1. (a) Physical characteristic:

(i) State of aggregation: solid
(ii) Texture: Crystalline or amorphous
(iii) Colour:
(iv) Odour:

(b) Melting Point:

(c) Systematic analysis for the detection of special elements:

Lassaigne's Test: A small amount of sample is fused with Na in a fusion tube. The fused mass is extracted with distilled water and then filtered. With the filtrate the following tests are performed.

Experiment	Observation	Inference
(i) Prussian blue test: Filtrate +freshly prepared $FeSO_4$ soln. + boiled, cooled + acidified with dil. H_2SO_4 soln.	Prussian blue or green ppt or colour.	N-present
(ii) Nitroprusside test: filtrate + NaOH soln. + sodium nitroprusside soln.	Violet or purple colour	S-present
(iii) Silver nitrate test: filtrate + conc. HNO_3, boiled, cooled, + $AgNO_3$ soln.	White ppt. which is soluble in dil. NH_4OH but reappears when acidified with HNO_3	Cl-present

Conclusion: From the above experiment, is concluded the supplied organic sample contain N, S, and Cl as special elements.

(d) Preliminary tests:

Experiment	Observation	Inference
(i) **Litmus test**: Blue litmus paper is treated with solution of sample in mixture of water and alcohol.	Blue litmus paper turns to red	Organic sample is acidic and may contain –COOH, Ar-OH or –SO_3H group.

(ii) **$NaHCO_3$ Test:** Saturated solution of sodiumbicarbonate under heating, cool + pinch of sample.	Effervescence of CO_2 gas.	-COOH, $-SO_3H$ group may be present
(iii) **Permanganate test:** Dil. KMnO4 solution is added to alcoholic sample solution drop by drop.	Pink colour of $KMnO_4$ is disappeared	Unsaturation (>C=C<) or easily oxidisable group may be present.
(iv) **Soda-lime test:** organic sample is heated with soda-lime.	(a)Characteristic smell of NH_3 (b) Characteristic smell of burnt sugar. (c) Characteristic smell of aniline.	(a) $-CONH_2$ may be present (b) Carbohydrate may be present (c) –CONHPh may be present.
(v) Ignition test : sample is burned	Yellow sooty flame	Sample may be aromatic

(e) Solubility test:

Cold H_2O	Hot H_2O	dil. HCl	dil. $NaHCO_3$	dil. NaOH	Conc. H_2SO_4

Conclusion:

- From solubility and melting point inference can be drawn for the given organic sample.
- If the sample is soluble in dil. NaOH. The compound may contain the functional group –COOH or phenolic –OH or –COOH and phenolic –OH both.

- As the sample effervesces with $NaHCO_3$ solution i.e the compound is soluble in $NaHCO_3$ solution then the sample may contains –COOH group with or without other functional groups.
- As the sample is soluble in only dil. HCl then the compound is amine with or without other functional groups.
- As sample is soluble in both dil. HCl and dil. NaOH the compound may be aminophenol or amino acid.
- As the sample is not soluble in dil acid or alkali but only soluble in concentrated H_2SO_4. The compound may contain $–NO_2$ and or >C=O group

(f) Systematic analysis of the functional groups:

1. Detection of Nitrogenous functional groups

Experiment	Observation	Inference
(i) Dye test: **Solution-I:** Sample is dissolved in dil. HCl and cooled in an ice-bath. **Solution-II:** Dil. $NaNO_2$ soln. is cooled in an ice-bath **Solution-III:** Alkaline β-napthol soln. is cooled in an ice-bath. The mixture of **solution-I** & **II** is cooled in an ice-bath .After some time alkaline β-napthol soln. is added to the mixture of **solution-I** & **II**.	Red or orange –red dye.	Ar-NH_2 group confirmed.
(ii) Mulliken and Barker's test: Alcoholic soln. of sample + solid NH_4Cl + Zn-dust boiled for few minutes, cooled allowed to stand for 5 minutes, and filtered. Filtrate + Tollen's reagent, warmed in a water bath.	Silver mirror or black or grey ppt.	Ar-NO_2 group confirmed.

(iii) **Hydrolysis test for amide group**: sample + Conc. NaOH solution, heated.	Characteristic smell of NH_3 which turns mercurous nitrate paper black.	-$CONH_2$ group confirmed.
(iv) **(a) Test for anilido (-CONHAr) group**: 0.05 g of sample is hydrolysed by 3 ml conc. HCl and 2 ml water. The hydrolysed product is diluted with 5 ml water, cooled then diazotized by cold dil. $NaNO_2$ solution and finally the mixture is added to cold alkaline solution of β-napthol. **(This test can only be performed when Ar–NH_2 group is absent.)**	Brilliant red or scarlet dye.	Anilido group (-CONHAr) present.
(b) Dichromate Test: sample + Concentrated H_2SO_4, shaken vigorously in a dry test tub + finely powdered $K_2Cr_2O_7$	Deep blue or bluish pink colour.	Anilido group Confirmed.

