Priyanka Sharma

M.Sc Final
(Chemistry)

Organic Chemistry

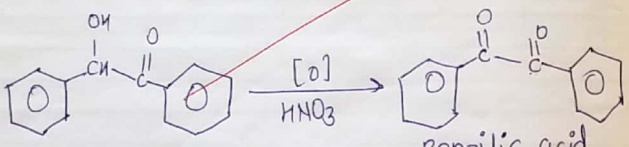
Index

Chemistry

Sr. No.	Name of the Experiment	Page No.	Date of Experiment	Date of Submission	Remarks
1.	To prepare benzilic acid from benzoin				
2.	To prepare benzanilide from benzophenone by the backmann-rearrangement.				
3.	To prepare 2-phenyl indole from phenyl hyrazine by Fisher-indole synthesis				
4.	To separate & identify the given mix. containing one liquid & two solid com-p. & pre. their suitable derivatives.				
5.	To separate & identify given mix. containing one liquid & two solids comp^{n} & prepare their suitable derivatives.				
6	To separate & identify the given mixture cont-aining one liquid & two solid comp^{n} prepare their suitable derivatives				

Chemical Rxn -

$2HNO_3 \longrightarrow H_2O + 2NO + 3[O]$



Benzilic acid

Date

Expt. No. _____ Page No. _____

Experiment = 1

Object:

To prepare benzilic acid from benzoin

Step I → Preparation of benzil from benzoin

Chemical Requirement -

(a) Benzoin → 8.5 gm
(b) Glacial acetic acid → 40ml
(c) Conc. HNO_3 → 20ml
(d) Ice

Procedure -

(i) In a round bottom flask place 8.5 gm of the benzoin, 40ml of glacial acetic acid & 20ml con. HNO_3.
(ii) Heat the mixture on a steam bath for two hours. Cool the flask in an ice bath add 150ml of water, mix throughly & allow the yellow ppt of benzile of settel.
(iii) Filter the product with Solution, waoh throughly with water & dry the product by pressing the crystal with a clean glass stopper.
(iv) Although again the benzil obtained is sufficiently pure for conversion to derivatives or to benzilic acid, it may be purified by crystalisation from methanol.

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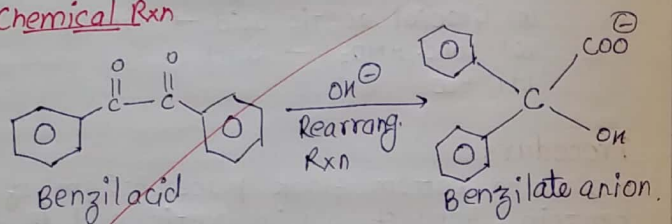
Chemical Rxn



Date

Expt. No. Page No.

Result -

Appearance → Yellow solid Crystal
Yield → 8 gm
M.P → 95°C.

Step - II → Preparation of benzilic acid from benzil.

Chemical Requirement -

(i) Benzil from I-step.
(ii) Potassium hydroxide - 5ml
(iii) Ethyl alcohol - 15 ml
(iv) Conc. HCl
(v) Ice.

Procedure -

(i) Dissolve 5gm of Potasium hydroxide pellets in 10ml of water in a 100ml flask.
(ii) Add 15ml of ethyl alcohol & mix well the resulting solution.
(iii) Add 5gm of pure benzil to the resulting solution.
(iv) Fit a reflux water consider to the flask & boil the contents on a water both for 10-15 minutes.
(v) Transfer the contents of the flask to a small beaker or a Procelain dish, cover with a watch glass all it to stand undisturbed for severed hours preferably overnight untill potassium benzolate is completely crystalised.

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Date

Expt. No. Page No.

