BGS SCIENCE ACADEMY & RESEARCH CENTER
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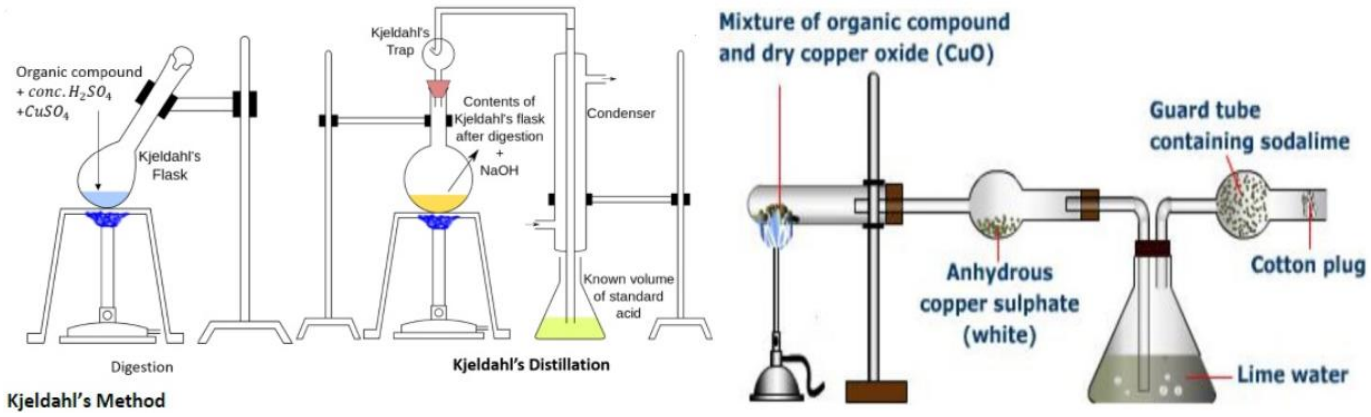
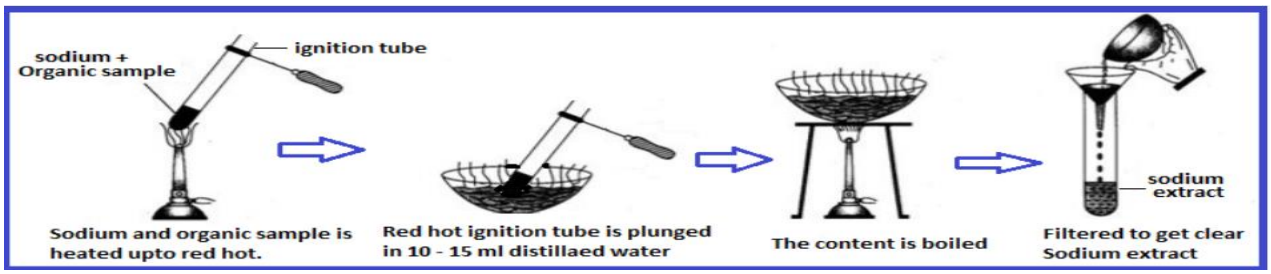
V Semester B.Sc.,
Paper-V
Organic Chemistry Laboratory Manual





SYSTEMATIC IDENTIFICATION OF ORGANIC COMPOUNDS

The systematic analysis of organic compounds involves the following steps.

I. Preliminary observations
II. Determination of physical constants such as melting points/boiling points
III. Detection of elements (Nitrogen, Halogens and Sulphur)
IV. Determination of solubility
V. Functional group tests
VI. Preparation of a suitable solid derivative

I. Preliminary observations

State: *Solid* (amides, carbohydrates, carboxylic acids etc.)
Liquid: (alcohols, aldehydes, ketones, esters, amines etc.)
Colour: *Yellow* (nitro compounds)
Brown to black amines and phenols)
Colourless (alcohols, aldehydes, ketones, acids, esters, amides etc).
Odour: *Pleasant* (alcohols, halogenated hydrocarbons)
Fruity (esters)
Fishy (amines)
Bitter almonds (benzaldehyde and nitrobenzene)

Experiment	Observation	Inference
Ignition test: Ignite a pinch of solid or a drop of a liquid on a nickel spatula.	The compound burns with sooty flame.	The compound is aromatic.
	The compound burns and chars with the smell of burnt sugar.	Compound may be carbohydrate.
	The compound does not burn but crackles leaving behind a white residue.	May be urea.
Test for unsaturation: *Bayer's test:* Few crystals or 2 drops of the compound+ $KMnO_4$ solution. Shake well.	Pink colour is discharged.	The compound is unsaturated or reactive or easily oxidisable.
	Pink colour is not discharged.	The compound is saturated or less reactive or not easily oxidisable.
Compound + 2 or 3 drops of bromine water. Shake well.	Orange red colour is discharged	Compound is unsaturated or reactive or easily oxidisable.
	Orange red colour is not discharged.	Compound is saturated or less reactive or not easily oxidisable.