2. Detection of Non-Nitrogenous functional groups:

Experiment	Observation	Inference
(i) $NaHCO_3$ Test: Saturated solution of sodium bicarbonate under heating, cool + pinch of sample.	Effervescence of CO_2 gas.	-COOH, -SO_3H group may be present
(ii) Esterification test: 1.0 g sample + 10 cc ethanol + 2-3 concentrated H_2SO_4, boiled in a water bath for 10 minutes. The mixture is then poured into 150 cc water.	A sweet smell of ester.	-COOH group is present and confirmed.

(iii) (a) Back dye test for phenolic-OH group: **Solution-I:** small amount of p-nitro aniline or aniline is dissolved in dil. HCl, cooled in ice-bath. **Solution-II:** $NaNO_2$ solution, cooled in ice-bath. **Solution-III:** alkaline solution of sample, cooled in ice-bath. The **solution-I** & **II** are mixed and kept for some time. The **solution-III** is added to the mixture of **solution-I** & **II**.	Orange-red dye.	Phenolic-OH group present and confirmed.
(b) $FeCl_3$ Test: alcoholic or aqueous solution of sample + $FeCl_3$ solution.	Violet or green or blue colouration.	Phenolic-OH group may be present.
(c) Libermann's Test: Sample + few crystal of sodium nitrite + Conc. H_2SO_4 (5 ml) The content of the test tube is added to 150 ml of water. The solution will appear red. Excess of NaOH soln. is added.	Green colour Red colour solution Green colour reappear	Phenolic-OH group present and confirmed
(iv) Test for carbonyl (>C=O) group: 2 g sample is dissolved in minimum quantity of ethanol + 5 cc saturated solution of 2,4-dinitrophenylhydrazone, warmed, scratched with glass road.	Yellow or orange ppt.	Carbonyl (>C=O or –CHO) group is present and confirmed.
(v) (a) Fehlings's test: sample + Fehlings's solution (Fehlings-I & Fehlings-II), heated in water bath. **(b) Tollen's Test:** sample + Tollen's reagent, heated in water bath.	Yellow or red ppt. Silver mirror or grey ppt.	-CHO group present and confirmed. -CHO group present and confirmed.

(vi) Hydroxamic acid test for ester group: sample + 5% hydroxylaminehydrochloride solution + saturated methanolic KOH solution until the mixture is alkaline, boiled, cooled + acidified with dil. HCl + 1-2 drops $FeCl_3$ solution.	Violet colouration.	-COOR group present and confirmed.
(vii) Test for unsaturation (>C=C<): (a) Alcoholic solution of sample + very dilute KMnO4 solution. (b) Sample solution in $CHCl_3$ +Br_2 solution.	Disappearance of pink colour. Disappearance of red colour of Br_2.	Unsaturation (>C=C<) or easily oxidisable group is present. Unsaturation (>C=C<) present and confirmed.

Highlights:

1. During Lassaigne's test a dark coloured sodium extract is obtained either due to incomplete fusion of the organic compound or when it is taken in excess of the sodium taken. Again if sodium is not used in excess during the experiment, on pouring the fusion tube into water no sodium hydroxide will be formed and the solution will not be alkaline. So green precipitate of ferrous hydroxide will not be observed during Prussian blue test and the test for N will fail.

2. If the solution is not alkaline sodium nitroprusside test will also fail.

   Above two tests can be performed by adding sodium hydroxide solution to the filtrate if the solution is not alkaline.

3. The solution will give a black precipitate on addition of ferrous sulphate if sulphur is present in the organic sample.

4. The ferrous sulphate solution should be freshly prepared. A little amount of ferrous sulphate is washed with distilled water to remove ferric sulphate is more soluble than ferrous sulphate. Next the sulphate is dissolved to get freshly prepared ferrous sulphate solution. A dilute $FeSO_4$ on standing is oxidized to basic Ferric Sulphate which becomes useless in analysis.

