

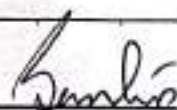
## ORGANIC QUALITATIVE ANALYSIS :-

### Lassaige's Test :-

A little amount of the sample was heated with a piece of sodium metal in a dry fusion tube at first slowly and then strongly for 2-3 min. till the lower part of the tube become red hot. then it was plunged into 5 ml of distilled water taken in a mortar and the broken mass was filtered with the filtrate the following tests were performed.

Experiment	Observations
<u>1. Test for S :-</u> To 1 ml of the filtrate 2-drops of sodium nitroprusside solution was added.	violet (Purple) coloration.
<u>2. Test for N :-</u> To 2ml of the filtrate 2ml of freshly prepared $\text{FeSO}_4$ sol <sup>n</sup> was heated to boil cooled and acidified with dil $\text{H}_2\text{SO}_4$ .	Prussian blue coloration (or precipitate)

Teacher's Signature



### Test for halogens (If N Present)

2 ml of Na extract was boiled with conc.  $\text{HNO}_3$  till the volume conc. to half then cooled and  $\text{AgNO}_3$  sol<sup>n</sup> was added.

liquor  $\text{NH}_4\text{OH}$  or  
A yellow ppt in solu-  
ble in hot liquor  
 $\text{NH}_4\text{OH}$  or a pale  
yellow ppt soluble  
in hot.

If the test 3 is positive the following test must be performed :-

2 ml of Na-extract was acidified with dil  $\text{H}_2\text{SO}_4$  and then few drops of  $\text{CCl}_4/\text{CHCl}_3$  was added. After that excess chlorine water was added and mixture was shaken.

Organic layer  
turned brown  
or violet.

### Test for non-nitrogenous functional groups :-

#### Experiment

Test for  $\text{COOH}$  group :-  
(a) To a saturated  $\text{NaHCO}_3$  sol<sup>n</sup> alcoholic sol<sup>n</sup> of the sample was added.

(b) To 0.5 gm of the sample 1 ml  $\text{MeOH}$  and few drops of conc.  $\text{H}_2\text{SO}_4$

#### Observation

Efferescence due to evolution of  $\text{CO}_2$ .

Sweet smell of ester.

Teacher's Signature

*Bunhu*

was containing  $\text{NaHCO}_3$   
taken in a beaker.

Test of Phenolic -OH group:-

(a) To an alcoholic solution  
of sample neutral  $\text{FeCl}_3$   
solution was added drop  
wise.

violet/blue/green  
red coloration.

(b) Back dye Test: Few  
drops of aniline was  
dissolved in dil HCl  
and cooled then freshly  
prepared  $\text{NaNO}_2$  sol<sup>n</sup>  
was carefully added to  
it. the resulting mixture  
was poured slowly to a  
cold alkaline solution  
of the sample.

red dye.

Test for carbonyl group:-

To an alcoholic solution of  
the sample in dry test tube  
excess 2,4 DNP solution  
was added and the contents  
of the tube was placed on  
a water bath for 5 minutes  
cooled and scratched with a  
glass rod.

Orange/yellow/  
red ppt

Test For Nitrogenous Functional groups :-Test for aromatic  $-NH_2$  group :-ExperimentsObservation

Diazo-Test : To a sample solution in dil HCl, freshly prepared  $NaNO_2$  solution was added and the mixture was poured into a cold alkaline  $\beta$ -naphthol solution was carefully added to it. The resulting mixture was poured slowly to a cold alkaline solution of the sample.

Immediate  
Scarlet on red  
Ppt.

Test for anilide ( $-CONHAr$ ) group:  
(If  $NH_2$  group is absent)

0.5 gm of the sample was dissolved in conc HCl and boiled for a few minutes. The mixture was cooled and dilute with water. Then the diazo-test was performed.

Immediate  
scarlet or red  
Ppt.

Test for aromatic  $NO_2$  group :-

(a) Mulliken Barker's test :-  
0.1 gm of the sample was mixed with a Pitch of

A black or grey  
Ppt on silver  
mirror.

Zn dust, 0.5 gm  $\text{NH}_4\text{Cl}$ , 2 ml of alc and a few drops of water. the mixture was boiled for 2-3 minute and then filtered hot in freshly prepared tallen's reagent.

( Tallen's Reagent: 1 ml  $\text{AgNO}_3$  sol<sup>n</sup> was taken in a test tube. few drops of dil  $\text{NaOH}$  was added to it. when a dark grey ppt appeared dilute  $\text{NH}_4\text{OH}$  was added till the dissolution of the ppt.

(b) When  $-\text{NH}_2$  or  $-\text{CONHAr}$  groups are absent the following test can be performed.

0.5 gm of the sample was mixed with 2 ml of conc  $\text{HCl}$  and few pieces of  $\text{Sn}$  or  $\text{Zn}$ . the mixture was boiled for 3-5 minutes and then cooled and the diazo-test was performed after dilution.

Immediate  
scarlet or red  
dye.

## Preparation of derivative for identification of a known organic compound.

### 1. Preparation of Benzoyl derivative (Amide gr)

Dissolve 1 gm of the substance in 5 ml of acetone in a 100 ml conical flask and add to it 2 ml of benzoyl chloride and 20 ml of 10% Sodium hydroxide sol<sup>n</sup> to the flask cork the flask and shake the contents vigorously until the odour of benzoyl chloride just disappears and a precipitate is formed. Filter the solid wash it with a little water and then crystallise from alcohol.

