



# R. K. GROUP OF COLLEGE

BEHIND KALWAR POLICE STATION, KALWAR, JAIPUR (RAJ.)





# CERTIFICATE

Name: Tanu Sain

Class: ~~III~~ Bsc III<sup>rd</sup> sem.

Roll No.:

Exam No.:

Institution \_\_\_\_\_

This is certified to be the bonafide work of the student in the \_\_\_\_\_

\_\_\_\_\_ Laboratory during the academic

year 20 \_\_\_\_ /20

No of practicals certified \_\_\_\_\_ out of \_\_\_\_\_ in the  
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Teacher in-charge

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Principal

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Examiner's Signature

Date : .....

institution Rubber stamp



# INDEX

S. No.	Name of Experiment	Page No.	Date of Experiment	Date of Submission	Remarks
1.	Preparation of Nickel dimethylglyoxime Complex.				
2.	In given organic compound identify one solid and one liquid functional, their BP/MP and its suitable derivative				
3.	Synthesis of acetanilide from aniline				
4.	To determine heat of neutralisation of strong acid and weak base.				
5.	To estimate copper as $CuSCN$ gravimetrically				
6.	In given organic compound identify one solid and one liquid functional group their BP/MP and its suitable derivatives.				
7.	Synthesis of meta-nitro aniline from meta dinitrobenzene				
8.	To study the distribution of benzoic acid between benzene and water.				
9.	Determination of Zinc as Zinc pyrophosphate				







Object :- Nickel dimethylglyoxime complexes  
 $[Ni(DMG)_2]$

Necessary Equipment :-

- (i) Nickel chloride - 6g,
- (ii) Di-methylglyoxime - 10%
- (iii) Ammonia solution - 1:1

Equipments :- Beaker, watch glass, test tube, Burner, Stirrer.

Method :-

One 400 ml. Beaker given nickel sulphate 6 gm Heat at  $72^{\circ}C - 80^{\circ}C$  and this solution put in 42-50 ml. Dimethylglyoxime. after this 5 in solution 1:1 ammonium solution drop by drop in watch glass.

In Beaker the water heat at  $20-30^{\circ}$  the down dropped read clear sum Di-methylglyoxime reagent mixed and testit. that the solution is complet or not. Beaker put for half-hour.

This is seperated by funnel and this is wased cold-water two three time and in the solution of wased 5 ml. alcohol. And dried it.







Result :-

Above method  $\text{Ni}(\text{DMG})$  prod used Read  
Colour solution.

Grained :-

3-4 gm

Precaution :- Beaker should be clean and  
~~dry~~ dried.

Beaker heat with the help of water  
reagent.



Object :-

In the given organic compound identify one solid and one liquid functional, their B.P / M.P and its suitable derivative.

Test for solid Compound (A)

Primary Test - (i) Physical state - Solid

(ii) Colour - white

(iii) Odour - Bad odour

(iv) Solubility - Soluble in boiling water

Observation table :-

S.No.	Test	Observation	Result
1.	Flame Test - Take compound on spatula & heat on burner	Burns with black flames	Compound is aromatic
2.	Litmus Test - Dip blue litmus paper in Sol <sup>n</sup> of compound	litmus paper turns red	Compound is acidic.
3.	Element test - L.S + freshly prepared $FeSO_4$ sol <sup>n</sup> + conc <sup>n</sup> $H_2SO_4$	No Colour or ppt obtained	Nitrogen is absent.
4.	Functional group test Compound + neutral $FeCl_3$ Sol <sup>n</sup>	Ppt obtained	Carboxylic group present.



5.	H.P. of compound H.P. of given compound	$\approx 121^\circ\text{C}$	Compound may be benzoic acid <chem>c1ccccc1C(=O)O</chem>
6.	<u>Confirmatory Test</u>		
i)	Compound + $\text{NH}_4\text{OH}$ sol <sup>n</sup> + neutral $\text{FeCl}_3$ sol <sup>n</sup>	ppt obtained	benzoic acid present.
ii)	Compound + conc. $\text{H}_2\text{SO}_4$ + alcohol + $\Delta$	funky smell obtained	benzoic acid present.

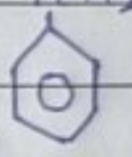
Test for liquid compound, (B) -

- Primary test -
- (i) Physical state - fluid
  - (ii) Colour - Colourless
  - (iii) Smell - Specific smell
  - (iv) solubility - soluble in water.

Observation Table

S.No.	Test	Observation	Result
1.	flame Test - Take compound on spatula & burn on luxmer	Burn without black flames	Compound is aromatic.
2.	litmus test - Dip red litmus paper in solution of compound.	litmus paper turns blue.	Compound is basic.



3. Element test - 1. S. + FeSO <sub>4</sub> Sol <sup>n</sup> freshly prepared + conc <sup>n</sup> H <sub>2</sub> SO <sub>4</sub>	Green ppt obtained	Nitrogen is present
4. Functional group test Compound + 2ml H <sub>2</sub> O + 1ml conc <sup>n</sup> HCl then cool it, new mix 3-5% NaNO <sub>2</sub> sol <sup>n</sup> + 2ml $\beta$ -Naphthal	Red-orange ppt is obtained	Amino group present.
5. B.P of compound	183° C	Aniline may be present 
6. Confirmatory Test		
i) 2 drop compound + ether + 10ml H <sub>2</sub> O + 1ml Sol <sup>n</sup>	purple colour obtained	Aniline confirm
ii) Compound + K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> + conc <sup>n</sup> H <sub>2</sub> SO <sub>4</sub>	Blue or black colour sol <sup>n</sup>	Aniline confirm
iii) dilute sol <sup>n</sup> of compound + solid NaNO <sub>2</sub> + basic $\beta$ -Naphthal solution	Red dye obtained	Aniline is present.



Solubility in  $\text{NaHCO}_3$

↓	↓
Soluble Compound (A)	Insoluble Compound (B)
Solid compound	Liquid compound
Carboxylic acid	Alcohol

formation of derivative :-

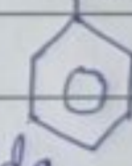
Solid Compound (A) - Take 0.5 gram carbon compound in a flask, then add 2 ml. Thionyl chloride ( $\text{SOCl}_2$ ) & heat for 20-30 minutes which leads to formation of acid chloride.

Now on adding 10 ml. concentrate ammonia solution drop by drop, there will be vigorous reaction. Now cool the mixture in ice, on cooling, white crystal of amide are obtained.

Liquid Compound (B) -

Take 1 gram aniline in a conical flask, add 10-15 ml. acetic acid and make it a solution. Now add bromine solution drop by drop till yellow colour obtained. Now heat it. Now slowly pour this solution in 50-100 ml water, to get separate bromo derivative.

Result :- In the carbon compound solid compound (A) is benzoic acid  $\text{C}_6\text{H}_5\text{COOH}$  where M.P =  $121^\circ\text{C}$



and its derivatives is amide.

Liquid compound (B)  $\rightarrow$  Aniline ( $\text{C}_6\text{H}_5\text{NH}_2$ )

B.P =  $183^\circ\text{C}$

Derivative = 2, 4, 6. tribromo aniline

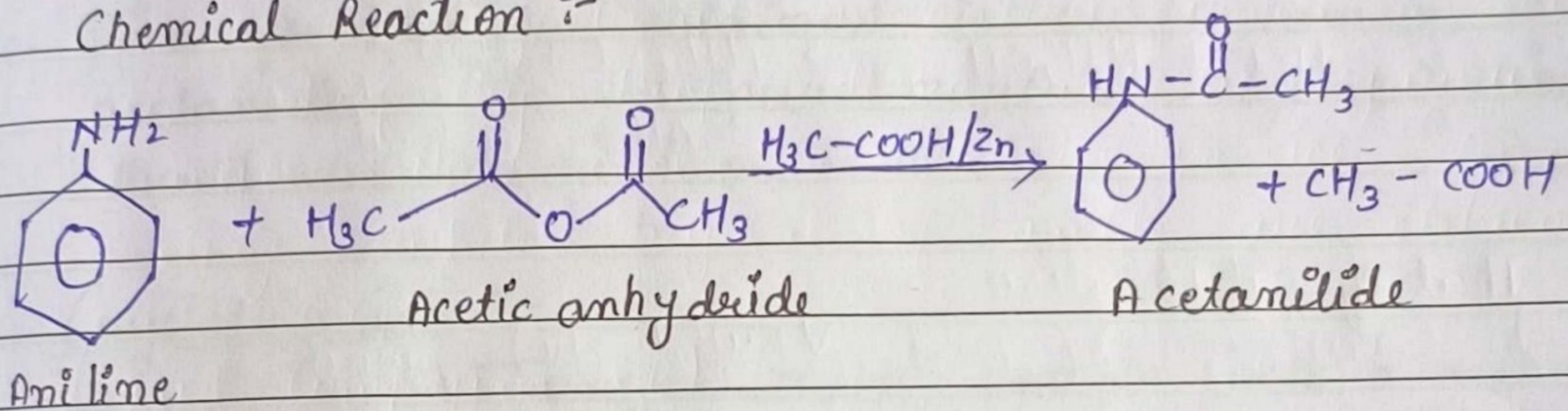


Object :- Synthesis of acetanilide from aniline.

Theory :-

Acetanilide is prepared from aniline when it reacts with acetic anhydride / glacial acetic acid in the presence of zinc dust.

Chemical Reaction :-



Material required :-

Aniline, Glacial acetic acid, acetic anhydride, zinc dust, distilled water, flask, beaker, pipette, reflux condenser, funnel, stirrer, burner, filter paper, Electronic balance.

Procedure :-

1. Wash all apparatus with distilled water before starting experiment.
2. Take flask and add 10 ml of aniline 20 ml of acetic anhydride, glacial acetic and mixture and zinc dust.
3. fix reflux condenser with flask & heat mixture gently for about 15-20 minutes in oil bath.



4. Pour hot mixture in beaker containing ice cold water with constant stirring & stir the mixture vigorously to hydrolyse excess of acetic anhydride.
5. Once all acetamide is precipitated collect & filter in funnel.
6. The precipitate obtained is crude sample of acetamide. To get pure crystals, crystallization should be carried out.

### Crystallization :-

Transfer crude sample into a beaker containing 20 ml water and heat gently. If the solution is coloured then add a small amount of activated carbon. Filter the hot solution with a funnel. Cool the mixture for 30 minute so that white shiny crystals of acetamide separate out. Filter off the crystals, wash them with water and dry in the folds of filter paper.