$4FeSO_4 + 2H_2O + O_2 \rightarrow 4Fe(OH)SO_4$

5. The addition of Ferric chloride solution and HCL is avoided during Prussian blue test as it forms a green precipitate or colour. Concentrated sulphuric acid, in turn, will give a fine blue precipitate or colouration as concentrated sulphuric acid will oxidise the excess ferrous sulphate present in solution to ferric sulphate and those ferric icons will yield Prussian blue in fine blue colour.

6. It is essential to boil the sodium extract with HNO_3 to remove CN^- as voilatole HCN. Otherwise it will give give ppt of AgCN similar to AgCl and misguide the analysis.

$$NaCN + AgNO_3 = AgCN + NaNO_3$$

$$NaCN + HNO_3 = HCN + NaNO_3$$

Beilstein's Test: A piece of stout copper wire, fitted with a cork to an end, is heated strongly in a non-luminous Bunsen flame till the copper wire fails to impact any coloured flame. The hot wire is then dipped into the O.S.to be tested and heated again in the non-luminous flame (The Carbon present in O.S. burns away with luminous flame) and then the appearance of green or bluish green flame indicates the presence of halogens (except fluorine). Other compounds such as urea, quinoline or pyridine derivatives, etc. impact green colour to the flame. Hence, the negative result indicates absence of halogens and the converse may not be true.

Test for Phenolic-OH Group: The following test (phenolphthalein test) can be done only when $NaHCO_3$ test for –COOH group or $–SO_3H$ group, Esterification test for –COOH group, litmus test are negative for the supplied organic sample.

Experiment	Observation	Inference
Solution-I: Very dilute NaOH solution + 2-3 drop phenolphthalein, pink colour solution is obtained. **Solution-II:** Organic sample is dissolved in ethyl alcohol. **Solution–I** is added to the solution-II drop by drop.	Pink colour of solution-I is disappeared.	Phenolic-OH group may be present.

Systematic Qualitative Analysis of Unknown Organic Sample

1. (a) Physical characteristic:

(v) State of aggregation: solid
(vi) Texture: amorphous
(vii) Colour: White
(viii) Odour: odoue less

(b) Melting Point: 158^{o}C

(c) Systematic analysis for the detection of special elements:

Lassaigne's Test: A small amount of sample is fused with Na in a fusion tube. The fused mass is extracted with distilled water and then filtered. With the filtrate the following tests are performed.

Experiment	Observation	Inference
(i) **Prussian blue test**: Filtrate +freshly prepared $FeSO_4$ soln. + boiled, cooled + acidified with dil. H_2SO_4 soln.	No Prussian blue or green ppt or colour.	N-absent
(ii) **Nitroprusside test:** filtrate + NaOH soln. +sodium nitroprusside soln.	No Violet or purple colour	S-absent
(iii) **Silver nitrate test:** filtrate + conc. HNO_3, boiled, cooled, + $AgNO_3$ soln.	No white ppt. or coloured ppt.	Cl, Br and I absent

Conclusion: From the above experiment, is concluded the supplied organic sample does not to contain any special element such as N, S, Cl, Br or I.

(c) Preliminary tests:

Experiment	Observation	Inference
(i)Litmus test: Blue litmus paper is treated with solution of sample in mixture of water and alcohol.	Blue litmus paper turns to red	Organic sample is acidic and may contain –COOH, Ar-OH or –SO_3H group.
(ii) $NaHCO_3$ Test: Saturated solution of sodiumbicarbonate under heating, cool + pinch of sample.	Effervescence of CO_2 gas.	-COOH, -SO_3H group may be present
(iii)Permanganate test: Dil. KMnO4 solution is added to alcoholic sample solution drop by drop.	No Pink colour of $KMnO_4$ is disappeared	Unsaturation (>C=C<) or easily oxidisable group may be present.
(iv)Soda-lime test: organic sample is heated with soda-lime.	No Characteristic smell of NH_3 or burnt sugar or aniline.	-$CONH_2$ group, Carbohydrate, or –CONHPh may be absent.
(v)Ignition test : sample is burned	Yellow sooty flame	Sample may be aromatic

(i)

(e) Solubility test with conclusion:

Cold H_2O	Hot H_2O	dil. HCl	dil. $NaHCO_3$	dil. NaOH	Conc. H_2SO_4
-	+	-	+	+	-

Conclusion:

From solubility and melting point inference can be drawn for the given organic sample.

(i) As the Sample is soluble in hot water but insoluble in cold water, the may be salicylic acid, benzoic acid or phenyl acetic acid.

(ii) The sample is soluble in dil. NaOH. The compound may contain the functional group –COOH or phenolic –OH or –COOH and phenolic –OH both.