### 2. Preparation of Benzoyl derivative :-

Dissolve 500 mg of the substance in 5 ml of a 100 ml conical flask and add to it 2 ml of benzoyl chloride and 20 ml of 10% NaOH sol<sup>n</sup> to the flask cork the flask and shake the contents vigorously until the odour of benzoyl chloride just disappears and a ppt is formed.

Filter the solid wash it with a little water and then crystallise from alcohol. Dry and determine the m.p of the benzoyl derivative of the Phenolic compound.

### 3. Preparation of amides and anilides derivative :-

Carboxylic acids may be converted to the corresponding acids chlorides by the action of PCl<sub>5</sub>.

#### ☐ Preparation of acid-chloride :-

organic acid and 5 ml of thionyl chloride in a 100 ml round bottom flask fitted with a water containing a calcium chloride guard tube. The mixture gently refluxed for 30 min. distill off excess of thionyl chloride under reduced pressure.

#### ☐ Preparation of Amide :-

To one part of the acid chloride add 2 ml of liquor  $\text{NH}_3$  in cold condition little of a time when vigorous reaction has ceased, stir cool and filter.

crystallise the product from minimum value of cold water or aq. alcohol. Filter, dry and determine the M.P. of the amide derivative.

#### 4. Preparation of 2,4-Dinitrophenyl hydrazone derivative :-

Dissolve 0.2 gm of the sample in minimum vol<sup>m</sup> of alc. then add 2 ml of 2,4-DNP hydrazine reagent in it. If the product does not ppt. out immediately or on scratching the side of the test tube. heat the mixture on steam bath for 5 min. adding one drop of conc.  $\text{H}_2\text{SO}_4$  filter the orange or red ppt wash it with cold water and crystallise the product from ethanol.

### 5. Preparation of Nitro derivative :-

Take 500 mg of the sample in a test tube, add dropwise with constant shaking about 5 ml of nitrating mix. heat the mixture for 15 mint on a water bath cooled and then pour the reaction product with stirring to 25 ml of ice cold water, Filter the solid under suction pump, wash with cold water till acid free re crystallise from di alcohol.

### 6. Preparation of Bromo derivative :-

Take a sol<sup>n</sup> of 1 gm of the substance in 5ml glacial acetic acid in a 100ml conical flask immersed in an ice bath and add to it a sol<sup>n</sup> of 1 ml bromine in 5ml of glacial acetic acid wise and with constant shaking keep the resulting mixture of room temperature for a few mint. and pour into a beaker containing crushed ice. Add just sufficient saturation sol<sup>n</sup> of  $\text{NaHCO}_3$  to destroy the excess of bromine if the resulting mixture is appreciably coloured, filter the solid separated wash with water and crystallise it from aqueous ethanol.

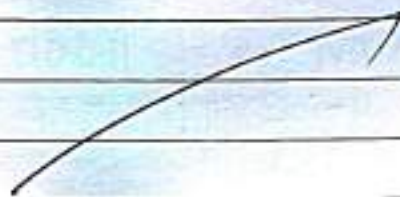
### 7. Hydrolysis of Amide :-

Amides are hydrolysed with sodium hydroxide to the corresponding acids and if it is solid serve as a derivative.

Heat a mixture of 1 gm of the substance 2 gm of sodium hydroxide and 5 ml of water



taken in a test tube for about 3 minute. Cool and acidity with dilute HCl filter the PPT, wash with water and crystallise from aqueous ethanol dry and note the M.P.



## Organic Qualitative Analysis for unknown organic compound :-

- ▲ Physical Character : 1. Colour - Brown  
2. Nature - crystalline  
3. Odour - No characteristic odour
- ▣ Melting Point :  $187-189^{\circ}\text{C}$

### Solubility :-

water	Dil HCl	Dil NaOH	sat $\text{NaHCO}_3$	Alc
-	-	+	+	+

### Conclusion :-

As the sample was soluble in water and sat  $\text{NaHCO}_3$ . It was strongly acidic in nature and may contains  $-\text{COOH}$  group.

### Lassaigne test :-

A little of the sample was heated with a piece of sodium metal in a dry fusion tube of first slowly and then strongly for 2-3 minutes till the lower part of the tube become red hot. then it was plunged into smel of distilled water taken in a mortar, and the broken mass was filtered with the filtrate the following tests were performed.

Experiment	Observation	Inferences
1 ml of the filtrate 2 drops of sodium nitroprusside sol <sup>n</sup> was added.	No violet colouration observed.	Sulphur absent.
2. Test for N: To 2 ml of the filtrate of freshly prepared $FeSO_4$ sol <sup>n</sup> was added heated to boil cooled and acidified with dil $H_2SO_4$ .	Prussian blue colouration observed.	Nitro-gen Present.
3. Test for halogens: 2 ml of Na extract was boiled with conc. $HNO_3$ till the volume come to half then cooled and $AgNO_3$ sol <sup>n</sup> was added.	NO appreciable change	Cl, Br, I absent.

Primary conclusion :-

Nitrogen is Present as special element  
then for both nitrogenous and non nitrogenous  
functional group. is performed.