II. Determination of physical constants

If the given compound is a solid, determine the melting point. Note down the temperature when the substance in the capillary tube has just melted.
If the compound is a liquid, determine the boiling point.
Correct boiling point = Observed boiling point + 3% correction.

III. Detection of elements: Lassaigne's test

Take a small piece of sodium metal in a sodium fusion tube. Warm the tube till the metal melts into silvery white. Cool the tube and add a pinch of the compound if it is a solid or a drop if it is a liquid. Heat the tube gently till it becomes red hot. Plunge it into about 5 ml of distilled water taken in mortar. Add two more such fusions into the same mortar. Grind the contents thoroughly with pestle. Transfer the contents into a small beaker. Heat nearly to boiling and filter. The filtrate is called *stock solution or sodium fusion extract.*

Experiment	Observation	Inference
Test for nitrogen: 1 ml of stock solution + few crystals of $FeSO_4$, boil, cool. Acidify with dil. H_2SO_4.	A blue or bluish green colouration.	Nitrogen is present.
Test for halogens: 1 ml of stock solution + 1ml of conc. HNO_3, boil, cool and add $AgNO_3$ solution.	White precipitate soluble in excess of NH_4OH. A pale yellow precipitate soluble in excess of NH_4OH. No precipitate.	Chlorine is present. Bromine is present. Halogens are absent.
Beilstein's test: Hold a copper wire in a non-luminous Bunsen flame until it does not impart colour to the flame. Cool, dip it in the liquid and ignite again.	Green or bluish green edged flame.	Halogen is present in the compound.
Test for sulphur: (a). 1 ml stock solution + dil. acetic acid + lead acetate solution	Black precipitate.	Sulphur is present.
(b). 1 ml stock solution + 2 or 3 drops of sodium nitroprusside solution.	Purple or violet coloured solution	Sulphur is present.

IV. Determination of solubility

The solubility of an organic compound in a solvent is determined to find out to the correct group the compound belongs. For this purpose different solvents such as water, ether, dil. HCl, 5% NaOH solution and conc. H_2SO_4 are used and the solubility is determined. This gives the group and the probable functional groups of organic compound belonging to that group.

In order to determine the solubility, about 2 ml of the solvent is taken in a test tube and a pinch of the given solid or 2 or 3 drops of the given liquid is added and shaken well. *If a homogenous solution is formed with or without change of colour then the compound is soluble. The formation of a precipitate also indicates the solubility. (ex. Benzyl alcohol mixed with conc. H_2SO_4). If oily drops are observed or two separate layers are formed or an emulsion is formed then the compound is insoluble.*

Note: 1. If the compound is insoluble in water, solubility in ether should not be tested.

2. If the compound contains nitrogen and it is insoluble in water and in dil. HCl, it belongs to VII group. Then the solubility in 5% NaOH and in conc. H_2SO_4 should not be carried out.

Soluble in water		Insoluble in water				
Soluble in ether	**Insoluble in ether**	**Soluble in dil. HCl**	**Soluble in 5% NaOH solution.**	**Soluble in conc. H_2SO_4**	**Insoluble in conc. H_2SO_4**	**Compound containing nitrogen & insoluble in water & in dil. HCl**
I	**II**	**III**	**IV**	**V**	**VI**	**VII**
Resorcinol(s)	Urea(s) & glucose(s)	amines.(l) *Ex.* aniline, *o(l), m(l) p-* toludines.(s)	Phenols, (l)carboxylic acids, (s)phenolic acids(s) *Ex:* phenol, *o, m, p-* cresols,(l) benzoic acid & salicylic acid(s)	Benzyl alcohol, (l)benzaldehyde(l), aceto-phenone,(l) methyl & ethyl benzoates(l)	Aromatic hydro-carbons & their halogen derivates. *Ex:* toluene(l), naphthalene, (s)chloro-benzene(l)	Amides & Nitro compounds *Ex:* benzamide(s) nitrobenzene (l) & *m-* dinitro-benzene(s)

V. Identification of functional groups

Analysis of I group organic compounds: *(Low molecular weight compounds)*

Experiment	Observation	Inference
1. Compound is dissolved in water + a few drops of neutral ferric chloride solution.	Violet or purple coloured solution.	It is a phenolic compound.
2. *Phthalein fusion test:* Compound in dry test tube + 1 or 2 pieces of phthalic anhydride + 2 drops of conc. H_2SO_4. Gently heat, cool and dilute with water. Pour it into NaOH solution.	A double coloured (green and red) solution is formed. i.e. green fluorescence.	Polyhydric phenol is confirmed. May be resorcinol.

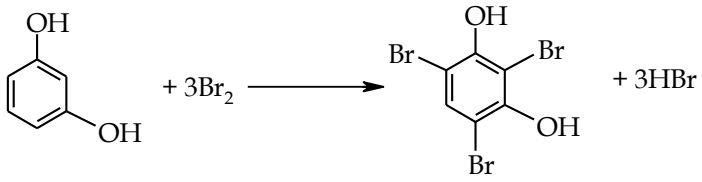
Preparation of derivative: *Bromo derivative:* Dissolve the compound in water taken in a conical flask. Add bromine water till the yellow colour persists. Pour the contents of the flask into a beaker of cold water. Filter the solid, wash with water and dry.

