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Identification of Organic Compound by Organic Qualitative Analysis

A Systematic Scheme for the identification of the organic compound is outlined below.

1. Preliminary Test
2. Solubility Test
3. Detection of Extra Elements
4. Detection of Functional Group
5. Determination of physical constant and Identification of the compound.
6. Confirmatory Test.
7. Derivatives preparation and its m.pt determination .

1. Preliminary Test

Test	Observation	Inferences
(a) Nature	**i)** Solid	Carbohydrate, acid , phenol, amine , higher hydrocarbon may be present.
	ii) Liquid	Alcohol , ketone , aldehyde, ester, phenol, amines may be present.
(b) Colour	**i)** Yellow -Solid	m- Dinitrobenzene, p- Nitro toluene, nitro phenol , nitro aniline.
	ii) Yellow -liquid	Nitrobenzene.
	iii) Brown	P – Toluidine , resorcinol.
	iv) Blackish	α – Naphthol ß – Naphthol
	v) Pink	
	vi) Buff or reddish	Aniline, phenol, Aromatic amine.
	vii) Colourless	Simple acid , alcohol, ester, ketone aromatic hydrocarbon.
(c) Odour	**i)** Carbolic	Phenol, cresol.
	ii) Fishy	Amine
	iii) Sweet pleasant	Ester, alcohol and halogen derivatives.
	iv) Bitter almonds	Nitrobenzene,Benzaldehyde
	v) Moth balls	Naphthalene
	vi) No particular smell	Aromatic acid ,amide, carbohydrate.
(d) Flame Test	**i)** Sooty flame	Aromatic compound or aliphatic compound containing small proportion of hydrogen e.g . $CHCI_3$,CCI_4
	ii) Non sooty flame	Aliphatic compound
	iii) Substance chars	Carbohydrate, sulphanilic acid.
(f) Test for unsaturation ***1*. $KMnO_4$ test.** Substance + 2 ml of water shake well + 2 drops of dilute $KMnO_4$ solution.	**i)** Decolourisation of $KMnO_4$	Unsaturated or easily oxidizable compound.
	ii) No decolourisation	Saturated compound.
***2*. Bromine water Test.** Substance + 2 ml of water shake well + 2 drops of bromine water.	**i)** Decolourisation of bromine water.	Unsaturated compound.
	ii) Decolourisation with formation of precipitate.	Easily substituted compound.
	iii) No decolourisation.	Saturated compound.

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2. Solubility Test

(A) Perform the following test only if the substance is soluble /miscible in water		
Test	**Observation**	**Inferences**
(a). 0.1gm of substance + 3ml of water shake well**.** **Test the solution with litmus paper.**	Substance dissolves	Lower member of alcohol, ester ketone, carbohydrate.
	i) **Blue** litmus paper turns **red**	Water soluble acid or phenol present.
	ii) **Red** litmus paper turns **blu**e	Water soluble base present
	iii) No action on the litmus paper	Water soluble neutral present
(b). 0.1gm of substance + Saturated $NaHCO_3$ solution. Strong effervescence and substance dissolves. To this clear solution add **conc. HCl**	**No solid appear**	Water soluble acid present
(c). 0.1gm of substance + water shake well**,** substance dissolves. To this clear solution add alcoholic $FeCl_3$ Solution.	Blue to violet colour	Water soluble phenol present
(B) Perform the following test only if the substance is insoluble / immiscible in water		
(a) 0.1gm of substance +3 ml Saturated $NaHCO_3$ solution. Shake well. The substance dissolves.	Strong effervescence	Carboxylic acid present.
To this clear solution add conc. HCl drop by drop.	A solid appear	Carboxylic acid confirmed.
(b) 0.1gm of substance +3 ml dilute NaOH solution.,Shake well. The substance dissolves. **To this clear solution add conc. HCl drop by drop.**	A solid or emulsion appear	Phenol present
(c) 0.1gm of substance +3 ml 1:1 HCl solution. Shake well. The substance dissolves. **To this clear solution add 20% NaOH solution drop by drop.**	A solid appear	Base confirmed
(d) If substance is insoluble in **$NaHCO_3$,NaOH, HCl** solution.	**…….**	Neutral compound present

3. Detection of Extra Elements

Sodium Fusion Test (Lassaigne's Test)

1. Take a small piece of dry sodium metal in a fusion tube and heat it gently till the metalmelts or fuses.
2. Add equal quantity of compound to this fused metal [If he compound is a liquid then add two drops of it with a capillary]
3. Heat it gently then strongly till it becomes red hot.
4. Plung the red hot tube in 10 ml or ¾ of a test tube of distilled water taken in a porcelain dish,covering it immediately with an asbestos sheet crush the fusion tube completely.
5. Carry out more fusion in the similar way. Boil the extract for five minutes reduce the volume to about 5 ml and filter. Perform fpllowing test using this filtrate.