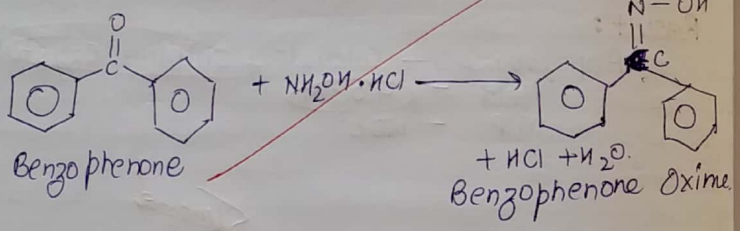
Filtered the product & wash it with ice cold ethanol.
Dissolve the potassium benzilate in about 50ml of water & then dissolve or add conc^n sol^n of HCl drop by drop till the precipitation of the free acid is complete.
Filter the product wash with water press dry & recrystalise from boiling water or benzene.

Result:

Appearance — Colourless shining crystal
Yield - 4.5 gm.
M.P - 150°C.

Signed
28/4/18

Chemical Rxn —



Date

Expt. No. _____ Page No. _____

Experiment - 2

Object:

To prepare benzanilide from benzophenone by the backmann - rearrangement.

Step - I : Preparation of benzophenone oxime from benzophenone

Chemical Requirement -

(i) Benzophenone - 5 gm
(ii) Hydroxylamine hydrochloride - 3 gm.
(iii) Rectified Sprit - 10ml
(iv) Sodium hydroxide (Pelets) - 5.6 gm
(v) Conc. HCl - 15 ml

Procedure -

(i) Place 5 gm of benzophenone, 3 gm of hydroxyl amine hydrochloride, 10 ml of rectified spirit & 2 ml of water in a flask.
(ii) Now add 5.6 gm of NaOH portion wise & with shaking & cool the flask. In a case than Rxn became to vigrous.
(iii) Boil the Sol^n under reflx condenser for 5 min.
(iv) Cool the Sol^n pour the contents to a Sol^n of 15ml of conc. HCl in 100 mgl of water in a 500 ml beaker.

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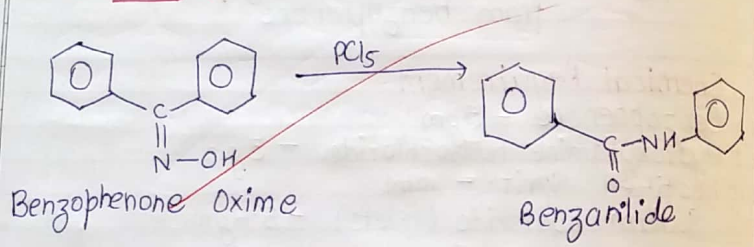
Chemical Rxn. :-



Benzophenone Oxime → Benzanilide

Date

Expt. No. Page No.

Result-

Appearance → Crystaline Solid
Yield – 5 gm
M.P – 142°c

Step-II : Preparation of benzalidine from Benzophenone oxime by backmann's Rearrangement.

Chemical properties-

(i) Benzophenone Oxime – 4 gm
(ii) Potasium penta chloride – 6 gm.
(iii) Anhydrous Ether – 40 ml

Procedure :

(i) Dissolve 4 gm of benzophenone oxime in 40 ml of anhydrous ether in a Conical flask.
(ii) Add 6 gm of powdered PCl5 & 6 ml of pure thionyl chloride to the Rxn mixture.
(iii) Shake well the contents & distil of the solvent on water bath.
(iv) Cool the residue, add 50 ml water & boil the resulting sol^n for Several min.

Result :

Appereance = White Crystalline Solid
Yield = 3 gm
M.P = 169°c.

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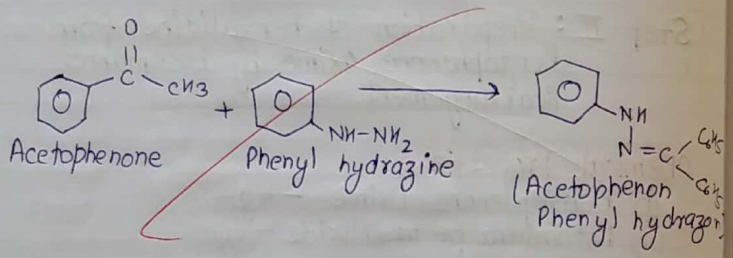
Chemical Rxn -



Acetophenone | Phenyl hydrazine | (Acetophenon Phenyl hydrazon)

Date

Expt. No. _____ Page No. _____

Experiment - 3

Object-

To prepare 2-Phenylindole from phenyl dhyzine by Fisher-Indole Synthesis

Step-I - Preparation of Phenyl hydrazin derivative of acetophenone.

