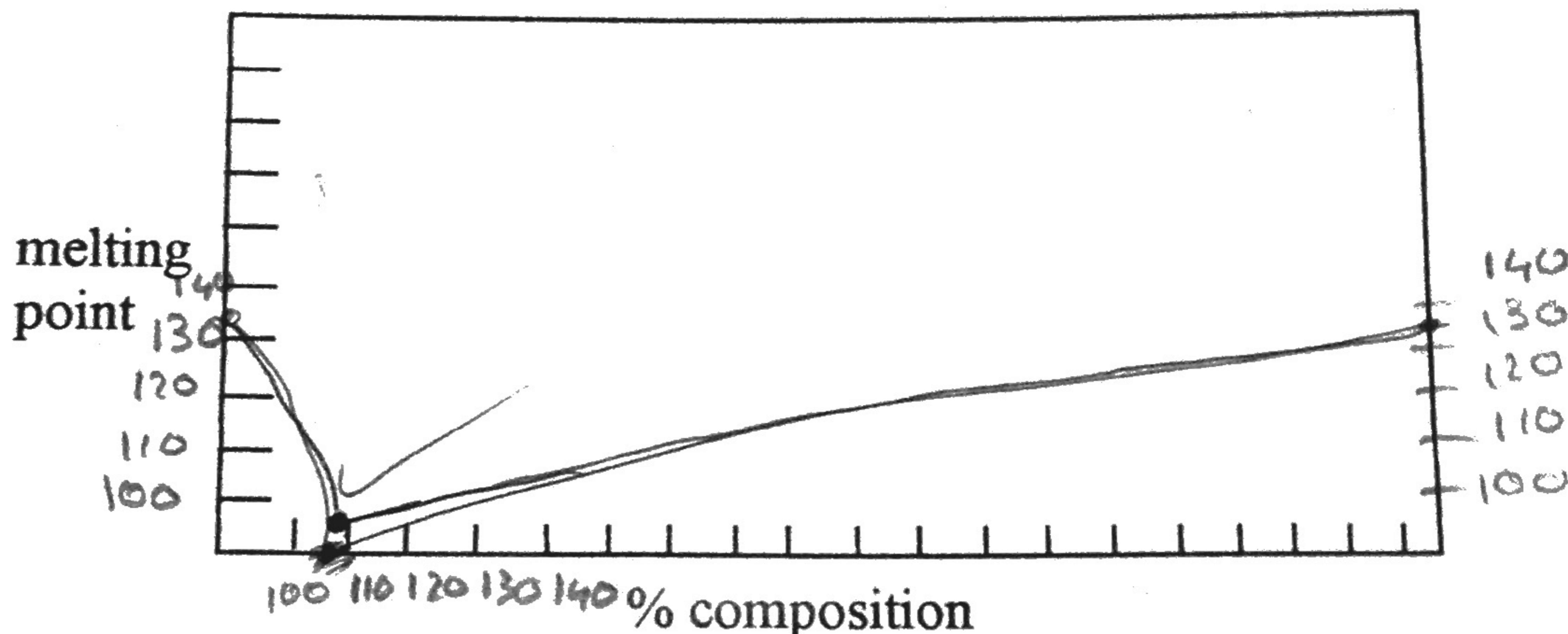


# Determination of Melting Points.

Compound	Start	End	m.p Range	Midpoint
Pure Compounds:				
1. Urea	130	132	✓ 2	131 ✓
2. Cinnamic acid	135	138	✓ 3	136 ✓
Mixtures:				
50 : 50 ✓	104	106	2 ↑	105
20 : 80				
80 : 20				

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



## Identification of an Unknown.

Unknown number: 71

Melting point of unknown:  $114 - 118^{\circ}\text{C}$

Possible Compounds:

Compound	Melting Point
1. Acetanilide	114
2. Mandelic acid	117
3. Benzoic acid	122

Record the melting point ranges for each of the following mixtures:

0.5 Melting point range for unknown + (1): 118 - 120 ✓

Melting point range for unknown + (2): 78 - 80 ✓

Melting point range for unknown + (3): 86 - 90 ✓

Unknown is: Acetanilide ✓

## QUESTIONS

1) What two effects do impurities have on the melting point of an organic compound?

- 1) Lower the melting point of the substance.
- 2) Broaden the melting point range.

2) For what two purposes are melting points routinely used?

- 1) identify of solid
- 2) check purity of organic compounds

3) What effects on the measured melting point would you expect in each of the following cases:

a) Presence of pieces of glass in the sample.

It will not be affected. ✓

b) Presence of solvent within the crystals.

The melting point will be lower. ✓

c) Rapid heating during melting point determinations.

~~The melt~~ It will be lower. higher ✓

d) Using too large a sample when determining the melting point?

It will be higher than the actual one. ✓

### Simple Distillation of Pure Acetone.

Boiling point of pure acetone found: 54

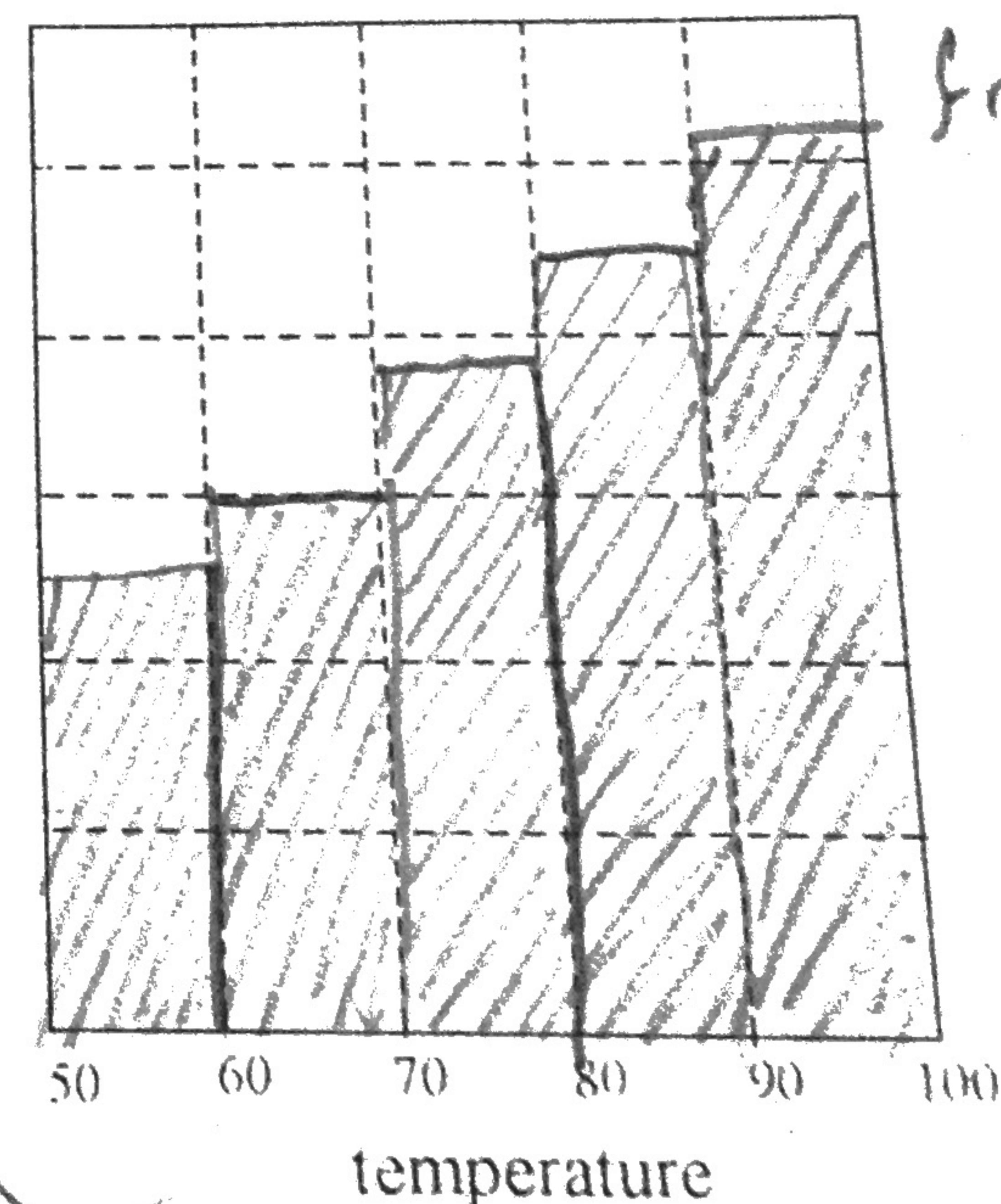
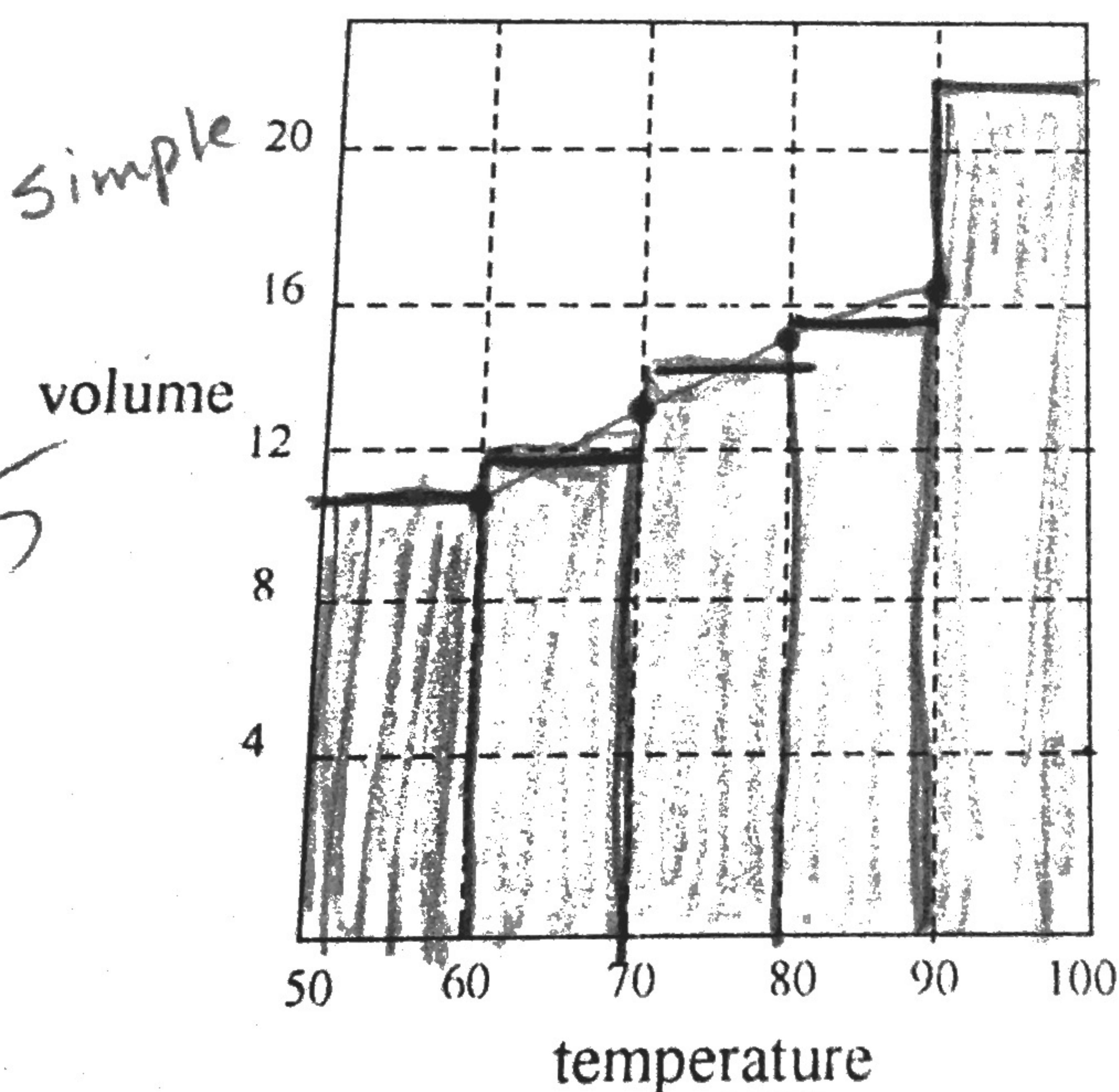
reported: 58

Account for any difference (if any) between the reported boiling point and the value obtained: 4

### Separation of a Mixture of Acetone and Water.

1. Fraction	Boiling Range	Volume of Distillate		Composition
		Simple	Fractional	
I	50 - 62	11 mL	17 mL	acetone + water
II	62 - 72	2 mL	1 mL	acetone + water
III	72 - 82	2 mL	3 mL	acetone + water
IV	82 - 95	1 mL	3 mL	acetone + water
V	residue	24 mL	22 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 distillation

### QUESTIONS

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

Because the liquid may have two substances with the same boiling point.