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Detection of Elements

Test	Observation	Inferences
Test for Nitrogen 1 ml of extract + 2-3 drops of NaOH solution tomake it alkaline + a few drops of freshly prepared $FeSO_4$solution ,boil for afew minutes ,cool and acidify it with by adding dil.HCl or dil. H_2SO_4.	Blue or green colour solution or Prussian blue coloration.	**Nitrogen present.**
Test for Sulphur **(i)** 1 ml of extract +1 ml of 2N Acetic Acid + 1 ml of Lead Acetate Solution.	A black precipitate.	**Sulphur present.**
(ii) 1 ml of extract + 1 drop of sodium nitroprusside solution.	A violet or purple coloration.	**Sulphur present.**
(iii) 1 ml of extract + 1 ml of aqueous $FeCl_3$ solution.	A blood red coloration	**Nitrog**en and Sulphur are present.
Test for Halogen **(i)** 1 ml of extract +1 ml of dilute HNO_3 (boil well if N and S are present) + 1 ml of 5% $AgNO_3$ solution.	A thick white precipitate.	**Halogen present**.
(ii) If halogen is present carry out the following test: 1ml of extract + 1ml of dilute H_2SO_4 +0.5ml of $CHCl_3$ and 0.5ml of chlorine water ,shake well and observe the colour of chloroform layer.	**(i)** Violet colour	Iodine present.
	(ii) Yellow or brown colour.	Bromine present.
	(iii) Colourless layer	Chlorine present.

Classify the given compound on the Basis of Element Present

On the basis of the elements present in the organic compound ,it belongs to one of the four groups,which may be further divided in subgroups are follows :

Group I : C, H, (O) **(i). Carboxylic acids** **(ii). Phenol** **(iii). Neutrals**	**Group III : C, H, (O),N and S** **(i). Acids** **(ii). Neutrals**
Group II : C, H, (O) and N **(i). Carboxylic acids** **(ii). Phenols** **(iii).Bases** **(iv).Neutrals**	**Group IV : C, H, (O) and Halogen** **(i). Neutrals**

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4. Detection of Functional Groups

Test	Observation	Inferences
Group I : C, H, (O) Carboxylic acids		
(a) 0.1gm of substance +3 ml Saturated $NaHCO_3$ solution. Shake well. The substance dissolves. **To this clear solution add conc. HCl drop by drop.**	Strong effervescence A solid appear	Carboxylic acid present. Carboxylic acid confirmed. **[F.G → —COOH]**
(b) 0.05 gm of compound + 1ml of water , shake well + 1-2 drops of alcoholic $FeCl_3$ solution.	**1.** Buff coloured precipitate	Benzoic acid or phthalic acid.
	ii. Violet coloured precipitate	Salicylic acid
	iii. Violet coloured precipitate obtain on heating the solution	Acetyl salicylic acid
	iv. Yellow coloured precipitate	Cinnamic acid
	v. Faint reddish coloured precipitate	Succinic acid
	vi. Deep yellow coloured solution	Citric acid
	vii. No change in FeCl3 solution.	Oxalic acid
Group I : C, H, (O) Phenol		
(a) 0.1gm of substance +3 ml dilute NaOH solution. Shake well. **To this clear solution add conc. HCl drop by drop.**	Compound dissolves completely. A solid or emulsion appear	Phenol present
(b).0.01gm of compound + 3ml of water or alcohol shake well and add a drop of alcoholic or neutral $FeCl_3$ solution.	i. Violet Colour	**Phenol Present**
	ii. Blue Violet Colour	Resorcinol
	Iii . White ppt slowly changing to pink, blue or violet	α - napthol
	iv. Green Colour	ß - napthol
(c). Phthalein test 0.01gm of compound + 0.01gm of phthalic anhydride + 2 drops of conc. H_2SO_4.Heat gently until the mixture fuses. Cool and pour it in a beaker containing 20 ml ogf very dilute NaOH solution.		
	Pink colour	**Phenol Present**
	Green or bluish green	α - napthol ß - napthol
	Yellowish-green flouresence	Resorcinol
(d).Liebermann test. 0.01gm of compound+1ml of conc. H_2SO_4 + 2 crystal of $NaNO_2$. Heat it gently. Dilute it with water , add 20% NaOH solution.	Red colouration Bluish greenish coloration	**[F.G → —OH (Phenolic)]**