Chemical required -

(i) Acetophenone - 10ml
(ii) Phenyl hydrazin - 12ml
(iii) Ethanal - 30ml
(iv) Acetic acid - 2-3ml

Procedure -

(i) In a round bottom flask. Place 10ml of acetoph-enone, 12ml Phenyl hydrazin, 30 ml ethanal containing 2-3 drops of glac. CH_3COOH.
(ii) The soln was warmed at 100°C for 15 min
(iii) After the poured the mix. into a beaker containing ice cool water.
(iv) The phenyl hydrazine derivative was filtered & washed with dil HCl

Result : Brown colour crystal form acetophenone phenyl hydrazone
Yield = 7.3 gm M.P = 106°C

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Date

Expt. No. Page No.

Step - II : To prepare 2-Phenyl indole from acetophenone Phenyl hydrazine.

Chemical requirement -

(i) Acetophenone phenyl hydrazine - 3gm
(ii) Polyphosphoric acid - 40ml
(iii) Water - 100ml.

Procedure -

Acetophenone phenyl hydrazon formed in step - I was heated with Polyphoric acid in boiling oil with 15min in round bottom flask.

Result :

Crystal of 2-Phenyl indole
Yield = 4.89 gm
M.P = 187°C

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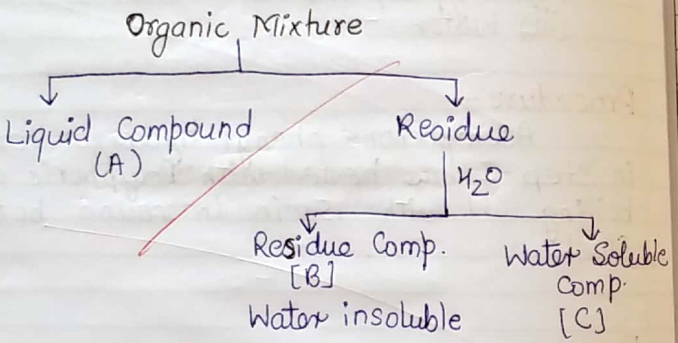

Date

Expt. No. Page No.

Experiment -4 (1)

Object:

To separate & identify the given mix. containing one liquid & two solid compound & prepare their suitable derivative.

Separation:

The given solid - liquid mixture are separated by distilation at 80°C liquid compound. A solid mix. of conc^n B & C was separated by H_2O.

[A] Identification of Compound "A":

Primary test:

(i) Physical State - liquid
(ii) Colour - Colourless
(iii) Odour - Specific order
(iv) Solubility - Unsoluble in water

Observation Table:

S.No.	Experiment	Observation	Inference
①	Ignition test: Compound take in spectula	Burn without smoky flem	Aliphatic
②	Litmus test: Litmus paper dip in compound sol^n	No effect on litmus paper	Compound is neutral.

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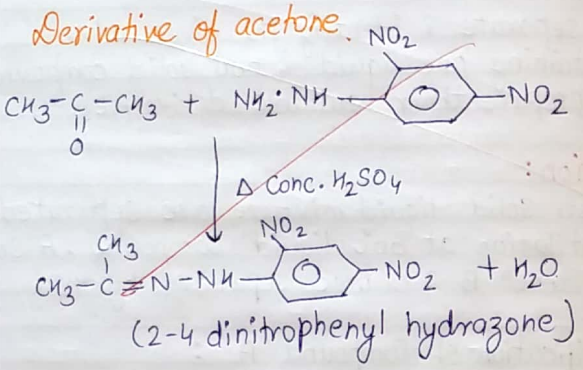
Derivative of acetone.



(2-4 dinitrophenyl hydrazone)

Date

Expt. No. Page No.