(iii) If the sample effervesces with $NaHCO_3$ solution i.e the compound is soluble in $NaHCO_3$ solution then the sample contains –COOH group with or without other functional groups.

(f) Systematic analysis of the functional groups:

1. Detection of Non-Nitrogenous functional groups:

Experiment	Observation	Inference
(i) $NaHCO_3$ Test: Saturated solution of sodiumbicarbonate under heating, cool + pinch of sample.	Effervescence of CO_2 gas.	COOH, $-SO_3H$ group may be present
(ii)Esterfication test: 1.0 g sample + 10 cc ethanol + 2-3 concentrated H_2SO_4,, boiled in a water bath for 10 minutes . The mixture is then poured into 150 cc water.	A sweet smell of ester.	-COOH group is present and confirmed.

(iii)(a) Back dye test for phenolic-OH group: **Solution-I:** small amount of p-nitro aniline or aniline is dissolved in dil. HCl , cooled in ice-bath. **Solution-II:** $NaNO_2$ solution, cooled in ice-bath. **Solution-III:** alkaline solution of sample, cooled in ice-bath. The **solution-I** & **II** are mixed and kept for some time. The **solution-III** is added to the **solution-I** & **II**.	Orange-red dye.	Phenolic-OH group present and confirmed
(b) $FeCl_3$ Test: alcoholic or aqueous solution of sample + $FeCl_3$ solution.	Violet or green or blue colouration.	Phenolic-OH group may be present.
(iv)Test for carbonyl (>C=O) group: 2.0 g sample is dissolved in minimum quantity of ethanol + 5 cc saturated solution of 2, 4-dinitrophenylhydrazone, warmed, scratched with glass road.	No yellow or orange ppt.	Carbonyl (>C=O or –CHO) group is absent.
(v) Test for (-CHO) group: **(a) Fehlings's test:** sample + Fehlings's solution (Fehlings-I & Fehlings-II), heated in water bath. **(b) Tollen's Test:** sample + Tollen's reagent, heated in water bath.	No yellow or red ppt. No silver mirror or grey ppt.	-CHO group absent. -CHO group absent.

(v)Hydroxamic acid test for ester group: sample + 5% hydroxylaminehydrochloride solution + saturated methanolic KOH solution until the mixture is alkaline, boiled, cooled + acidified with dil. HCl + 1-2 drops $FeCl_3$ solution.	No violet or wine colouration.	-COOR group absent.
(vi)Test for unsaturation (>C=C<): (a)Alcoholic solution of sample + very dilute KMnO4 solution. (b)Sample solution in $CHCl_3$ +Br_2 solution.	No disappearance of pink colour. No disappearance of red colour of Br_2.	Ethylenic unsaturation (>C=C<) or easily oxidisable group is absent. Ethylenic unsaturation (>C=C<) or easily oxidisable group is absent.

2. Detection of Nitrogenous functional groups:

Experiment	Observation	Inference
(i) Dye test: **Solution-I:** Sample is dissolved in dil. HCl and cooled in an ice-bath. **Solution-II:** Dil. $NaNO_2$ soln. is cooled in an ice-bath **Solution-III:** Alkaline β-napthol soln. is cooled in an ice-bath. The mixture of **solution-I** & **II** is cooled in an ice-bath .After some time alkaline β-napthol soln. is added to the mixture of **solution-I** & **II**.	Red or orange –red dye.	Ar-NH_2 group confirmed.

(ii). Mulliken and Barker's test: Alcoholic soln. of sample + solid NH_4Cl + Zn-dust boiled for few minutes, cooled allowed to stand for 5 minutes, and filtered. Filtrate + Tollen's reagent, warmed in a water bath.	Silver mirror or black or grey ppt.	Ar-NO_2 group confirmed.
(iii). Hydrolysis test for amide group: sample + Conc. NaOH solution, heated.	Characteristic smell of NH_3 which turns mercurous nitrate paper black.	-$CONH_2$ group confirmed.
(iv). **Test for anilido (-CONHAr) group**: 0.05 g of sample is hydrolysed by 3 ml conc. HCl and 2 ml water. The hydrolysed product is diluted with 5 ml water, cooled then diazotized by cold dil. $NaNO_2$ solution and finally the mixture is added to cold alkaline solution of β-napthol.	Brilliant red or scarlet dye.	Anilido group (-CONHAr) present.

(g) Preparation of the derivative with methods of derivatisation and melting point of the prepared derivative.