Result :- white shining crystal of aniline acetamide are obtained.

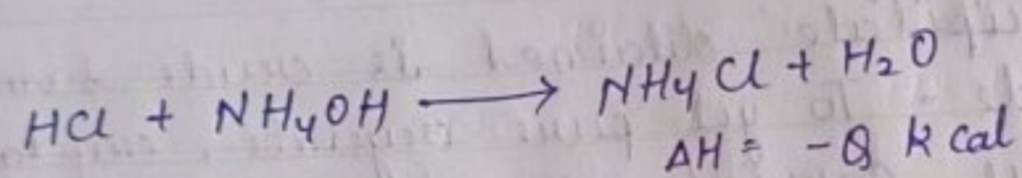
Yield of Acetamide = 12 gm

Shape of crystals = Plate shaped.

melting point =  $114^{\circ}\text{C}$

Colour of crystals = Colourless crystal





$$\text{Heat of neutralisation} = \frac{V+w(T_s-T_4)}{N_1 V_1} \times 1000$$

where

- $w$  = equivalent weight of water
- $V$  = Volume of mixture of acid & base
- $T_4$  = temperature of acid & base
- $T_s$  = final temperature of mixture.
- $V_1$  = Volume of acid
- $N_1$  = Normality of acid

PAGE NO. ....

Experiment = 4

DATE .....

Object :-

To determine heat of neutralization of strong acid (HCl) and weak base (NH<sub>4</sub>OH)

Principle :- Heat released in neutralization of 1 gram equivalent of strong acid and 1 gram equivalent of weak base is called heat of neutralisation. Takes place in two steps :-

- i) In the first step ionisation of weak acid takes place.
- ii) In second step combination of H<sup>+</sup> & OH<sup>-</sup> gives H<sub>2</sub>O. i.e. some amount of heat is absorbed during ionisation lowering value of heat of neutralization 13.7 kcal.

Heat of neutralisation

$$\text{Heat of neutralisation} = \frac{V+w(T_s-T_4)}{N_1 V_1} \times 1000$$

where

- $w$  = equivalent weight of water
- $V$  = Volume of mixture of acid & base
- $T_4$  = temperature of acid & base
- $T_s$  = final temperature of mixture
- $V_1$  = Volume of acid
- $N_1$  = Normality of acid



### Calculation

1) Equivalent weight of  $H_2O$  in thermos flask

$$w = \frac{100(t_2 - t_3)}{t_3 - t_1} - 100$$

$$w = 100 \frac{(73 - 47)}{47 - 25} - 100$$

$$w = \frac{100 \times 26}{26} - 100 = 118.18 - 100$$

$$w = 18.18 \text{ Calorie/degree}$$

2) Calculation of heat of neutralization

$$= \frac{V + w(T_5 - T_4) \times 1000}{\text{Volume of acid \& base} \times \text{Normality of acid \& base}}$$

Volume of acid & base  $\times$  Normality of acid & base

$$V = 100 + 100 = 200 \text{ ml}$$

$$T_4 = 18.1^\circ \text{C}$$

$$T_5 = 24^\circ \text{C}$$

$$w = 18.18 \text{ Calorie}$$

$$= \frac{(200 + 18.18(24^\circ \text{C} - 18.1)) \times 100}{100 \times 1}$$

$$= \frac{218.18 \times 5.9 \times 100}{100}$$

$$= 128.8 \text{ Kcal}$$

PAGE NO. ....

DATE .....

Required apparatus - Thermos flask, two thermometer ( $0.1^\circ \text{C}$ ), heater, a glass stirrer, luxette etc.

Required solution - HCl sol<sup>n</sup> of N-normality  
NH<sub>4</sub>OH sol<sup>n</sup> of N-normality  
Phenolphthalein.

### Observation -

1. Equivalent weight of water in thermos flask  
Volume of cold water - 100 ml  
temperature of cold water -  $t_1 = 25^\circ \text{C}$   
temperature of warm water -  $t_2 = 73^\circ \text{C}$   
temperature of mixture -  $t_3 = 47^\circ \text{C}$

2. Heat of neutralization  
temperature of sol<sup>n</sup> of N-HCl & N-NH<sub>4</sub>OH  
 $t_4 = 19.2 + 17.02 = 18.1^\circ \text{C}$   
temperature of mixture -  $t_5 = 24^\circ \text{C}$

Result - Heat of neutralisation of strong acid (HCl) & weak base is 128.8 kcal.



Aim :-

To estimate copper as  $\text{CuSCN}$  gravimetrically

Apparatus Required

Desiccates, sintered glass crucible (G4), wire gauze, Reagent Beaker (400ml), black watch glass, Bunsen Burner, Tripod stand, vacuum filtration apparatus, Stirrer.

Chemical Required

Copper sulphate pentahydrate solution, freshly prepared sulphurous acid solution, Ammonium thiocyanate solution, wash liquid (200 cc distilled water + Ammonium thiocyanate (24, 10%) + Sulphurous acid (10-15 drops) & Alcohol (20%)

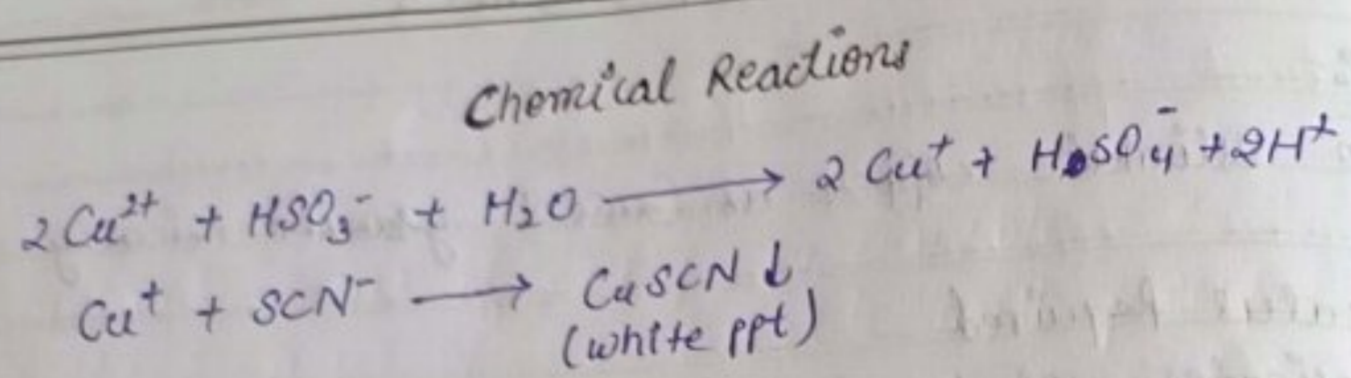
Theory

When cupric ions are treated with  $\text{NH}_4\text{SCN}$  in a solution containing  $\text{H}_2\text{SO}_3$  (reducing agent) the copper (I) thiocyanate is precipitated which can be weighed after drying at  $110^\circ\text{C}$

The essential experimental conditions are :-

- \* The presence of reducing agent such as  $\text{H}_2\text{SO}_3$  or Ammonium hydrogen sulphate to reduce  $\text{Cu(II)}$  to  $\text{Cu(I)}$
- \* Solution should be slightly acidic since in strong acidic medium  $\text{CuSCN}$  ppt dissolves  $\text{HNO}_3$  should not be used since it oxidise  $\text{Cu(I)}$  to  $\text{Cu(II)}$
- \* Slight excess of  $\text{NH}_4\text{SCN}$  should be added since the large excess increases the solubility of copper due to the





Observation Table

Mass of empty G4 crucible

$$m_1 = 22.6370 \text{ g}$$

$$m_2 = 22.4122 \text{ g}$$

$$m_3 = 22.4188 \text{ g}$$

$$\text{since } m_2 - m_3 = 0.0004 \text{ g}$$

$$\text{constant mass of empty G4 crucible} = 22.4188 \text{ g}$$

mass of G4 crucible with precipitate

$$m_1 = 23.0177 \text{ g}$$

$$m_2 = 22.6841 \text{ g}$$

$$m_3 = 22.6837 \text{ g}$$

$$\text{since } m_2 - m_3 = 0.0004 \text{ g}$$

$$\therefore \text{constant mass of G4 crucible with precipitate} = 22.6837 \text{ g}$$

PAGE NO. ....

DATE .....

formation of a complex copper thiocyanate ion.

\* The ppt should be dried at  $110^\circ\text{C}$  for 2-4 hrs since  $\text{CuSCN}$  is stable upto  $200^\circ\text{C}$ . Above  $300^\circ\text{C}$  the ppt is converted to  $\text{Cu}_2\text{S}$ .

\* The ppt are curdy & its readily coagulated by boiling & is washed with  $\text{NH}_4\text{SCN}$  & a little  $\text{H}_2\text{SO}_3$  acid or ammonium hydrogen sulphate is added to washed solution to prevent any oxidation of  $\text{Cu(I)}$  ion.

Procedure:-

20 cc of  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  solution was taken in 400 cc beaker few drops of dil.  $\text{HCl}$  was added followed by slight excess of the freshly prepared sulphurous acid solution (35-40 cc). The solution was diluted with distilled water upto 150 cc. It was heated nearly to boiling & then a slight excess of ammonium thiocyanate solution was added with constant stirring completion of preparation was checked. It was heated for 1-2 min and kept filtered through pre-standardized G4 sintered glass crucible. The precipitate was washed several times with wash liquid (200 cc distilled water + 10% 2cc ammonium thiocyanate + 10-15 drops of freshly prepared saturated sulphurous acid) & finally with 20% alcohol. It was heated in oven, cooled & weighed. The process was repeated till the constant weight was obtained.



observations & Calculations

Constant mass of empty Cu crucible = 22.4188g  
constant mass of Cu crucible with precipitate = 22.6837g

mass of ppt obtained = 22.6837 - 22.4188g = 0.2649g

121.63 (mole) of  $\text{CuSCN}$  contains 63.55 (mole) of copper ions  
 $\therefore$  1g of  $\text{CuSCN}$  would contain  
 $= \frac{63.55}{121.63}$  g of  $\text{Cu}^+$  ion = 0.5224g

xg of  $\text{CuSCN}$  would contain =  $\frac{63.55}{121.63} \times$  xg of  $\text{Cu}^+$  ion

2000 cc of solution would contain =  
 $= \frac{63.55}{121.63} \times$  xg of  $\text{Cu}^+$  ions  
 $= 0.1383$  g of  $\text{Cu}^+$  ions.

