

RPS DEGREE COLLEGE

BALANA (MAHENDERGARH)-123029



Lab Manual

Chemistry (B.Sc.3rd & 4th Semester)

Department of Chemistry

ORGANIC CHEMISTRY

1. To analyze the given organic compound in a systematic way i.e. detection of extra element ,functional group , determination of melting point , preparation of solid derivatives of following compounds : Naphthalene, oxalic acid, β -Naphthol ,benzophenone , phthalic acid , aspirin, p-nitro toluene, anthracene ,m-dinitrobenzene , benzyl chloride , glucose, fructose, urea, cinnamic acid , benzamide.

EXPERIMENT:-1

AIM :- Detection of organic compounds.

I. PRELIMINARY TESTS FOR ORGANIC COMPOUNDS

PHYSICAL CHARACTERSTICS

(a) Colour – from the colour of organic compounds following observation can be drawn

Experiment	Observation	Inference
(a) Note the colour of compound	I. Yellowish II. Pale yellow liquid III. Deep orange yellow	Nitro compounds Nitrobenzene Nitro aniline, nitrophenols.

(b)Odour:- From the odour of compound following observation can be drawn

Experiment	Observation	Inference
(a) Note the odour of	(a) Fruity smell	Esters

the compound	(b) Vinegar like smell (c) Pungent smell (d) Wine like smell (e) Fishy smell	Acetic acid Benzoyl chloride Alcohol Amines
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(c) Ignition Test:- On burning organic compounds give following information

Experiment	Observation	Inference
(a) Take a small amount of compound on a nickel spatula and heat.	a) Burns with a smoky flame. b) Burns with non smoky flame. c) Burnt with sugar smell. d) Ammonical smell. e) Irritating smell with coughing. f) rotten eggs smell	Aromatic compounds Non aromatic compounds Carbohydrates Urea Benzoic acid, succinic acid, salicyclic acid Sulphur present

(d) Solubility Test:- The organic compound either dissolved in water or NaOH or dil. HCl gives following information

Experiment	Observation	Inference
a) Take a small amount of compound in a clean test tube and add 3 – 4 ml water. Shake well. b) Test the compound with litmus. c) Test the solubility in dil. HCl d) Test the solubility in NaOH	a) Sparingly soluble or insoluble b) Soluble c) Turns blue litmus to red d) Turns red to blue e) Soluble and reprecipitated on adding alkali f) Soluble but reprecipitated on adding acid	Hydrocarbons, esters, ethers etc. Lower alcohols, aldehydes, ketones, acids etc. Acids, phenols Lower amines Base Phenol

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PRELIMINARY CHEMICAL TESTS

1. **Sodalime test:-** On mixing organic compound with sodalime following observation are seen

Experiment	Observation	Inference
a) Take about 0.5 gm of solid compound with 2 gm of sodalime (NaOH + CaO) in a test tube and heat it	a) Ammonical smell b) Burnt sugar smell c) Smell of phenol d) Smell of bitter almonds	Urea, acetamide Carbohydrates Phenolic acid Benzaldehyde derivatives

2. **Ferric chloride test:-** with ferric chloride following observation are seen

Experiment	Observation	Inference
a) Dissolve 0.5 gm of compound in 2 – 3 ml of water and add 4- 5 drops of neutral FeCl ₃ solution	(a) Violet (b) Blue (c) Blue violet (d) White ppt. changing to violet	Phenols, salicyclic acid P-cresol Resorcinol, m-cresol α -naphthol

3. **Conc. H₂SO₄ test:-**

Experiment	Observation	Inference
a) Heat 0.5 gm of compound with 2 – 3 ml of conc. H ₂ SO ₄	a) Dissolves with violent reaction. b) Soluble in cold water. c) Dissolves without charring and CO is given out. d) Charring with no gas is evolved. e) Dissolves	Alcohols Ethers Formic acid or oxalic acid Aldehydes, ketones and aromatic hydroxy acid Urea, oxalate

	without charring and CO ₂ is given out.	Aromatic
	f) Dissolves slowly but not precipitated on dilution.	Carbohydrates
	g) Dissolves on heating with charring CO ₂	

4. Sodium bicarbonate test:-

Experiment	Observation	Inference
a) Mix the compound in water and add solid NaHCO ₃	a) Evolution of CO ₂ with effervescence.	Acid
	b) Dissolves with no evolution of CO ₂ gas	Phenols

5. Action of KMnO₄ solution:-

Experiment	Observation	Inference
a) Add a very dil. Solution of KMnO ₄ and dil. H ₂ SO ₄ to the solution of compound	a) Decolorised immediately	Unsaturated compounds
	b) Decolorised slowly	Aldehydes

DETECTION OF EXTRA ELEMENTS

1. For detection of extra elements we have to prepare Lassaigne's extract

a. Preparation of lassaigne's extract:-

Cut small pieces of dry sodium metal into ignition test tube now heat it till the pieces changes to silver globule. Now add a pinch of organic compound into this test tube again heat it till the tube becomes red hot. Now take a china dish containing 10 ml water now break the hot tube into this china dish. Now the solution is heated and filtered. The filtrate is known as lassaigne's extract. (L.E.) or sodium extract (S.E.)

