆H₅CH₂-CO-CH₃
 - c) C₆H₅CH(OH) CH₃
 - d) C₆H₅CH₂-CO-OCH₃
 - e) C₆H₅-CO-CBr₃
 - f) C₆H₅CH(OH)CH₂CH₃

ESTERIFICATION OF ALCOHOLS AND PHENOLS

Name Leen Flouzer Section 3

mass 1 = 5.531 9

Tilter = 3.10 9

Write an equation for the preparation of aspirin:

Theoretical yield of aspirin: 2.56 q

Actual yield: 2.431 9

Percentage yield: $\frac{2.431}{2.56} \times 100 = 94.96\%$

Ferric Chloride Test

Color with Salicylic acid: Violet

Color with Aspirin: Volet

II. Preparation of Methyl Benzoate

Write the overall equation for the preparation of methyl benzoate.

Theoretical yield of methyl benzoate:

Actual yield:

Percentage yield:

QUESTIONS

1)	What is the	he function	of the	acid	in	the	preparation	of methyl
	benzoate?	(give equat	ions).					

2) What experimental means may be used to drive an esterification towards completion?

3) Tell how a mixture of benzoic acid and methyl benzoate may be separated?

4) What impurities are most likely to be present in the aspirin you have prepared? Show (by equations) how they are formed.