1000 cc of solution would contain  
 $= \frac{63.55}{121.63} \times x \times \frac{1000}{20}$  g of  $\text{Cu}^+$  ions  
 $= 0.1383 \times 50g = 6.915$  g

molar mass of  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O} = 436.18$ g  
63.55g of copper ions present in 436.18g of  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$

$\left[ \frac{63.55}{121.63} \times x \times \frac{1000}{20} \right]$  g of Cu ions are present in  
 $= \left[ \frac{436.18}{63.55} \times \frac{63.55}{121.63} \times x \times \frac{1000}{20} \right]$  g/L  
 $= 47.4616$  g/L

Result

Amount of copper present in the given solution was 47.4616 g/L

Precautions

- \* Excess of HCl should not be added
- \* Large excess of ammonium thiocyanate should not be used.



Object :-

In the given compound, identify one solid organic and one liquid functional group, their BP/MP and its suitable derivative.

Test for liquid compound (A)

- Primary test - (i) Physical state - Aqueous  
 (ii) Colour - Colourless  
 (iii) Odour - specific smell  
 (iv) solubility - Soluble in water

Observation Table

S.No.	Test	Observation	Result
1.	flame test - Take compound on spatula heat of burner	Burns without black flames	Compound is aliphatic.
2.	Litmus Test - Dip red and blue litmus paper in aq. sol <sup>n</sup>	Colour is unaffected	Compound is neutral.
3.	Element Test L.S + freshly prepare $FeSO_4 \cdot 7H_2O$ + conc <sup>n</sup> $H_2SO_4$	No colour or ppt	Nitrogen is absent.
4.	Functional group test i) Compound + ceric Sodium nitrate	Sol <sup>n</sup> turns red	Alcoholic group

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ii) Compound + Na metal	H <sub>2</sub> gas evolved	Alcoholic group is present
5. B.P of compound	≈ 78°C	May be C <sub>2</sub> H <sub>5</sub> OH
6. Confirmatory test		
i) compound + acidic acid + conc <sup>n</sup> H <sub>2</sub> SO <sub>4</sub>	fruity smell	Ethyl alcohol is present.
ii) 1 ml compound + 2 ml I <sub>2</sub> + drop by drop NaOH sol <sup>n</sup> + Δ	Brown colour of I <sub>2</sub> disappear yellow ppt form	C <sub>2</sub> H <sub>5</sub> OH is confirmed.

Test for solid compound (B) -

- Primary test -
- (i) Physical test - Solid
  - (ii) Colour - Purple
  - (iii) Odour - Phenolic
  - (iv) Solubility - Insoluble in H<sub>2</sub>O

Observation Table

S.No.	Test	Observation	Result
1.	Flame Test - Take compound on spatula & heat on burner	Burns with black flames	compound is aromatic.
2.	Litmus Test - Dip red and blue litmus paper in sol <sup>n</sup>	litmus paper turns red.	compound is acidic

Teacher's Signature .....



3. Element test - L.S + freshly sol <sup>n</sup> of FeSO <sub>4</sub> + conc <sup>n</sup> . H <sub>2</sub> SO <sub>4</sub>	No colour or ppt	Nitrogen is absent.
4. functional group present compound + neutral FeCl <sub>3</sub> sol <sup>n</sup>	Purple colour sol <sup>n</sup>	phenol group present.
5. M.P for compound	≈ 94°C	Compound may be naphthol.
6. Confirmatory test		
1) Compound + H <sub>2</sub> O + FeCl <sub>3</sub> sol <sup>n</sup>	white ppt	2-naphthol present.
2) 2 gm compound + 2 ml NaOH sol <sup>n</sup> + 2 drop CHCl <sub>3</sub> + Δ	Blue colour sol <sup>n</sup>	2-naphthol present.

#### -Formation of Derivative :-

Liquid Compound (A) :- In small beaker compound + 3,5-dinitrobenzoic acid chloride + Δ + NaHCO<sub>3</sub> then cool, it, we obtain 3,5-dinitrobenzoate derivative obtained.



Solid compound (B) -

In a test tube shake a-  
Sgtl compound + 1ml benzene sol<sup>n</sup> + 2ml  
picric acid, then stir it, yellow solid is of  
picrate.

Result :-

In the given organic compound liquid  
component (A) -  $C_6H_5OH$

B.P =  $78^\circ C$

derivative = 3,5 dinitrobenzoate

Solid component (B) - phenol

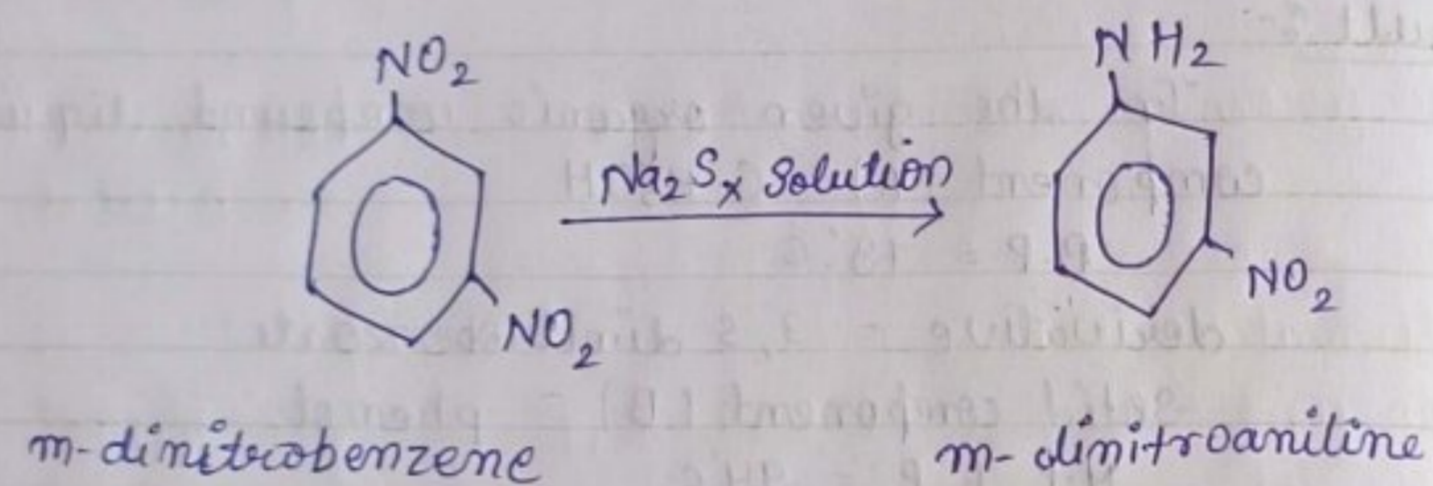
M.P B.P =  $94^\circ C$

derivative - picrate.

Teacher's Signature.....



Reaction -



Object:- Synthesis of meta-nitro aniline from meta dinitrobenzene.

Reagents required:-

- (i) m-dinitrobenzene = 5 gm
- (ii) Sodium Sulphide = 8 gm
- (iii) Sulphur powder = 3 gm
- (iv) ice (v) Jantav kaumla.

Equipment : Two beakers, dropping funnel.

Method -

Sodium sulphide and 8 gm of sulphur powder are dissolved in 30 ml water in a beaker. It is 3 gm of heated slowly till a solution of sulphur is obtained. This water sulphide solution is taken in a dropping funnel. In another beaker, 5 gm of m-dinitrobenzene and 50 ml of Jantav are heated on a sand heater and boiled. In this solution polysulphide sol<sup>n</sup> is added slowly through a dropping funnel by continuously stirring it rapidly. This mixture is boiled for about half an hour. Thereafter, the mixture is quickly filtered through a sieved funnel and the sieved mixture is cooled in ice. Yellow crystals of metanitroaniline are formed. After dissolving the crystals in sufficient quantity of hot water, a small animal is used to remove the crystals.



Add konla and boil the mixture ~~Result~~.

Result:-

m-nitroaniline is obtained by the above method.

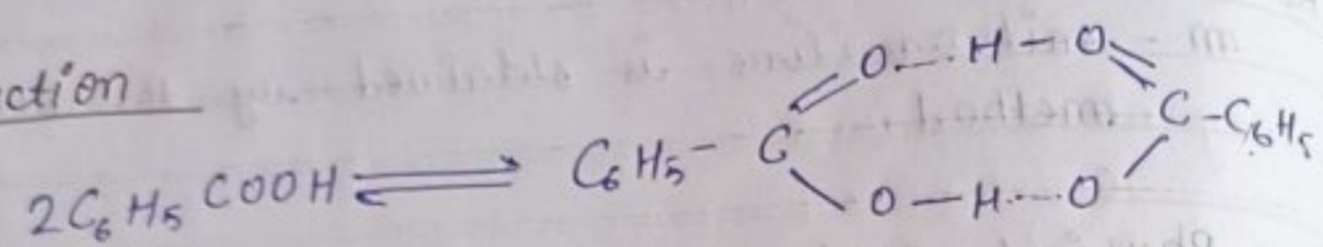
Physical state - Yellow coloured shiny crystals

Yield - 2.5 gm

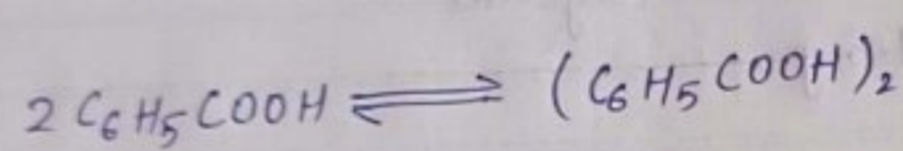
Melting point - 114°C



Reaction



or



$$K = \frac{\text{CH}_2\text{O}}{\sqrt{\text{C}_6\text{H}_6}}$$

PAGE NO. ....

Experiment - 8

DATE .....

Object:- To study the distribution of benzoic acid between benzene and water.

Equipment - Beaker, Burette stand, reagent bottle, separating funnel, Pipette.

Chemical - Benzoic acid, benzene, phenolphthaleine, NaOH, Distilled water

Theory:-

Benzoic acid is dissolved in two acid solvents benzene and water. The benzene and benzoic acid combination takes place in.

