

SUBJECT: PHARMACEUTICAL ORGANIC CHEMISTRY- I

II SEMESTER B.PHARM (1st YEAR)

PRACTICAL LAB MANUAL

EXPERIMENT: 1

DATE:

BASIC LABORATORY APPARATUS

AIM: To observe and write description of various apparatus used in lab
For various experiments.

Apparatus:

Beaker, Conical flask, Burette, funnel, Separating funnel, Buchner funnel, volumetric flask, Specific gravity bottle, Round bottom flask, Reflux condenser, Watch glass, Measuring cylinder, China dish.

Observation:

All the apparatus have their own use with some advantages and disadvantages. The observation done with some description this apparatus is given as follows:

i) Beaker:

It is a cylindrical glass material with an opening which is circular and marking level of volume of substances taken it is also given.

Uses: It is used to make solutions of definite volumes.

It is used in some experiments to evaporate some volatile substances.

ii) Conical flask:

It is made up of glass, which is in conical shape. It have broad at base and becomes narrow to the top, and the narrow end is like a tube. It also has some marking of definite volumes.

Uses: It is used in titrations. It is used in performing the experiments which needs more space & less exposure

iii) Burette:

It is a long cylindrical glass tube, with measured units on it whose end is narrow tip with a knob.

Uses: It is mainly used in titration experiments in which are reagent is taken in it standardized with other in conical flask.

It is used to get volumes of unknown weight of one of the reagent by taking average values.

Precautions: Before using any burette first it should be washed with water, and then it

should be rinsed with the reagent which is going to be added in it.

iv) Funnel:

It is a conical structured glass, whose open ends vary in diameter, the small open end is continued as tube like.

Uses: It was used in transferring of substances easily into a narrow apparatus like conical flask, Burette etc., used in filtering the reagents.

Disadvantage: The substances are not completely filtered out.

v) Separating Funnel:

It is a conical glass, consists of a funnel with an opening of two ends. The upper end is large from it enlarges, going narrow it to the Bottom. It has a knob to let out of the substance near the out let.

Uses: It is used in the separation of two ends immerse the liquid, liquid like oil & water. At first we put liquid in the funnel and shake gently, since the density of oil is less it floats on water surface and then water is filtered using bottom knob and oil will remain in the separating funnel.

vi) Buchner funnel:-

It is a funnel made up of porcelain & has perforated plate to support a filter paper. It is a funnel which filters substances through pressure the side arm of the funnel is attached by means of thick walled rubber tubing via a suitable top to a water pump.

Uses: It is more advantageous than simple funnel because in Buchner funnel due to suction all the water amount is taken away so dried extract can be obtained.

vii) Volumetric flask:-

It is a long necked flask with an enlarged round bottom.

Uses: It is used in making definite volume of a substance with a definite weight of substances. It is used in volumetric titration reactions.

viii) Specific gravity bottle:-

It is a small specific bottle which is a narrow neck & a pear shaped body.

Uses: It is a very small bottle used to weight any substances. First weight of specific gravity bottle is weighed. Then the substance is added and then total weight is measured. Then weight of the bottle is subtracted from the total weight. Thus the weight of the substance is known.

IX) Round Bottom flask (RBF):-

It is a flask with round bottomed and short necked.

Uses: It is a flask which is used to heat at very high temperatures.

X) Reflux Condenser:-

It is a long glass tube which has two openings up & down which are connected to tap and another out slid. It is structured in such a way that heat is minimum released.

Uses: It is used to condenser the reaction going on in RBF at a very high temperature. The precise design of condensers depends up on the volatility of the reaction liquids, low boiling liquids ($< 60^{\circ}\text{C}$) require the use of the double surface condenser.

XI) Watch Glass:

It is used for keeping powders and used in mixing solids watch glass is used to carry out some experiments. It has a concave surface.

XII) Measuring Cylinder:-

It is used to measure the solution or liquid reagents during various reactions; it is a glassy apparatus with scale on it.

XIII) China dish:

It is used to take test solutions and powdered reagents. It is also used to melt or heat solid reagents.

REPORT:

EXPERIMENT: 2**DATE:****DETERMINATION OF MELTING POINT****Aim:**

To determine the melting point of given sample.