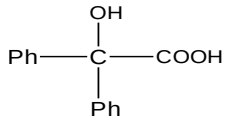
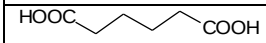
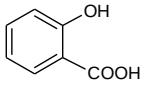
Preparation of Amide (-$CONH_2$) derivative for Carboxylic acid (-COOH):

A mixture of 0.5 gm (approximate) of organic sample and 2 gm (approximate) of phosphorous pentachloride (4 gm) is taken in a clean and dry mortar and ground thoroughly by a pestle until it becomes liquid. An inverted small funnel is placed over the liquid and 10 ml of liquor ammonia is added cautiously to it along the side of the funnel. The mixture is stirred and allowed to cool after the vigorous reaction has subsided. The product is filtered, recrystallised from dilute alcohol (50:50) and M.P is determined.

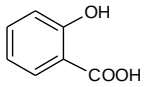
$$RCOOH + PCl_5 \longrightarrow RCOCl \xrightarrow{NH_3} RCONH_2$$

M.P of the derivative = 139°C

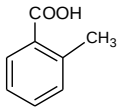
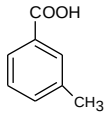
(h) Overall conclusion with Literature Survey:

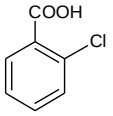
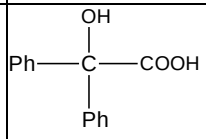
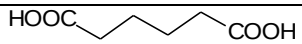
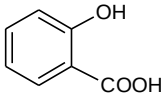
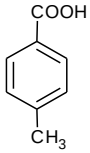
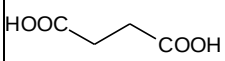
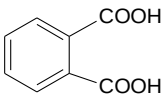
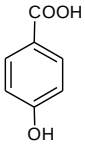
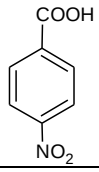
Probable Compound	Literature M.P of the probable compound. (°C)	Literature M.P of the derivative (amide) of probable compound. (°C)	M.P of the supplied organic sample. (°C)	M.P of the derivative (amide) of the supplied organic sample. (°C)
 Benzilic acid	150	154	158	139
 Adipic acid	151	220		
 Salicylic acid	158	139		
m-chloro benzoic acid	158	134		

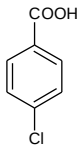
From the above experiments and literature survey, it is concluded that the supplied organic sample may be Salicylic acid. The Structure of the Salicylic acid is given bellow.



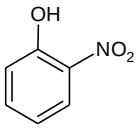
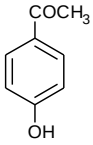
1. <u>Carboxylic Acid (-COOH)</u>:

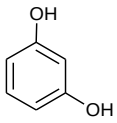
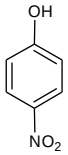
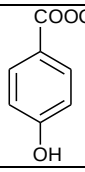
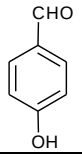
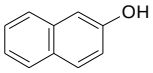
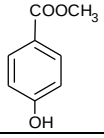
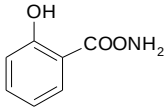
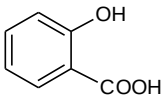
M.P (°C)	Name of the compounds	Properties	M.P of the Derivative (°C)
76	$Ph\text{-}CH_2\text{-}COOH$ Phenyl acetic Acid	Soluble in hot water	**Amide = 154**
104	 O-toluic Acid		**Amide = 142**
110	 m-toluic Acid		
122	$Ph\text{-}COOH$ Benzoic Acid		**Amide = 128**
133	$Ph\text{-}CH = CH\text{-}COOH$ Cinnamic acid		**Amide = 147** **Dibromo derivative = 195** **Acid devivative = 122** **(Oxidation with alkaline $KMnO_4$ followed by acidification with dil HCl yield benzoic acid)**

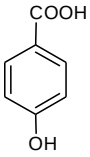
140	 o-chloro benzoic acid		**Amide = 142**
150	 Benzilic acid		**Amide = 154**
151	 Adipic acid		**diamide = 220**
158	 Salicylic acid		**Amide = 139**
180	 p- toluic Acid		**Amide = 158**
185	 Succinic acid		**Amide = 228**
195	 Phthalic acid		**diamide = 219** **(Phthalamide)**
213	 4-hydroxy benzoic acid		**Amide = 1162**
241	 4-Nitrobenzoic acid		**Amide = 201**

243 **(240)**	 4-Chloro benzoic acid		**Amide = 179**

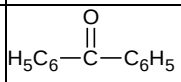
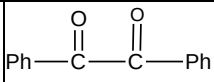
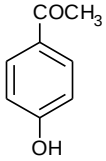
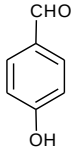
2. Phenolic –OH group:

M.P (°C)	Name of the compounds	Properties	M.P of the Derivative (°C)
45	 2-nitrophenol		**Benzoate = 142**
80	Vanillin		**DNP = 271** **Bromo derivative=160**
97	m-nitrophenol		
109	 4-Hydroxy acetophenone		**DNP =261** **Benzoate =134**
	Catechol $C_6H_4(OH)_2$ (1,2)		**Bromoderivative (Tetra)=192** **3,5-dinitrobenzoate**

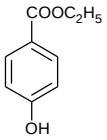
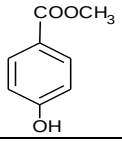
			derivative (di)=152
110-113	 Resorcinol		**Bromoderivative(di)=112** **3,5-dinitrobenzoate derivative (di)=201**
114	 4-nitrophenol		**Benzoate = 142** **Bromoderivative(di)=142** **3,5-dinitrobenzoate derivative =186**
116	 Ethyl-4-hydroxybenzoate		**Acid Derivative = 213**
117	 4-Hydroxybenzaldehyde		**DNP = 280** **Semicarbazone = 224**
123	 β-napthol		**Benzoate = 107**
131	 Methyl-p-hydroxybenzoate		**Acid Derivative = 213**
139	 Salicylamide		**Acid = 158**
140	Meta-Nitro Benzoic acid		**Amide = 143**
158	 Salicylic acid		**Amide = 139**

200	m-hydroxybenzoic acid		
213	 4-Hydroxybenzoic acid		**Amide = 162**

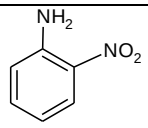
3. Carbonyl Group (-CO-), aldehyde or ketones:

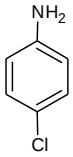
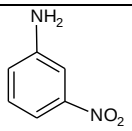
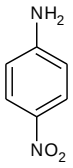
M.P (°C)	Name of the compounds	Properties	M.P of the Derivative (°C)
48	 Benzophenone		**DNP = 238**
95	 Benzil		**DNP (di) = 189**
109	 4-Hydroxy acetophenone		**DNP =261** **Benzoate = 134**
117	 4-Hydroxybenzaldehyde		**DNP = 280**

4. Ester (-COOR):

M.P (°C)	Name of the compounds	Properties	M.P of the Derivative (°C)
116	 Ethyl-4-hydroxybenzoate		**Acid derivative = 213**
131	 Methyl-p-hydroxybenzoate		**Acid derivative = 213**

5. <u>Aromatic amino (-NH_2) group</u>:

M.P (°C)	Name of the compounds	Properties	M.P of the Derivative (°C)
45	p-Toluidine		**Benzoyl= 158**
50	α-Napthyl amine		
57	4-Methoxyaniline(p-Anisidine)		**Benzoyl= 154**
71	 2-Nitro aniline		**Benzoyl= 98**

71	 4-Chloroaniline		**Benzoyl = 193**
114	 3-Nitroaniline		**Benzoyl = 155**
147	 4-Nitroaniline		**Benzoyl = 199**
174	3-Aminophenol		
184	4-Aminophenol		**3,5-dinitrobenzoate derivative=178**
186	4-Aminobenzoicacid		**Benzoyl = 278**
198	Aniline hydrochloride		
300	Sulphanilic acid		

6. Aromatic nitro ($-NO_2$) group:

M.P (°C)	Name of the compounds	Properties	M.P of the Derivative (°C)
44	o-Nitrophenol		**Benzoate = 142**
44	o-Nitro benzaldehyde		
58	m-Nitrobenzaldehyde		

71	2-Nitro aniline		**Benzoyl= 98**
83	p-Nitrochlorobenzene		
90	m-Dinitrobenzene		
106	p-Nitrobenzaldehyde		
114	3-Nitroaniline		**Benzoyl = 155** **Picrate = 143**
114	p-Nitrophenol		**Benzoate = 142**
118	o-Dinitrobenze		
140	m-Nitrobenzoic acid		**Anide = 143** **Anilide = 153**
143	m-Nitrobenzamide		
144	o-Nitrobenzoic acid		**Anilide = 155**
147	4-Nitroaniline		**Benzoyl = 199**
176	o-Nitro benzamide		
201	p-Nitro benzamide		
210	p-Nitro acetanilide		
241	p-Nitrobenzoic acid		**Amide =201**

7. Anilido Group (-NHAr) :

M.P (°C)	Name of the compounds	Properties	M.P of the Derivative (°C)
161	**Benzanilide (PhNHCOPh)**		**Acid derivative= 122**