Hence the value of distribution coefficient -

$$K = \frac{\text{CH}_2\text{O}}{m\sqrt{\text{C}_6\text{H}_6}} = \frac{\text{CH}_2\text{O}}{(\text{C}_6\text{H}_6)^{1/m}}$$

where  $m$  = number of atoms which get combined, two atoms of benzene get combined in benzene.

So, it will have  $n = 2$

Teacher's Signature.....



Method:-

- (i) 10% benzene solution in four different bottles or flasks and add water and benzene to it and prepare the mixture as per the table.
- (ii) Each mixing bottle or flask must be corked. After applying it, shake it well by hand or by any 20-30 minutes. Keep the bottle aside for 5-10 min. So that equilibrium is established.
- (iii) In the bottle two layers (one of benzene and one of aqueous) get separated, these are separated by separating funnel.
- (iv) Take 10 ml of the benzene layer of bottle (1) in a conical flask and add saturated water to it. Add 0.1 or 0.01 g of phenolphthalein and get a consistent ml with 2-3 reading. Titrate 20-30. Similarly, other
- (v) Similarly, other bottles are also separated from 2, 3 and both the layers are titrated. It is written in the table of observation.

Observation table:-

S.No.	10% volume of benzoic acid (ml)	Volume of pure benzene (ml)	Volume of distilled water (ml)	Total (ml)
1.	10	40	50	100
2.	20	30	50	100
3.	30	20	50	100
4.	40	10	50	100

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Calculation :-

(i) The concentration of valeric acid in the vein layer

$$N_1 V_1 = N_2 V_2$$

$$N_1 = \frac{N_2 V_2}{V_1} = \frac{0.1 \times 22.1}{10} = 0.221 N$$

$$C_{C_6H_6} = \frac{0.1 \times 22.1 \times 122}{10}$$

$$= 26.96 \text{ moles/l}$$

$$\sqrt{C_{C_6H_6}} = 5.19$$

(ii) Concentration of valeric acid in the aqueous layer

$$N_1 V_1 = N_2 V_2$$

$$N_1 = \frac{0.01 \times 23}{10} = 0.023 N$$

$$C_{H_2O} = \frac{0.01 \times 23 \times 122}{10} = 2.806 \text{ mole/l}$$

(iii) Distribution coefficient  $k$  is

$$k = \frac{C_{H_2O}}{\sqrt{C_{C_6H_6}}} = \frac{2.806}{5.19} = 0.5406$$

$$k \cdot D = \frac{0.5406 + 0.320 + 0.211 + 0.153}{4}$$

$$= 0.306$$

PAGE NO. ....

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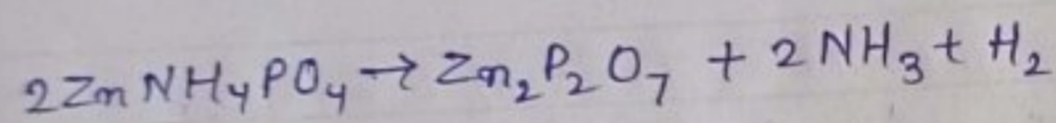
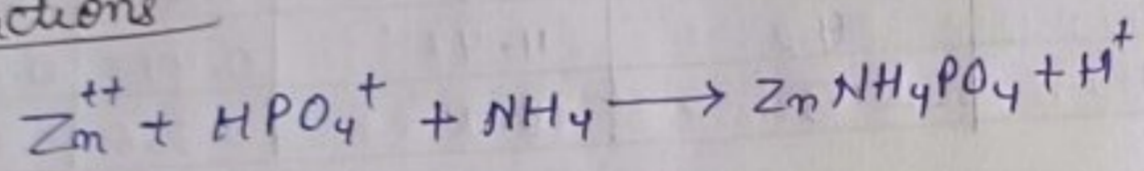
Bottle No.	10ml for benzene layer NaOH conc <sup>n</sup> volume of 0.01	For 10ml aqueous volume, 0.01 N NaOH is used	Benzoic acid in the benzene layer	The conc <sup>n</sup> of acid is greater than that of the aqueous layer	$k = \frac{C_{H_2O}}{\sqrt{C_{C_6H_6}}}$	$k = \frac{C_{H_2O}}{\sqrt{C_{C_6H_6}}}$
1.	23	22.1	26.93	2.806	0.100	0.5406
2.	17	18	10.3	1.03	0.1	0.320
3.	11	9.2	4.47	0.447	0.1	0.211
4.	4.7	3	5.71	0.366	0.06	0.153

Result :- Benzoic acid is found as a diatomic molecular in benzene. its the value of distribution coefficient is 2 and value is 0.306.

Teacher's Signature.....



Reactions



Object :- Determination of zinc as Zinc pyrophosphate.

Reagents :-

1. Zinc Carbonate solution.
2. 10% Diammonium Hydrogen Phosphate.
3. Dilute ammonia solution.

Reaction Observation :-

Weight of the empty crucible = 32.02 gm

Calculation :-

Weight of the empty crucible = 32.02 gm

Weight of the empty crucible = 32.23 gm

Weight of ppt ( $\text{Zn}_2\text{P}_2\text{O}_7$ ) = 32.23 - 32.02 = 0.21 gm

Molecular weight of  $\text{Zn}_2\text{P}_2\text{O}_7$  = 304 gm

Amount of Zn in  $\text{Zn}_2\text{P}_2\text{O}_7$  = 130 gm

(0.21) x 304 gm of  $\text{Zn}_2\text{P}_2\text{O}_7$  contains =  $(130/304) \times 0.21$  gm of zinc  
= 0.089 gm

25 ml of Zinc salt solution contains = 0.089 gm of zinc.

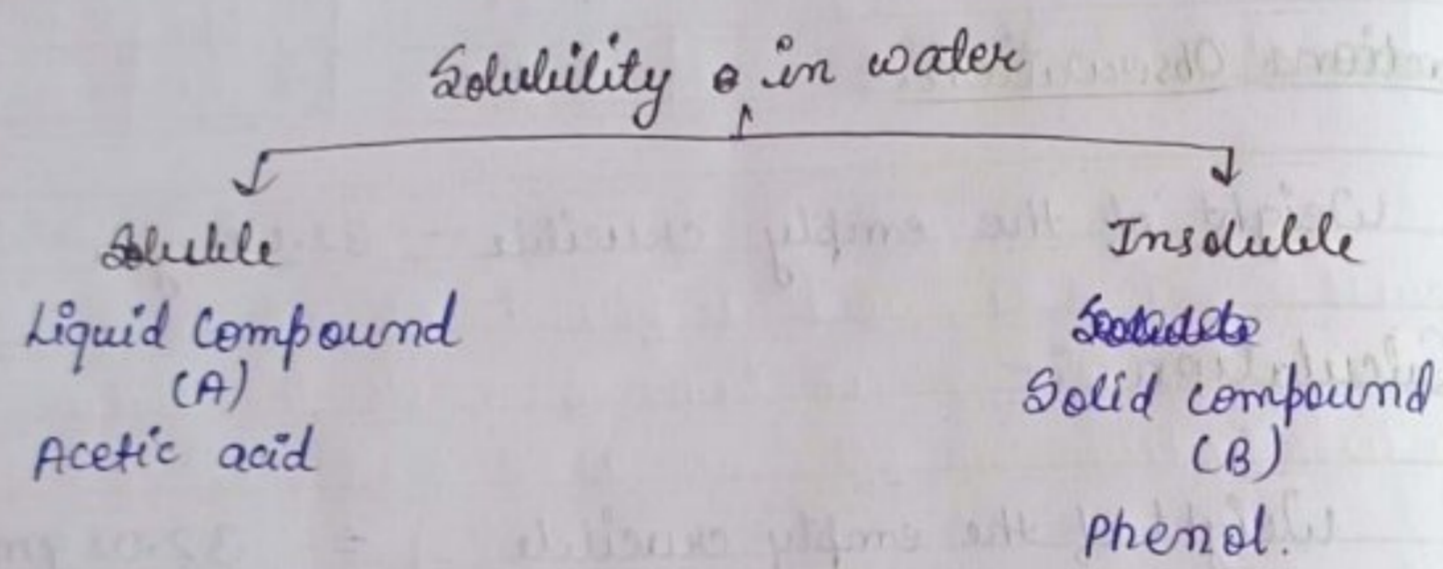
1000 ml of zinc salt solution contains =  $(0.089/25) \times 1000$   
gm of zinc

= 3.59 gm of zinc.

Result :-

The amount of zinc is found to be 3.59 gm.





**Object:-** To identify two functional groups (one solid & one liquid) in given organic compound. Also find M.P/B.P & suitable derivative.

**Test for liquid compound (A)**  
 Primary test  
 (i) Physical state - Aqueous  
 (ii) Colour - Colourless  
 (iii) Odour - Vinegar like  
 (iv) Solubility - soluble in water

Observation table

S.No.	Test	Observation	Result
1.	Flame test - Take compound on spatula & heat on burner	Burns with black flames	Compound is aromatic.
2.	Litmus Test - Dip blue litmus paper in sol <sup>n</sup>	Litmus paper turns red.	Compound is acidic.
3.	Element test - L.S + freshly prepared FeSO <sub>4</sub> Sol <sup>n</sup> + conc <sup>n</sup> H <sub>2</sub> SO <sub>4</sub>	No colour or ppt	Nitrogen absent.
4.	Functional group test Compound + C <sub>2</sub> H <sub>5</sub> OH + conc <sup>n</sup> H <sub>2</sub> SO <sub>4</sub> + Δ + cool + water	fruity smell	Carboxyl group present.

Teacher's Signature.....



5. B.P of compound	≈ 118°C	Compound is CH <sub>3</sub> COOH.
6. Confirmatory Test		
i) Compound neutral sol <sup>n</sup> neutral FeCl <sub>3</sub>	Sol <sup>n</sup> turns red	Acetic acid is confirmed.
ii) 1 ml acid + 10 drops of alcohol + 5 drops conc <sup>d</sup> H <sub>2</sub> SO <sub>4</sub> + Δ + cool.	fruity smell	CH <sub>3</sub> COOH is confirmed.

Test for solid compound (B)

- Primary test - (i) Physical State - Solid  
 (ii) Colour - Colourless  
 (iii) Odour - Phenolic  
 (iv) Solubility - Insoluble in H<sub>2</sub>O.

Observation table

S.No.	Test	Observation	Result
1.	Flame test - Take compound on spatula & heat on burner	Burns with black flame	Mixture is aromatic
2.	Litmus test - Dip red and blue litmus paper in sol <sup>n</sup>	litmus paper turns blue to red.	Compound is acidic.