Reactions of resorcinol

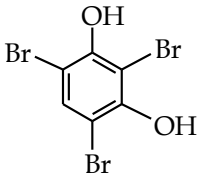
1 Neutral ferric chloride test

$$6ArOH + FeCl_3 \longrightarrow [Fe(OAr)_6]^{3+}.Cl^- + 6H^+$$

2. Bromination



2 Structure of bromoderivative



Analysis of II group organic compounds: *(Amides and Carbohydrates)*

Experiment	Observation	Inference
1. Compound + 20% NaOH solution. Boil.	Pungent smelling gas is evolved.	May be amide.
Expose a glass rod dipped in conc. HCl to the vapours.	Dense white fumes are formed.	May be amide
2. *Biuret test:* Compound in a dry test tube, heat till it melts and solidifies. Cool, add water, a few drops NaOH solution and a drop of dilute $CuSO_4$ solution.	Violet colouration.	Urea is confirmed.

Preparation of derivative: *Urea nitrate derivative:* Dissolve urea in minimum amount of water. Add conc. nitric acid till a white crystalline solid appears. Filter and dry the solid.

Reactions of urea

1 Biuret test

2 . Structure of urea nitrate derivative

$NH_2CONH_2.HNO_3$

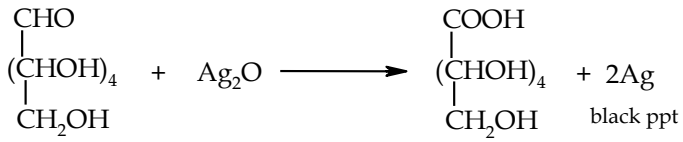
Experiment	Observation	Inference
Tests for Carbohydrates: 1. Compound + conc. H_2SO_4. 2. *Molisch test:* Solution of the compound in water + α- naphthol in alcohol + a few drops of conc. H_2SO_4 carefully down the sides of the test tube. 3. *Tollen's test:* Solution of the compound in water + Tollen's reagent and heat gently without shaking the test tube. 4. *Fehling's test:* Solution of the compound in water + equal volumes of Fehling's A and Fehling's B solutions. Heat on a water bath.	A black residue. A violet ring is formed. A black precipitate is formed. No black precipitate. Red precipitate is formed. No red precipitate.	It is a carbohydrate. The compound is a carbohydrate. It is a reducing sugar. It is a non-reducing sugar. It is a reducing sugar. It is a non-reducing sugar.

Preparation of derivative: *Osazone derivative:* Dissolve the compound in water taken in a test tube. Add 1 ml of glacial acetic acid and 3 ml of phenyl hydrazine. Heat the contents in a beaker containing boiling water. A yellow crystalline solid is formed. Filter the solid, wash and dry.

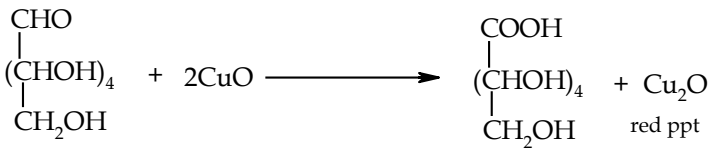
Reactions of carbohydrate

Example: glucose

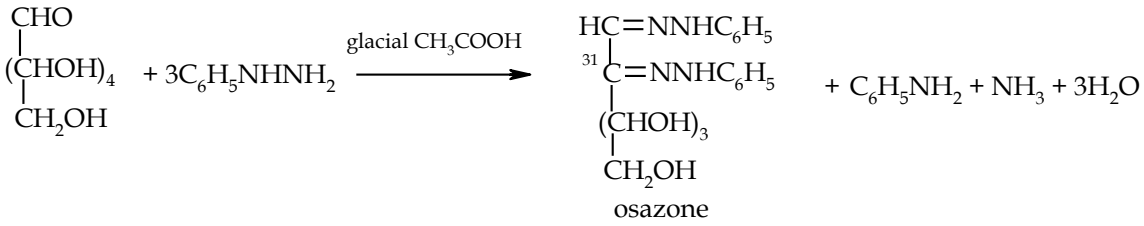
1 ***Tollen's test***



2 ***Fehling's test***



3. Structure of phenyl hydrazine derivative of glucose/fructose



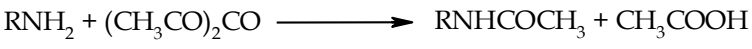
Analysis of III group organic compounds: *(Amines)*

Experiment	Observation	Inference
1. 2 or 3 Drops compound in a dry test tube + 4 or 5 drops acetic anhydride, warm, cool and pour the mixture into a test tube containing cold water.	A white solid is formed.	The compound is primary or a secondary amine.
2. *Carbyl amine test:* Compound + chloroform + alcoholic KOH solution and warm.	A foul smelling compound is formed.	The compound is a primary amine.
3. *Diazotisation & Coupling:* Dissolve the compound in dil. HCl, cool in ice. Cool a solution of sodium nitrite in water. Mix the two solutions.	Pale blue or yellow coloured solution.	Primary amine is confirmed.
Add a small portion of the mixture to a solution of β-naphthol in NaOH solution.	A bright orange red dye is formed.	Primary amine is confirmed.