- 2) What is the effect of each of the following on the observed bp?

- a) The thermometer is not kept moist with condensate.

The boiling point will be higher.

- b) The presence of a non-volatile impurity.

The boiling point will be higher

- 3) What effect does a reduction of the external pressure have on the boiling point?

The boiling point will be lower

- 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 keep filled with water during experiment.

- 5) During a distillation why should the distilling flask be filled to two thirds of its capacity only?

prevent bumping.

## Selection of Recrystallizing Solvent

(a.5) ✓

Compound	water		Solubility		ligroin		Suitable Solvent
	cold	hot	cold	hot	cold	hot	
salicylic acid	-	+	+	+	-	-	water
anthracene	-	-	-	-	-	+	ligroin
sodium benzoate	+	+	-	+	-	-	alcohol

(6)

### Recrystallization of an Unknown

Unknown No.: A

Solvent	water		alcohol		ligroin	
	cold	hot	cold	hot	cold	hot
Solubility	+	+	+	+	-	-

Suitable recrystallizing solvent:

Mass of crude unknown : 1.016

(3)

Mass of the purified unknown : 0.313

% yield : 30.99 %.

Melting point of crude unknown :

Melting point of pure unknown :

### QUESTIONS

- 1) Why is it preferable to allow the hot solution to cool gradually instead of chilling immediately in an ice-bath?

To have large amounts of crystals with suitable size.

- 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?

- 1) Dissolve large amount of solid in high temp.
- 2) Dissolve readily impurities at low temp or not at all even at the b.p.
- 3) Not react with substance to be purified.

3) For what purpose is charcoal used in recrystallization?

to remove colored impurities.

4) How are insoluble impurities removed during recrystallization?

By hot gravity filtration.

5) Why must the flask and funnel be heated before the hot solution is filtered?

to prevent precipitation of the substance to be purified on filter paper

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

Because there will be super saturation and there will be premature crystals.

## Isolation of Caffeine from Tea Leaves

Mass of tea leaves: 10 g

(X) (1)

Mass of extracted caffeine: 0.03

Percentage of caffeine in tea leaves: 0.06 %.

(1)

## 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?

to release the excess amount of pressure which builds up. To allow air to displace liquid

- 2) What are the properties of a suitable solvent for the extraction of an organic solute from an aqueous solution?

- 1) Immiscible with the liquid in which the solute is present.
- 2) Readily dissolve the solute to be extracted.
- 3) Extract little or none of the impurities and other compounds.

(2)

- 3) What is the role of sodium carbonate in the extraction of caffeine from tea leaves?

Salting out effect

(react with tannic acid),  
and convert to  
water soluble

(1)

(2)

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

It will make the  $K_D$  low and the extraction will not effective.

(1)

- 5) The distribution coefficient,  $K_D$  ( $C_{\text{ether}}/C_{\text{water}}$ ), 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?

$$K_D = \frac{C_{\text{ether}}}{C_{\text{water}}} = \frac{\frac{m_1}{V_1}}{\frac{m_2}{V_2}} = \frac{90 V_1}{10 V_2} = 10$$

(1)

$$\frac{V_2}{V_1} = \frac{90}{10} = 10$$

$$\frac{V_1}{V_2} \times \frac{9}{10}$$

$$10 \text{ v ether} = 9 \text{ v water}$$

$$\frac{V_e}{V_w} = \underline{\underline{0.9 : 1}}$$

(1)

## Steam Distillation of Bromobenzene.

Fraction Number	Boiling Range	Volume of Water	Volume of Bromobenzene
1	94	5.2	4.8
2	94	5.2	4.8
3	94	5.3	4.7

### Isolation of Essential Oils.

Name of spice used: anise

(A)

Mass of ground spice: 10 g

(3)

Mass of essential oil: 0.02

Percentage of essential oil in the spice: 0.2%

### 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).

① In simple fractional distillation the b.p. of the mixture is between the b.p. of water and acetone but in steam distillation the b.p. is lower.

② The composition of the two liquids in simple fractional distillation is different but in steam distillation it is constant

③ In simple fractional distillation  $X_{\text{acetone}} + X_{\text{water}} = 1$

In Steam distillation  $X_{\text{water}} = X_{\text{bromobenzene}} \approx 1$

2) What are the advantages and disadvantages of steam distillation as a method of purification?

~~Advantages: Steam distillation is convenient for the purification of high b.p. compounds by low-temperature distillation and so replaces the vacuum distillation.~~

~~Disadvantages:~~

- 1) Steam volatile those that have appreciable vapour pressure at the temperatures of steam distillation.
- 2) Immiscible with water.
- 3) Inert toward steam and stable under the conditions of steam distillation.

3) Suggest another possible method that might be used to obtain essential oils from the spices.

vacuum distillation

solid-liquid extraction



B  
4) At  $95.5^{\circ}\text{C}$ , the vapor pressure of water is 641 mm, and that of A 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).

$$\frac{\text{moles of A}}{\text{moles of B}} = \frac{\text{mass of A}/\text{mwt A}}{\text{mass of B}/\text{mwt B}} = \frac{P_A^{\circ}}{P_B^{\circ}}$$

$$= \frac{641}{119} = 5.38$$

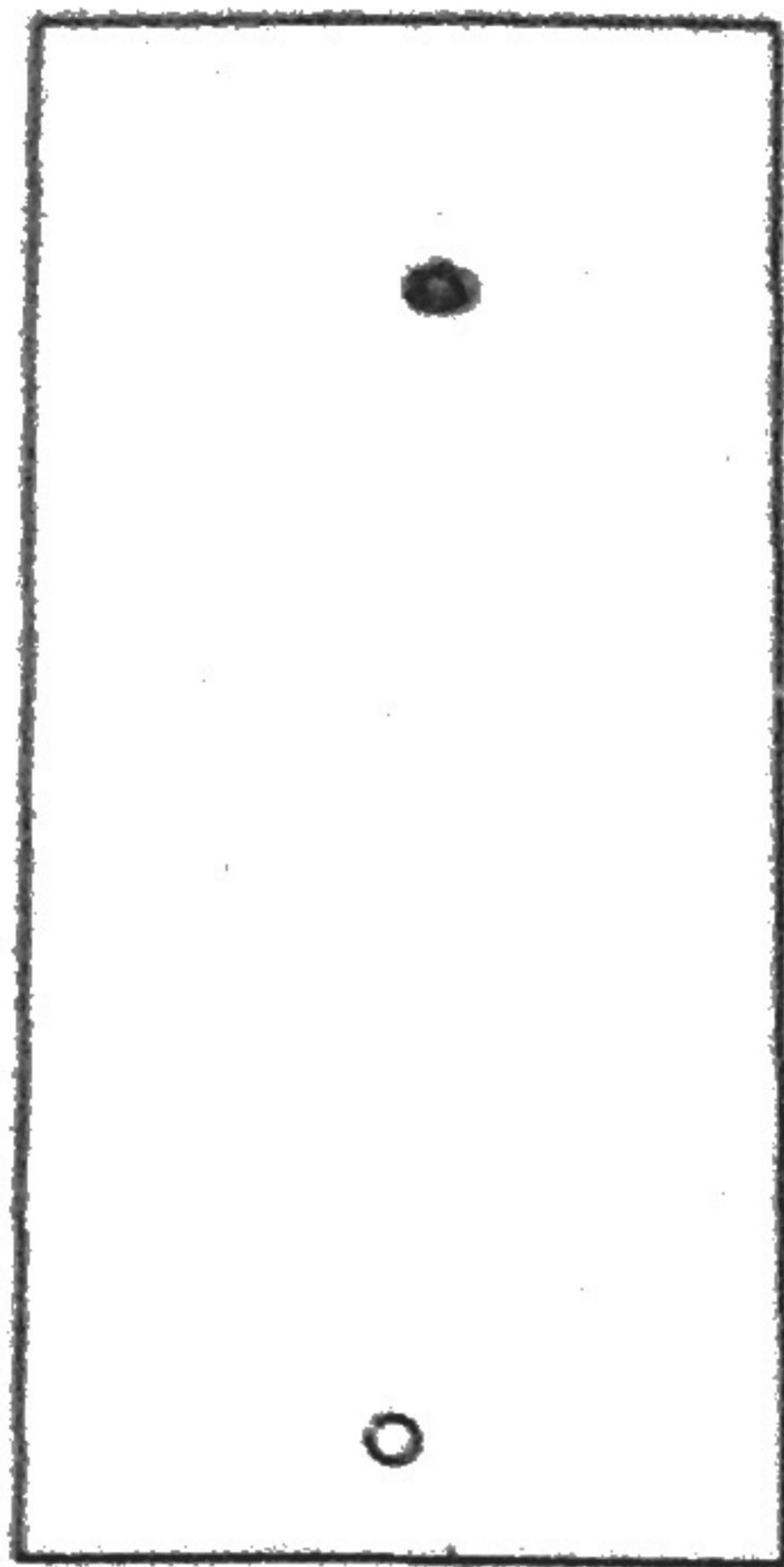
$$\frac{n_{\text{Bromo}}}{n_{\text{water}}} = \frac{119}{641} = 0.186$$

$$n = \frac{m}{\text{mwt}} \quad \frac{m_{\text{Bromo}}}{m_{\text{water}}} = \frac{119 \times 157}{641 \times 18} = 1.62$$