Apparatus:

Thermometer, capillary tube, Burner, Stand, Thread

Chemicals Required:

Liquid paraffin wax, and sample substance

Principle:

Melting point is defined as the temperature at which solid becomes into liquid substances under a pressure of one atmosphere is called melting point.

Melting point is determined one of the most common technique used to characteristic the organic compound and to check the state of purity. Melting point of a crystalline solid is the temperature at which solid begins to change into liquid state. The purity of the compound has sharp melting point due to which the change from solid to liquid is quick.

Impure sample has lower melting point than that of pure. Its melting range is wide. Both temperature and sharpness of the melting (range) point are the useful criteria of purity.

Procedure:

One end of the capillary tube is sealed by heating, it in the non-luminous portion of the flame as well as continuously rotating heating until it is closed. The open end of the capillary tube is pushed into a small amount of completely dried and finally powdered organic compound which is under examination. The powder is shaking by tapping the sealed end of the capillary tube on the bench. The procedure is repeated until the length of the powder material is 3-4 mm outside of the capillary tube wiped clean.

A thermometer is inserted into a one hold rubber stopper. The capillary tube is tied to the thermometer with a rubber band and a thread. The capillary tube is tied in such a way that it's sealed end & indirect contact with the bulb of thermometer.

The tube is filled with liquid paraffin. The thermometer with the capillary tube is immersed in liquid paraffin in such a way that the open end of the capillary tube and rubber band should be above the level of the liquid

paraffin the side arm of the tube is heated at a uniform rate. The flame of the burner & adjusted in such a way that the temperature at which the last crystal is disappeared and this melting point is reported.

Report:

The given sample melts at

EXPERIMENT: 3

DATE:

DETERMINATION OF BOILING POINT

Aim:

To determine the boiling point of given sample.

Apparatus:

Distillation flask, thermometer, stand, burner

Principle:

Boiling point of the liquid is the temperature at which liquid begins to boil and gets converted into its vapor form. This is usually a characteristic of liquid or solvent in its pure form.

Boiling point involves breaking of oppositely charged ions. This occurs when temperature is reached at which thermal energy of the particle is great enough to overcome cohesive force that hold the molecules. Generally when reasonable amount of liquid compounds are available boiling point is determined by slowly distilling the material from a sphere shaped flask & regarded the boiling point at the temperature at which the liquids starts distilling for small quantity of liquid. The material should be distilled using boiling point apparatus.

Procedure:

Transfer the given liquid into a distillation flask and add 1 or 2 fragments of porcelain. Arrange the apparatus in such a way that the bulb of the thermometer should be in the centre of the flask & slightly below the side tube heat the flask from a flame and adjust the flame in such a way that the distillate is collected at the rate of 1 or 2 drops / Sec. The temperature will rise rapidly until it is near the boiling point of the liquid then slowly and finally it remains constant. Record the temperature when it remains the constant. Collect the liquid and continue distillation until only a small volume of liquid remains in the flask. Observe the boiling point.

Report:

The given liquid boils at ...

Systematic general procedure for analysis of unknown organic substance:

No.	Experiment	Observation	Inference
I – Primary reaction			
1. Physical stage :		a. Solid	May be aromatic acid or aromatic amines or carbohydrate
		b. Liquid	May be ester or aldehyde or ketone
2. Color :		a. Colorless or white	May be acid or ester or aldehyde or ketone or alcohol or carbohydrate or hydro carbon
		b. Reddish brown or dark color	Aromatic amine may be presence
3. Odor :		a. Pleasant smell	May be ester or ketone
		b. Phenolic smell	May be phenol or Napthol
		c. Aniline like smell	May be aromatic amine
		d. Alcoholic like smell	May be alcohol
		e. Pungent smell	Aliphatic acid may be present
		f. Ammonia smell	Aliphatic amine may be present
4. Solubility : A small quantity of the substance is taken well with 5ml of distill water & if the substance dose not dissolve is heated to boil. The solution is then tested with litmus paper.		a. soluble in cold water	May be carbohydrate, di carboxylic acid (or) urea
		B. Soluble in hot water and re-crystallisation on cooling.	Aromatic mono carboxylic acid may be present.
		c. The solution answers to litmus paper.	Acid or phenol may be present.

5. Reaction with sodium hydroxide :

Take a small quantity of the substance & shake with 5ml of 20% sodium hydroxide solution & then heated to boil.