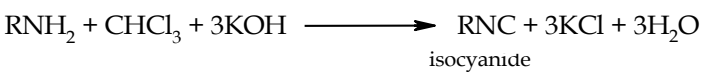
Preparation of derivative: *Acetyl derivative:* Take the compound in a dry test tube. Add a drop of glacial acetic acid and 1 ml acetic anhydride. Warm, cool and transfer the contents into ice cold water taken in a beaker. A white solid separates out. Filter, wash and dry the solid.

Reactions of primary aromatic amines

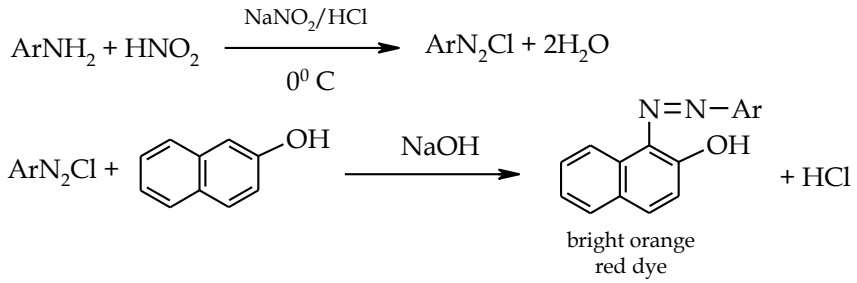
1. Acetylation



2. Carbylamine reaction



3. Diazotisation and coupling



4. Structure of acetyl derivative for primary amines

$$R-NH-CO-CH_3$$

5. Structure of benzoyl derivative for primary amines

$$R-NH-CO-C_6H_5$$

Analysis of IV group organic compounds: *(Phenols, Carboxylic acids and Phenolic acids)*

Experiment	Observation	Inference
1. Compound + saturated $NaHCO_3$ solution.	Mild effervescence takes place.	The compound is an acid.
	No effervescence.	The compound is a phenol.
Tests for carboxylic acids: *2. Esterification:* Compound + 3-4 drops of alcohol + 2-3 drops of conc. H_2SO_4. Heat and pour the mixture containing water.	Fruity odoured compound is formed.	The compound is a carboxylic acid.
3. Compound + water, shake well and add neutral $FeCl_3$ solution.	Purple colouration.	Phenolic acid is confirmed.
	No purple colouration.	It is not a phenolic acid.

Preparation of derivative: Amide derivative for benzoic acid: Take the given compound in a dry test tube, add PCl_5, heat and cool. Add liquor ammonia drop wise carefully. A vigorous reaction takes place with the formation of a white solid. Filter, wash and dry the solid.

Bromo derivative for salicylic acid: Take the given compound in a test tube. Add bromine water and pour the contents in to a beaker containing ice cold water. Filter the white solid that separates out, wash and dry.

Acetyl derivative for salicylic acid: Take the given compound in a dry test tube. Add a few drops of conc. sulphuric acid and 2 ml of acetic anhydride. Heat and pour the contents in a beaker containing ice cold water. Filter the white solid that separates out, wash and dry.

Reactions of carboxylic acid

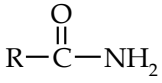
1 Reaction with $NaHCO_3$ solution

$$RCOOH + NaHCO_3 \longrightarrow RCOONa + CO_2 + H_2O$$

2 Esterification

$$RCOOH + C_2H_5OH \xrightarrow[\text{heat}]{\text{conc. } H_2SO_4} RCOOC_2H_5 + H_2O$$

3 Structure of amide derivative of carboxylic acid



Reactions of phenolic acid

1 Reaction with $NaHCO_3$ solution

$$Ar(OH)COOH + NaHCO_3 \longrightarrow Ar(OH)COONa + CO_2 + H_2O$$

2 Esterification

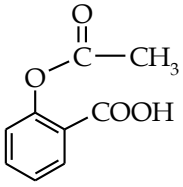
$$Ar(OH)COOH + C_2H_5OH \xrightarrow[\text{heat}]{\text{conc. } H_2SO_4} Ar(OH)COOC_2H_5 + H_2O$$

3 Neutral ferric chloride test

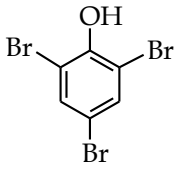
$$6Ar(OH)COOH + FeCl_3 \longrightarrow [Fe(OAr\text{-}COOH)_6]^{3+}.Cl + 6H^+$$

4 Structure of acetyl and bromo derivatives of phenolic acid

(a) acetyl derivative



(b) bromo derivative



Experiment	Observation	Inference
Tests for phenols: 1. Compound + excess of water, shake well and add neutral $FeCl_3$ solution.	Violet (or purple or green or blue) colouration.	The compound is a phenol.