③	**Element test:**		
(a)	N-Teot: L·S + few drops (1-2) of NaOH Sol^n + freshly prepared $FeSO_4$ Sol^n + Δ + cool + add conc. H_2SO_4	Green/blue ppt Sol^n is not obtained.	N-absent
(b)	S-Teot: L·S + dil CH_3COOH + Few drops of $(CH_3COO)_2Pd$.	Black ppt. is not obtained	S-absent
(c)	X-teot: L·S + dil HNO_3 + $AgNO_3$ Sol^n	White & Yellow ppt is formed	X-halogen is present
④	**Functional gp. test** Comp^n + 2ml alcohol + (1-2) Pellets KOH + Δ + cool + dil HNO_3 + $AgNO_3$ Sol^n	White & Yellow ppt. is formed	X-halogen is present
5	**Determination of boiling point** B·P of Compound	61°C	Chloroform may be
6	**Compound test:**		
(i)	Comp.+ Crystal of resorcinal + aq. NaOH + Δ	Red colour is obtained	Chloroform Confirms.

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Date

Expt. No. Page No.

B] Identification of Solid Comp. (B):

Primary test:

(i) Physical State – Solid Crystaline
(ii) Colour – Yellow
(iii) Odour – Specific Colour
(iv) Solubility – Insoluble in water

Observation Table –

S.No.	Experiment	Observation	Inference
1	**Ignition test:** Comp^n take on spectula	Compound burns with smoky flame	Comp^n is aromatic
2	**Litmus test:** Comp^n Sol^n + Litmus paper	No effect on the litmus paper	Comp. is neutral
3	**Element test:**		
(a)	N-test: L.S + NaOH Sol^n + $FeSO_4$ Sol^n + Δ + Cool + Conc. H_2SO_4	Green & blue ppt. is not obtained	N is absent
(b)	S-test: L.S + dil CH_3COOH + $(CH_3COO)_2Pd$ Sol^n	Black ppt. is not obtained	"S" is absent
(c)	X-test: L.S + conc. HNO_3 + $AgNO_3$ Sol^n	White ppt is not obtained	X-absent

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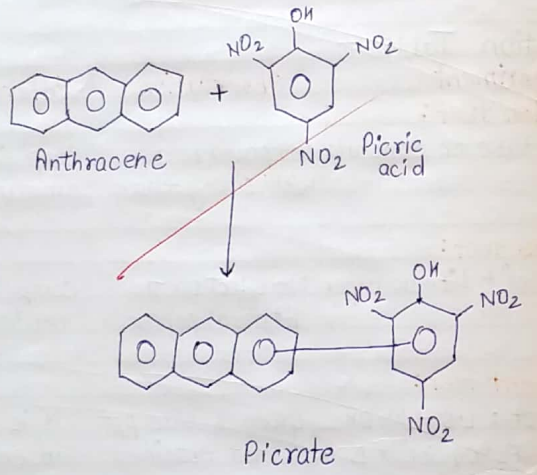
Derivative :⇒



Date

Expt. No. _____ Page No. _____

4 Functional gp. test: Substrate + benzen sol^n + dil sol^n of iodine + Shake.	Sol^n remains violet	Aromatic hydrocarbon confirms.
5. Melting pt. M·P of the compound	216°C	Anthracene may be
6. Compound test: Comp^n + Substrate sol^n of picric acid in the benzene + Δ.	Deep red colour solid is obtain	Anthracene () is confirmed.
7. Derivative – Comp^n + ethanol + saturated sol^n of Picric acid + Δ + cool + Filter	Red colour solid is obtained	Picrate
8. M·P of derivative M·P of Picrate	138°C.	Picrate conformed

(C) Identification of Solid Compound 'C' :-

Primary test:

(i) Physical state - Crystaline Solid
(ii) Colour - Brown.
(iii) Odour - Carbolic Odour
(iv) Solubility - Poorly Soluble in water

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Date

Expt. No. Page No.