8. Amide ($-CONH_2$) Group:

M.P (°C)	Name of the compounds	Properties	M.P of the Derivative (°C)
128	Benzamide($PhCONH_2$)		Acid derivative= 122

Preparation of derivative

(1) **Preparation of Amide ($-CONH_2$) derivative for Carboxylic acid (-COOH):**

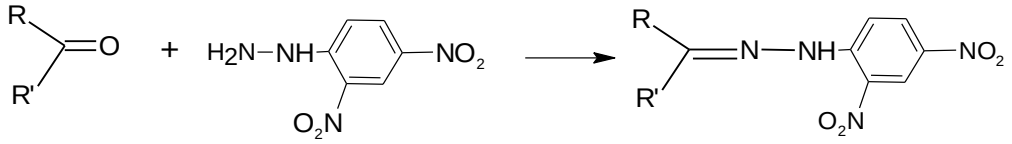
A mixture of 0.5 gm (approximate) of organic sample and 2 gm (approximate) of phosphorous pentachloride (4 gm) is taken in a clean and dry mortar and ground thoroughly by a pestle until it becomes liquid. An inverted small funnel is placed over the liquid and 10 ml of liquor ammonia is added cautiously to it along the side of the funnel. The mixture is stirred and allowed to cool after the vigorous reaction has subsided. The product is filtered, recrystallised from dilute alcohol (50:50) and M.P is determined.

$$RCOOH + PCl_5 \longrightarrow RCOCl \xrightarrow{NH_3} RCONH_2$$

M.P of the derivative =

(2) **Preparation of 2,4-dinitro phenyl hydrazone derivative for aldehyde or ketone** :

0.4 gm of organic sample is dissolved in minimum quantity of ethyl alcohol in a test tube. Then 10 ml of saturated alcoholic solution of 2,4dinitro phenylhydrazine (containing 2 ml H_2SO_4) is added to the alcoholic solution of sample. The mixture is heated in a water bath for 10 minutes and is then cooled to room temperature. After some times, solid crystals of 2,4-dinitro phenyl hydrazone derivative is filtered and washed with water. The derivative is crystallized from ethyl alcohol.



M. P of the derivative =

(3) **Preparation of Benzoyl derivative for phenolic-OH group:**

1.0 g of compound is dissolved in 15 cc of 10% aqueous solution of NaOH in a test tube. Then 2 cc of benzoyl chloride is added and a cork is placed at the mouth of the test tube tightly. The mixture is shaken vigorously for 10 minutes. The benzoyl derivative is filtered, washed with water and is recrystallised from rectified spirit.

$$ArOH + NaOH \xrightarrow{PhCOCl} ArOCOPh$$

M.P of the derivative =

(4) **Preparation of Benzoyl derivative for Ar-NH_2 group:**

1.0 gm of compound is dissolved in 15 cc of 10% aqueous solution of NaOH in a test tube. Then 2 cc of benzoyl chloride is added and a cork is placed at the mouth of the test tube tightly. The mixture is shaken vigorously for 10 minutes. The benzoyl derivative is filtered, washed with water and is recrystallised from rectified spirit.

$$ArNH_2 + NaOH \xrightarrow{PhCOCl} ArNHCOPh$$

M.P of the derivative =

(5) **Preparation of derivative by hydrolysis for –$CONH_2$ group:**

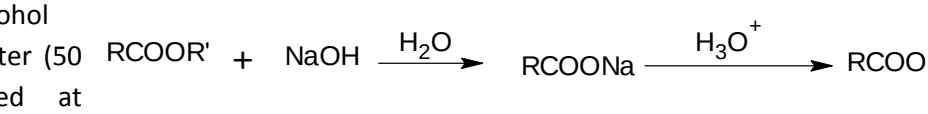
The mixture of 1 gm of organic sample and 20 ml of 20% NaOH solution are boiled for 15 minutes in a 50 ml conical flask and the cooled. The mixture is then acidified with dilute HCl. The white solid is filtered at the pump, washed with cold water, recrystallised from alcohol and water (50 : 50) , dried at 100 °C.

$$RCONH_2 + NaOH \xrightarrow{H_2O} RCOONa \xrightarrow{H_3O^+} RCOOH$$

M.P of the derivative =

(6) **Preparation of derivative by hydrolysis for –COOR group:**

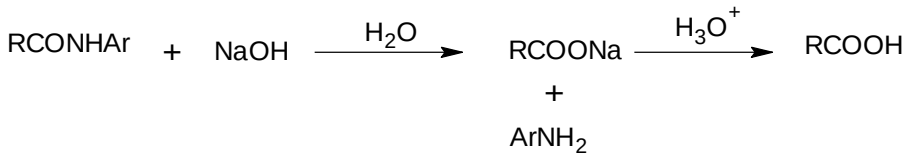
The mixture of 1 gm of organic sample and 20 ml of 20% NaOH solution are boiled for 15 minutes in a 50 ml conical flask and the cooled. The mixture is then acidified with dilute HCl. The white solid is filtered at the pump, washed with cold water, recrystallised from alcohol and water (50 : 50) , dried at 100 °C.