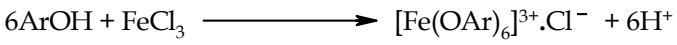
2. *Phthalein fusion test:* Compound + 1 or 2 flakes of phthalic anhydride + 2 or 3 drops of conc. H_2SO_4. Heat till the solid melts and pour a small amount of this to NaOH solution.	Pink (or green or red or blue) colouration.	The compound is a phenol.
3. *Libermann's test:* Compound + a few crystals of sodium nitrite + 3 or 4 drops of conc. H_2SO_4, warm, cool and dilute with water. Add excess of NaOH solution.	Red colouration. Green or blue colouration.	Phenol is confirmed.

Preparation of derivative: *Benzoate derivative for phenol, m-cresol and p-cresol:* Dissolve about 1 ml of phenol in half test tube of 5% NaOH solution taken in a conical flask. Add about 2 ml of benzoyl chloride slowly. Cork the flask and shake well for about 10 minutes. Pour the contents into cold water. Filter the solid, wash and dry.

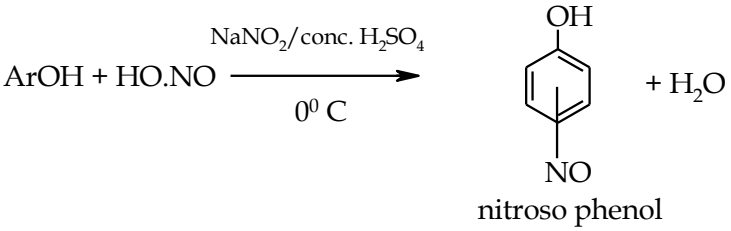
Preparation of picrate derivative for o-cresol*:* Prepare a strong solution of cresol in about 3 ml of benzene and picric acid in about 3 ml of benzene separately. Mix the two solutions and evaporate to dryness. Yellow crystalline solid separates out. Filter, wash and dry.

Reactions of phenols

1 Neutral ferric chloride test

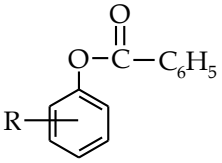


2 Libermann's nitroso reaction:



nitroso phenol

3 Structure of benzoyl derivative



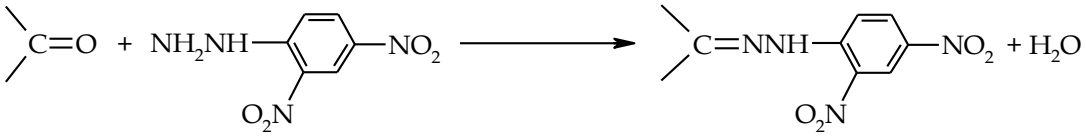
Analysis of V group organic compounds: *(Aromatic alcohol, Aldehydes, Ketones & Esters)*

Experiment	Observation	Inference
Tests for aldehydes and ketones: 1. 2 drops of the compound in a dry test tube + 1 ml of 2, 4-DNP and shake well.	Yellow or orange red solid separates out.	The compound may be an aldehyde or a ketone.
.2. *Schiff's test:* Compound + Schiff's reagent. Shake well.	Pink colouration. No pink colouration.	Compound is an aldehyde. Compound is a ketone.
3. Tollen's test: Compound + Tollen's reagent. Heat the test tube in a water bath without shaking.	A bright silver mirror or a black precipitate. No black precipitate.	Aldehyde is confirmed.
4. Compound + sodium nitroprusside solution + NaOH solution. Shake well.	Red colouration.	Ketone is confirmed. The compound is a methyl ketone (CH_3–C=O).
5. Compound + iodine solution till yellow colour persists + excess of NaOH solution. Heat and cool.	Yellow solid separates out.	Methyl ketone is further confirmed.

Preparation of derivative: Phenyl hydrazone derivative of benzaldehyde and Acetophenone: Take a few drops of the given organic compound in a dry test tube. Add a few drops of glacial acetic acid and about 2 or 3 ml phenyl hydrazine. Shake well. Yellow solid separates out. Filter, wash and dry the solid.