(4) Functional gp. test-

(i) Comp^{n} + $FeCl_3$ Sol^{n} — Violet colour obtained — Phenol may be. +nt.

(ii) Libermann - nitrous test:

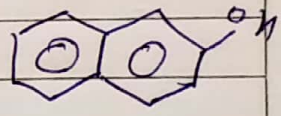
(a) Comp. + Na Nitrate + conc. H_2SO_4 — Green colour confirms.

(b) Dil it with H_2O

(c) Then add it NaOH. Sol^{n} — Red colour obtain. It convert into blue colour — Phenol confirms.

(5) Determination of M.P:

M.P of compound — 110°C



β-Napthol may be.

(6) Compound test:

(i) Comp. + NaOH Sol^{n} + $CuCl_3$ + △ — Blue colour is obtained — β-napthanol confirms.

Derivative-

Comp. + benzene + Picric acid + Share + crystal is brown warm benzene. — Yellow crystal obtained — Picrate

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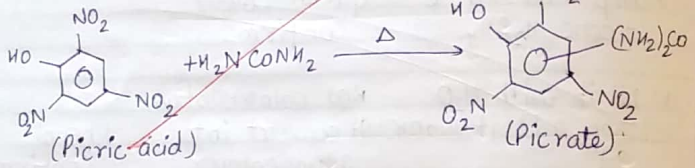

(Picric acid) (Picrate)

Date

Expt. No. Page No.

M·P of Picrate 156°C Picrate confirmed

Result:

In the given organic mixture the following comp. are present.

Compound - A:

Comp. (A) is Chloroform ($CHCl_3$)

B·P of Comp^n – 61°C

Derivative – 2,4 dinitro derivative

Compound - B:

Comp (B) is anthracene ()

M·P of Comp – 216°C

Derivative – Picrate

M·P of derivative – 138°C

Compound - C:

Comp. "C" is β-napthol ()

M·P of Comp. – 110°C

Derivative – Picrate

M·P of derivative – 156°C

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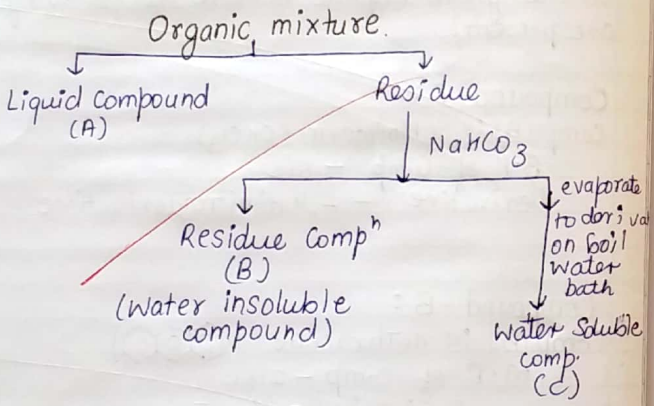

Date

Expt. No. _____ Page No. _____

Experiment = 5

Object:

To separate & identity the given mix. containing one liquid & two solid comp^n & prepare their suitable derivatives.

Separation:

The given solid-liquid mix. are separated by distillation of 80°c liq. compound A & Solid mix. of comp^n B & C was separated by H_2O.

Identification of Compound (A):

Primary test:

① Physical State - liquid
② Colour - Colourless
③ Odour - Specific odour
④ Solubility - Soluble in water

Observation Table:

S.No	Experiment	Observation	Inference
	Comp. take in the Spetulla	Comp.^n burn with smoky flame	Comp^n is aliphatic

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Date

Expt. No. _____ Page No. _____

Litmus Test:		
Litmus paper dip in comp^{n} sol^{n}	No effect on the litmus paper	Comp. is Neutral.
Element test:		
(a). N-test: L·S + NaOH + $FeSO_4$ + Δ + cool + conc. H_2SO_4	Green & blue ppt/ sol^{n} not obtained	N-absent
(b) S-test: L·S + dil CH_3COOH + $(CH_3COO)_2Pd$ sol^{n}	Black ppt is not obtained	S-absent
(c) X-test: L·S + dil HNO_3 + $AgNO_3$ sol^{n}	White ppt. is not obtained	X-absent
Functional gp. test:-		
Ceric ammonium mixture test:		
Comp. + Few drops ceric ammonium nitrate + Shake	Red colour obtained	Alcohol gp may be.
Determination of B·P:		
B·P of comp^{n}	78°C	Ethyl alcohol may be.