Teacher's Signature

*Senthil*

## Test for nitrogenous functional group :-

Experiment	Observation	Interference
Test for Ar-NH <sub>2</sub> gr: Diazo-Test: To a sample sol <sup>n</sup> in dil HCl freshly prepared NaNO <sub>2</sub> sol <sup>n</sup> was added and the mixture was cooled to 0-5°C then the mixture was poured into cold alkaline β-naphthol sol <sup>n</sup> .	Red colouration ppt observed.	Definitely NH <sub>2</sub> gr Present.

## Test for amide (-CONHAr) gr :-

As-NH<sub>2</sub> group is present the test for amide (-CONHAr) group was not performed.

Experiment	Observation	Interference
Test for Ar-NH <sub>2</sub> group :- Mulliken Barker's Test :- 0.1 gm of the sample was mixed with a pinch of Zn dust 0.5 gm of NH <sub>4</sub> Cl, 2 ml of alcohol and a few drops of water. The mixture was boiled for 2-3 min. and then filt-hot in a freshly prepared Tollens's reagent.	No grey ppt or silver mirror found.	-NO <sub>2</sub> gr absent.

Experiment	Observation	Interence
Test for amide gr: To a little sample 2 half beads of NaOH and 1 ml of water were added and the mixture was heated strongly till evolution of fumes	NO smell of $\text{NH}_3$	$-\text{CONH}_2$ gr. absent

Test for non-nitrogenous function gr:

Experiment	Observation	Interence
Test for $-\text{COOH}$ gr: (a) To a saturated $\text{NaHCO}_3$ sol <sup>n</sup> a/c. sol <sup>n</sup> of the sample added.	Effervesence due to evolution of $\text{CO}_2$ .	$-\text{COOH}$ gr Present.
(b) To 0.5 gm of sample 1 ml MeOH and few drops of conc. $\text{H}_2\text{SO}_4$ was added then the sol <sup>n</sup> mixture was warmed was and poured into water containing <del>water</del> $\text{NaHCO}_3$ taken in a beaker.	Sweet smell at ester.	Definitely $-\text{COOH}$ gr Present.

### Test for Phenolic OH gr:

(a) To an alcoholic sol<sup>n</sup> of the sample neutral FeCl<sub>3</sub> sol<sup>n</sup> was added drops wise.

Red dry  
colouration  
observed  
due to pre-  
sence of amino  
acid

-OH gr  
absent

(b) Back dye test: Few drops of aniline was dissolved in dil HCl and cooled then freshly prepared NaNO<sub>2</sub> sol<sup>n</sup> was carefully added to it. The resulting mixture was poured slowly to a cold alkaline sol<sup>n</sup> of the sample.

No-red  
dye  
observed

-OH gr  
absent.

### Test for carbonyl gr:-

To an alcoholic sol<sup>n</sup> of the sample in dry test tube, excess 2,4 DNP sol<sup>n</sup> was added and the contents of the tube was placed on a water bath for 5 min cooled and scratched.

Experiment	Observation	Inference
with a glass rod as the above test in negative the test for aldehyde with Tollen's reagent was not performed.	NO orange yellow/red ppt	Carbonyl gr absent.
Test for unsaturation a) To a sample sol <sup>n</sup> in alcohol bromine water was added.	Decolorisation of bromine colour.	unsaturation absent.
b) To an acetone sol <sup>n</sup> of the sample dilute aqueous $KMnO_4$ sol <sup>n</sup> was added.	Decolouration of $KMnO_4$ colour	unsaturation absent.

Conclusion :-

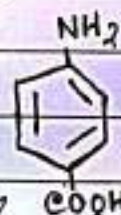
Special element Present

N

Functional gr Present

$-NH_2$ ,  $-COOH$

As the compound display M.P  $187-189^\circ$  and has  $-NH_2$  and  $-COOH$  functional group, so from lit survey it can be concluded that unknown compound is p-amino benzoic acid.



Teacher's Signature

Bunika

# Organic Qualitative Analysis for unknown compound :-

Physical character :-

1. Colour - white
2. Nature - crystalline
3. Odour - No characteristic odour.

Melting Point :  $113^{\circ} - 115^{\circ}$

Solubility :-

Water	Dil HCl	Dil NaOH	Sat $\text{NaHCO}_3$	Alc
-	-	-	-	+

Solubility classification :-

As the sample was not soluble in any solvent. It was neutral in nature and may contains  
 (a) Carbonyl (if N absent) (b) Nitro/amido (if N Present)

▣ Lassigne's Test : A little of sample was heated with a piece of sodium metal in dry fusion tube at first slowly and then strongly for 2-3 minutes till the lower part of the tube become red hot. then it was plunged into 5ml of distilled water taken in a mortar and the broken mass was filtered with the filtrate the following tests were performed.



Experiment	Observation	Interference
1. Test for S: 1 ml of the filtered 2 drops of Sodium nitroprusside sol <sup>n</sup> was added.	No violet colouration observed	Sulphur absent.
2. Test for N: To 2 ml of the filtrate 2 ml of freshly prepared FeSO <sub>4</sub> sol <sup>n</sup> was added heated to boil cooled and acidified with dil H <sub>2</sub> SO <sub>4</sub> .	Prussian blue colouration observed.	Nitrogen Present.
3. Test for halogens: 2 ml of Na extract was boiled with conc. HNO <sub>3</sub> then cooled and AgNO <sub>3</sub> sol <sup>n</sup> was added.	No appreciable change.	halogen absent.