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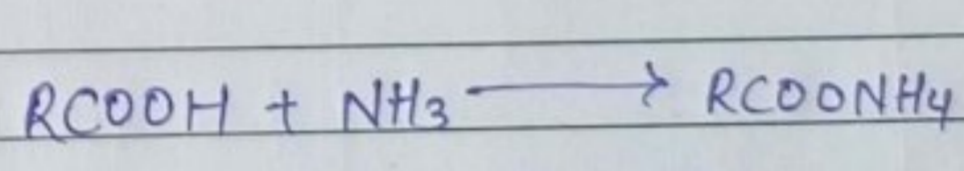


*[Faint handwritten notes on the left page, including a table with columns for 'Name of compound', 'Molecular weight', and 'Molar mass']*

3. Element test	L.S + freshly prepared $FeSO_4$ Sol <sup>n</sup> + conc <sup>n</sup> $H_2SO_4$	No colour or ppt	Nitrogen is absent.
4. functional group test	Compound + neutral $FeCl_3$	Purple colour Sol <sup>n</sup>	Phenol group present.
5. B.P. of compound		182°C	Phenol present
6. Confirmatory Test			
i)	Phenol + Thallic acid (2:1) + A + NaOH	solution turns pink	Phenol is confirmed.
ii)	Compound + solid Sodium nitrate + conc <sup>n</sup> $H_2SO_4$	green ppt which on dilution turns red	Phenol is confirmed.

formation of derivative

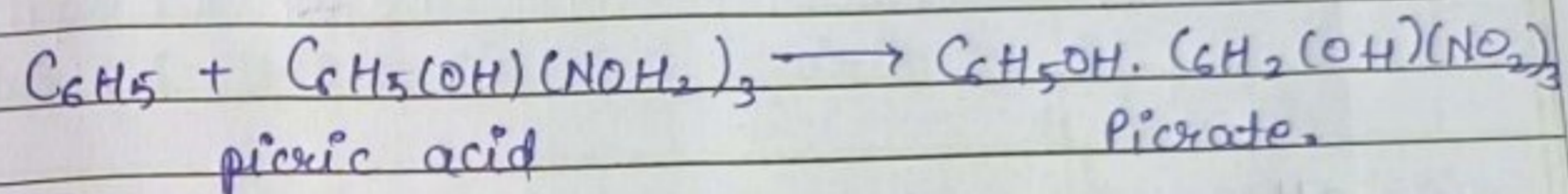
Liquid Compound (A) = In 1 ml acid add  $NH_4OH$  Sol<sup>n</sup> slowly, till smell of ammonia obtained. Then on burner heat the Sol<sup>n</sup>, obtained ~~Sol<sup>n</sup>~~ solid is ammonia.





Solid Compound (B) -

In a test tube take 0.5 gm compound & dissolve in 1 ml benzene then 2 ml of picric acid, solid obtained is picrate.

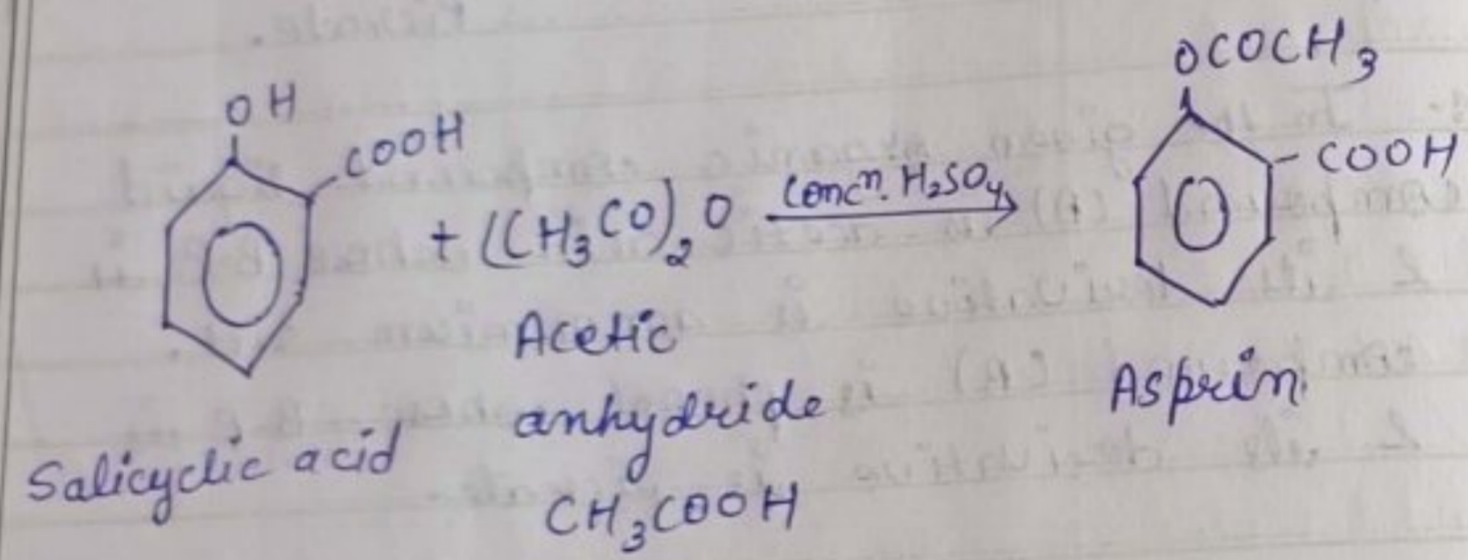


Result:- In the given organic compound liquid compound (A) is acetic acid where B.P is 118°C & its derivative is ammonium salt. Solid compound (B) is phenol where B.P is 182°C & its derivative is picrate.

Liquid Compound (A) = In benzene and 2 ml of 10% solution of picric acid, the solid obtained is picrate.

Solid Compound (B) = In benzene and 2 ml of 10% solution of picric acid, the solid obtained is picrate.





Aim:-

Synthesis aspirin by acetylation of salicylic acid and compared it with the ingredient of an aspirin tablet by TLC.

Step 1:- Preparation of aspirin:-

Aspirin is prepared in the laboratory by acetylation of salicylic acid with acetic anhydride in the presence of concentrated sulphuric acid or syrupy phosphoric acid.

The reaction involved is given as under -

Requirement:-

- Salicylic Acid - 5g
- Acetic anhydride - 5g
- Conc. H<sub>2</sub>SO<sub>4</sub> - 5g

Procedure:-

Take 5g of Salicylic acid in a small round bottomed flask. Add 6-7 ml of acetic anhydride and 5/6 drops of concentrated sulphuric acid to it. Shake well so that the reagents get mixed thoroughly. Fix an air condenser into the mouth of

Signature .....



flask and warm the contents to about  $50^{\circ}\text{C}$  in a water bath for 5-10 minutes with occasional shaking.

To the warm mixture add 10-15 ml of ice cold water to decompose excess of acetic anhydride.

Pour the contents of the flask with stirring into crushed ice contained in a beaker, stir well by means of a glass rod. White crystal of aspirin separate out. If a white solid does not appear (due to the presence of excess of acetic anhydride), scratch the walls of the beaker with a glass rod. Filter the crude product and wash it with cold water. Dry by pressing between folds of filter paper.

#### Purification:-

Recrystallise the crude products aspirin from equal volume of water and glacial acetic acid. Pure white product is obtained.

#### Result:-

yield - 5-8 g  
melting point -  $136-137^{\circ}\text{C}$  (with decomposition)



Object :-

To find out the distribution coefficient of iodine in water and organic chloride, carbon monoxide or chloroform, carbon disulphide etc.

Principle :- Iodine remains in the same intrinsic state in water and organic solvent carbon tetrachloride. Hence the value of distribution coefficient does not depend on the solubility of the solution. Hence the coefficient is calculated using the following formula

$$K = \frac{C_{\text{org}}}{C_{\text{H}_2\text{O}}}$$

where the solubility of iodine in organic solvent is  $C_{\text{org}}$  and in water is  $C_{\text{H}_2\text{O}}$ .

Equipment :- Burette, pipette, conical flask in four bottles, sterilising funnel, conical flask

Chemicals :-

- |  |  |
|--|--|
| (i) Iodine                                 | (ii) Potassium Iodide KI                                   |
| (iii) Starch                               | (iv) Sodium thiosulphate $\text{Na}_2\text{S}_2\text{O}_3$ |
| (v) organic solvent (Carbon tetrachloride) |  |



Observation Table :-

B.No.	Titration of aqueous layer			Titration of the ccl <sub>4</sub> layer		
	Initial Reading	Final Reading	% Hypo Solution value	B.No.	Initial Reading	Final Reading
I	2	2.9	1.8	I	1	4.5
II	2.2	3.7	1.5	II	1	4
III	3	9	1	III	3	10
IV	1.5	2.5	0.8	IV	2	8

Calculation -

→ Concentration of iodine in the aqueous layer

• Calculation bottle - I

$$N_1 V_1 = N_2 V_2$$

$$N_1 \times 5 = \frac{1}{100} \times V_2$$

$$N_1 = \frac{V_2}{500}$$

$$N_1 = \frac{1.8}{500} = 0.0036$$

$$N_2 = \frac{N}{100}$$

$$[V_2 = 18]$$

• Calculation bottle - II

$$N_1 V_1 = N_2 V_2$$

$$N_2 = \frac{1.5}{500} = 0.003$$

$$[V_2 = 1.5]$$

• Calculation bottle - III

$$N_3 = \frac{1}{500} = 0.002$$

$$[V_2 = 1]$$

• Calculation bottle - IV

$$N_4 = \frac{0.8}{500} = 0.0016$$

$$[V_2 = 0.8]$$

PAGE NO. ....

DATE .....

Required solution :- 10% dissolution of iodine in carbon tetrachloride meaning - 100 ml of total iodine in CCl<sub>4</sub>

(ii) Solution - 100 gm of 10 molar iodine is dissolved in 100 ml water.

(iii) N/20 Sodium tetrathionate (Hypo solution) N/10 for bisulfate sol<sup>n</sup>, wgt 3.1 gm in the bisulfate and prepare a 250 ml graduated sodium flask. Dissolve

(iv) N/200 sodium bisulfate (Hypo) sol<sup>n</sup> - above sol<sup>n</sup> is prepared by dilution method in 400.