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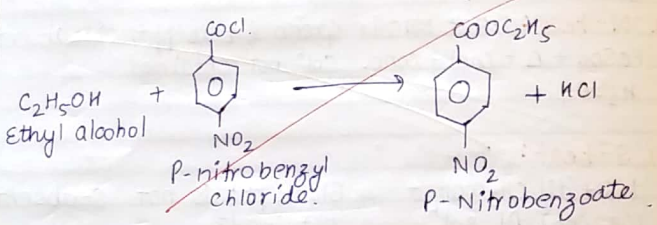

Expt. No. _____ Page No. _____

Compound test :		
Comp. + I_2 Sol^n + NaOH + Δ.	Yellow colour obtained	Ethyl alcohol confirms.
Derivatives –		
Alcohol + p-nitro benzoyl Chloride + Δ + $NaH CO_3$ Sol^n + Cool.	Solid p-nitro benzoate is obtained	p-nitrobe-nzoate.
M.P.		57°C

Identification of Solid Comp(B) –

Primary Test :

① Physical State – Crystaline solid
② Colour – White
③ Odour – specific odour
④ Solubility – Soluble in hot water

Observation Table –

S.No.	Experiment	Observation	Inference
①	**Ignition test :** Comp. take in the spechulla	Comp. burns with Smoky Flame	Comp. is aromatic

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Date

Expt. No. _____ Page No. _____

Litmus test:		
Comp. Sol^n + Litmus paper	No effect	Comp. is neutral
Element test:		
(a) **N-test:**		
L.S + NaOH + $FeSO_4$ Sol^n + Δ + Cool + Conc. H_2SO_4	Green / blue ppt. / Sol^n are obtain	N - present
(b) **S-test:**		
L.S + dil CH_3COOH + $(CH_3COO)_2Pd$ Sol^n	Black ppt is not obtained	S-absent
(c) **X-test:**		
L.S + Conc. HNO_3 + $AgNO_3$ Sol^n	White ppt. is not obtained	X-absent
Functional gp. Test:		
Comp. + H_2O + Shake + H_2O_2 Sol^n + Δ + Cool + $FeCl_3$ Sol^n	Red / blue colour obtain	Aromatic amide may be
M.P of Compound		
M.P of Compound	129°C	Benzamide may be
Compound Test:		
Comp. + dil NaOH + Δ + Cool + dil HCl	White ppt. is obtain	Benzamide is confirm

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Date

Expt. No. _____ Page No. _____

Derivations :

Comp. + aniline + Δ	White ppt. Obtain	
M.P of derivative	162°C	Benzaldylide confirms.

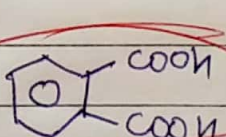
Result:

In the organic mix. following comp. are +nt

Compound A :

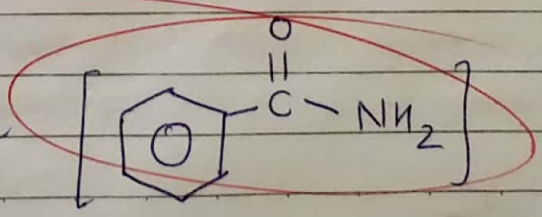
Comp. (A) is ethyl alcohol
B.P of Comp - 78°C
Derivatives - p-nitrobenzoate
M.P of Derivatives - 57°C

Compound B:

Comp. (B) is Pthalic acid 

M.P of the comp. - 195°C
Derivative - Amide
M.P of derivative - 220°C.