A. Soluble in cold alkali & reappearance on acidifying.	Aliphatic or aromatic acid or phenol may be present.
b. Dissolves boiling for few minutes	Ester or anhydrous may be present.
c. Turns yellow or brown on boiling.	Aldehyde or carbohydrate may be present
d. The mixture on boiling evolves ammonia.	Amides or diamides may be present.
e. The color of the solution turns yellow to red.	Nitro phenol may be present.

6. Reaction with dil. Hydrochloric acid : Take a small quantity of the substance in a dry test tube & shake with 1ml hydrochloric acid.	Dissolved & reappearance on acidifying.	Basic substance may be present.
7. Reaction with Concentrated Sulphuric acid : A small quantity of the substance is treated with Conc. Sulphuric acid in a dry test tube & heat gently.	The mixture chars with the smell burnt sugar with effervescence.	Sugar may be present.
8. Reaction with Soda lime : About 2g of substance is mixed with 2g of soda lime heat the mixture strongly & gently in test tube.	a. Ammonia vapor is evolved.	a. Amid or urea may be present.
	b. Aniline vapor is evolved.	b. Aniline may be present.
	c. Charring with the smell of burnt sugar.	c. Carbohydrate may be present.
9. Reaction with neutral ferric Chloride : To aqueous solution of the substance add few drops of neutral ferric chloride solution.	a. Solution turns blue, green or violet color.	a. Phenol may be present.
	b. Buff color precipitate.	b. Aromatic acid may be present.
	c. No Characteristic change.	c. Absence of Phenol & Aromatic acid.
[Test for Saturated & Unsaturated]		
1. Reaction with bromine water: A small amount of substance shaken with a few ml of freshly prepared bromine water.	a. Bromine water is decolorized & no precipitate is form.	a. Unsaturated compound may be present.
	b. Bromine water is decolorizing with a formation of precipitate.	b. Phenol or aromatic amine may be present.

	c. No decolourisation	c. Saturated compound may be present.
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2.	Reaction with alkaline Potassium per manganate: A small quantity of the substance is added with alkaline Potassium per manganate & shakes well.	a. Permanganate color decolorizes.	a. Unsaturated compound or aldehyde or reducing sugar may be present.
		b. No decolourisation.	b. Saturated compound may be present.

[Test for aliphatic or aromatic Compound]		
1. Flame test : A small quantity of the substance is heated in Nickel Spectrum.	a. Burnt with luminous flame.	a. Aliphatic compound may be present.
	b. Burnt with luminous Smokey flame.	b. Aromatic or highly unsaturated aliphatic compound may be present.
	c. Burnt with the smell of turn's sugar.	c. Carbohydrate or hydroxyl acid may be present.
	d. Burnt with green color flame.	d. Halogen may be present.
2. Nitration Test : To a small quantity of the substance add 5ml of Conc. Sulphuric acid and 5ml of Conc. Nitric acid. In dry test tube, keep test tube in boiling water both for few minutes. The test tube is cooled & the condense is pour into a cold water in a beaker.	a. Yellow oil precipitate separate out.	a. Aromatic compound may be present.
	b. No yellow oil precipitate.	b. Aliphatic compound may be present.

SPECIAL ELEMENTS [N, S, Halogens]:

Lassaignes Test:

Preparation of Sodium fusion extract melt a small piece of dry sodium metal in a small fusion tube add 1g of the substance [3 drops of the liquid] in a fusion tube & heat gently at first then to red hotness & quickly and carefully plunge red hot end of the tube in 10ml of the distill water in a china dish.

Stir the solution and boil for few minutes & filter through a filter paper. This filtrate is a sodium fusion extract.

1.	Test for Nitrogen : To a few ml of extract add a drop of freshly prepared ferrous sulphate [Crystal Salt are can be used] boil the solution & cool add 2ml of dilute sulphuric acid.	Prussian blue or green color is precipitate.	Nitrogen is present.
2.	Test for Halogen To 1 ml of the fusion extract and add 1ml of dilute Nitric acid boil & then cool. Then add 1 drop of Silver nitrate solution.	a. White precipitate [soluble in ammonium hydroxide]	a. Chloride is present.
		b. Pale yellow precipitate [partially soluble in NH_4OH]	b. Bromide is present.
		c. Deep yellow precipitate [insoluble in NH_4OH]	c. Iodine is present.
3.	Test for Sulphur : a) Lead acetate Test : To 1ml of fusion extract add 1ml of dilute Acetic acid & few drops of lead acetate solution are added.	Black precipitate is obtained.	Presence of Sulphur.
	b) Sodium Nitro prusside: To 1ml of fusion extract add 1ml of freshly prepare sodium nitro prusside is added.	Violet color is obtained.	Presence of Sulphur.