# TLC Examination of Isomeric Nitroanilines.

5.5

4.6

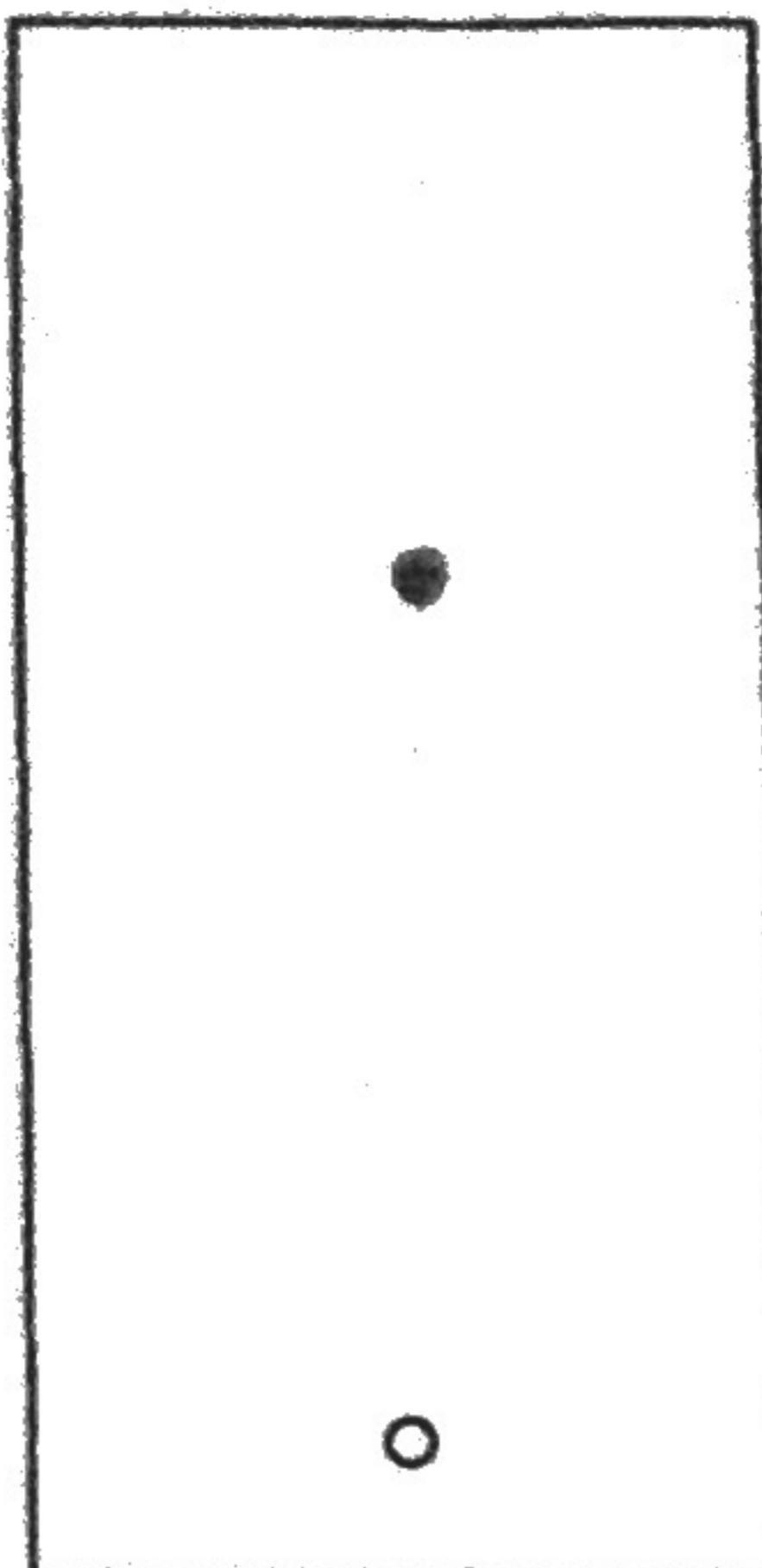


*ortho*-isomer

$$R_f = \frac{4.6}{5.5} = 0.84$$

1.5

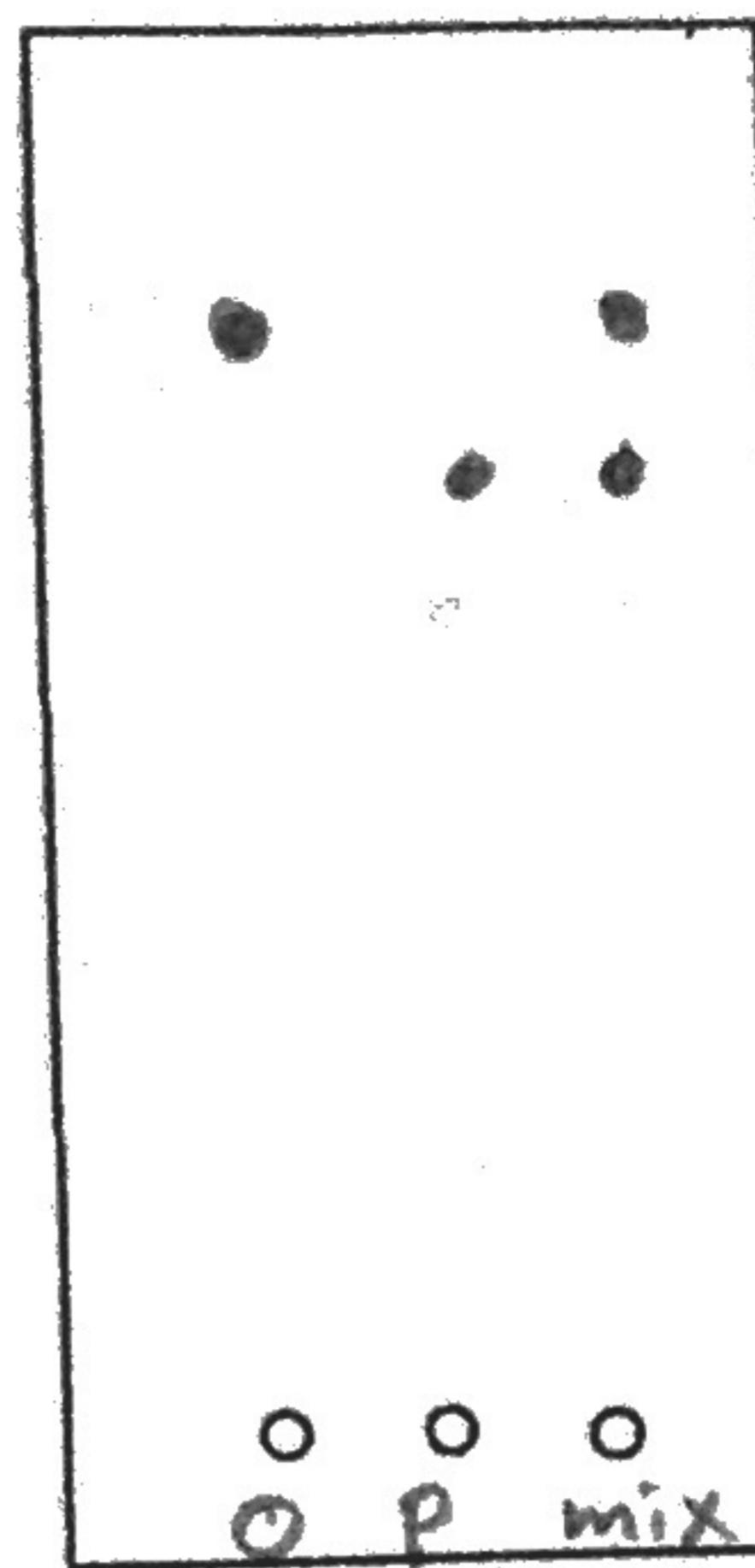
4.1



*para*-isomer

$$R_f = \frac{4.1}{5.5} = 0.75$$

✓



mixture

$$R_f = \frac{4.6}{5.5} = 0.84$$

$$R_f = \frac{4.1}{5.5} = 0.75$$

Too  
Large

## 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:  $\frac{1.6}{3.5} = 0.45$

$R_f$ -value for the blue dye:  $\frac{3.1}{3.5} = 0.85$

✓

(2)

## QUESTIONS

1) How will the following affect the TLC separation?

a) too much sample applied.

Too much sample makes too large spots and overlapping will occur between spots. 0.5

b) Stationary phase is too active.

$R_f$  will be lower so we can't observe the separation. 0.5

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

The spots will become outer of the slide so we can't determine  $R_f$  and we can't observe the separation. 0.5

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

We will not be able to observe separation spots because the ~~dye~~<sup>spots</sup> will be dissolved in the solvent. 0.5

e) Polarity of the solvent being too high.

The distance will be higher so the  $R_f$  will be higher. 0.5

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

The blue dye is more soluble as its distance is larger so the  $R_f$  is higher. 1.5

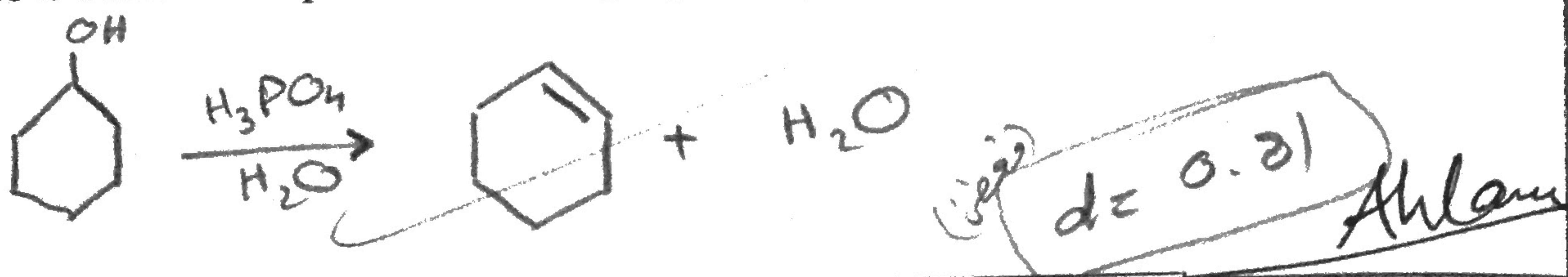
3) Which compound is more strongly adsorbed on silica, ortho- or para-nitroaniline? Correlate the  $R_f$  values with the structures. ?

Para-nitroaniline is more strongly adsorbed because of its more intermolecular forces.

in O-nitroaniline it has intramolecular forces (H-bonding) so  $R_f$  will be higher. 1.5

## I (or II). Preparation of Alkenes

Write a balanced equation for the preparation of the alkene.

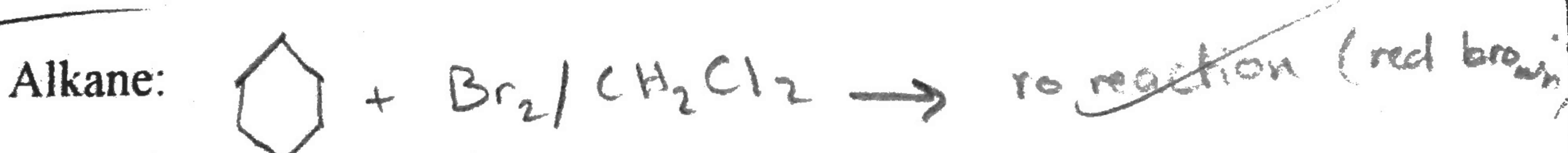
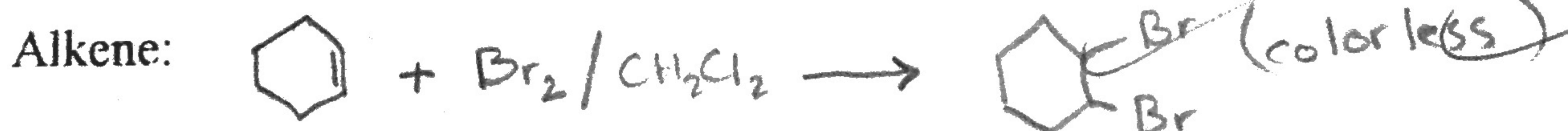


2

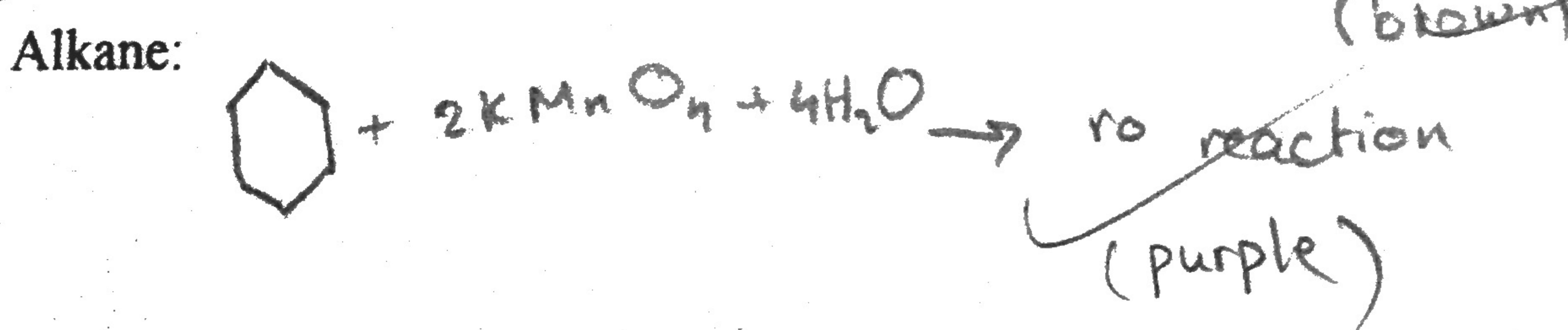
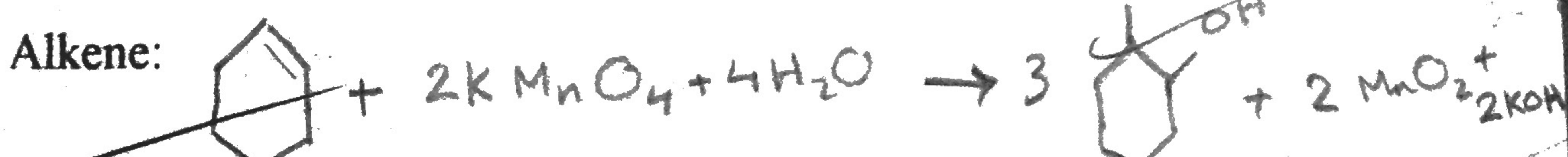
Alcohol	Alkene	$V = 6.4 \text{ ml}$
molecular weight 100	molecular weight 82	
grains used 9.6	moles expected 0.096	
moles used $n = \frac{m}{M_{\text{WT}}}$ 0.096	grams expected $n \times M_{\text{WT}}$ (theoretical yield) 7.872	
	grams obtained (actual yield) 5.184	$d = \frac{m}{V}$
percentage yield = $\frac{\text{actual yield}}{\text{theoretical yield}} \times 100$	65.85 %	

## III. Tests for Unsaturation

a) Bromine test: (equations and observations)



b) Baeyer test: (equations and observations)



## QUESTIONS:

- 1) Dehydration reactions are acid-catalyzed. What is the function of the acid in such reactions?