Reactions of aldehydes and ketones

1 Reaction with 2,4-DNP



2 Tollen's test

$$RCHO + Ag_2O \xrightarrow{\text{heat}} RCOOH + 2Ag\downarrow$$

black ppt

3 Fehling's test

$$RCHO + 2CuO \xrightarrow{\text{heat}} RCOOH + Cu_2O\downarrow$$

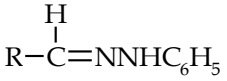
red ppt

4. Iodoform reaction for methyl ketone

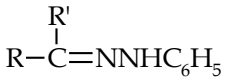
$$RCOCH_3 + 3I_2 + 4NaOH \longrightarrow RCOONa + 3NaI + CHI_3\downarrow + 3H_2O$$

yellow ppt

5. Structure of phenyl hydrazone derivative for aldehydes



6. Structure of phenyl hydrazone derivative for ketones

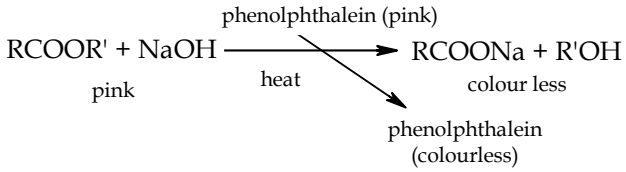


Experiment	Observation	Inference
Tests for Esters: 1. A drop of NaOH solution + excess of water + a drop of phenolphthalein. Add two drops of the compound and warm with shaking. 2. *Hydroxamic acid test:* Compound + alcohol + a few crystals hydroxyl amine hydrochloride + NaOH solution. Boil and cool. Acidify with excess of conc. HCl and add a drop of neutral $FeCl_3$ solution.	Pale pink coloured solution Pink colour gradually disappears. Purple colouration.	 The compound is an ester. Ester is confirmed.

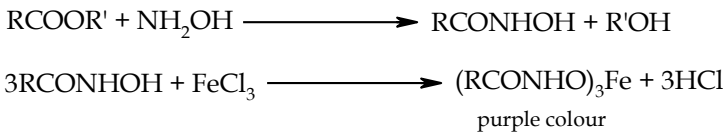
Preparation of derivative: *Acid derivative - Saponification:* Take a few drops of the given compound in a beaker. Add dilute sodium hydroxide solution (add small porcelain bits to avoid spurting). Boil until all the oily drops completely disappear and no pleasant smell persists. Cool and acidify with conc. HCl. A white solid separates out. Filter, wash and dry the solid.

Reactions of esters

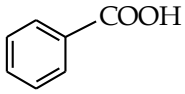
Phenolphthalein test



1 Hydroxamic acid chloride test



2 Structure of acid derivative



Experiment	Observation	Inference
Tests for Alcohols: 1. Compound in a *dry* test tube + a piece of dry sodium metal.	Brisk effervescence takes place and the metal dissolves.	The compound may be alcohol.
Esterification: 2. Compound + few drops of glacial acetic acid + 2 drops of conc. H_2SO_4. Warm and pour the contents into a beaker containing Na_2CO_3 solution.	A fruity smelling compound is formed.	Alcohol is confirmed.

Preparation of derivative: Acid derivative - Oxidation: Take a few drops of the given organic compound in a beaker. Add 10 ml water, a pinch of sodium carbonate crystals and two spatulas of $KMnO_4$ crystals (add small porcelain bits to avoid spurting). Boil until the pink colour is discharged and cool. Filter *the* brown precipitate and acidify the filtrate *(if the filtrate is pink add a pinch of sodium bisulphite crystals to make it colourles)* with conc. HCl. A white solid separates out. Filter, wash and dry the solid.

Reactions of aromatic alcohols

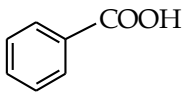
1 Reaction with sodium metal

$$2RCH_2OH + 2Na \longrightarrow 2RCH_2ONa + H_2$$

2 Esterification

$$RCH_2OH + CH_3COOH \xrightarrow[\text{heat}]{\text{conc. } H_2SO_4} RCH_2OCOCH_3 + H_2O$$

3. Structure of acid derivative



Analysis of VI group organic compounds: *(Aromatic hydrocarbons and their Halogen derivatives):*

Experiment	Observation	Inference
Tests for aromatic hydrocarbons: 1. Compound + conc. H_2SO_4. Heat strongly without shaking.	The compound dissolves forming a homogeneous solution.	The compound is an aromatic hydrocarbon.

2. Compound + dimethyl sulphate. Heat the solution with shaking. Add NH_4OH to the solution.	A homogenous solution. A white turbidity.	Aromatic hydrocarbon is confirmed.
Tests for halogen compounds 1. Compound + alcoholic $AgNO_3$ solution. Boil and cool.	White precipitate. No precipitate.	The compound is a side chain halogen compound. The compound is a nuclear halogen compound.

Preparation of derivative: Picrate derivative for naphthalene and biphenyl: Dissolve the given organic compound in 2 ml benzene in a test tube. Dissolve equal amount picric acid in 2 ml benzene in another test tube. Mix the two solutions. Filter the yellow solid and dry.

Nitro derivative for toluene: Take 2 ml of conc. H_2SO_4 in a dry test tube. Add a few drops of the given organic compound, 1 ml conc. HNO_3 and 1 ml conc. H_2SO_4. Shake well for a few minutes and pour the contents into ice cold water. Filter the yellow solid and dry.