Primary conclusion: As nitrogen is Present as special element then test for both nitrogenous and non nitrogen functional group is performed.

☐ Test for Nitrogenous function gr:

Experiment	Observation	Inference
<p>Test for Ar-NH<sub>2</sub> gr:-</p> <p>To a sample sol<sup>n</sup> in dil HCl freshly prepared NaNO<sub>2</sub> sol<sup>n</sup> was added and the mixture was cooled to 0-5°C. then the mixture was poured into a cold alkaline β-naphthol sol<sup>n</sup>.</p>	<p>No appreciable change.</p>	<p>Ar-NH<sub>2</sub> group absent.</p>
<p>Test for anilide (CONHAr) gr:</p> <p>(a) 0.5 gm of the sample was dissolved in conc HCl and boiled for a few minutes. the mixture was cooled and dilute with water then the diazo test performed.</p>	<p>Immediate red ppt observed.</p>	<p>CONHAr group Present.</p>
<p>(b) 0.5 gm of sample was dissolved in 75% H<sub>2</sub>SO<sub>4</sub> and boiled for a few minute. the mixture was cooled and dilute with water. then diazo test performed.</p>		

Experiment	Observation	Inference
<p>Test for <math>\text{NH}_2</math> gr:</p> <p>Mulliken Barker's Test:</p> <p>0.1 gm of the sample was mixed with a pinch of <math>\text{Zn}</math> dust 0.5 gm of <math>\text{NH}_4\text{Cl}</math> 2ml of alcohol and few drops of water.</p> <p>The mixture was boiled for 2-3 min, and <del>the</del> filtered hot in a freshly prepared Tollen's Reagent.</p>	<p>NO Black or grey ppt observed.</p>	<p>- <math>\text{NO}_2</math> group absent.</p>
<p>Test for amide gr:-</p> <p>To a little sample 2 half pieces of <math>\text{NaOH}</math> and 1ml of water were added and the mixture was heated strongly till evolution of fumes.</p>	<p>No smell of <math>\text{NH}_3</math></p>	<p>- <math>\text{CONH}_2</math> gr. absent</p>

Test for non-nitrogenous function group:-

Experiment	Observation	Inference
<p>Test for <math>-\text{COOH}</math> gr:</p> <p>To a sat. <math>\text{NaHCO}_3</math> sol<sup>n</sup> alcoholic sol<sup>n</sup> of the sample added.</p>	<p>No evolution of <math>\text{CO}_2</math></p>	<p>- <math>\text{COOH}</math> gr. absent.</p>

Expt. No. \_\_\_\_\_

Test for Phenolic OH gr.  
To an alcoholic sol<sup>n</sup> of the sample neutral FeCl<sub>3</sub> sol<sup>n</sup> was added dropwise.

No evolution of CO<sub>2</sub>.  
No appreciable change.

Phenolic -OH gr absent.

Test for carbonyl gr.  
To an alcoholic sol<sup>n</sup> of the sample in dry test tube excess 2,4-DNP sol<sup>n</sup> was added and placed in water bath.

No appreciable change.

Carbonyl group absent.

Test for unsaturation:

(1) To a sample sol<sup>n</sup> in alcohol bromine water was added.

No decoloration on of Bromine colour.

Unsaturation absent.

(2) To an acetone sol<sup>n</sup> of sample add dil. KMnO<sub>4</sub>.

No decoloration of KMnO<sub>4</sub> colour.

Unsaturation absent.

Conclusion: In the supplied sample.

Special element Present

N

Functional gr Present

-CONHAr

As the compound exhibits M.P 113-115°C and it contains -CONHAr functional group, so from lit survey it can be concluded that unknown compound is Acetanilide.



Teacher's Signature

*[Signature]*

Organic Qualitative Analysis for unknown compound:

- Physical Characters :-
- ① Texture - crystalline
  - ② Colour - light green
  - ③ odour - Pungent

Melting Point : ~~97~~ - 100°C

Solubility:

Water	Dil HCl	Dil NaOH	Sat NaHCO <sub>3</sub>	Alc
-	-	+	+	+

Solubility classification:

As the sample was soluble in NaOH the sample may be acids or nitrophenol.

Lassaigene's Test :- A little of the sample was heated with a piece of Sodium metal in a dry fussion Tube at first slowly and then strongly for 2-3 min. till the lower part of the tube become red hot. then it was plunged into 5ml of distilled water taken in a mortar and the broken mass was filtered with the filtrate the following tests were performed.

Experiment	Observation	Inference
1. Test for N: To 2ml of the filtrate 2ml of freshly prepared FeSO <sub>4</sub> sol <sup>n</sup> was added, heated to boil cooled and acidified with H <sub>2</sub> SO <sub>4</sub> .	Prussian blue colouration observed.	Nitrogen Present.
2. Test for S: To 1ml of the filtrate 2 drops of Na <sub>2</sub> [Fe(CN) <sub>5</sub> NO] sol <sup>n</sup> was added.	No violet colouration observed.	Sulphur absent.

Teacher's Signature \_\_\_\_\_

*Sanku*

Test for Halogen:

2ml of Na extract was boiled with conc.  $\text{HNO}_3$  till vol<sup>m</sup> come to half then cooled and  $\text{AgNO}_3$  sol<sup>n</sup> added.

No appreciable changed.

Cl, Br, I absent.

Primary conclusion: As ~~the~~ nitrogen is Present as special element then test for both nitrogenous and non-nitrogenous group is performed.