Acid catalyst like "H<sub>2</sub>SO<sub>4</sub> and H<sub>3</sub>PO<sub>4</sub>" give anions (HSO<sub>4</sub><sup>-</sup> and H<sub>2</sub>PO<sub>4</sub><sup>-</sup>) which are weak nucleophiles and therefore minimize substitution products.

- 2) Could hydrochloric acid be an acceptable substitute for the acid used in this experiment? Explain.

~~No. Because the nucleophilic Cl<sup>-</sup> will convert alcohols to alkyl halides instead of alkenes.~~

- 3) Why was it necessary to wash the crude alkene with a solution of aqueous base?

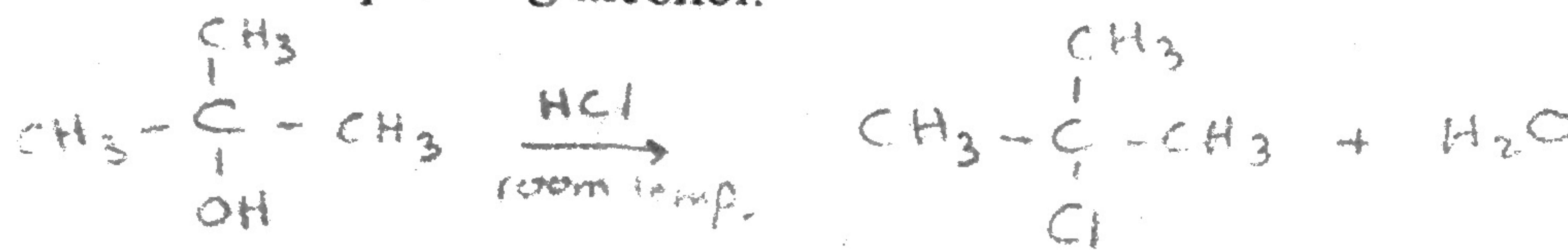
To neutralise the excess (H<sub>3</sub>PO<sub>4</sub>)

which would catalyse the conversion of alkene back to the starting alcohol.

# I (or II). Preparation of Alkyl Halide

1. 2 ✓

Write an overall equation for the preparation of the alkyl halide from the corresponding alcohol.



Theoretical yield of alkyl halide:  $n \cdot \text{mwt} = 0.075 \times 92.5 = 6.9375$

Actual yield:  $v \cdot d = 0.84 \times 1.2 = 1.008$

Percentage yield:  $\frac{\text{actual}}{\text{theoretical}} \times 100 = 14.53\%$

## III. Relative Reactivities of Alkyl Halides

Alkyl Halide	Reaction Time	
	$\text{AgNO}_3$	$\text{NaI}$
1-chlorobutane	No reaction	
2-chlorobutane	slow	
2-chloro-2-methylpropane	fast	
allyl chloride	—	
chlorobenzene.	No reaction	

Order of reactivity towards  $\text{AgNO}_3$ :

2-chloro, 2-methylpropane > 2-chlorobutane > 1-chlorobutane  
 $3^\circ \qquad \qquad \qquad 2^\circ \qquad \qquad \qquad 1^\circ \qquad >$   
 chlorobenzene

Order of reactivity towards  $\text{NaI}$ :

## QUESTIONS

1) Write all the steps in the purification of the alkyl halide mentioning the purpose of each.

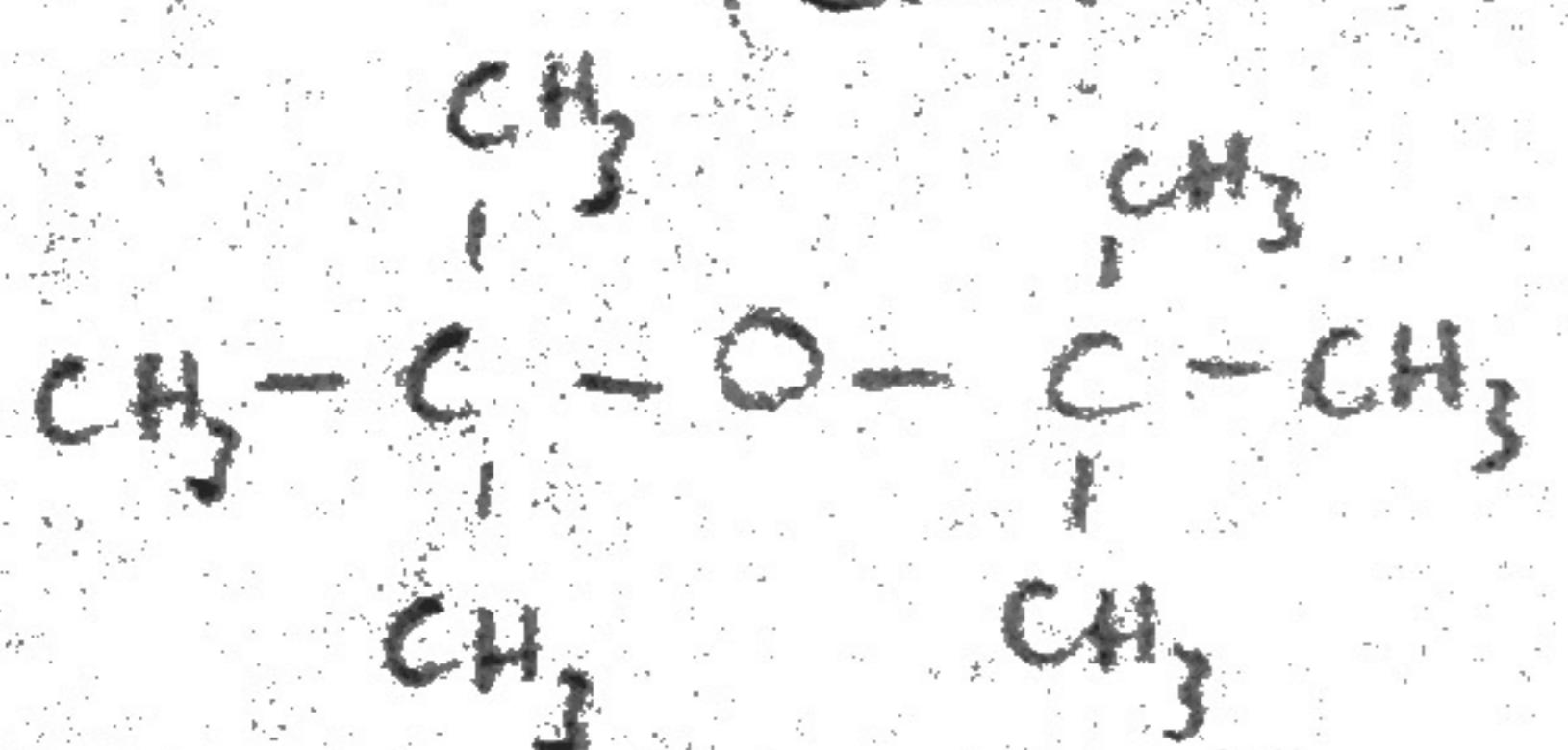
1) Separation : To separate the organic layer from the aqueous layer.

2) Washing : To react with  $H_2O$  and remove any traces of HCl.

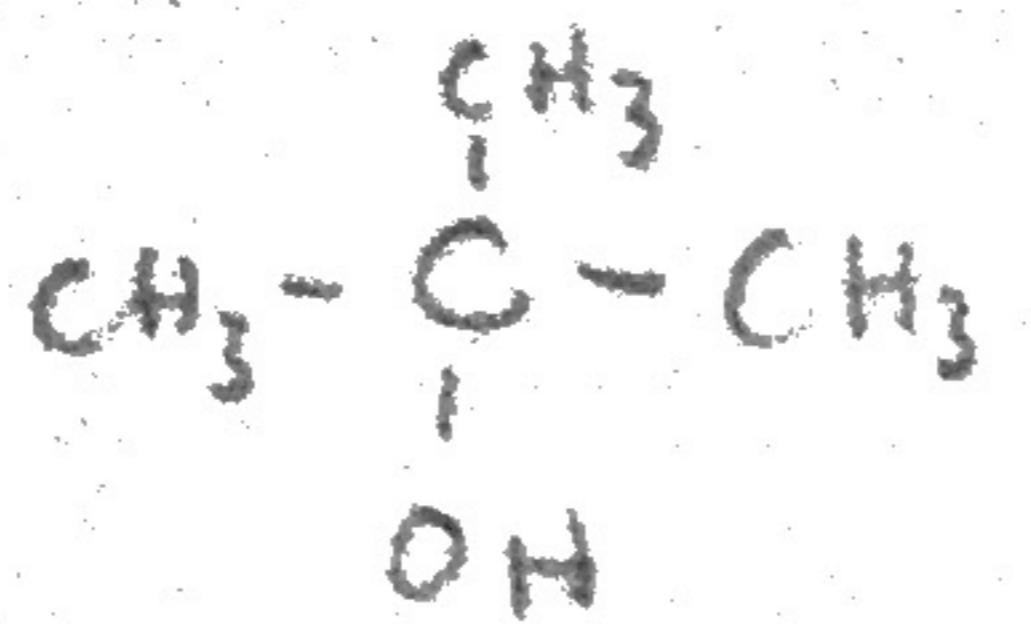
3) Drying agent : To get rid of water traces /

2) Write the structure of all possible by-products that may form during the preparation of the alkyl halide.

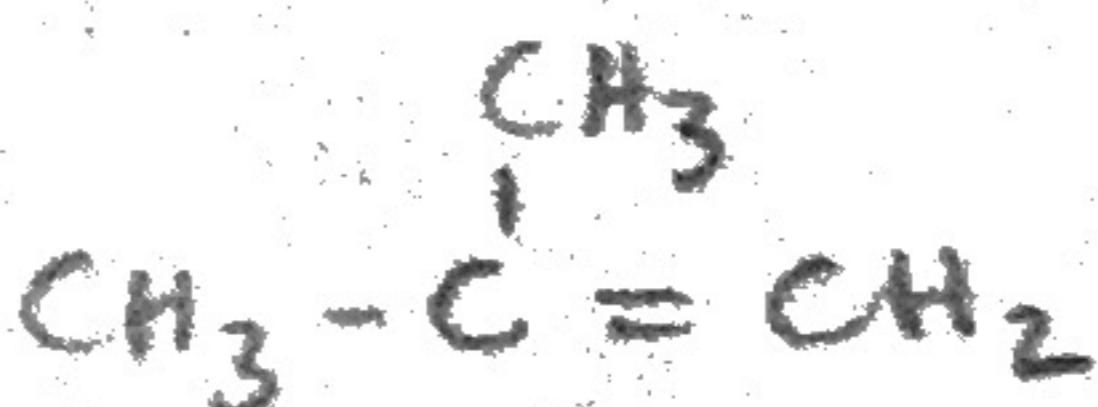
(ether)



(alcohol)



(alkene)



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

Because the Aromatic compound Chlorobenzene is stable. Carbo cation is not stable so the reaction will not form.