B. Neutral test solution: 0.5g or 0.5 ml of compound + 1ml water +1 drop of phenolphthalein solution + 0.2 ml of ammonia solution then boil to remove excess of ammonia, cool and add few drops of aqueous ferric chloride solution.	Buff color precipitate Yellow color precipitate Violet color precipitate Faint reddish color Deep yellow color solution No change in color Red color	Benzoic acid or phthalic acid Cinnamic acid Salicylic acid Acetyl salicylic acid Succinic acid or acetic acid Oxalic acid Acetic acid
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[Test for Functional group]			
	1. Test for acids : 0.2g or 0.2 ml of the substance +2-3ml saturated sodium bicarbonate solution.	Effervescence of CO ₂ and then substance dissolves. Reappearance of the substance by adding dilute HCl. (till acidic)	Carboxylic acid is present

A. Esterification Test : To 1g of the substance add 2ml of alcohol & add 5drops of Conc. Sulphuric acid. Heat this mixture on a boiling water bath and pour into the beaker containing sodium carbonate solution.	A fruity odor obtained.	Presence of carboxylic acid confirmed
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C. Fluorescein test: 0.2 g compound + 0.2 g resorcinol+2drops of concentrated sulphuric acid in dry test tube. Heat it and add dilute NaOH solution drop wise in excess.	Bright green color of fluorescence	Phthallic acid is confirmed
2. TEST FOR AMIDES/IMIDES AND NITRILES: A. Alkaline hydrolysis test: 0.2g or 0.2 ml of compound + 5 ml of 10% NaOH solution and boil it	Evolution of ammonia which can be tested with a rod dipped in concentrated Hcl, white fumes occur	Amide, imide or nitrile group is confirmed.
B. Hydroxamic test: 0.2g of compound +1 ml hydroxylamine Hcl in ethyl alcohol boil on a water bath for 5 mins, cool and add 4 - 5 drops of aqueous ferric chloride	Wine red/violet color	Aliphatic amide is confirmed
C. Biuret test(urea) Heat 0.2 g compound in dry test tube till the melted compound solidifies and evolution of ammonia ceases, cool it, dissolve the solid in 2 ml dilute NaOH solution and add 1-2 ml of dilute copper sulphate solution drop wise.	Formation of pink/violet, blue and green color	Urea is confirmed
D. Hydrogen peroxide test: 0.2g compound +1 ml water add about 7-8 drops of hydrogen peroxide solution heat to boiling cool and add few drops of ferric chloride solution	Bluish color or brown color	Aromatic amide confirmed

E. Saturated KOH solution test:(imide): 0.2 g compound+ 1-2 ml of saturated KOH solution	White precipitate	Imide is confirmed
F. Tafel's test: 0.2 g compound + 3 ml Con Hcl + 0.3 g of potassium dichromate	Red or violet changing to green	Anilide is confirmed
G.2,4dinitro chlorobenzene test: Take a paper soaked with 2, 4 dinitro chlorobenzene and put little organic compound on it.	Intense color is produced	Anilide is confirmed

3. TEST FOR AMINES :		
A, NITROUS ACID TEST: 0.2 G of compound or 0.2 ml of compound +2 ml of water in a boiling tube .Cool the solution in an ice bath to 0°C - 5°C .To this add a cooled solution in an ice sodium nitrite drop wise and mix well. Test for the presence of free nitrous acid. (starch iodide paper turns blue)	Vigorous evolution of nitrogen Compound dissolves, but re-precipitates. Clear solution. An oily product separates or solution is cloudy. Deep orange red solid.	May be aliphatic primary amine May be tertiary aliphatic or heterocyclic amine. May be aromatic primary amine Secondary amine Tertiary aromatic amine
B. HINSBERG TEST: 0.2 g or 0.2 ml compound +2 ml pyridine +2 ml of freshly prepared 2% aqueous NaOH solution .Shake well and add 2 drops of benzene sulphonyl chloride.	Yellow after shaking. Orange color. Deep red color or purple color.	Primary amine is confirmed. Secondary amine is confirmed Tertiary amine confirmed.
C. CARBYLAMINE TEST : 0.2 gm +0.2 ml alcoholic KOH solution +2-3 drops of chloroform solution. Warm it carefully.	A foul smell of isocyanides	A primary amine or aromatic amine is confirmed.
D. AZO DYE TEST: Take three test tubes add 0.2 g or 0.2 ml of compound + dil HCl cool to 0°C-5°C. In the second test tube take 0.2 g of sodium nitrite+2ml of water. Cool to 0°C - 5°C.Mix these to test tubes. To this add a well cooled solution of 0.2 g of β naphthol in 5 ml 10 % NaOH solution drop wise stir well.	Formation of orange -red dye.	Aromatic primary amine is confirmed.