Compound C :

Comp. (C) is benzamide 

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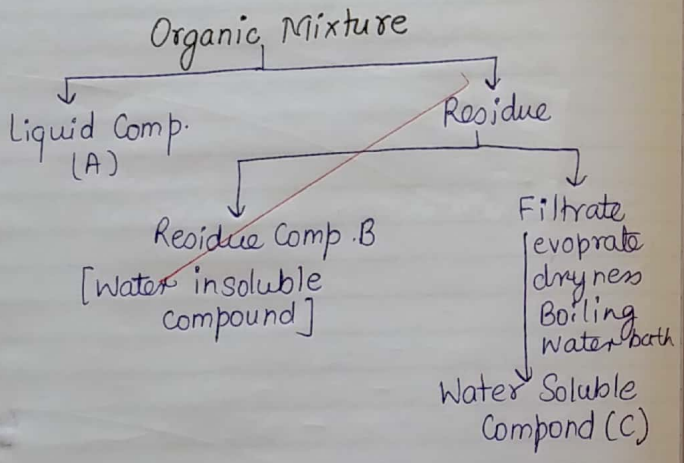
Date

Expt. No. _____ Page No. _____

M.P of Comp. – 129°C
Derivative – Benzaldehyde
M.P of derivative – 162°C.



Date

Expt. No. _____ Page No. _____

Experiment = 6

Object:

To separate & identify the given mixture containing one liquid & two solid comp^n prepare their suitable derivatives.

Separation:

The given solid - liquid mixture are separated by distilation at 80°c liquid comp [A] & solid mix. of comp. (B) & (C) was separated by H_2O.

Identification of Compound (A):

Primary test:

① Physical State - liquid
② Colour - Colourless
③ Odour - Specific odour
④ Solubility - Soluble in water

Observation table:

S.No.	Experiment	Observation	Inference
(1)	Ignition test: Comp. take in the spechulla	Comp. burns with non-smoky flame	Comp. is aliphatic

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Date

Expt. No. _____ Page No. _____

Litmus test:		
Litmus paper dip in comp. sol^n	No effect	Comp. is neutral
Element test:		
(a) N-test:		
L.S + Few drops of NaOH + freshly prep. $FeSO_4$ + Δ + Cool + Conc. H_2SO_4	Green / blue ppt. is not obtained	N-absent
(b) S-test:		
dil CH_3COOH + $(CH_3COO)_2Pd$ sol^n	Black Ppt. not obtained.	S-absent
(c) X-test:		
L.S + dil HNO_3 + $AgNO_3$ sol^n	White ppt. not obtained	~~X-absent Alcohol may be~~.
Functional gp. test:		
Cerric ammonium Nitrate		
Comp. + Ceric ammo-nium nitrate + Shake	Red Colour obtained	alcohol may be
Melting pt. of Compound:		
M.P of comp^n	65°C	methyl alcohol may be

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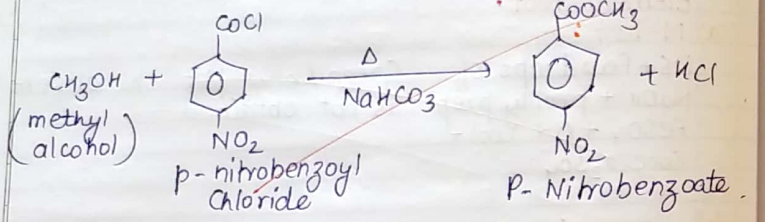

Date

Expt. No. Page No.

Compound test:

Comp. + Salicylic acid + conc. H_2SO_4 + Δ + H_2O	Smell of winger green oil	CH_3OH confirms

Derivatives:

Alcohol + p-nitrobenzo-yal Chloride + Δ + $NaHCO_3$ + Cool	Crystal of p-nitro-benzoate obtain	p-nitroben-zoate confirmed

Determination of M.P:

M.P of Comp.	96°C	p-nitrobenzoate confirmed

Identification of Solid Compound "B":-

Primary test:

1. Physical State - Crystaline solid
2. Colour - Specific odour
3. Odour - Colourless
4. Solubility - Soluble in water.

Observation table:

S.No.	Experiment	Observation	Inference
	Ignition test: Comp. taken in the spechulla	Burns with non-smoky flames.	Comp. is aliphatic

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Date

Expt. No. Page No.