## ~~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.

	<i>o</i> -Nitrophenol	<i>p</i> -Nitrophenol
Theoretical yield		
Actual yield		
Percentage yield		

### **QUESTIONS**

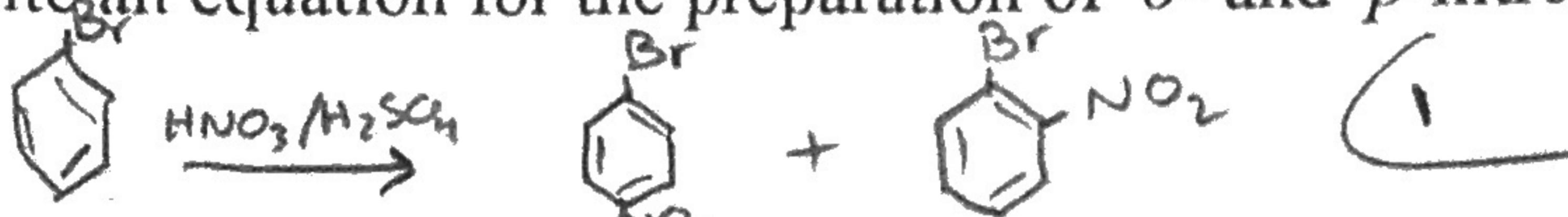
- 1) Write equations that show the mechanism for the nitration of phenol.

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.



Theoretical yield of *p*-nitrobromobenzene:  $n \times \text{mwt}$

Actual yield: 1.89

Percentage yield: ~~60%~~

$$\frac{1.89}{4.04} = 47\%$$

### QUESTIONS

1) What has happened to the *o*-nitrobromobenzene that was formed in this experiment? How can it be recovered?

It dissolved in the mother liquor because it is soluble in ethanol  
we can get it from evaporation.

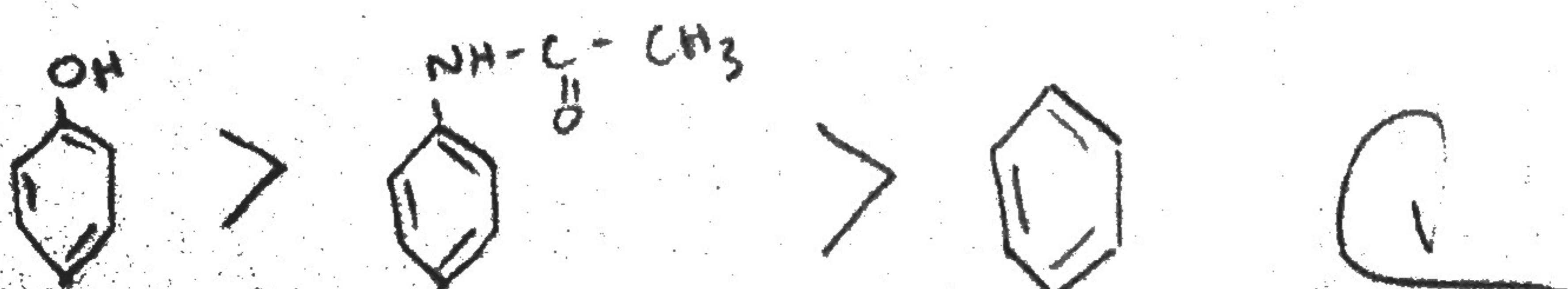
2) Why should the temperature be kept between 45-50 during the experiment? To prevent di and tri nitration.

### IV. Relative Bromination Rates.

compound	Structure	Reaction time
benzene		No reaction
acetanilide		slow
phenol		fast

5/20/08  
2

From your results, arrange the groups: NH-CO-CH<sub>3</sub>, H and OH in decreasing order of ring activation towards bromination.



# I. ALCOHOLS

## 1. Solubility of Alcohols in Water

Alcohol	Structure	Solubility
1-butanol	$\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OH}$	Not soluble
2-methyl-2-propanol	$\begin{array}{c} \text{CH}_3 \\   \\ \text{CH}_3\text{C}\text{H}-\text{OH} \\   \\ \text{CH}_3 \end{array}$	Soluble
cyclohexanol	$\begin{array}{c} \text{CH}_3 \\   \\ \text{C}_6\text{H}_5\text{OH} \end{array}$	Not soluble
ethylene glycol	$\begin{array}{c} \text{CH}_2-\text{CH}_2 \\   \quad   \\ \text{OH} \quad \text{OH} \end{array}$	Soluble

What general conclusions can you draw concerning the solubility of alcohols in water? Alcohols of low molecular weight are soluble in water, solubility in water decreases with increasing m.wt but increases with branching and with the number of OH group

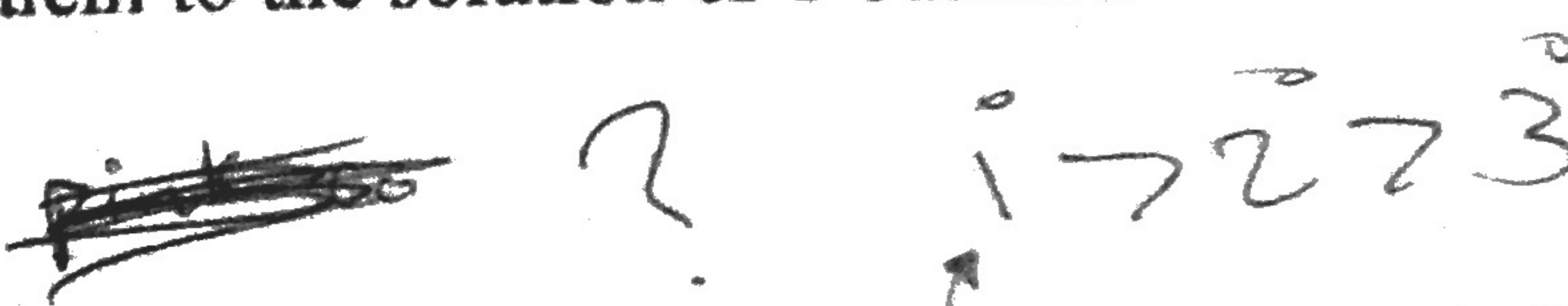
## 2. Acid Properties of Alcohols

Alcohol	Reaction Equation	Observations
1-butanol	$2\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OH} + 2\text{Na} \rightarrow 2\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{O}^-\text{Na}^+ + 2\text{H}_2$	fast
2-butanol	$2\text{CH}_3-\text{CH}_2-\text{CH}_2-\text{OH} + 2\text{Na} \rightarrow 2\text{CH}_3-\text{CH}_2-\text{O}^-\text{Na}^+ + 2\text{H}_2$	slow
2-methyl-2-propanol	$\text{CH}_3-\overset{\text{CH}_3}{\underset{\text{CH}_3}{\text{C}}}-\text{OH}$	very slow

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



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



The pink color will be strong

The color of the indicator changes to pink because the media is basic due to  $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{O}^-\text{Na}^+$  salt which is strong base.

### 3. Oxidation of Alcohols with Chromic Acid

Alcohol	Reaction Equation	Observations
1-butanol	$\text{K}_2\text{Cr}_2\text{O}_7 + \text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OH} \xrightarrow{\text{H}_2\text{SO}_4} \text{CH}_3\text{CH}_2\text{CH}_2\text{COH}$	green
2-butanol	$\text{K}_2\text{Cr}_2\text{O}_7 + \text{CH}_3\text{CH(OH)CH}_2\text{CH}_3 \xrightarrow{\text{H}_2\text{SO}_4} \text{CH}_3\text{C}(\text{OH})\text{CH}_2\text{CH}_3 + \text{Cr}_2\text{O}_5$	green
2-methyl-2-propanol	$\text{CH}_3\text{C}(\text{CH}_3)_2 + \text{K}_2\text{Cr}_2\text{O}_7 \xrightarrow{\text{H}_2\text{SO}_4} \text{no rxn.}$	orange no rxn.

Seeers

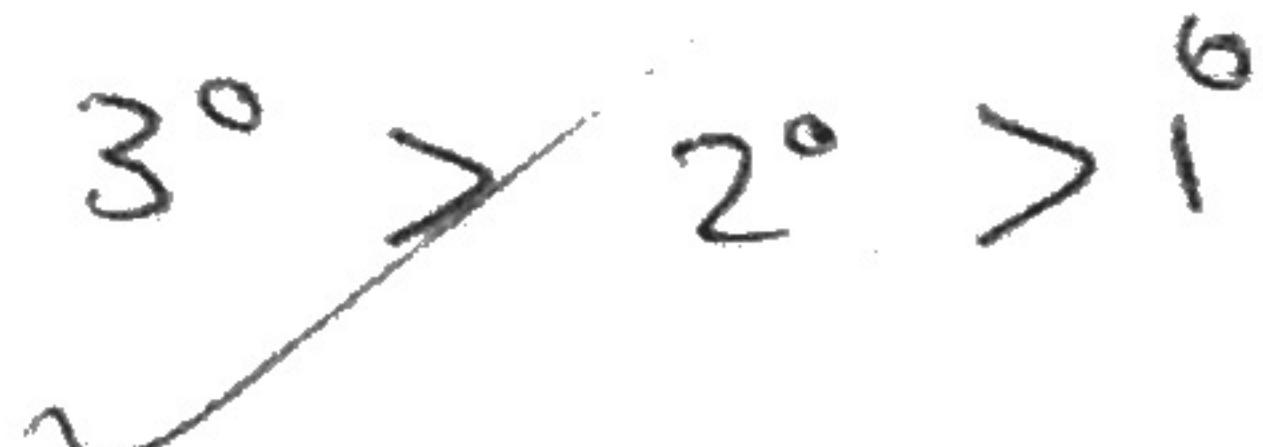
### 4. Lucas Test

Alcohol	Structure of Product	Observations
1-butanol	$\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{Cl}$	no rxn.
2-butanol	$\text{CH}_3\text{CH}(\text{Cl})\text{CH}_2\text{CH}_3$	slow
2-methyl-2-propanol	$\text{CH}_3\text{C}(\text{CH}_3)\text{CH}_2\text{Cl}$	very fast

(several hours  
very very slow)

Soar

Arrange the three alcohols according to their rates of reaction with the Lucas reagent:



### 5. Iodoform Test

Alcohol	Structure of Product	Observations
1-butanol	$\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OH}$	no rxn.
2-butanol	$\text{CH}_3\text{CH}_2\text{CH}(\text{OH})\text{CH}_3$	yellow ppt.
2-methyl-2-propanol	$\text{CH}_3\text{C}(\text{CH}_3)_2\text{OH}$	yellow ppt.

## II. PHENOLS

### 1. Acidity of Phenols

Compound	Structure	Solubility	
		Water	NaOH
cyclohexanol		not soluble	not soluble
phenol		not soluble	soluble
p-cresol		not soluble	soluble

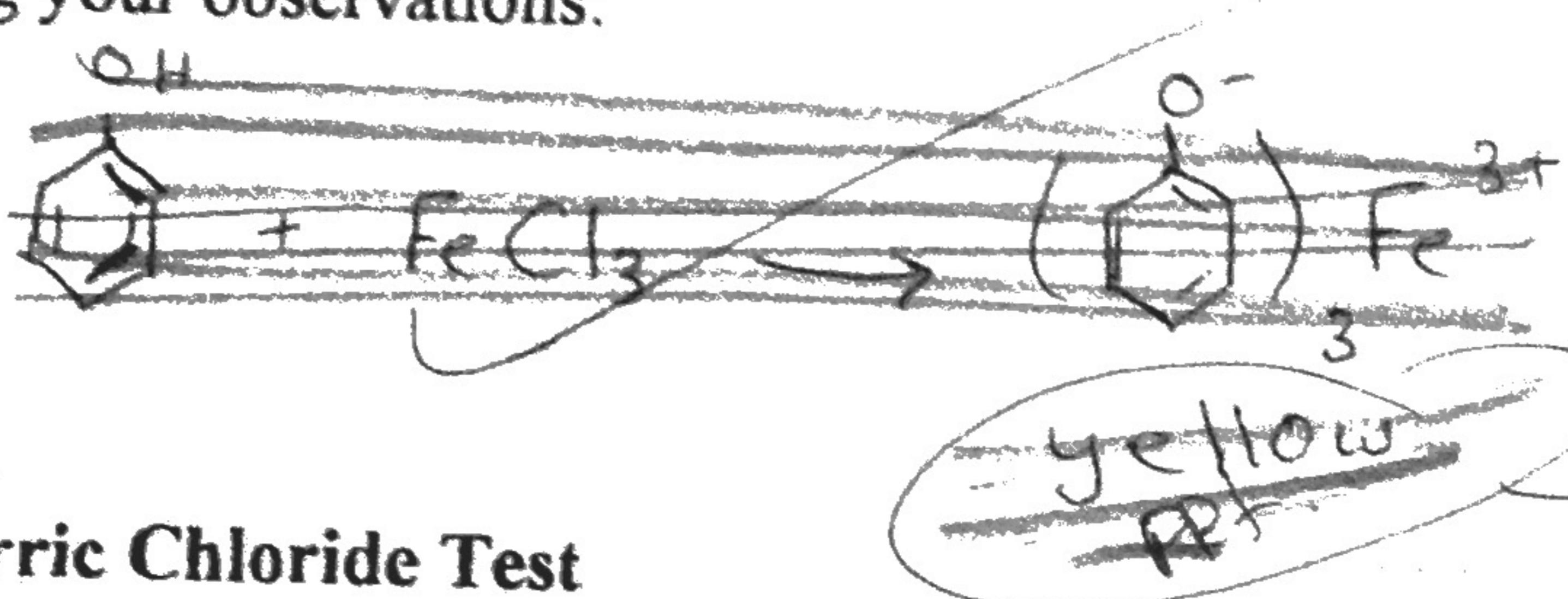
Write your conclusion about the solubility of alcohols and phenols in

water and NaOH solutions: ~~The phenol only is soluble in water~~

~~cyclohexanol and p-cresol are soluble in NaOH~~  
 cyclo alcohols and phenols are insoluble in water but phenols are strong acids and react with NaOH, Cyclohexane don't react with NaOH so it is insoluble in it.