E. LIBERMAN NITROSO TEST:

0.2 g or 0.2 ml dilute HCl & cool 0°C. Add at about 1 ml ice cold solution of sodium nitrite. After 5 minutes add 5 ml of ether, shake and keep for 2 minutes. Remove ethereal layers & evaporate it on electrical water bath. Now to the residue add 2 drops of phenol, warm & add few drops of concentrated sulphuric acid.

Intense green color or blue color on addition of NaOH solution.

A secondary amine is confirmed.

4. TEST FOR ALCOHOL: A. Sodium metal test: 2ml compound +a small dry piece of sodium metal	Vigorous effervescence due to liberation of hydrogen gas.	Alcoholic hydroxyl group is present.
B Cerric ammonium nitrate test: 0.5 ml compound +few ml drop of cerric ammonium nitrate reagent.	Appearance of red color	Alcoholic group is confirmed.
C. Lucas test: (to distinguish between primary,secondary, tertiary alcohol) 1ml of compound+5-7 ml Lucas reagent .Shake well and note the time required for the separation of two distinct layers.	Layers separate immediately Layer separation 5-10 minutes A clear homogeneous solution is obtained	Tertiary alcohol Secondary alcohol Primary alcohol

<p>5.Test for Aldehyde: A.2,4 dinitro phenyl hydrazine test: To the alcoholic solution of the substance add 5 drops of 2,4 dinitro phenyl hydrazine hydrochloride in Conc. Sulphuric acid. Heat the solution in boiling water bath for few minutes and then cool.</p>	<p>Color change from yellow or orangered precipitate is obtained.</p>	<p>Presence of Aldehyde and ketones. (Carbonyl group confirmed).</p>
<p>B.Schiff's Test : To the little of the substance is dissolve in alcohol and add 2 ml of Schiff's reagent and shake well</p>	<p>The solution turns pink color.</p>	<p>Presences of pink color develop slowly when aromatic aldehyde is present. (Negative in ketones and vanillin)</p>
<p>C.Tollen's test : To a small quantity of the substance add about 5ml of Tollen's reagent and heat the test tube for 5 min in a boiling water bath for few minutes.</p>	<p>Silver mirror is formed.</p>	<p>Aldehyde or reducing sugar may be present.</p>
<p>D.Fehling's Test : To a little of the solution add Fehling solution I and II and it heated on the water bath.</p>	<p>Reddish brown precipitate of cuprous oxide.</p>	<p>Aldehyde may be present (Reduces Fehling's solution to red cuprous oxide.)</p>
<p>E.Benedict's test: To a little of the solution add benedicts reagent and it heated on the water bath.</p>	<p>Reddish brown precipitate of cuprous oxide.</p>	<p>Aldehyde may be present (Reduces Fehling's solution to red cuprous oxide.)</p>

6.Test for Ketone : Sodium nitro prusside test:		
a) TO a little solution add 1 ml of Sodium nitro prusside solution & 5 drops of 10% Sodium hydroxide solution.	Red color develops. Yellow color at the walls of the test tube.	Presence of ketone. Benzophenone is present.
b) IODOFORM TEST: To the little of the substance, solution of Iodine is added followed by sodium hydroxide.	Yellow crystalline precipitate separate.	Ketones are present. RCO group confirmed
c)Borsch Test : To a small quantity of the substance add a drop of hydrochloric content is heated and cool. To the 2 ml of above solution add freshly prepared saturated solution of Sodium bi Sulphate& shake well. It is cooled under water.	White crystalline.	Presence of ketone.