Litmus test:		
Litmus paper dip in comp. Sol^n	Blue change in red	acidic in nature.
Element test:		
(a) **N-test:**		
L.S + few drops of + NaOH Sol^n + $FeSO_4$ Sol^n + Δ + Cool + conc. H_2SO_4	Blue/Green ppt. is not obtain	N-absent
(b) **S-test:**		
L.S + CH_3COOH + $(CH_3COO)_2Pd$ Sol^n	Black ppt. is not obtain	S-absent
(c) **X-test:**		
L.S + dil. HNO_3 + $AgNO_3$ Sol^n	White ppt. is not obtain	X-absent
(4) **Functional Gp. test:**		
Sodium bicarbonate test:		
Comp. + $NaHCO_3$ + Δ	CO_2 gas evolved	-COOH gp. may be.
(5) **M.P of Compound:**		
M.P of Comp.	185°C	Succinic acid may be.

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Expt. No. _____ Page No. _____

Compound test:		
Comp. + Resorcinal + conc. H_2SO_4 + Δ,	Red-brown colour obtain	
To mix + NaOH.	Organge-green colour obtain	Succinic acid confirmed
Derivatives:		
Comp. + $SOCl_2$ + Δ + cool + conc. NH_3 Sol^n + cool it once cube	White crystal obtain	Amide confirm
Determination of M.P:		
M.P of derivative comp.	260°C	Amide confirms

Identification of Solid Compound (C):

Primary test:

1. Physical State - Solid
2. Colour - Colourless
3. Odour - Specific odour
4. Solubility - Insoluble in water

Observation Table:

S.No.	Experiment	Observation	Inference
	Ignition test:		
	Comp. take in spectula	Comp. burns with smoky flames	Comp. is aromatic

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Date

Expt. No. _____ Page No. _____

Litmus Test:		
Litmus paper dip in comp Sol^n	No effect	Comp. is neutral in nature
Element test:		
(a) **N-test:**		
L.S + few drops of NaOH Sol^n + freshly prepared $FeSO_4$ Sol^n + Δ + cool + Conc. H_2SO_4	Green / blue ppt. Sol^n is obtained	N-present
(b) **S-test:**		
L.S + CH_3COOH + $(CH_3COO)_2$ Pd Sol^n	Black ppt is not obtain	S-absent
(c) **X-test:**		
L.S + dil. HNO_3 + $AgNO_3$ Sol^n	White ppt. is not obtain	X-absent
Functional gp. test: Mulliken & Baker test:		
Alcohol + $CaCl_2$ + Zinc dust powder + Δ + cool + tollen reagent	Brown/ black colour ppt. Obtain	Nitro gp. may be
M.P of compound		
M.P	90°C	m-dinitroben-zene may be

Teacher's Signature _____

Expt. No. _____ Date _____

Page No. _____

Compound test:

(i) Comp. + NaOH + Δ	Voilet colour obtain	m-dinitro-benzene confirms
(ii) Comp. + dil NaOH + Δ glucose.		

Determination of M.P:

M.P of derivative comp.	90°C	m-dinitro-benzene confirms

Result:

In the following compound (organic mix.) present –

Compound (A) –

Compound (A) is methanol (CH_3OH)

M.P – 65°C

Derivative – p-nitrobenzoate

M.P of derivative – 96°C

Compound (B) –

Compound (B) is succinic acid

M.P – 185°C

Derivative – Amide

M.P of derivative – 260°C

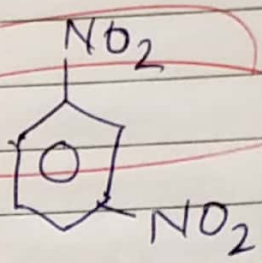
Signature

Date

Expt. No. Page No.

Compound - "C":

Comp. "C" is dinitrobenzene



M.P of Comp. - 90°C.

28/4/18