Method :-

1. First of all, prepare the following four mixtures with 1% iodine sol<sup>n</sup> and make the total volume of all the mixture equal by mixing proper, four mixtures can be made as per the table.

S.No of Bottle or Plastic	Volume of iodine sol <sup>n</sup> (ml)	Volume of pure ccl <sub>4</sub> (ml)	Volume of distilled water (ml)	Total volume (ml)
1	50	0	100	150
2	40	10	100	150
3	30	20	100	150
4	20	30	100	150

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→ what is the concentration of iodine in carbon dioxide chloride Sol<sup>m</sup>?

$$N_1 V_1 = N_2 V_2 \quad \left[ V_2 = \frac{N_1}{20} \right]$$

$$N_1 = \frac{N_2 V_2}{20 \times V_1}$$

• Calculation Bottle-I

$$N_I = \frac{10.5}{20 \times 5} = 0.10 \quad \left[ \begin{matrix} V_1 = 5 \\ V_2 = 10.5 \end{matrix} \right]$$

• Calculation Bottle-II

$$N_{II} = \frac{8}{100} = 0.08 \quad \left[ \begin{matrix} V_1 = 5 \\ V_2 = 8 \end{matrix} \right]$$

• Calculation Bottle-III

$$N_{III} = \frac{7}{100} = 0.07 \quad \left[ V_2 = 7 \right]$$

• Calculation Bottle-IV

$$N_{IV} = \frac{6}{100} = 0.06 \quad \left[ V_2 = 6 \right]$$

calculation of the distribution coefficient

S.No.	Concentration of iodine in the aqueous layer	Iodine conc <sup>n</sup> in carbon tetrachloride layer	The distribution coefficient K $K = \frac{C_{CCl_4}}{C_{H_2O}}$
I	0.0036	0.10	$K_1 = 27.7$
II	0.003	0.08	$K_2 = 26.6$
III	0.002	0.07	$K_3 = 35$
IV	0.0016	0.06	$K_4 = 37.5$

$$K_D = \frac{K_1 + K_2 + K_3 + K_4}{4} = \frac{27.7 + 26.6 + 35 + 37.5}{4} \Rightarrow \frac{126.8}{4} \Rightarrow 31.7$$

(i) Each bottle or flask is corked and shaken well with a cork or any other instrument for about 20-30 min. After this, it kept 15-20 min. so that equilibrium can be established in it.

(ii) Two layers are separated in the bottle. These are separated by a separating layer of water.

(iv) Bottle number (1) obtained from CCl<sub>4</sub> layer and add 10 ml of 10% solution to it. Titrate this solution with  $\frac{N}{20}$  Na<sub>2</sub>C<sub>2</sub>O<sub>3</sub>. This is done with the help of an indicator. The blue colour disappears at the last point, repeat the reading corresponding action. Till 10 ml of the aqueous layer obtained from the bottle is taken in a conical flask, 10 ml KI solution is added to it. No Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution and titration is done using 1% starch as indicator from the solution. The final point is repeat till the blue colour disappears. also different from the layer of blue colour are the corresponding readings. Titrate other bottles in the same manner.

write the observation in the table.

Distribution Coefficient :-  $K = \frac{C_{CCl_4}}{C_{H_2O}}$

And find the value of K by using CCl<sub>4</sub> and CH<sub>2</sub>O

Result :- The distribution coefficient of iodine in the organic H<sub>2</sub>O layer CCl<sub>4</sub> and 31.7 g equivalent/liter.

Teacher's Signature.....



## Practical assignment work

ques-1 Estimate zinc as Zinc ammonium phosphate.

ques-2<sup>(a)</sup> Identification of organic compound (solid or liquid) through element detection (N and S) melting point, boiling point, functional group analysis with preparation of suitable derivative  
(A - acetic acid, B - Phenol)

(b) Synthesis of Aspirin from Salicylic acid

ques-3 To determine the heat of neutralisation of given acid and base.

OR

~~ques-4~~ To determine partition coefficient iodine between water and  $\text{CCl}_4$ ,  $\text{CHCl}_3$ ,  $\text{CS}_2$  at room temperature.

Teacher's Signature.....



Object :- Determination of zinc as Zinc <sup>diammonium</sup> phosphate.

Reagents :-

1. Zinc Carbonate solution.
2. 10% Diammonium Hydrogen Phosphate.
3. Dilute ammonia solution.

Observation :-

Weight of the empty crucible = 32.02 gm

Calculation :-

Weight of the empty crucible = 32.02 gm

Weight of the empty crucible = 32.23 gm

Weight of ppt ( $Zn_2P_2O_7$ ) =  $32.23 - 32.02 = 0.21$  gm

Molecular weight of  $Zn_2P_2O_7$  = 304 gm

Amount of Zn in  $Zn_2P_2O_7$  = 130 gm

$(0.21) \times 304$  gm of  $Zn_2P_2O_7$  contains =  $(130/304) \times 0.21$  gm of zinc  
= 0.089 gm

25 ml of Zinc salt solution contains = 0.089 gm of zinc.

1000 ml of zinc salt solution contains =  $(0.089/25) \times 1000$   
gm of zinc  
= 3.59 gm of zinc.

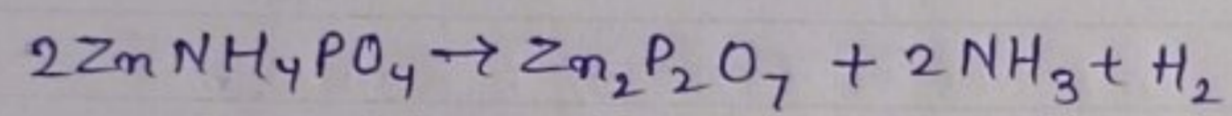
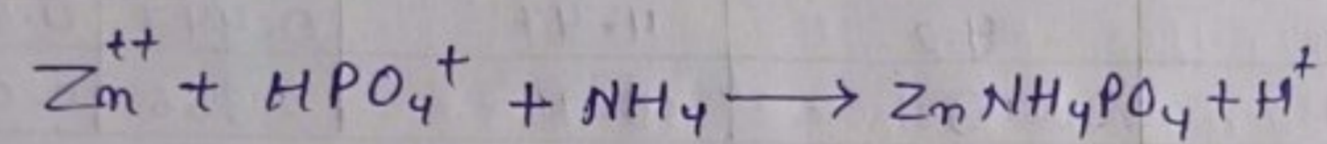
Result :-

The amount of zinc is found to be 3.59 gm.

Teacher's Signature .....



### Reactions



PAGE NO. ....

Experiment - 1

DATE .....

Object :- Determination of zinc as Zinc <sup>ammonium</sup> phosphate.

### Reagents :-

1. Zinc Carbonate solution.
2. 10% Diammonium Hydrogen Phosphate.
3. Dilute ammonia solution.

### Observation :-

Weight of the empty crucible = 32.02 gm

### Calculation :-

Weight of the empty crucible = 32.02 gm

Weight of the empty crucible = 32.23 gm

Weight of ppt ( $\text{Zn}_2\text{P}_2\text{O}_7$ ) = 32.23 - 32.02 = 0.21 gm

Molecular weight of  $\text{Zn}_2\text{P}_2\text{O}_7$  = 304 gm

Amount of Zn in  $\text{Zn}_2\text{P}_2\text{O}_7$  = 130 gm

(0.21) x 304 gm of  $\text{Zn}_2\text{P}_2\text{O}_7$  contains =  $(130/304) \times 0.21$  gm of zinc  
= 0.089 gm

25 ml of zinc salt solution contains = 0.089 gm of zinc.

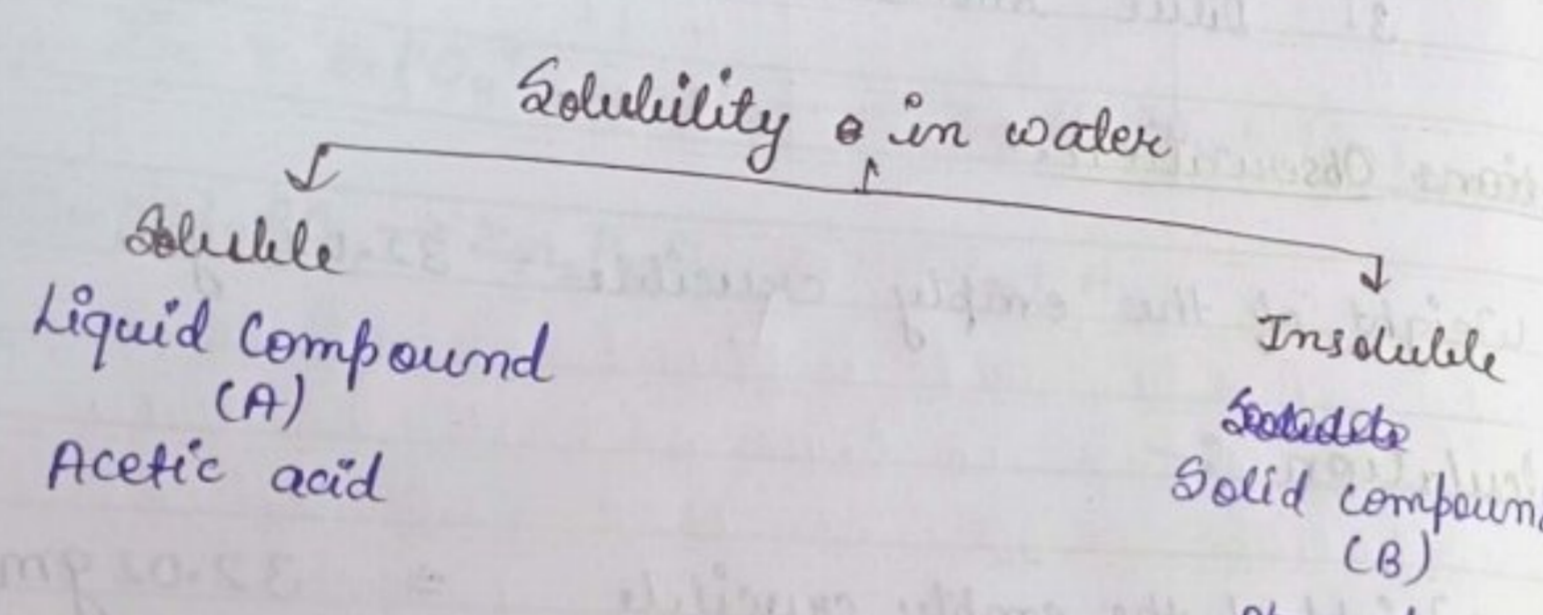
1000 ml of zinc salt solution contains =  $(0.089/25) \times 1000$   
gm of zinc  
= 3.59 gm of zinc.