Test for nitrogenous functional group:

Experiment	Observation	Inference
<u>Test for Ar-NH<sub>2</sub> gr:</u> Diazo Test: To a sample sol <sup>n</sup> in dil HCl freshly prepared $\text{NaNO}_2$ sol <sup>n</sup> was added and the mixture was cooled to $0-5^\circ\text{C}$ then the mixture was poured into cool alkaline $\beta$ -naphthol sol <sup>n</sup> .	No appreciable changed.	Ar-NH <sub>2</sub> group absent.
<u>Test for anilide (-CONHAr):</u> 0.5 gm of the sample was dissolved in conc. HCl and boiled for a few minute the mixture was cooled and dilute with water then the diazo test performed.	No appreciable changed.	-CONHAr group absent

Test for aromatic-NO<sub>2</sub>

Group: Mulliken Box-Ker's Test :- 0.1 gm of the sample was mixed with a pinch of Zn dust, 0.5 gm of NH<sub>4</sub>Cl, 2 ml of alcohol and a few drops of water. The mixture was boiled for 2-3 min and then filtered hot in a freshly prepared Jollen's reagent.

A black ppt observed

Definitely -NO<sub>2</sub> group Present.

(b) 0.5 gm of the sample was mixed with 2ml of conc. HCl and few pieces of Sn or Zn the mixture was boiled for 3-5 minutes and then cooled and the diazo test was performed.

immediate scarlet ppt observed.

-NO<sub>2</sub> group Present.

Test for amino group :-

To a little sample 2 half beads of NaOH and 1 ml of water were added and the mixture was heated strongly till evolution of fumes.

No smell of NH<sub>3</sub>

-CONH<sub>2</sub> group absent

Teacher's Signature \_\_\_\_\_

*[Handwritten Signature]*

### Test for non-nitrogenous function group:

Experiment	Observation	Inference
<p>Test for -COOH gr: To an alcoholic sol<sup>n</sup> of the sample neutral FeCl<sub>3</sub> sol<sup>n</sup> was added.</p>		
<p>(b) To 0.5 gm of the sample 1 ml MeOH and few drops of conc. H<sub>2</sub>SO<sub>4</sub> was added then sol<sup>n</sup> mixture was warmed and poured into water containing NaHCO<sub>3</sub> taken in a beaker.</p>		
<p>Test for Phenolic-OH gr: To aq sol<sup>n</sup> of the sample neutral FeCl<sub>3</sub> sol<sup>n</sup> was added dropwise. Back dye test: few drops of aniline was dissolved in dil HCl and cooled then freshly prepared NaNO<sub>2</sub> sol<sup>n</sup> was carefully added to it. The resulting mixture was warmed and poured water containing NaHCO<sub>3</sub>.</p>	<p>winered colouration observed.</p>	<p>Phenolic-OH group Present.</p>
	<p>No red dye observed.</p>	<p>As NO<sub>2</sub> is strong electron withdrawing gr. It deactivates the ring so the test was -ve.</p>

Teacher's Signature Sanku



### Test for carbonyl gr:-

To an alcohol sol<sup>n</sup> of the sample in dry test tube excess 2,4 DNP sol<sup>n</sup> was added and the contents of the tube was placed on a water bath for 5 min cooled and scratched with a glass rod.

No appreciable change.

carbonyl group absent.

### Test for unsaturation:-

(a) To a sample sol<sup>n</sup> in alcohol bromine water was added.

No decolouration of Bromine colour.

unsaturation absent

(b) To an acetone sol<sup>n</sup> of the sample dilute aqueous  $KMnO_4$  sol<sup>n</sup> was added

No decolouration of  $KMnO_4$  colour.

unsaturation absent.

In the supplied sample

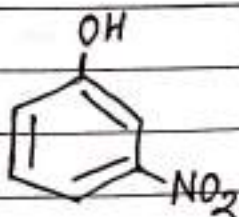
Special element Present

N

Functional group Present

$-NO_2$ , OH

From lit survey and from M.P study it can be concluded that unknown compound is m-nitrophenol.



Teacher's Signature

*Sanku*

14

# Organic qualitative analysis for unknown organic compound.

Physical characteristics:

- ① Texture - Amorphous
- ② colour - white
- ③ odour - NO characteristic odour.

M.P -  $237 - 239^{\circ}\text{C}$

## Solubility

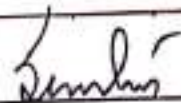
Water	Dil HCl	Dil NaOH	Sat $\text{NaHCO}_3$
-	-	+	+

Solubility classification: As the sample was insoluble in both water and dil HCl and soluble in dil NaOH and sat  $\text{NaHCO}_3$  sol<sup>n</sup>, it was strongly acidic in nature and may contain acids, nitrophenols.

✓ Lassaigne's Test :- A little of the sample was heated with a piece of Na-metal in a dry fusion tube at first slowly and then strongly for 2-3 min till the lower part of the tube become red hot then it was plunged into 5 ml distilled water taken in a mortar and the broken mass was filtered with the filtrate the following tests were performed.

Experiment	Observation	Interference
1. Test for S: 1 ml of filtrate + drops of Na-nitroprusside sol <sup>n</sup> added.	No violet colouration observed	Sulphur absent.