M.P of the derivative =

(7) **Preparation of derivative by hydrolysis for –CONHAr group:**

The mixture of 1 gm of organic sample and 20 ml of 20% NaOH solution are boiled for 15 minutes in a 50 ml conical flask and the cooled. The mixture is then acidified with dilute HCl. The white solid is filtered at the pump, washed with cold water, recrystallised from alcohol and water (50 : 50) , dried at 100 °C.



M.P of the derivative =

(8) **Preparation of Nitro derivative for –CONHAr group:**

The mixture of organic sample (2.0 gm) and 5 ml glacial acetic acid is taken in a 250 ml beaker and Stirred. Then add 10 ml of concentrated H_2SO_4 is added to the mixture. The reaction mixture is cooled in a freezing mixture of ice and salt and stirred the solution. When the temperature of the solution falls to 0-2 °C, the mixture acids (2.5 ml conc. HNO_3 and 1.5 ml conc. H_2SO_4) is added gradually or drop wise with constant stirring. The temperature is maintained bellow 10°C. After all the acid mixture has been added, the beaker is removed from the freezing mixture, allowed it to stand at room temperature for one hour. The reaction mixture is into 50gm of crushed ice (or into 100 ml cold water) and allow for 15 minutes. The derivative is filtered under suction on Buchner funnel, washed thoroughly with cold water until free from acids. Recrystallise from ethanol.

M.P of the derivative =

Organic Preparation:

(1) Preparation of p-nitro acetanilide from acetanilide:

The mixture of acetanilide (5.0 gm) and 5 ml glacial acetic acid is taken in a 250 ml beaker and Stirred. Then add 10 ml of concentrated H_2SO_4 is added to the mixture. The reaction mixture is cooled in a freezing mixture of ice and salt and stirred the solution. When the temperature of the solution falls to 0-2 $^{\circ}$C, the mixture acids (2.5 ml conc. HNO_3 and 1.5 ml conc. H_2SO_4) is added gradually or drop wise with constant stirring. The temperature is maintained bellow 10°C. After all the acid mixture has been added, the beaker is removed from the freezing mixture, allowed it to stand at room temperature for one hour. The reaction mixture is into 50gm of crushed ice (or into 100 ml cold water) and allow for 15 minutes. The derivative is filtered under suction on Buchner funnel, washed thoroughly with cold water until free from acids. Recrystallise from ethanol.

Experimental Data:

Appearance	Melting Point of Crystalline product	yield
Pale yellow crystalline solid	**214°C**	**4.0 g**

(2) Preparation of Phthalimide from phthalic anhydride:

The mixtures of 6.5 g phthalic anhydride (finely powdered) and urea (1.5 gm) are take in a 500 ml round bottomed flask fitted wit an air condenser and heated the flask at 130-135 C on a sand bath for 15-20 minutes. The reaction begins with the melting of the contents, effervescences commences which gradually increases in vigorous, and thereafter, the mass suddenly froths up and temperature rises spontaneously to 160 C. Then the mass is allowed to cool for 40 minutes and added water to disintegrate the solid in a flask. The solid is filtered at the pump, wash with a little water and dry at 100 ^{o}C. Recrystallise 0.5 gm of the product from alcohol.

Experimental Data:

Appearance	Melting Point of Crystalline product	yield
White crystalline solid	**234 °C**	**4.0 g**

(3) Preparation of Benzoic Acid from ethyl benzoate:

5.0 g of ethyl benzoate and 60 ml 10 % aqueous NaOH solution is taken in a 250 ml round bottomed flask. The flask is the fitted with water condenser and refluxed the solution for 30 minutes. The mixture is cooled and then acidified with concentrated HCl. The mixture is diluted with cold water and filtered the solid at the pump, washed with ice cold water and dry at 100 C. Recrystallise 0.5 gm of the product from hot water.

Experimental Data:

Appearance	Melting Point of Crystalline product	yield
White crystalline solid	**122 °C**	**4.0 g**