### Result :-

The amount of zinc is found to be 3.59 gm.

Teacher's Signature .....





Object:- To identify two functional groups (one solid & one liquid) in given organic compound. Also find M.P/B.P & suitable derivative.

- Test for liquid compound (A)
- Primary test (i) Physical state - Aqueous
  - (ii) Colour - Colourless
  - (iii) Odour - Vinegar like
  - (iv) Solubility - Soluble in water

Observation table

S.No	Test	Observation	Result
1.	Flame test - Take compound on spatula & heat on burner	Burns with black flames	Compound is aromatic.
2.	Litmus Test - Dip blue litmus paper in sol <sup>n</sup>	Litmus paper turns red.	Compound is acidic.
3.	Element test - L-S + freshly prepared FeSO <sub>4</sub> Sol <sup>n</sup> + conc <sup>n</sup> H <sub>2</sub> SO <sub>4</sub>	No colour or ppt	Nitrogen absent.
4.	Functional group test Compound + C <sub>2</sub> H <sub>5</sub> OH + conc <sup>n</sup> H <sub>2</sub> SO <sub>4</sub> + Δ + cool + water	Fruity smell	Carboxyl group present.

Teacher's Signature.....



Handwritten notes on the left page, including an observation table with columns for Test, Observation, and Result. The text is mirrored and difficult to read due to bleed-through from the reverse side.

5. B.P. of compound	≈ 118°C	compound is CH <sub>3</sub> COOH.
6. Confirmatory Test		
i) Compound neutral sol <sup>n</sup>	Sol <sup>n</sup> turns red	Acetic acid is confirmed.
ii) 1 ml acid + 10 drops of alcohol + 0 drops conc <sup>n</sup> H <sub>2</sub> SO <sub>4</sub> + Δ + cool.	fruity smell	CH <sub>3</sub> COOH is confirmed.

Test for solid compound (B)  
 Primary test - (i) Physical state - Solid  
 (ii) Colour - Colourless  
 (iii) Odour - Phenolic  
 (iv) Solubility - Insoluble in H<sub>2</sub>O.

Observation table

S.No.	Test	Observation	Result
1.	Flame test - Take compound on spatula & heat on burner	Burns with black flames	Mixture is aromatic
2.	Litmus test - Dip red and blue litmus paper in sol <sup>n</sup>	Litmus paper turns blue to red.	Compound is acidic.

Teacher's Signature.....

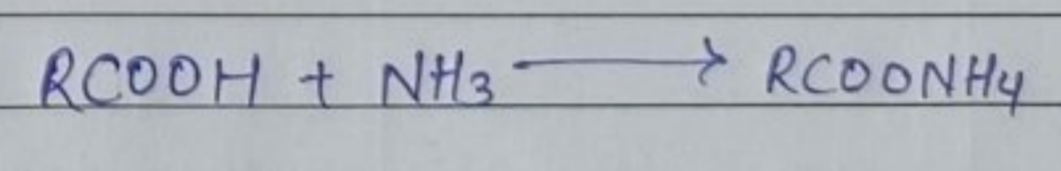


(A) Liquid compound (B) Solid compound  
 (i) Colour - colourless (ii) Solubility - soluble in water  
 (iii) Solubility - insoluble in water  
 (iv) Solubility - soluble in water

3. Element test	L.S + freshly prepared $FeSO_4$ sol <sup>n</sup> + conc <sup>n</sup> $H_2SO_4$	No colour or ppt	Nitrogen is absent.
4. functional group test	Compound + neutral $FeCl_3$	Purple colour sol <sup>n</sup>	Phenol group present.
5. B.P of compound		182°C	Phenol present
C. Confirmatory Test			
i)	Phenol + Thalic acid (2:1) + $\Delta$ + NaOH	solution turns pink	Phenol is confirmed.
ii)	Compound + solid Sodium nitrate + conc <sup>n</sup> $H_2SO_4$	green ppt which on dilution turns red	Phenol is confirmed.

formation of derivative

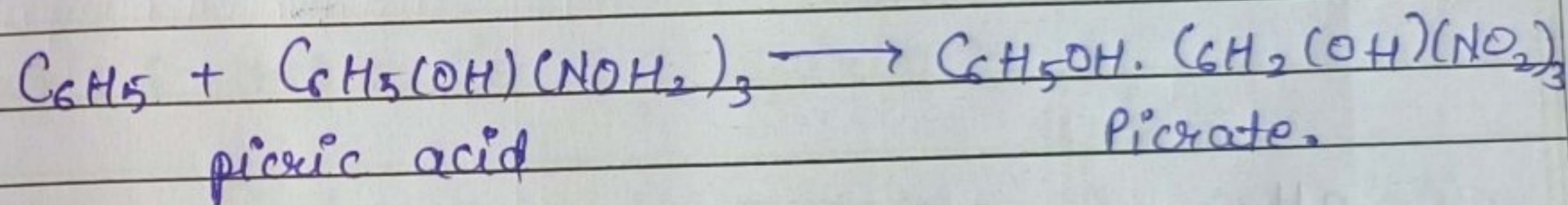
Liquid Compound (A) = In 1 ml acid add  $NH_4OH$  sol<sup>n</sup> slowly, till smell of ammonia obtained. Then on burner heat the sol<sup>n</sup>, obtained ~~sol<sup>n</sup>~~ solid is ammonia.





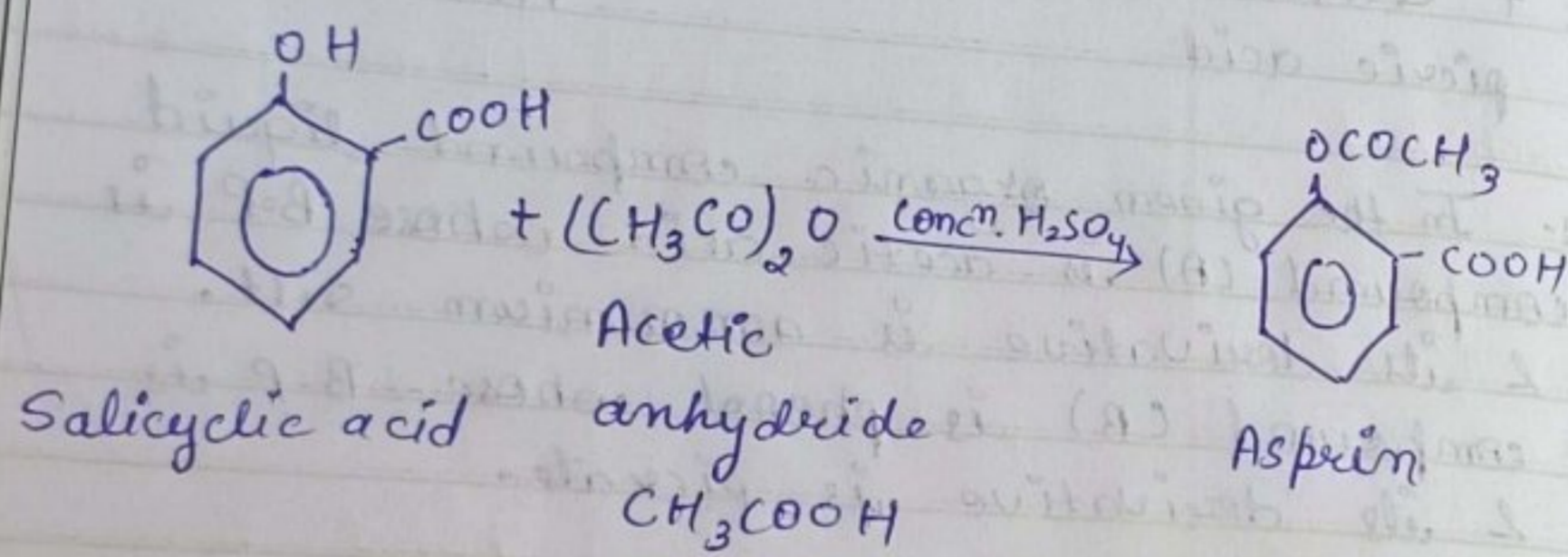
Solid Compound (B) -

In a test tube take 0.5 gm compound & dissolve in 1 ml benzene then 2 ml of picric acid, solid obtained is picrate.



Results:- In the given organic compound liquid compound (A) is acetic acid whose B.P is  $118^\circ\text{C}$  & its derivative is ammonium salt.  
Solid compound (B) is phenol whose B.P is  $182^\circ\text{C}$  & its derivative is picrate.





PAGE NO. ....

Experiment - 2(B)

DATE .....

Aim:-

Synthesis aspirin by acetylation of salicylic acid and compared it with the ingredient of an aspirin tablet by TLC.

Step 1:- Preparation of aspirin:-

Aspirin is prepared in the laboratory by acetylation of salicylic acid with acetic anhydride in the presence of concentrated sulphuric acid or syrupy phosphoric acid.

The reaction involved is given as under-

Requirement:-

Salicylic Acid - 5g  
Acetic anhydride - 5g  
Conc. H<sub>2</sub>SO<sub>4</sub> - 5g

Procedure:-

Take 5g of salicylic acid in a small round bottomed flask. Add 6-7 ml of acetic anhydride and 5-6 drops of concentrated sulphuric acid to it. Shake well so that the reagents get mixed thoroughly. Fix an air condenser into the mouth of



flask and warm the contents to about  $50^{\circ}\text{C}$  in a water bath for 5-10 minutes with occasional shaking.

To the warm mixture add 10-15 ml of ice cold water to decompose excess of acetic anhydride.

Pour the contents of the flask with stirring into crushed ice contained in a beaker, stir well by means of a glass rod. White crystal of aspirin separate out. If a white solid does not appear (due to the presence of excess of acetic anhydride), scratch the walls of the beaker with a glass rod. Filter the crude product and wash it with cold water. Dry by pressing between folds of filter paper.

#### Purification:-

Recrystallize the crude products aspirin from equal volume of water and glacial acetic acid. Pure white product is obtained.