Teacher's Signature



Test for amide gr: - 0.5 gm of the sample was dissolved in conc. HCl and boiled cooled and dil with water. then diazo test was performed.

No immediate scarlet red ppt observed.

- CONHAr group absent.

Test for Ar-NO<sub>2</sub> gr: -

(i) 0.5 gm of the sample was mixed with 2ml of conc. HCl and few pieces of Zn or 2n. The mixture was boiled, cooled and the diazo-test was performed.

Immediate scarlet for red ppt.

- NO<sub>2</sub> gr Present

(ii) Mulliken Baskar's Test: 0.1 gm of the sample was mixed with a pinch of Zn dust 0.5 gm of NH<sub>4</sub>Cl, 2ml of alcohol and water the mixture was freshly prepared Tollen's reagent.

A black grey ppt observed

Definitely - NO<sub>2</sub> gr Present.

## Test for non-nitrogenous functional gr:-

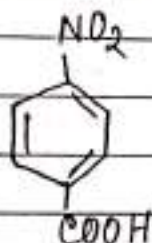
Experiment	Observation	Inference
Test for -COOH group:-		
(a) To a saturated $\text{NaHCO}_3$ sol <sup>n</sup> alcoholic sol <sup>n</sup> of the sample was added.	Effervescence due to $\text{CO}_2$ .	-COOH gr. Present.
(b) To 0.5 gm of the sample 1 ml MeOH and few drops of conc. $\text{H}_2\text{SO}_4$ was added then the sol <sup>n</sup> mixture was warmed and poured into water containing $\text{NaHCO}_3$ taken in Beaker.	Sweet smell of ester.	Definitely -COOH gr Present.
Test for Phenolic -OH gr:-		
To an alcoholic sol <sup>n</sup> of the sample neutral $\text{FeCl}_3$ sol <sup>n</sup> was added dropwise.	No violet <del>blue</del> colouration observed.	Phenolic -OH gr absent
Test for carbonyl gr:-		
To an alc sol <sup>n</sup> of the sample in dry test tube excess 2,4 DNP sol <sup>n</sup> was added and the contents of the tube was placed on a water bath for 5 min, cooled and scratched.	No appreciable change.	Carbonyl group absent.

Conclusion :- In this supplied sample

Special element Present  
Nitrogen

Functional gr. Present  
-NO<sub>2</sub> and -COOH  
group.

From lit survey of m.p studies it can be concluded that the unknown compound is p-nitrobenzoic acid.



Experiment	Observation	Inference
2. To 2 ml of the filtrate 2 ml of freshly prepared $\text{FeSO}_4$ sol <sup>n</sup> was added, heated, cooled and acidified with dil. $\text{H}_2\text{SO}_4$ .	Russian blue colouration observed.	N - Present.
3. Test for halogen:- 2 ml of Na-extract was boiled with conc. $\text{HNO}_3$ till the vol <sup>m</sup> came to half then cooled and $\text{AgNO}_3$ sol <sup>n</sup> was added.	NO appreciable change.	Cl, Br, I halogen absent.

Primary conclusion:-

The sample was amphoteric in nature and nitrogen (N) and sulphur (S) was present in it as a special element.

Detection of function groups:-

As nitrogen (N) is present as special element test for both nitrogenous and non-nitrogenous functional group were performed.

Test for nitrogenous functional groups:-

Experiment	Observation	Inference
<p>Test for Ar-NH<sub>2</sub> gr:</p> <p>To a simple sol<sup>n</sup> in di H<sub>2</sub>O, freshly prepared NaNO<sub>2</sub> sol<sup>n</sup> was added and the mixture was cooled to 0-5°C. Then the mixture was poured into a cold alkaline β-naphthol sol<sup>n</sup>.</p>	<p>Immediate scarlet or red ppt. observed.</p>	<p>Ar-NH<sub>2</sub> group Present.</p>
<p>Test for Ar-NO<sub>2</sub> gr:-</p> <p>0.1 gm of the sample was mixed with a pinch of Zn dust 0.5 gm of NH<sub>4</sub>Cl, 2 ml of alc. and a few drops of water. The mixture was boiled for 2-3 min and then filtered hot in a freshly prepared Tollen's reagent.</p>	<p>No black ppt or No grey ppt.</p>	<p>Ar-NO<sub>2</sub> group absent.</p>
<p>Test for Amide group:</p> <p>To a little sample 2 half beads of NaOH and 1 ml of water were added and the mixture was heated strongly till evolution.</p>	<p>No smell of NH<sub>3</sub></p>	<p>-CONH<sub>2</sub> group absent.</p>

Teacher's Signature: Sunil

### Test for -COOH group:

To a saturated  $\text{NaHCO}_3$  sol<sup>n</sup> alc. sol<sup>n</sup> of the sample was added.

effervescence  
due to evolution of  $\text{CO}_2$

-COOH gr  
may  
present

■ To 0.5 gm of the sample 1 ml  $\text{MeOH}$  and few drops of conc.  $\text{H}_2\text{SO}_4$  was added then the mixture was warmed and poured into water containing  $\text{NaHCO}_3$  taken in a beaker.

No smell  
of acetic

-COOH  
absent  
(The effervescence due to  $\text{SO}_3\text{H}$  gr)

### Test for Phenolic -OH gr:

To alcoholic sol<sup>n</sup> of the sample neutral  $\text{FeCl}_3$  sol<sup>n</sup> was added.

No appreciable  
change.