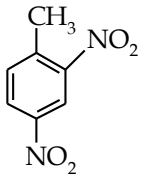
Nitro derivative for chlorobenzene and benzyl chloride: Take 2 ml of conc. H_2SO_4 in a dry test tube. Add a few drops of the given organic compound, 2 ml conc. HNO_3 and 2 ml conc. H_2SO_4. Heat the contents in a water bath for about 10 - 15 minutes. Cool the content and transfer into crushed ice. Filter the yellow solid and dry.

Reactions of aromatic hydrocarbons:

1. **Reaction with concentrated sulphuric acid**

$$ArH + HO.SO_3H \xrightarrow{heat} Ar.SO_3H + H_2O$$

2. Structure of nitro derivative of toluene:



Reactions of halogenated hydrocarbons

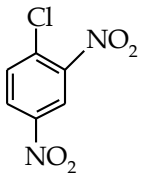
1 Reaction of nuclear halogen derivative with alcoholic silver nitrate:

$$\underset{\text{alcohlolic}}{ArCl + AgNO_3} \xrightarrow{heat} \text{No precipitate}$$

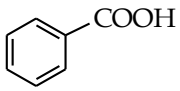
2 Reaction of side chain halogen derivative with alcoholic silver nitrate:

$$\underset{\text{alcohlolic}}{ArCH_2Cl + AgNO_3} \xrightarrow{heat} \underset{\text{white ppt}}{ArCH_2NO_3 + AgCl}$$

3 Structure of nitro derivative for nuclear halogen derivative



4. Structure of acid derivative for side chain halogen compound:



Analysis of VII group organic compounds: *(Amides and Nitro compounds):*

Experiment	Observation	Inference
Tests for Amides:		
1. Compound + 20% NaOH solution, boil.	Pungent smelling gas is evolved. Dense white fumes.	The compound is an amide.
Expose a glass rod dipped in conc. HCl to the fumes.	No pungent smelling gas.	The compound is not an amide. May be nitro compound.
2. Acidify the above solution with conc. HCl.	A white solid separates out.	Amide is confirmed.

Preparation of derivative: *Acid derivative for amide:* Take the compound in a beaker. Add 20% NaOH solution. Heat till the smell of ammonia ceases. Cool and acidify with conc. HCl. A white solid separates out. Filter and dry the solid.

Experiment	Observation	Inference
Tests for Nitro compounds:		
1. Compound + zinc dust + excess of conc. HCl, heat till the compound dissolves and filter. Cool the filtrate in ice for about 2 minutes. Prepare a solution of $NaNO_2$ and cool it in ice for about 2 minutes. Mix the two solutions. Add two drops of this solution to β-naphthol in NaOH solution.	A bright orange red dye is formed.	Aromatic nitro compound is confirmed.
2. *Neutral reduction:* Compound + 10% NH_4Cl solution + zinc dust. Boil for few minutes and filter into Tollen's reagent.	A black precipitate is formed.	It is an aromatic nitro compound.
3. Compound + acetone + NaOH solution. Shake well.	Pink or violet colouration. No change.	It is dinitro compound. It is mononitro compound.

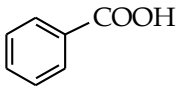
Dinitro derivative for nitrobenzene: Take 2 ml of conc. H_2SO_4 in a dry test tube. Add a few drops of the given organic compound, 2 ml conc. HNO_3 and 2 ml conc. H_2SO_4. Heat the contents in a water bath for about five minutes. Cool and pour the mixture into ice water. Filter the yellow solid and dry.

Reactions of amides

1 Hydrolysis

$$RCONH_2 \xrightarrow[2.\ HCl,\ acidification]{1.\ 20\%\ NaOH,\ heat} RCOOH + NH_3 + HCl$$

2 Structure of acid derivative



Reactions of mononitro compounds

1 Reduction with Zn/dil. HCl

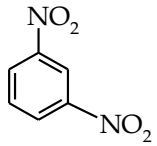
$$ArNO_2 + 6[H] \xrightarrow{Zn/HCl} ArNH_2 + 2H_2O$$

Neutral reduction:

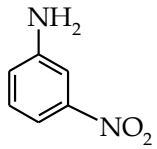
$$ArNO_2 + 4[H] \xrightarrow{Zn/NH_4Cl} \underset{\text{aryl substitued hydroxyl amine}}{ArNHOH} + H_2O$$

$$ArNHOH + 2[Ag(NH_3)_2]OH \longrightarrow \underset{\text{black ppt}}{RNO} + 2Ag + 4NH_3 + 2H_2O$$

Structure of dinitro derivative of mononitro compound:



2 Structure of amino derivative of dinitro compound:



Physical constants

Solids		Liquids	
1. Resorcinol	110 °C	1. Aniline	184 °C
2. Glucose	144 °C	2. Ortho toludine	199 °C
3. Para toludine	44 °C	3. Meta toludine	202 °C
4. Urea	132 °C	4. Phenol	182 °C
5. Benzoic acid	121 °C	5. Ortho cresol	203 °C
6. Salicylic acid	158 °C	6. Meta cresol	202 °C
7. Naphthalene	80 °C	7. Para cresol	203 °C
8. Diphenyl	70 °C	8. Benzaldehyde	179 °C
9. Benzamide	128 °C	9. Acetophenone	202 °C
10. m-Dinitro benzene	90 °C	10. Benzyl alcohol	205 °C
		11. Ethyl benzoate	213 °C
		12. Toluene	110 °C
		13. Chlorobenzene	132 °C
		14. Nitro benzene	210 °C

MODEL PROCEDURE

Systematic identification of the given organic compound

I. Preliminary observations

State : liquid

Colour: colourless

Odour: Smell of Bitter almonds

Experiment	Observation	Inference
Ignition test: A small amount of the organic compound is ignited on a nickel spatula.	The compound burns with a sooty flame.	The compound is aromatic.
Test for unsaturation:		
1. Compound + $KMnO_4$ solution. Shaken well	Pink colour is discharged	The compound is reactive or unsaturated.
2. Compound + bromine water. Shaken well.	Orange red colour is discharged	The compound is reactive or unsaturated.

II. Determination of boiling point of the given liquid

The observed boiling point = 174°C

3% Correction = 5.22 °C

The boiling point of the liquid = 174 °C + 5.22 °C = 179.22°C

III. Detection of elements: Lassaigne's test: (preparation of sodium fusion extract)

A small piece of dry sodium metal is introduced in a fusion tube. It is heated gently until the solid melts into silvery white. It is cooled. A drop of the organic compound is added, heated gradually and strongly until the tube became red hot. It is then plunged into 5 ml distilled water taken in a mortar. Two such fusions are carried out. The contents are grinded with a pestle, transferred into a beaker, heated and filtered. The filtrate is called sodium fusion extract.

Experiment	Observation	Inference
Test for nitrogen: 1 ml of the solution + a few crystals of $FeSO_4$. Boiled, cooled and acidified with dil. H_2SO_4.	No green or blue colouration.	Nitrogen is absent.
Test for halogen: 1 ml of the solution + conc. HNO_3, boiled, cooled and treated with $AgNO_3$ solution.	No white or pale yellow precipitate.	Halogen is absent.
Test for sulphur: 1 ml solution + dil. Acetic acid + lead acetate solutions.	No black precipitate	Sulphur is absent.

IV. Solubility test

Water soluble & ether soluble	Water soluble & ether insoluble	dil. HCl	5% NaOH	conc. H_2SO_4		Compound with N not belonging upto groups III
				soluble	insoluble	
I	II	III	IV	V	VI	VII
–	–	–	–	+	×	×

The given organic compound forms a precipitate with conc. H_2SO_4. Hence it is soluble conc. H_2SO_4 and belongs to V group. The compound may be an aldehyde, a ketone, an ester or an alcohol.

V. Functional group identification

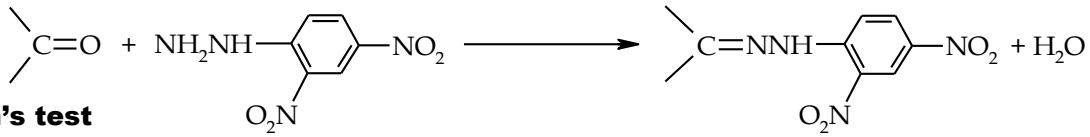
Experiment	Observation	Inference
Tests for aldehydes and ketones: 1. 2 drops of the compound in a dry test tube + 1 ml of 2, 4-DNP and shake well.	Yellow or orange red solid separates out.	The compound may be an aldehyde or a ketone.
.2. *Schiff's test:* Compound + Schiff's reagent. Shake well.	Pink colouration. No pink colouration.	Compound is an aldehyde. Compound is a ketone.
3. Tollen's test: Compound + Tollen's reagent. Heat the test tube in a water bath without shaking.	A bright silver mirror or a black precipitate.	Aldehyde is confirmed.

VI. Preparation of derivative: ***Preparation of derivative: Phenyl hydrazone derivative of benzaldehyde***

Take a few drops of the given organic compound in a dry test tube. Add a few drops of glacial acetic acid and about 2 or 3 ml phenyl hydrazine. Shake well. Yellow solid separates out. Filter, wash and dry the solid.

Reactions of aldehydes and ketones

1 Reaction with 2,4-DNP



2 Tollen's test

$$RCHO + Ag_2O \xrightarrow{heat} RCOOH + 2Ag\downarrow$$

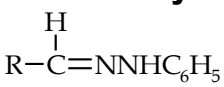
black ppt

3 Fehling's test

$$RCHO + 2CuO \xrightarrow{heat} RCOOH + Cu_2O\downarrow$$

red ppt

4. Structure of phenyl hydrazone derivative for aldehydes



Report : The given organic compound is *an aromatic aldehyde*
Boiling point: 179.22°C.