Group I : C, H, (O) Neutrals

Test	Observation	Inferences
(a)Teat for Carbohydrates **Molish Test** : (Perform this test only if the compound is colorless and soluble in water) Dissolve 0.5 gm of the compound in 2ml of water + 2/3 drops of 10% α-napthol dissolved in ethyl alcohol, add carefully 1 ml of conc.H_2SO_4 along the sides of the test tube.	A Violet ring appears at the junction of two layers.	**Carbohydrate present**
(b)Test for Aaldehydes and Ketones. **(i)** 0.05 gm of the compound + 3 ml of 2,4 dinitrophenyl hydrazine. Shake well.	Yellow or orange red crystalline precipitate	**Aldehyde or Ketone Present**
If this test is positive, perform the following test to distinguish between aldehyde and ketone.		
Test for Aldehydes : **(i) Schiff's Test** 0.05 gm of the compound + 2/3 ml of Schiff's Reagent .Shake well.	Viotet colour immediately develops	**Aliphatic aldehyde present**
	Pink colour slowly develops	**Aromatic aldehyde present**
(ii) Tollen's Test OR Silver Mirror test: 0.1 gm of the compd +2-3mlTollents reagent (i.e. Ammonical silver Nitrate solution) + Heat it on a boiling water bath.	A silver mirror is formed on the inner sides of the test tube	**Aldehyde present** **[F.G → −CHO]**
(iii) Fehling Solution Test 0.1gm of the compd + **1ml Fehling A** + **1ml Fehling B** solution .Heat it gently	Formation of red ppt of Cuprous oxide	
(iv) Benedict's test 0.1gm of the compd + Benedicts solution + Heat it gently.	Formation of red ppt Cuprous oxide	
Teat for Ketones : 0.1gm of compd + 2ml of sodium nitroprusside solution + 2 drops of NaOH	Wine Red colour or Orange red colour CH_3-CO- gr gives this test	**Ketone present** **[F.G → C = O]**
(c). Test for Esters Dissolve 0.1gm or 0.5ml of compound in 1 ml of ethyl alcohol + a drop of phenolphthalein + 2 drops of very dilute NaOH solution. Heat on a boiling water bath.	Pink colour disappears	**Ester present**
(d).Test for Alcohols (i). Take a small piece of dry Na metal in a fusion tube and add a few drops of compound.	Rapid evolution of H_2	**Alcohol present**
(ii). 1ml of acetyl chloride in a dry test tube + drops of the compound.	Strong effervescence	**Alcohol present**
e). Test for Hydrocarbons 0.5ml of compound + 1 ml of iodine in carbon disulphide, shake well	Purple colour of CS_2 layer changes to brown colour	**Ether present**
(f). Test for Hydrocarbons 0.1gm of compound + 1-2 ml of water, shake well + 1-2 drops of very very dilute $KMnO_4$ solution. Shake again.	If all the above tests fail	**Hydrocarbon present**
	Decolourisation	**Unsaturated hydrocarbon present**
	No decolourisation	**Saturated hydrocarbon present**

Group II : C, H, (O) and N Carboxylic acids

Test	Observation	Inferences
i) 0.1 gm of the compd + 2/3 ml of sat $NaHCO_3$ solution. Shake well . To this clear solution add conc. HCl drop by drop	Strong effervescences and compound dissolves.	**Nitro-Carboxylic acid or amino carboxylic acid present**
	Solid appears	**Nitro-Carboxylic acid present**
	No solid appears	**Amino carboxylic acid present**
ii) If nitro carboxylic acid is present, perform the following test of nitro group – **a)** Test for nitro group – 0.2 gm of the compd + 2 ml of ethyl alcohol + 0.1 gm of solid NH_4Cl + 0.1 gm of Zn dust. Boil for 5min and filter. Filter + Tollen's Reagent	Black or grey precipitate	**Nitro group present.** **[F.G → $-NO_2$]**
iii) If amino carboxylic acid present ,perform the following test for amino gr- 0.5 gm of the compd +3 / 4 ml of 1:1 HCl, shake well. Cool and add few drops of 2% $NaNO_2$ solution	A clear solution is obtained which when added to a cold solution of alkaline ß-napthol give orange red dye	**Aromatic primary amino group is present** **[F.G → $-NH_2$]**

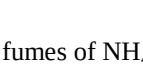
Group II : C, H, (O) and N Phenols

i)0.1 gm of the compd + dilute NaOH solution Add conc HCl drop by drop	Compd dissolves producing deep yellow or orangecolour	**Nitro phenol or Amino phenol is present**
	Solid reappears	**Nitrophenol present**
	No solid reappears	**Aminophenol present**
ii) If nitro phenol present perform the test for $-NO_2$ group as written above.	Black or grey ppt	**Nitro group present**
iii) If Aminophenol present, perform the test for $-NH_2$, group as written above.	Orange red dye	**Aromatic Primary amino group present**