Phenolic  
-OH gr  
absent.

### Test for carbonyl gr:

To an alcoholic sol<sup>n</sup> of the sample in dry test tube, excess 2,4 DNP sol<sup>n</sup> was added and the contents of the tube was placed on a water bath for 5 min, cooled and scratched. Test for

No appreciable  
change  
observed.

carbonyl  
gr. absent.

Teacher's Signature: Bunlin



## Unsaturation.

① To a sample sol<sup>n</sup> of the sample in alc bromine water was added.

No decolouration of bromine colour.

unsaturation absent.

② To an acetone sol<sup>n</sup> of the sample dilute aqueous  $KMnO_4$  sol<sup>n</sup> was added.

No decolouration of  $KMnO_4$  colour

unsaturation absent.

## Conclusion:

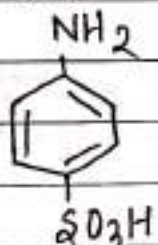
Special element Present

N.S Present

Functional gr. Present

Ar-NH<sub>2</sub> gr and SO<sub>3</sub>H gr Present.

□ From lit survey of m.p studies it can be concluded that unknown compound is sulfanilic acid



Teacher's Signature :

*[Signature]*

# Organic Qualitative Analysis of unknown organic compound.

## Physical characteristics :-

1. Texture - crystal
2. colour - Brown
3. odour - No characteristic odour
4. M.P -  $158^{\circ} - 160^{\circ}C$

## Solubility :-

Water	Dil HCl	Dil NaOH	sat $NaHCO_3$	Alc
+	-	-	-	+

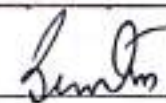
## Solubility classification :-

As the sample was soluble in water so the compound was Polar in water.

## Detection of special element :-

Lassaige's test :- A little of the sample was heated with a piece of Na-metal in a dry fussion tube at ~~at~~ first slowly then strongly until the lower part of the tube become red hot. then the tube is Plu-  
nged into distilled water taken in mortar. Broken mass was filtered with the ~~the~~ filtrate the following Test were Performed.

Experiment	Observation	Inference.
To 1ml of filtrate 2 drops of sodium nitroprusside $3cm^3$ added.	No violet colouration observed.	Sulphur absent.

Teacher's Signature : 

Test for N: To 2ml of the filtrate freshly prepared  $\text{FeSO}_4$  sol<sup>n</sup> was added and then heated to boil then cooled and acidified with dil  $\text{H}_2\text{SO}_4$ .

No Prussian blue colouration observed.

N - absent.

Test for Halogen: 2ml of Na-extract was acidified with dil  $\text{HNO}_3$  and cooled then  $\text{AgNO}_3$  sol<sup>n</sup> was added.

No white ppt observed.

Cl, Br, I absent.

Primary conclusion: Since the above three tests are -ve for the supplied sample, there is no special element in the supplied sample.

Since nitrogen was absent as special element test for nitrogenous functional gr ( $-\text{NH}_2$ ),  $-\text{CONH}_2$ ,  $-\text{CONHAr}$ ,  $\text{NO}_2$  were not performed.

Experiment	Observation	Interence
To a sat $\text{NaHCO}_3$ sol <sup>n</sup> alcoholic sol <sup>n</sup> of the sample was added to 0.5 gm of the sample 1 ml MeOH and few drops of conc. $\text{H}_2\text{SO}_4$ was	Effervescence due to evolution of $\text{CO}_2$ .	$-\text{COOH}$ gr. Present.

Teacher's Signature :

*Sanku*

added then the sol<sup>n</sup> mixture was warmed and Poured into water containing  $\text{NaHCO}_3$  taken in a beaker.

Sweet smell of ester.

-COOH gr definitely Present.

☑ Test for Phenolic -OH gr:-

① To an alcoholic sol<sup>n</sup> of the sample neutral  $\text{FeCl}_3$  added dropwise.

green colouration observed.

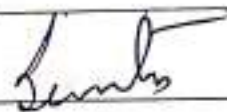
Phenolic -OH group Present.

② Few drops of aniline was dissolve in dil HCl and cooled then freshly prepared  $\text{NaNO}_2$  sol<sup>n</sup> was carefully added to it. the resulting mixture was Poured slowly to a cold alkaline sol<sup>n</sup> of the sample.

~~green~~  
red dye observed.

Definitely -OH gr Present

Teacher's Signature :

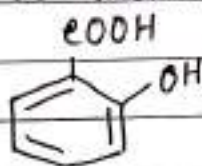


Conclusion :- In the supplied sample.

Special element Present  
No special element  
Present.

Functional group Present  
- COOH gr and Phenolic  
- OH gr. Present.

From lit survey of M.P studies we concluded the unknown sample is salicylic acid.



Teacher's Signature :

# Organic qualitative analysis of unknown organic compound :-

Physical character: 1. Texture - yellowish  
2. colour - Powdery  
3. odour - Bad odour.

Melting Point -  $187 - 189^{\circ}\text{C}$

Solubility :-

Water	Dil HCl	Dil NaOH	Sat $\text{NaHCO}_3$	Alc
+	-	-	-	+

Solubility classification :-

As the sample was soluble in water it is Polar in nature and they may contain low mass aliphatic acid Poly Phenols or salts.