<p>7.Test for Carbohydrate</p> <p>A.Molisch test: To 1 ml of aqueous solution of the substance add few ml of molisch reagent then add 2 ml of Conc. Sulphuric acid along the side of the test tube.</p>	<p>Violet ring is formed</p>	<p>Presence of Carbohydrate.</p>
<p>B.Barfoed's Test : To 1 ml of aqueous solution of the substance add 1 ml of Barfoed's reagent heat the mixture in a boiling water bath.</p>	<p>Red precipitate is formed.</p>	<p>Presence of Carbohydrate Monosaccharide is confirmed.</p>
<p>C. Fehling's Test: To a little of the substance Fehling's solution I and II and boiling with water bath.</p>	<p>Reddish brown precipitate.</p>	<p>Presence of Carbohydrate</p>
<p>D. Tollen's Test : To a small quantity of the substance add 5 ml of Tollen's reagent heat the test tube for a few minutes in boiling water bath.</p>	<p>Silver mirror is formed.</p>	<p>Presence of Carbohydrate</p>
<p>E. Seliwanoff's test: 0.2 gm compound +2 later +0.5ml concentrated Hcl+a crystal of resorcinol is added. Heat it and observe the color.</p>	<p>Cherry red color is formed.</p>	<p>Ketones confirmed</p>
<p>F. Osazone Test : To 5g of phenyl hydrazine in hydrochloride and 15g of Sodium acetate. It is</p>	<p>Bright yellow crystalline is formed.</p>	<p>Presence of Monosaccharide</p>

dissolved in 100 ml of water. To this add 1 ml of aqueous solution of Substance & glacial acetic acid and it is heated in a boiling water bath for 10 minutes and cooled.

7. Test for Phenol

Phthalein Test :

About 0.1 g of Substance and twice the amount of phthalic anhydride (2ml) and few drops of Sulphuric acid heat gently for 2 minutes and then cooled.

Add the solution in the beaker containing 20 ml of dilute Sodium hydroxide solution.

Red or pink colour is formed.

Yellowish green colour.

Presence of monohydric phenol.

Resorcinol [dihydric phenol]

Dye Test

(Azodiformation):

Dissolve 3 drops of aniline in 4ml of dil. hydrochloric acid cooled. The solution under ice. To this add 1ml of Saturated 20% Sodium nitrite solution cooled it. Add ice cold aqueous solution of organic substance to the above mixture. After that add a few drops of Sodium hydroxide.

Orange red dye is formed.

Presence of phenol

Liebermann's Test :		
<p>To a small quantity of the substance mixed with 0.1g of Sodium nitrite in dry test tube and gently heat for two minutes and cool. Add 5 drops of Conc. Sulphuric acid and shake well.</p>	<p>Deep violet color.</p>	<p>Presence of phenol.</p>
<p>The above solution is poured into a beaker containing 100ml of cold water.</p>	<p>Red color is formed</p>	<p>Presence of phenol.</p>
<p>The content of the above beaker is made alkaline with dilute Sodium hydroxide solution</p>	<p>The red color solution turns to blue or green</p>	<p>Presence of phenol.</p>

8. TEST FOR NAPHTHOL To 1ml of alcoholic solution of the substance add a saturated Solution of picric acid	Orange crystals are found	Presence of alpha naphthol
To 1 ml of the alcoholic solution of the substance add neutral Ferric Chloride solution.	White precipitate is formed.	Presence of alpha naphthol.
A small quantity of the substance add is dissolve Conc. Hydrochloric acid and heated for 5 minutes and then it is poured into 5 ml of water	Yellow precipitate is obtained.	Presence of alpha naphthol.
A small quantity of the substance is dissolve in 10% of sodium hydroxide add Chloroform and boil.	The color change from blue to yellow and then colorless.	Presence of beta naphthol.
Warm the aqueous solution of the substance and add few drops of Ferric Chloride.	The color change from green to white.	Presence of beta naphthol.
A small quantity of the substance was added with picric acid and alcohol then boiled and cooled.	Orange crystals are obtained.	Presence of beta naphthol.