Experiment	Observation	Inference
a) Test for nitrogen:- To about 2ml of the S.E., added a few drops of NaOH	No deep blue or blush green colour	Nitrogen absent

ml alcohol and 2 -3 drops of conc. H ₂ SO ₄		
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2. Test for phenolic group:-

Experiment	Observation	Inference
a) Litmus test:- add few drops of blue litmus solution to aqueous compound	a) Blue litmus turned red	-COOH or Phenolic group present
b) Ceric ammonium nitrate test:- To aqueous solution of organic compound add few drops of ceric ammonium nitrate.	b) Brown ppt.	Phenolic group confirmed

3. Test for Ketonic group $>C=O$:-

Experiment	Observation	Inference
a) Sodium nitroprusside test:- add a pinch of organic compound in alkaline sodium nitroprusside solution.	a) Presence of red colour	Presence of $>C=O$ group
b) Schiff's reagent test:- add small amount of organic compound to 1 – 2 ml of schiffs reagent and shake.	b) Pink colour	Presence of $>C=O$ group Or Aldehyde present

4. Test for Carbohydrates:-

Experiment	Observation	Inference
a) Molisch test:- add few drops of alcoholic α -naphthol solution to about 1 ml of aqueous solution of organic compound	a) Deep violet ring at the junction	Carbohydrates present
		Carbohydrates

b) Conc. H_2SO_4 test:- add about 1 ml of con. H_2SO_4 to the given compound and warm.	b) Charring	present
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5. Test for esters:-

Experiment	Observation	Inference
a) Hydrolysis Test:- To about 1 ml of substance, a drop of phenol phthalein and few drops of dil NaOH are added. Heat the contents on a water bath.	a) Pink colour disappears	Ester group present

6. Test for amide ($-\text{CONH}_2$) group:-

Experiment	Observation	Inference
a) HNO_2 test:- add a pinch of organic compound to ice cold solution of NaNO_2 and dil. CH_3COOH	a) Effervescence	$-\text{CONH}_2$ group present
b) NaOH test:- Heat a small amount of compound with NaOH solution	b) NH_3 (g) Evolved	$-\text{CONH}_2$ group present

7. Test for $-\text{NH}_2$ group:-

Experiment	Observation	Inference
a) HNO_2 test:- To ice cold solution of compound in dil. HCl . Add few drops of saturated NaNO_2 solution.	a) Brisk effervescence	Aliphatic $-\text{NH}_2$ group present
b) Dye test:- To about 0.5 g of compound add 2-3 ml of water	b) Orange-red dye	Aromatic D($-\text{NH}_2$) group present

containing 1 ml of conc. HCl. Then cool the contents in ice, add 2 ml of 10% NaNO ₂ solution dropwise with constant stirring then add ice cold solution of alkaline β -naphthol		
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8. Test for anilides:-

Experiment	Observation	Inference
a) NaOH Test:- Heat a small amount of compound with 1-2 ml concentrated solution of NaOH	a) Peculiar smell	-NHCOR (anilide) group present

9. Test for –NO₂ group:-

Experiment	Observation	Inference
a) Dye Test:- Heat the given compound with 1 ml of conc. HCl and few pieces of granulated tin and heat the mixture for about 5 minutes in a boiling water bath. Filter and cool the filtrate in ice bath, then add 1, 2 – ml of NaNO ₂ solution followed by 1, 2 – ml of ice cold alkaline β -naphthol solution.	a) Orange red	-NO ₂ group

10. Test for halogen group:-

Experiment	Observation	Inference
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a) Boil about 0.2 or 2-3 ml of compound with 2-3 ml of KOH or NaOH (alc.) for 5 minutes. Cool and add dil. HNO ₃ and AgNO ₃ .	a) Ppts. Formed	Halogen group is present
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11. Test for –OH (alcoholic) group:-

Experiment	Observation	Inference
a) Ester test:- add 1g of CH ₃ COONa to about 1-2 ml of compound and then 2-3 drops of conc. H ₂ SO ₄	a) Fruity smell	Alcoholic group present
b) Ceric ammonium nitrate test:- To few drops (10-15), add 2 ml of ceric ammonium nitrate solution	b) Red or pink colour	Alcoholic group confirmed

12. Test for – CHO group:-

Experiment	Observation	Inference
a) Fehling's solution test:- add a small amount of organic compound to 2-3 ml of fehling's solution. Heat the contents on a water bath.	a) Red ppts.	CHO group present
b) Tollen's reagent test:- Warm 4-5 ml of Tollen's reagent with small amount of organic compound on a water bath.	b) Silver mirror is formed	-CHO group present
c) Sodium bisulphite test:- To about 1		-CHO group

ml of organic compound, add 1-2 ml of saturated sodium bisulphite solution.	c) White ppt.	$> C = O$ group present
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PREPARATION OF SOLID DERIVATIVES

1. DERIVATIVES OF PHENOLS:-

- a. Picrates: - Mix equal amounts of saturated solution of compound and picric acid in benzene (say 3 ml each). Shake the contents vigorously. Filter the precipitates formed and recrystallise from benzene (if required).

2. DERIVATIVES OF CARBOXYLIC ACIDS:-

- a. S-Benzylisothiuronium salts :- Dissolve 0.5 g of given compound in 5-6 ml of water by heating. Add a drop of phenolphthalein indicator and then add NaOH till it becomes pink. Add 1-2 drops of HCl. Dissolve approximately 2.0 g of S-Benzyl isothiuronium chloride in 5-6 ml of water. Mix both the solution stir and cool the precipitates formed are filtered and crystallized from hot water.

3. DERIVATIVES OF CARBOHYDRATES:-

- a. Osazones :- Shake about 1 g of compound, 2 g of phenyl hydrazine hydrochloride and 3g of sodium in a boiling water bath. Yellow mass separates out, recrystallise from alcohol. Glucose, fructose and sucrose form osazone in approximately 2, 5 and 30 minutes respectively.

4. DERIVATIVES OF ALDEHYDES AND KETONES:-

- a. 2,4- Dinitrophenyl hydrazone: - Add approximately 5 ml of 2, 4- Dinitro hydrazine to 1g or 1ml of the compound in a dry test tube. Then add 1-2 drops of conc. H_2SO_4 Shake the contents and heat for few minutes. Cool, filter and recrystallise from alcohol or benzene.