Lassaigne's test :-

A little of the sample was heated with a piece of sodium metal in a dry fusion tube at first slowly and then strongly for 2-3 minutes till the lower part of the tube become red hot. Then it was plunged into 5 ml of distilled water taken in a mortar and the broken mass was filtered with the filtrate the following tests were performed.

Teacher's Signature :

*Sanjay*

Experiment	Observation	Inference
Test for S <sup>2-</sup> : To 1 ml of the filtrate 2-drops of Na-Nitro-Prusside sol <sup>n</sup> was added.	No violet colouration observed.	Sulphur absent.
Test for N <sup>3-</sup> : To 2 ml of the filtrate 2 ml of freshly Prepared FeSO <sub>4</sub> sol <sup>n</sup> was added, heated to boil cooled and acidified with dil H <sub>2</sub> SO <sub>4</sub>	Prussian blue colouration observed.	Nitrogen Present.
Test for Halogen: 2 ml of Na-extract was boiled with conc. HNO <sub>3</sub> till the vol <sup>m</sup> come to half then cooled and AgNO <sub>3</sub> sol <sup>n</sup> added.	No white or yellow ppt.	Halogen absent.

Conclusion :- As the sample Polar in nature and nitrogen was Present as an special element. As nitrogen was Present as special element then the test for both nitrogenous and non-nitrogenous functional group must be performed.

## Test for nitrogenous functional groups :-

Experiment	Observation	Inference
<p><b>Test for Ar-NH<sub>2</sub> gr:-</b>  <b>Diazo Test:</b> To a sample sol<sup>n</sup> in dil HCl freshly prepared NaNO<sub>2</sub> sol<sup>n</sup> was added and the mixture was cooled to 0-5°C. then the mixture was poured into a cold alkaline β-naphthol sol<sup>n</sup>.</p> <p>As the Ar-NH<sub>2</sub> gr is (+)ve then the following test for amide gr must be (-ve)</p> <p><b>Test for Ar-NO<sub>2</sub> gr:-</b>  <b>Mulliken Barker's Test:</b>            0.1 gm of the sample was mixed with a pinch of Zn-dust, 0.5 gm of NH<sub>4</sub>Cl, 2ml of alcohol and a few drops of water. the mixture was boiled for 2-3 min, and then filtered hot in a freshly prepared Tollen's reagent.</p>	<p>Immediate scarlet or red ppt.</p> <p>No appreciable change observed.</p>	<p>Ar-NH<sub>2</sub> group present.</p> <p>Ar-NO<sub>2</sub> group absent.</p>

Teacher's Signature : \_\_\_\_\_

Zunbi



Test for amide gr:-

To a little sample 2 half beads of NaOH and 1 ml of water were added and the mixture was heated strongly till evolution of fumes.

No Smell of NH<sub>3</sub>

- CONH<sub>2</sub> group absent.

Test for non-nitrogenous - functional gr:-

Experiment

Test for -COOH group:

(a) To a sat NaHCO<sub>3</sub> sol<sup>n</sup> alcoholic sol<sup>n</sup> of the sample was added.

(b) To 0.5 gm of the sample 1 ml MeOH and few drops of conc. H<sub>2</sub>SO<sub>4</sub> was added then the sol<sup>n</sup> mixture was warmed and poured into water containing NaHCO<sub>3</sub> taken in a beaker.

Observation

NO evolution of CO<sub>2</sub>

No sweet smell observed,

Inference.

-COOH group absent.

-COOH gr absent confirmed.

Experiment		
<p>Test for Phenolic-OH gr:-            (a) To an alc. sol<sup>n</sup> of the sample neutral FeCl<sub>3</sub> sol<sup>n</sup> was added dropwise.</p>	<p>red colouration observed</p>	<p>-OH gr may be Present.</p>
<p>(b) Back-dye Test:- few drops of aniline was dissolved in dil HCl and cooled then freshly prepared NaNO<sub>2</sub> sol<sup>n</sup> was carefully added to it. The resulting mixture was poured slowly to cold alkaline sol<sup>n</sup> of the sample.</p>	<p>red dye colouration observed.</p>	<p>definitely -OH gr Present.</p>
<p>Test for carbonyl gr:-            To an alc sol<sup>n</sup> of the sample in dry Test tube excess 2,4-DNP sol<sup>n</sup> was added and the contents of the tube was placed on a water bath for 5 mint. cooled and scratched with a glass rod.</p>	<p>No appreciable changed.</p>	<p>Carbonyl group absent.</p>

Teacher's Signature :

Sanku

As the carbonyl gr is absent the test for Tollen's reagent for aldehyde gr is not performed.

Test for unsaturation:

(a) To a sample sol<sup>n</sup> in alcohol bromine water was added.

No decolouration of bromine.

unsaturation absent.

(b) To an acetone sol<sup>n</sup> of the sample aq.  $KMnO_4$  sol<sup>n</sup> added.

No decolouration of  $KMnO_4$ .

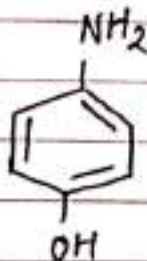
unsaturation absent.

Conclusion:

Special element Present  
N

Functional group Present  
 $-NH_2$ ,  $-OH$  grs

From lit survey of m.p studies it can be concluded the unknown sample is P-amino Phenol.



EXAMINED  
B.Sc. Pt. III/III  
Practical Exam  
Signature of External Examiner

13.12.19

Teacher's Signature:

*[Signature]*