<p>9. Test for Ester :</p> <p>A small quantity of the substance is hydrolyzed with 10% Sodium hydroxide solution and acidifies with Conc. hydrochloric acid</p>	<p>White precipitate is formed.</p>	<p>Presence of Ester.</p>
<p>To a little of the substance add a drop of phenolphthalein add dilute sodium hydroxide drop by drop till the pink color appearance. Now heat it in a boiling water bath.</p>	<p>Disappearance of pink color.</p>	<p>Presence of Ester.</p>
<p>To a few drops of the substance add 5g of hydroxyl amine hydrochloride and add 5ml of 10% Sodium hydroxide solution and boil gently, cooled and acidified with dilute hydrochloric acid and Ferric chloride.</p>	<p>Red or violet brown color forms immediately.</p>	<p>Presence of Ester.</p>

10. TEST FOR NITRO COMPOUNDS: 1.Reaction with sulphuric acid: Shake 0.2 gm +0.2 ml compound + 2 ml fuming sulphuric acid added cautiously.	Soluble Insoluble	Aromatic hydrocarbon is present Saturated aliphatic & cyclic compound is present
A. NEUTRAL REDUCTION TEST: 0.2 g or 0.2 ml compound +2- 3 ml 50% alcohol +1ml Calcium chloride solution+ pinch of Zn dust, boil the contents and filter into Tollen's reagent.	Black or gray precipitate	Nitro group is confirmed
B. Ferrous hydroxide test: 0.2 g or 0.2 ml compound +2ml freshly prepared 5% solution of ferrous ammonium sulphate + a drop of dilute sulphuric acid +excess of KOH solution .Shake the test tube	Red brown precipitate	Nitro group is confirmed

EXPERIMENT: 11

DATE:

QUALITATIVE ANALYSIS OF UNKNOWN SAMPLE - I

No.	Experiment	Observation	Inference
I – Primary reaction			
1.	Physical stage : State:		
2.	Colour:		
3.	Odor :		
4.	Solubility :		

Group-1	Group-2	Group-3A	Group-3B	Group-4	Group-5	Group-6	Group-7
Soluble in water and ether	Soluble in water insoluble in ether	Soluble in NaHCO_3	Soluble in NaOH	Soluble in dil HCl	Soluble only in Conc H_2SO_4 (N,S absent)	Insoluble in Conc. H_2SO_4 (N,S, absent)	Not containing group 1-6 (N,S present)

[Test for Saturated & Unsaturated]		
1.	Reaction with bromine water: A small amount of substance shaken with a few ml of freshly prepared bromine water.	
2.	Reaction with alkaline Potassium per manganate: A small quantity of the substance is added with alkaline Potassium per manganate & shaken well.	

[Test for aliphatic or aromatic Compound]		
1.	Flame test : A small quantity of the substance is heated in Nickel Spatula.	
2.	Nitration Test : To a small quantity of the substance add 5ml of Conc. Sulphuric acid and 5ml of Conc. Nitric acid. In dry test tube, keep test tube in boiling water both for few minutes. The test tube is cooled & the condensed is pour into cold water in a beaker.	

SPECIAL ELEMENTS

Lassaignes Test :

Preparation of Sodium fusion extract melt a small piece of dry sodium metal in a small fusion tube add 1g of the substance [3 drops of the liquid] in a fusion tube & heat gently

at first then to red hotness & quickly and carefully plunge red hot end of the tube in 10ml of the distill water in a china dish . Stir the solution with broken end of the tube boil for few minutes & filter through a filter paper. This filtrate is a sodium fusion extract.

	<p>Test for Nitrogen : To a few ml of extract add a drop of freshly prepared ferrous sulphate [Crystal salt can be used] boil the solution & cool add 2ml of dilute Sulphuric acid.</p>		
	<p>Test for Sulphur :</p> <p>a) Lead acetate Test : To 1ml of fusion extract add 1ml of dilute Acetic acid & few drops of lead acetate solution are added.</p> <p>b) Sodium Nitro pruside : To 1ml of fusion extract add 1ml of freshly prepared sodium nitro pruside is added.</p> <p>Test for Halogen To 1 ml of the fusion extract and add 1ml of dilute Nitric acid boil & then cool. Then add 1 drop of Silver nitrate solution.</p>		

[Test for Functional group]

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REPORT

a. Saturated or unsaturated -----.

B. Aliphatic or aromatic -----.

c. Special element -----.

d. Functional group -----.

The given unknown organic sample contains -----