### 2. Reaction of Phenols with Bromine Water

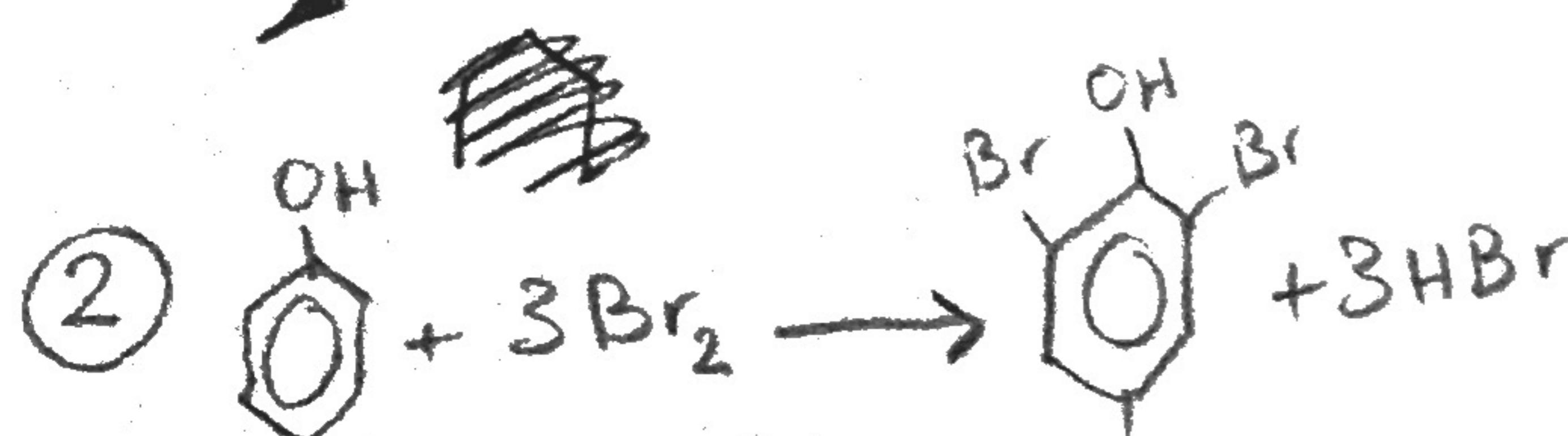
Write an equation for the reaction of phenol with bromine water giving your observations:



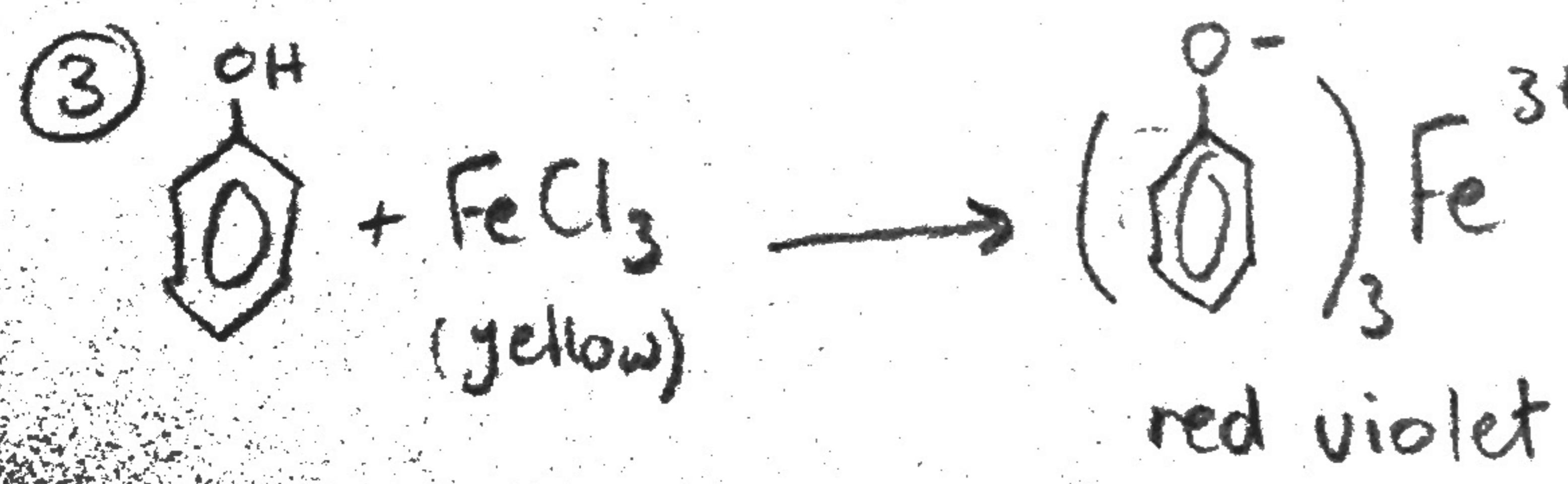
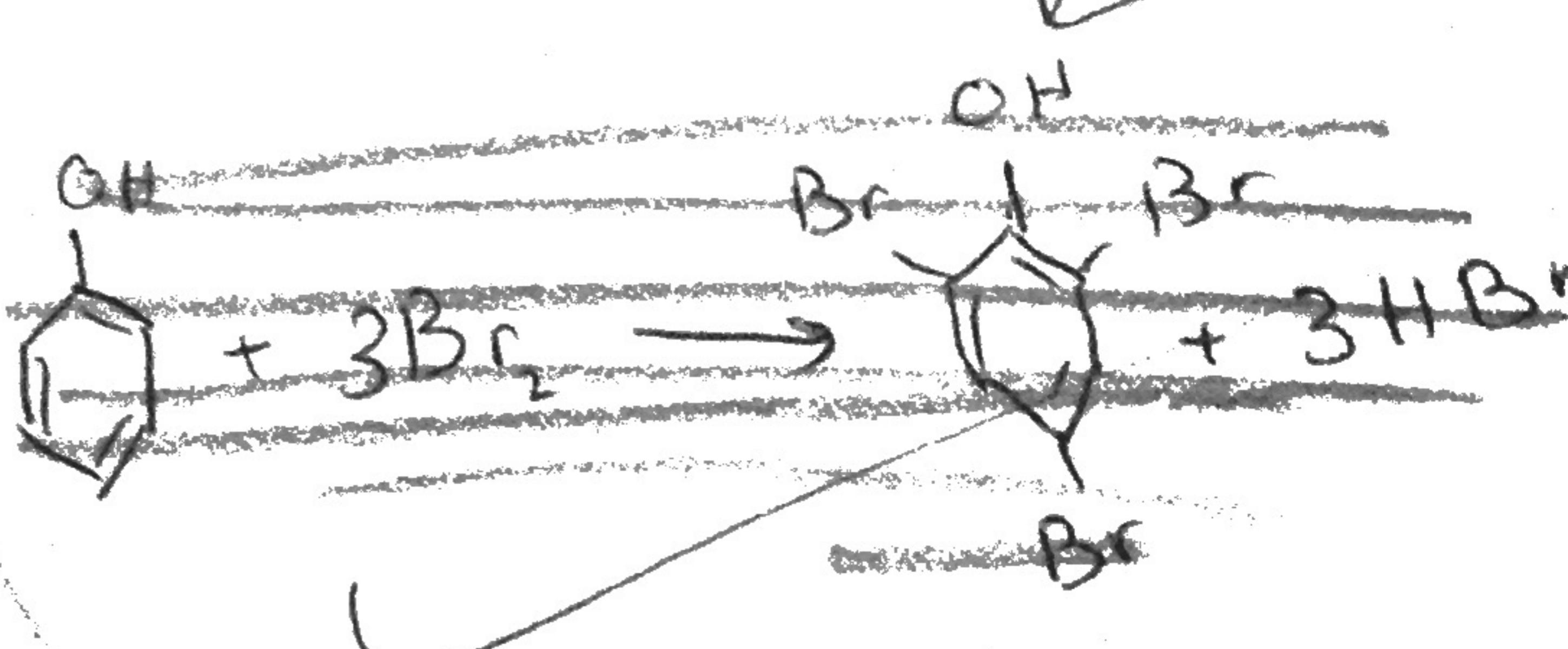
### 3. Ferric Chloride Test

Alcohol	Structure	Observations
cyclohexanol		no rxn still yellow
phenol		green-violet

Write an equation for the reaction of phenol with  $\text{FeCl}_3$ :



Red brown color of bromine is discharged and white ppt. is formed



## 1. 2,4-Dinitrophenylhydrazine Test

Compound	Structure	Observations and Results
acetone	$\text{CH}_3-\overset{\text{O}}{\underset{\text{H}}{\text{C}}}-(\text{H}_3)$	Orange ppt fast
benzaldehyde	$\text{C}_6\text{H}_5-\overset{\text{O}}{\underset{\text{H}}{\text{C}}}-\text{H}$	Orange ppt slow

## 2. Tollens' Test

Compound	Structure	Observations and Results
formaldehyde	$\text{H}-\overset{\text{O}}{\underset{\text{H}}{\text{C}}}-\text{H}$	silver mirror slow
benzaldehyde	$\text{C}_6\text{H}_5-\overset{\text{O}}{\underset{\text{H}}{\text{C}}}-\text{H}$	silver mirror
acetone	$\text{CH}_3-\overset{\text{O}}{\underset{\text{H}}{\text{C}}}-\text{CH}_3$	no rxn

## 3. Fehling's or Benedict's Tests

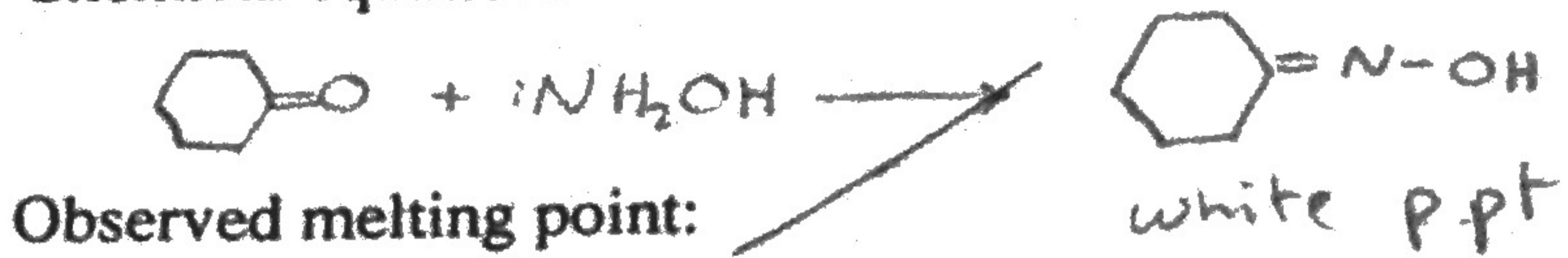
Compound	Structure	Observations and Results
formaldehyde	$\text{H}-\overset{\text{O}}{\underset{\text{H}}{\text{C}}}-\text{H}$	red
benzaldehyde	$\text{C}_6\text{H}_5-\overset{\text{O}}{\underset{\text{H}}{\text{C}}}-\text{H}$	red
acetone	$\text{CH}_3-\overset{\text{O}}{\underset{\text{H}}{\text{C}}}-\text{CH}_3$	no rxn