#### Result:-

Yield - 5-8 g  
melting point -  $136-137^{\circ}\text{C}$  (with decomposition)



Object :-

To find out the distribution coefficient of iodine in water and organic chloride, carbon monoxide or chloroform, carbon disulphide etc.

Principle :- Iodine remains in the same intrinsic state in water and organic solvent carbon disulphide. Hence the value of distribution coefficient does not depend on the solubility of the solution. Hence the coefficient is calculated using the following formula

$$K = \frac{C_{\text{org}}}{C_{\text{H}_2\text{O}}}$$

where the solubility of iodine in organic solvent is  $C_{\text{org}}$  and in water is  $C_{\text{H}_2\text{O}}$ .

Equipment :- Burette, pipette, conical flask in four bottles, sterilising funnel, conical flask

Chemicals :-

- |   |  |
|---|--|
| (i) Iodine                              | (iii) Potassium Iodide KI                                  |
| (ii) Starch                             | (iv) Sodium thiosulphate $\text{Na}_2\text{S}_2\text{O}_3$ |
| (v) organic solvent (carbon disulphide) |  |



Observation Table :-

Titration of aqueous layer			Titration of the c.cly layer				
B.No.	Initial Reading	Final Reading	%100 Hypo Solution value	B.No.	Initial Reading	Final Reading	%10 Hypo Solution value
I	1	2.9	1.8	I	1	4.5	10.5
II	2.2	3.7	1.5	II	1	4	8
III	3	9	1	III	3	10	7
IV	1.5	2.5	0.8	IV	2	8	6

Calculation -

→ Concentration of iodine in the aqueous layer

• Calculation bottle - I

$$N_1 V_1 = N_2 V_2$$

$$N_1 \times 5 = \frac{1}{100} \times V_2$$

$$N_1 = \frac{V_2}{500}$$

$$N_1 = \frac{1.8}{500} = 0.0036$$

$$N_2 = \frac{N}{100}$$

$$[V_2 = 1.8]$$

• Calculation Bottle - II

$$N_1 V_1 = N_2 V_2$$

$$N_2 = \frac{1.5}{500} = 0.003$$

$$[V_2 = 1.5]$$

• Calculation Bottle - III

$$N_3 = \frac{1}{500} = 0.002$$

$$[V_2 = 1]$$

• Calculation Bottle - IV

$$N_4 = \frac{0.8}{500} = 0.0016$$

$$[V_2 = 0.8]$$



Required solution is 10% dissolution of Iodine  
in carbon tetrachloride  
meaning - 100 ml of total iodine in  $CCl_4$

(ii) Solution - 100 gm of 10 modulated is dissolved  
in 100 ml water.

(iii)  $N/20$  Sodium bisulfate (Hyo solution)  $N/20$  for  
bisulfate sol<sup>n</sup>, wgt 3.1 gm in the bisulfate and  
prepare a 250 ml graduated sodium flask. Dissolve.

(iv)  $\frac{N}{200}$  sodium bisulphate (hypo) sol<sup>n</sup> - above sol<sup>n</sup> is  
prepared by dilution method in 400.

method :-

1. first of all, prepare the following four mixture with  
1% iodine sol<sup>n</sup> and make the total volume of all  
the mixture equal by mixing puzzles, four mixture  
can be made as per the table.

S. No of Bottle or Plastic	Volume of iodine sol <sup>n</sup> (ml)	Volume of pure $CCl_4$ (ml)	Volume of distilled water (ml)	Total volume (ml)
			100	150
1	50	0	100	150
2	40	10	100	150
3	30	20	100	150
4	20	30	100	150

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→ what is the concentration of iodine in carbon dioxide chloride sol<sup>n</sup>?

$$N_1 V_1 = N_2 V_2 \quad \left[ V_2 = \frac{N_1}{20} \right]$$

$$N_1 = \frac{N_2 V_2}{20 \times V_1}$$

• Calculation Bottle - I

$$N_I = \frac{10.5}{20 \times 5} = 0.10 \quad \left[ \begin{array}{l} V_1 = 5 \\ V_2 = 10.5 \end{array} \right]$$

• Calculation Bottle - II

$$N_{II} = \frac{8}{100} = 0.08 \quad \left[ \begin{array}{l} V_1 = 5 \\ V_2 = 8 \end{array} \right]$$

• Calculation Bottle III

$$N_{III} = \frac{7}{100} = 0.07 \quad [V_2 = 7]$$

• Calculation Bottle IV -

$$N_{IV} = \frac{6}{100} = 0.06 \quad [V_2 = 6]$$

Calculation of the distribution coefficient

S. No.	Concentration of iodine in the aqueous layer	Iodine conc <sup>n</sup> in Carbon tetrachloride layer	The distribution coefficient $K$ $K = \frac{C_{CCl_4}}{C_{CH_2O}}$
I	0.0036	0.10	$K_1 = 27.7$
II	0.003	0.08	$K_2 = 26.6$
III	0.002	0.07	$K_3 = 35$
IV	0.0016	0.06	$K_4 = 37.5$

$$K_D = \frac{K_1 + K_2 + K_3 + K_4}{4} = \frac{27.7 + 26.6 + 35 + 37.5}{4} \Rightarrow \frac{126.8}{4}$$

$$\Rightarrow 31.7 \text{ g/l}$$

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(i) Each bottle or flask is corked and shaken well with a cork or any other instrument for about 20-30 mint. After this, it keep 15-20 mint. so that equilibrium can be established in it.

(ii) Two layers are separated in the bottle. These are separated by a separating layer of water.

(iv) Bottle number (1) obtained from  $CCl_4$  layer and add 10 ml of 10% solution to it. Titrate this solution with  $\frac{N}{20} Na_2C_2O_3$ . This is done with the help of an indicator. The blue colour disappears at the last point, repeat the reading corresponding action. Till 10 ml of the aqueous layer obtained from the bottle is taken in a conical flask, 10 ml KI solution is added to it.  $\frac{N}{100} Na_2S_2O_3$  solution and titration is done using starch as indicator from the solution. The final point is repeat till the blue colour disappears. also different from the layer of blue colour are the corresponding readings. Titrate other bottles in the same manner. write the observation in the table.

Distribution Coefficient :-  $K = \frac{C_{CCl_4}}{C_{H_2O}}$

And find the value of K by using  $CCl_4$  and  $C_{H_2O}$

Result :- The distribution coefficient of iodine in the organic  $H_2O$  layer  $CCl_4$  and 31.7 g equivalent/liter.

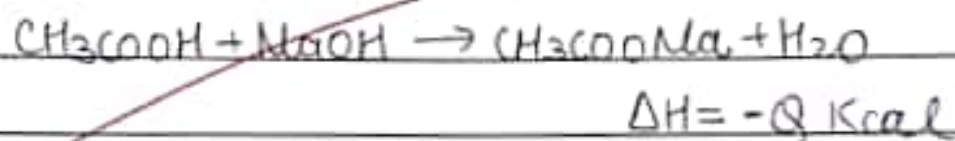


Object :-

To determine heat of neutralisation of weak acid ( $\text{CH}_3\text{COOH}$ ) & strong base ( $\text{NaOH}$ )

Principle :- The value of heat of neutralisation of weak acid & strong base is less than  $13.7 \text{ Kcal/mole}$ . As such the neutralisation takes place in two steps :-

- i) In the first step ionisation of weak acid takes place.
- ii) In the second step combination of  $\text{H}^+$  &  $\text{OH}^-$  gives  $\text{H}_2\text{O}$  i.e. some amount of heat is absorbed during ionisation, lowering the value of heat of neutralisation from  $13.7 \text{ Kcal}$ .



Heat of neutralisation,

$$= \frac{(V+w)(T_s - T_f) \times 1000}{N_1 V_1}$$

where,  $w =$  equivalent weight of water in flask

$V =$  volume of mixture of acid & base

$T_f =$  temperature of acid & base

$T_s =$  Final temperature of mixture

$V_1 =$  volume of acid

$N_1 =$  Normality of acid.



Calculation

(1) Equivalent weight of water in thermal flask

$$W = 100 \frac{(t_2 - t_3)}{t_3 - t_1} - 100$$

$$W = 100 \frac{74 - 48}{48 - 26} - 100$$

$$W = \frac{100 \times 26}{22} - 100$$

$$W = 118.18 - 100$$

$$W = 18.18 \text{ Calorie/degree}$$

2) Calculation of heat of neutralisation

$$= \frac{V + W (T_s - T_u) \times 1000}{\text{Volume of acid \& base} \times \text{Normality of acid and base}}$$

$$V = 100 + 100 = 200 \text{ ml}$$

$$T_u = 19.2^\circ \text{C} \quad T_s = 25^\circ \text{C}$$

$$W = 78.18 \text{ Calorie}$$

$$= \frac{(200 + 78.18)(25 - 19.2) \times 1000}{100 \times 1}$$

$$= \frac{218.18 \times 5.8 \times 1000}{100} = 12654.44 \text{ Kcal}$$

Heat of neutralisation = 12.65 Kcal.



Required apparatus - Thermos flask, two thermometers, beaker, burette

Required solution -

- i)  $\text{CH}_3\text{COOH}$  sol<sup>n</sup> of N normality.
- ii)  $\text{NaOH}$  sol<sup>n</sup> of N normality.
- iii) Phenolphthalein.

Observation :-

1) Equivalent weight of water in thermos flask.  
 Volume of cold water = 100 ml  
 Temperature of cold water -  $t_1 = 26^\circ\text{C}$   
 Temperature of warm water -  $t_2 = 74^\circ\text{C}$   
 Temperature of mixture -  $t_3 = 48^\circ\text{C}$

2) Heat of neutralisation

Volume of N- $\text{CH}_3\text{COOH}$  sol<sup>n</sup> = 100 ml  
 Volume of N- $\text{NaOH}$  sol<sup>n</sup> = 100 ml  
 Volume of N- $\text{CH}_3\text{COOH}$  sol<sup>n</sup> =  $20.1^\circ\text{C}$   
 Temperature of N- $\text{NaOH}$  sol<sup>n</sup> =  $18.3^\circ\text{C}$   
 Temperature of N- $\text{CH}_3\text{COOH}$  & N- $\text{NaOH}$  sol<sup>n</sup>  
 $T_4 = 20.418$   $T_5 = 25^\circ\text{C}$

Result :- Heat of neutralisation of weak acid ( $\text{CH}_3\text{COOH}$ ) and strong acid ( $\text{NaOH}$ ) is 12.65 K.calorie.

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 18/04/2021