Group II : C, H, (O) and N Bases

0.5 gm of the compd +3 / 4 ml of 1:1 HCl, shake and filter. Filtrate + 20% NaOH drop by drop	Compd dissolves	**Amines present**
	Solid reappears or emulsion obtained	**Amines confirmed**
0.5 gm of the compd +3 / 4 ml of 1:1 HCl, shake well. Cool and add few drops of 2% $NaNO_2$ solution	Deep yellow, red or green colour	**Aromatic tertiary amino group (-N-) present**
	White or yellow emulsion	**Aromatic secondary amino group (-NH-) present**
	A clear solution is obtained which when added to a cold solution of alkaline ß-napthol give orange red dye	**Aromatic primary amino group ($-NH_2$) present**

Group II : C, H, (O) and N Neutrals

Test	Observation	Inferences
(1).Test for Diphenylamine 1/2 crystal of compd + 1 ml of conc.H_2SO_4 + Conc HNO_3. or one drop of dilute $NaNO_2$ solution (If this test is positive,perform group test listed under C,H,(O) and N Basic to confirm the functional group	Deep Blue Colour	**Diphenylamine present**
(2) Test for Amides 0.2 gm of compound + 3ml of 20% NaOH sol .Boil for 1-2 min.Test the gas evolved -	Evolution of NH_3(confirm with the smell)	**Amide group present**
i). Bring a moist red litmus paper over the mouth of test tube	Red litmus paper turns blue	**Amide group confirmed**
ii). Dip a glass rod in conc HCl and hold this over the mouth of the test tube	Dense white fumes of NH_4Cl are evolved on the glass rod	**[F.G** →  **]** **Amide**
(3) Test for Anillide 0.1gm of the compd + 1ml of conc HCl. Boil for 2 min, cool and add 5 ml of water + a few drops of cold $NaNO_2$ sol. And mix well. Add this solution to a cold solution of alkaline ß-napthol.	Orange red dye	**Anilide Group present** **[F.G → Ar -** NH – CO – **R]** **R** → $-CH_3$, $-C_6H_5$
(4). Test for Nitro group **(a). <u>Mulliken's Test</u>** 0.2 gm of the compd + 2 ml of ethyl alcohol + 0.1 gm of solid NH_4Cl + 0.1 gm of Zn dust. Boil for 5min and filter. Filter + Tollen's Reagent	Black or grey precipitate	**Nitro group present.** **[F.G → – NO_2]**
(b). <u>Azo-Dye Test</u> (If primary amino group absent, then only perform this test) 0.5 gm of the compd + 0.5 gm of Tin metal + 2 ml of conc HCl. Boil or 3 min, cool filter and dilute with about 5 ml of water +few drops of $NaNO_2$. Add this sol to a cold solution of alkaline ß-napthol.	Orange red dye	**Nitro group present.** **[F.G → – NO_2]**
(c). <u>Test for Dinitro compound</u> 0.5 g of the compound + 1-2 ml of acetone, shake well to dissolve the com pound+ 1-2 drops of dilute NaOH solution.	Dark purple or Violet colour	**Dinitro compound present**

Group III : C, H, (O) and N & S - Acids

This class includes amino sulphonic acid

1) Test for Amino Sulphonic Acid **a)** 0.2 gm of the compound + 3 -4 ml of saturated $NaHCO_3$ solution.	Effervescences and compound dissolves	**Amino Sulphonic acid present**
b) Perform test for **–NH_2 group** as above.	**Orange red dye** is formed	**Aromatic primary amino group present**

Group III : C, H, (O) and N & S - Neutral		
This class includes Thiourea		
Test for Thiourea Thiourea is soluble in water and neutral to litmus paper 0.1 gm of comp + 2ml oof 20% NaOH solution, boil ,cool and add a few drops of lead acetate solution.	Black ppt is obtained.	**Thiourea is confirmed** **[F.G** → S ‖ − C - **NH_2]** **Thioamide**
Group IV : C, H, (O) and Halogen - Neutral		
0.2 gm of compd + 2-3 of ml of dilute NaOH solution Boil for a few min, cool + dilute HNO_3 till acidic + 1ml of $AgNO_3$ solution.	White or yellow ppt No Precipitate.	Aliphatic halide like $CHCl_3$,CCl_4 present Chlorobenzene or bromobenzene present

5. Determination of physical constant and Identification of the compound.

Take organic compound into one end sealed capillary and determine m.pt /B pt

6.Confirmatory Test

Test	Observation	Inference

7.Derivatives preparation and its m.pt determination .

Results.

From the above step by step analysis of given organic compound shows that ,it is

State :
Colour :
Odour :
Aromatic / Aliphatic :
Saturated /unsaturated :
Solubility :
Extra elements :
Functional group :
M.pt/B . pt