## 4. Iodoform Test

Compound	Structure	Observations and Results
acetone	$\text{CH}_3-\overset{\text{O}}{\underset{\text{H}}{\text{C}}}-\text{CH}_3$	yellow ppt
2-propanol	$\text{CH}_3\overset{\text{OH}}{\underset{\text{H}}{\text{C}}}-\text{CH}_3$	yellow ppt
2-pentanone	$\text{CH}_3\overset{\text{O}}{\underset{\text{H}}{\text{C}}}-\text{CH}_2\text{CH}_2\text{CH}_3$	yellow ppt
3-pentanone	$\text{CH}_3\text{CH}_2\overset{\text{O}}{\underset{\text{H}}{\text{C}}}-\text{CH}_2\text{CH}_3$	no rxn

## 5. Cyclohexanone Oxime

Chemical equation:



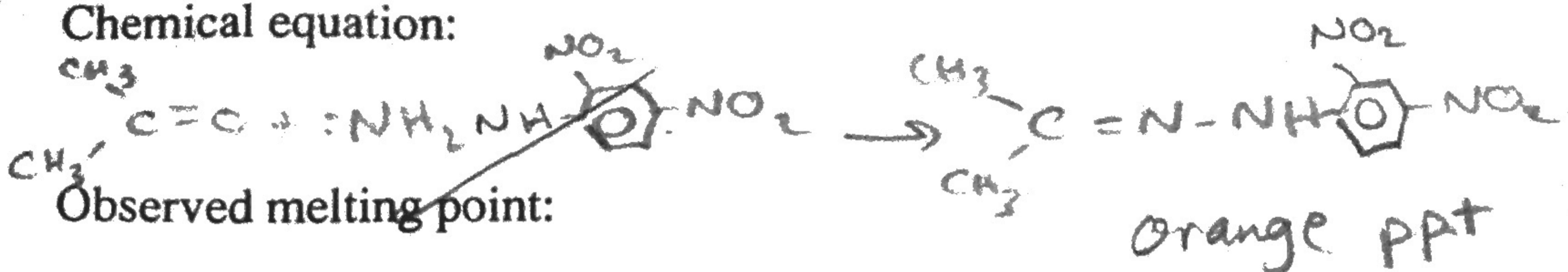
## 6. Cyclohexanone Phenylhydrazone

Chemical equation

Observed melting point:

## 7. Acetone 2,4-Dinitrophenylhydrazone

Chemical equation:



## 8. Cyclohexanone Semicarbazone

Chemical equation

Observed melting point:

## 9. Identification of Unknown

Unknown No.:

Tollen's Test:

Iodoform Test :

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

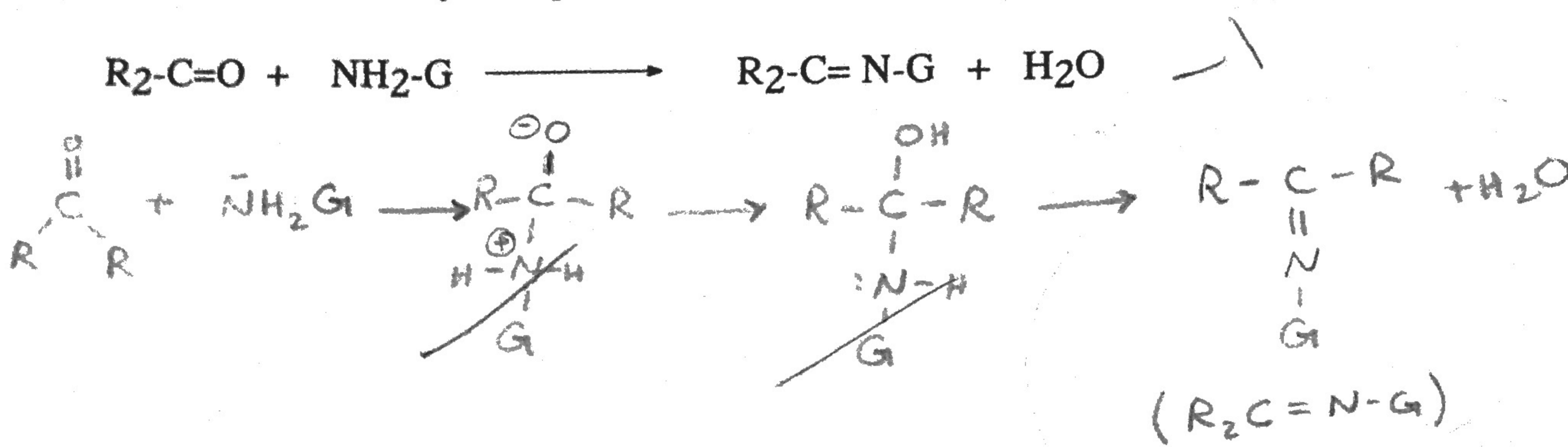
2) ..... mp:.....

3) ..... mp:.....

Unknown is:

**QUESTIONS**

- 1) Write a detailed general mechanism for the preparation of derivatives of carbonyl compounds:

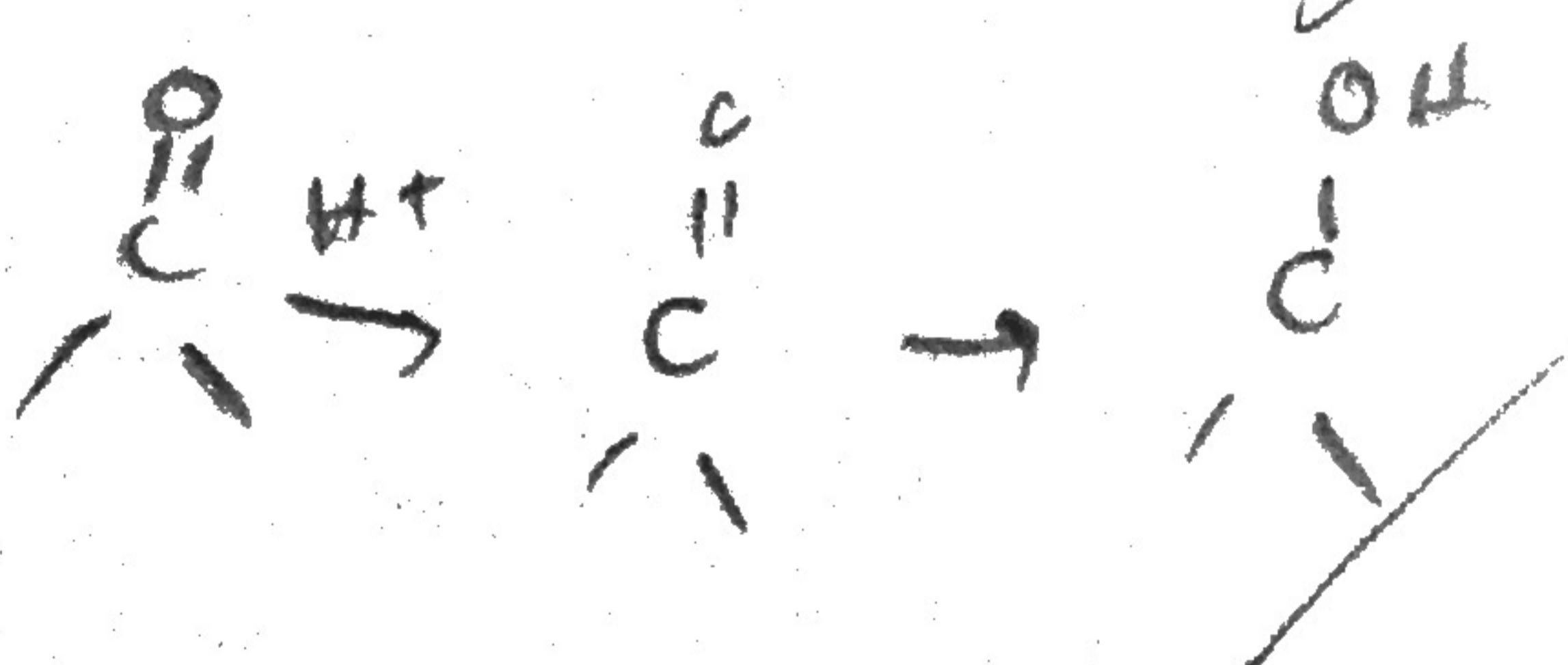


- 2) Which of the following compounds gives a positive iodoform test?

- a)  $\text{CH}_3\text{-CH}_2\text{-CH}_2\text{-CO-CH}_2\text{-CH}_3$
- b)  $\text{CH}_3\text{-CH}_2\text{-CH}_2\text{-CH(OH)-CH}_3$  ✓
- c)  $\text{CH}_3\text{-CH}_2\text{-CH}_2\text{-CH}_2\text{-CHO}$

- 3) With the help of a chemical equation, suggest a role for the acid in the formation of 2,4-dinitrophenylhydrazones.

$\text{H}_2\text{SO}_4$  is used to catalyze the rxn by increasing the electron deficiency of the carbon rate increase



convert OH from bad leaving group to good  
One  $\text{OH}^-$

## I. Benzoic Acid by Hydrolysis of Benzonitrile

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



Theoretical yield of benzoic acid:

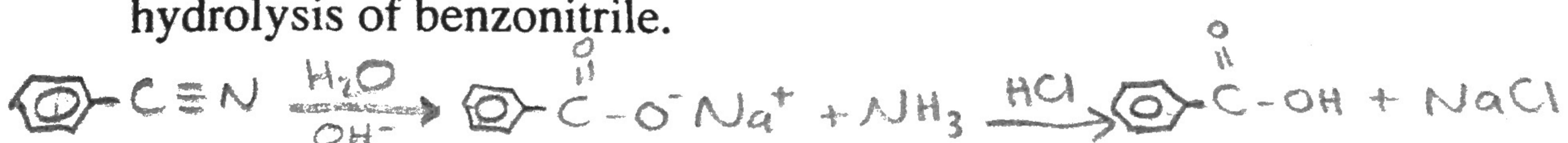
Actual yield:

Percentage yield:

Melting point of benzoic acid:

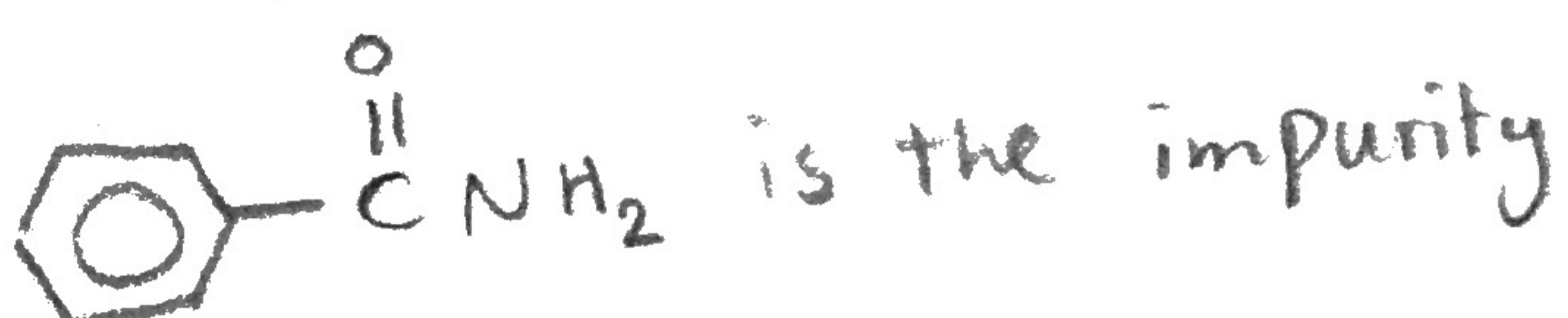
### QUESTIONS

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



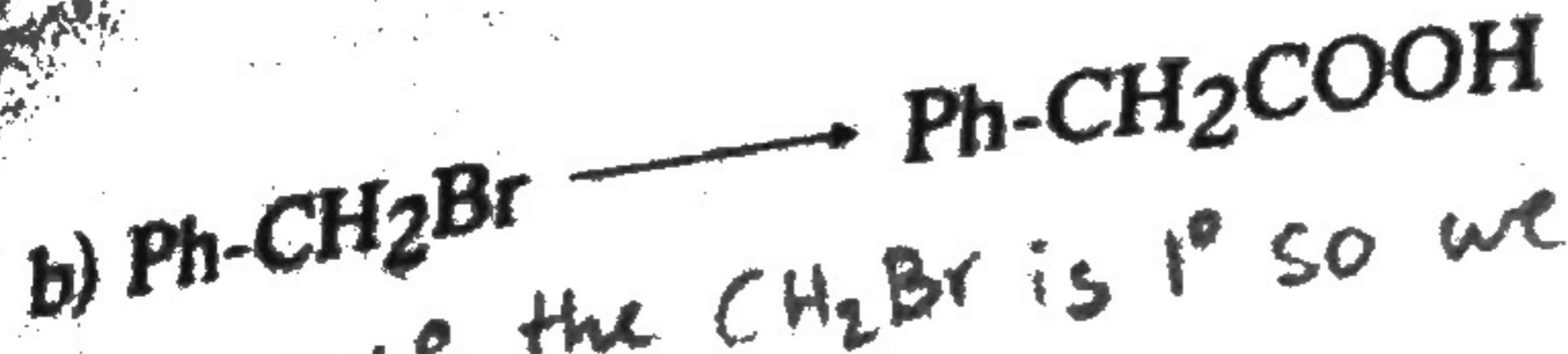
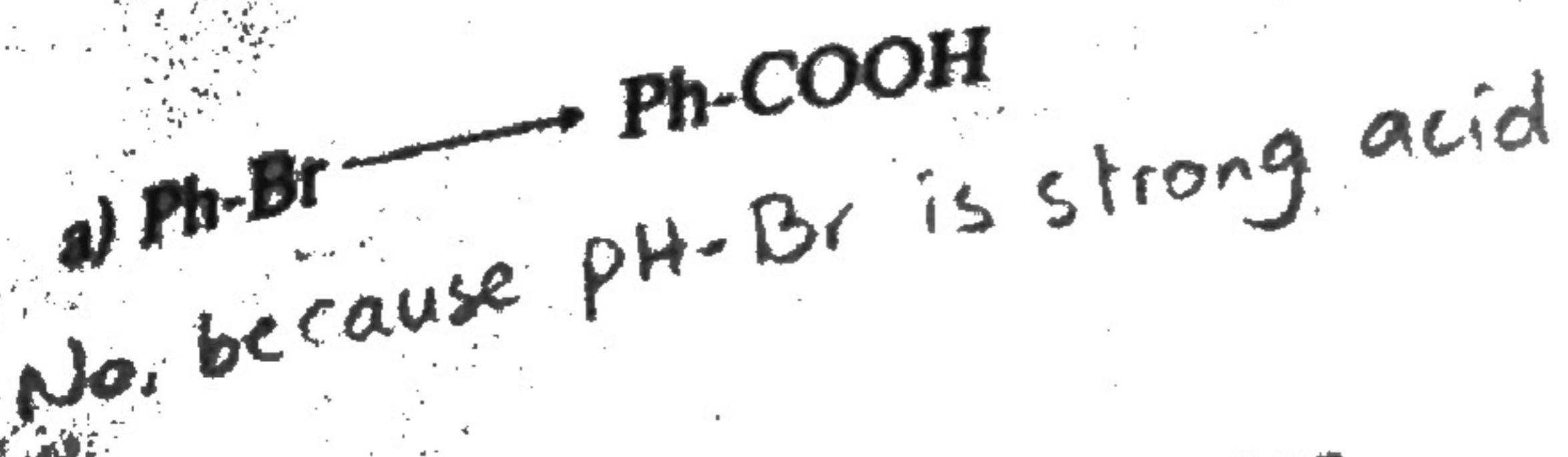
The base will convert the acid into water-soluble salt from which the free acid may be regenerated by acidification with a mineral acid.

- 2) What impurity might be found in the benzoic acid prepared from benzonitrile? How does this impurity arise?

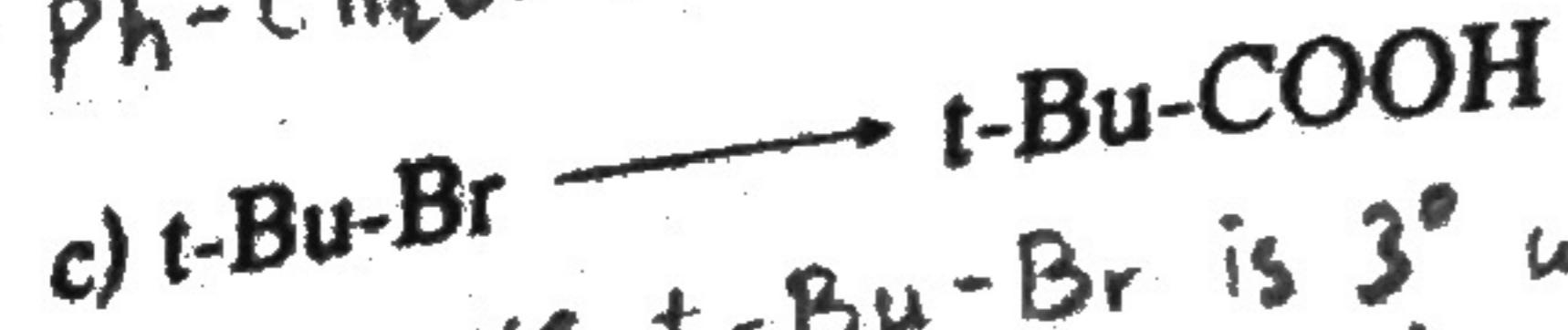
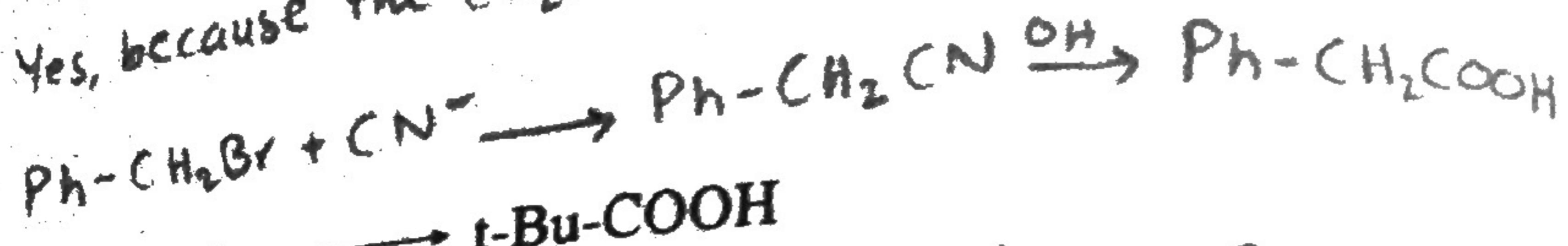


amide  $\text{CO}_2$  of incomplete hydrolysis

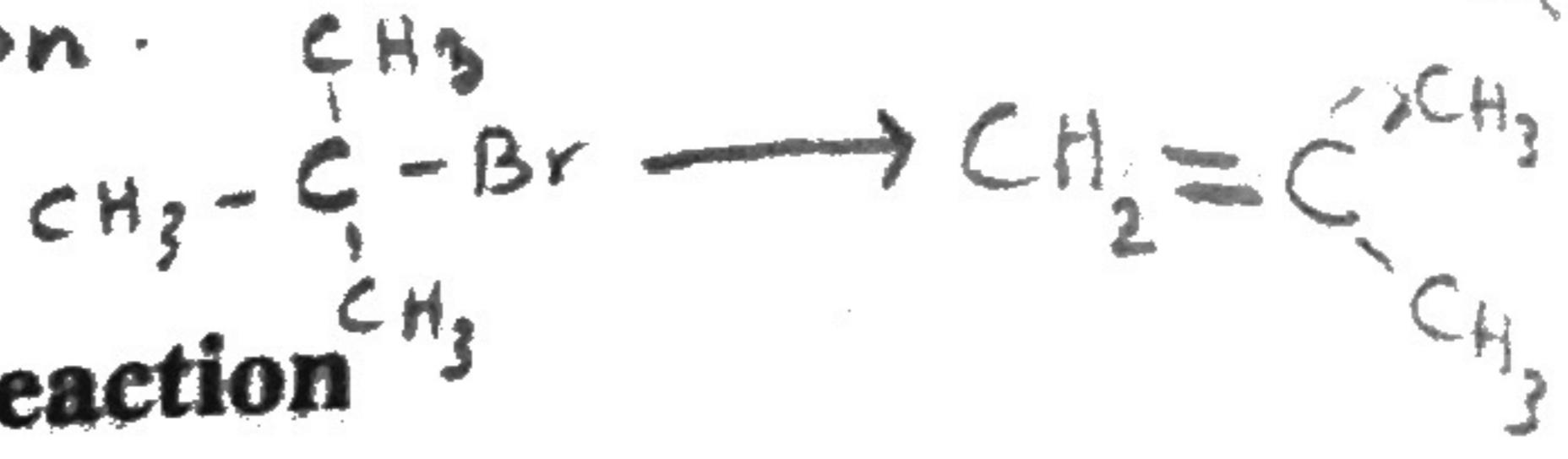
- 3) Would it be possible to use the nitrile method to achieve the following conversions? Illustrate with equations.



Yes, because the  $\text{CH}_2\text{Br}$  is  $1^\circ$  so we can use  $\text{SN}_2$



No, because  $\text{t-Bu-Br}$  is  $3^\circ$  we can't use  $\text{SN}_2$  and the rxn doesn't go substitution.



## II. Benzoic Acid by the Haloform Reaction

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

Theoretical yield of benzoic acid:

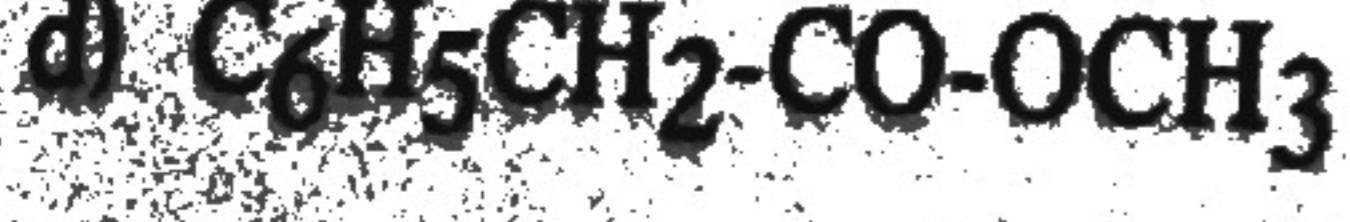
Actual yield:

Percentage yield:

Melting point of benzoic acid:

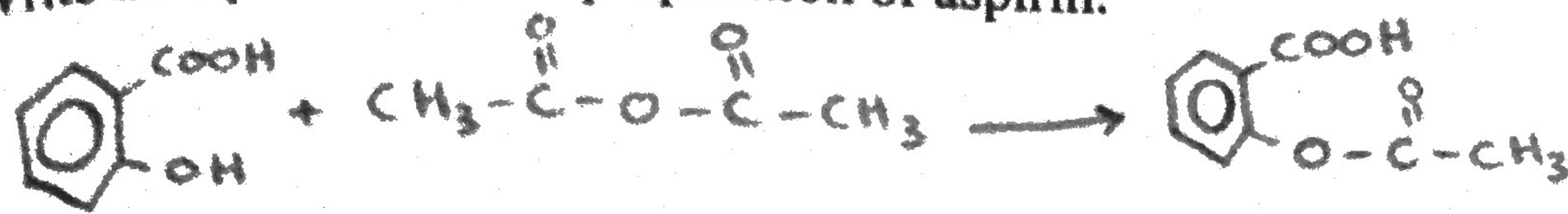
## QUESTIONS

1) Which of the following compounds will give the haloform reaction?



## I. Preparation of Aspirin

Write an equation for the preparation of aspirin:



Theoretical yield of aspirin:

Actual yield:

Percentage yield:

(A)

### Ferric Chloride Test

Color with Salicylic acid:

violet

~~Score~~ Color with Aspirin:

colorless

## X II. Preparation of Methyl Benzoate

Write the overall equation for the preparation of methyl benzoate.

Theoretical yield of methyl benzoate:

Actual yield:

Percentage yield: