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KARPAGAM ACADEMY OF HIGHER EDUCATION

(Deemed to be University Established Under Section 3 of UGC Act 1956) Coimbatore – 641 021.

SYLLABUS DEPARTMENT OF CHEMISTRY B. Sc CHEMISTRY

Semester-IV

16CHU413ORGANIC SPECTROSCOPY PRACTICAL4H2C

Instruction Hours/week:L:0 T:0 P:4 Total:100

Marks: Internal: 40 External: 60

Scope

This lab course deals with the spectroscopy practical's involving the Identification of simple organic compounds by IR spectroscopy and NMR spectroscopy, qualitative analysis of organic compounds preparation of formaldehyde resin, extraction of caffeine and preparation of methyl orange.

Objectives

The lab course enables the student to

- 1. Extraction of caffeine from tea leaves.
- 2. Preparation of urea formaldehyde resin
- 3. Understand the qualitative analysis of unknown organic compounds
- 4. Identify simple organic compounds by IR spectroscopy and NMR spectroscopy
- 5. Prepare of methyl orange

Methodology

Spectroscopic methods UV, IR and NMR

1.Extraction of caffeine from tea leaves.

2. Preparation of urea formaldehyde resin.

3.Qualitative analysis of unknown organic compounds containing monofunctional groups (carbohydrates, aryl halides, aromatic hydrocarbons, nitro compounds, amines and amides) and

simple bifunctional groups, e.g. salicylic acid, cinnamic acid, nitrophenols etc.

4.Identification of simple organic compounds by IR spectroscopy and NMR spectroscopy (Spectra to be provided).

5.Preparation of methyl orange.

Suggested Readings

Text Books:

- 1. Vogel, A.I. (2012). Quantitative Organic Analysis. Part 3. Pearson.
- 2. Mann, F.G. & Saunders, B.C. (2009). *Practical Organic Chemistry*. Pearson Education
- 3. Furniss, B.S., Hannaford, A.J., Smith, P.W.G. & Tatchell, A.R. (2012). *Practical Organic Chemistry*. 5th Ed. Pearson.

Reference Books

- 1. Ahluwalia, V.K. & Aggarwal, R. (2000). *Comprehensive Practical Organic Chemistry: Preparation and Quantitative Analysis*. University Press.
- 2. Ahluwalia, V.K. & Dhingra, S. (2000). *Comprehensive Practical Organic Chemistry: Qualitative Analysis*. University Press.

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Batch: 2016-2019

Experiment. No: 1

PREPARATION OF METHYL ORANGE

Aim:

To prepare a pure sample of Methyl Orange by treating sulphanilic acid and dimethylaniline.

Principle:

Sulphanilic acid is diazotized by allowing sodium salt to react with a mixture of sodium nitrate and HCl at ice cool condition. This diazotized salt readily complexes with nitrogen, N-Dimethyl Aniline to yield methyl orange. It is precipitated as it sodium salt by adding sufficient quantity of sodium hydroxide solution.

Chemicals Required:

Sulphanilic acid-5.3 g Anhydrous sodium carbonate-1.5 g Sodium nitrite-1.9 g Con.HCl - 5ml Dimethyl aniline-4.2 ml Acetic Acid- 1.5 ml 20% NaOH – 20 ml Sodium chloride -5 g

Procedure:

5.3 g of Sulphanilic acid, 1.5 g of anhydrous sodium carbonate and 50 ml water are taken in a 150ml conical flask and warmed to get a clear solution. The solution is cooled under tap water. The solution of sodium nitrite (1.9 g of sodium nitrite dissolved in minimum quality of water) is added into it. Now the flask is cooled in a glass trough containing crushed ice or freezing mixture. So that the temperature is maintained around 5°C. The solution thus prepared is poured into a beaker containing 5.3 ml of Con.HCl and a little amount of crushed ice kept at cooled condition. Diazotization takes place producing crystals of diazobenzene sulphonates. In a separate test tube 3.2 ml of pure dimethyl aniline is dissolved in 1.5 ml of glacial acetic acid cooled to 0-5°C. This solution of dimethyl aniline is slowly added to the diazotized solution with vigorous strring. The mixture is allowed to stand for 15 minutes. Then addition of about 20 ml of 20% NaOH solution is undertaken. An orange colour sodium salt of methyl orange separates as fine particles.

In heating the mixture to about near boiling condition. The methyl orange will get dissolved. To saltout the methyl orange as its salt. 5g of sodium chloride is added to the solution and warmed on a water bath. When the mixture is allowed to cool, initially in the air followed by ice water. Fine powder of methyl orange settles down at the bottom of the beaker. It is filtered at the pump washed with a dilute solution of NaCl and dried.

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Recrystalisation

About 1g of the sample is recrystalised from hot water. Red orange crystals of methyl orange are separated and dried.

Report:

The yield of the methyl orange = -----

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NH-CH₂OH

Experiment. No: 2

PREPARATION OF UREA FORMALDEHYDE RESIN

AIM: -

To prepare urea formaldehyde and phenol formaldehyde resins.

Apparatus required: -

Beaker, glass rod, funnel, filter paper and chemical balance.

Chemicals: -

Urea, formaldehyde sol., conc. H₂SO₄, distilled water.

Theory: -

Amino resins are condensation products obtained by the reaction of formaldehyde with nitrogen bearing compounds such as aniline, amides for ex:- melamine formaldehyde, urea formaldehyde etc.

Urea formaldehyde is prepared by condensation reaction between urea and formaldehyde in acidic or alkaline medium.

The first product formed during the formation of resin is monomethylol and dimethylol ureas.

NH-CH₂OH

 $\begin{array}{c|cccc} NH_2CONH_2+ & HCHO & & C = O \\ NH_2 & & & \\ Urea & Formaldehyde & Monomethylol \\ & & & Urea \end{array} \begin{array}{c} HCHO & & \\ C = O \\ NH-CH_2OH \\ Dimethylol \\ Urea \\ \end{array}$

Polymerization can take place from mono or dimethylol urea or possibly through both, with the formation of long chains.



NH-CH₂OH Prepared by Mr. R Kumar and Dr. M. Makeswari, Assistant Professor, Department of Chemistry, KAHE 3/24

COURSE NAME: ORGANIC SPECTROSCOPY PRACTICAL COURSE CODE: 16CHU413 Batch: 2016-2019 - H₂O $\dot{n} C = O$ $\dot{C} = O$ Polymerization NH-CH₂OH - N-CH2n Dimethylol urea Cross-linked polymer

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A fully cross-linked urea formaldehyde resin can be represented as:-

Urea formaldehyde resin (cross-linked polymer)

CO

Procedure:-

- 1. Place about 5 ml of 40% formaldehyde solution in 100 ml beaker.
- 2. Add about 2.5 g of urea with constant stirring till saturated solution is obtained.
- 3. Add a few drops of conc. H2SO4, with constant stirring.
- 4. A voluminous white solid mass appears in the beaker.
- 5. Wash the white solid with water and dry it in the folds of filter paper.
- 6. Weight the yield of product

Precautions:-

- 1. While adding concentrated H2SO4, it is better to stay little away from the beaker since the reaction sometimes becomes vigorous.
- 2. The reaction mixture should be stirred continuously.

Observations:-

Mass of the beaker (W1) = ----g. Mass of the beaker with urea formaldehyde (W2) = -----g. Therefore mass of urea formaldehyde (W2 - W1) = -----g.

Result:-

The yield of urea formaldehyde = -----g

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Experiment 3:

EXTRACTION OF CAFFEINE FROM TEA LEAVES

Aim:

To extract

Caffeine –an alkaloid, from

Tea leaves.

Materials Required:

Tealeaves, sodiumcarbonate, methylenechloride, sodiumsulphate, Beakers (100ml) funnel, glassrod whatmann filter paper no.40, hotplate

Theory

Caffeine(1,3,7-trimethylxanthine) is an example of alkaloids, a natural product, generally produced by plants. These compounds usually contain C, H, O and N and some weak bases. In this experiment, you will extract caffeine from tea leaves using dichloromethane. Here we take advantage of the same principles we used in the thin layer chromatography, especially "like dissolves like". Methylenechloride is a liquid but it is not like water so they form two layers just like vinegar and oil. The caffeine molecules dissolve in methylenechloride layer. The two layers then can be separated, and the methylenechloride which has a very low boiling point can be evaporated in the hood, leaving a residue of caffeine.

Procedure

- 1. Add 5g of tealeaves to 50ml of boiling water.Allow to stand for 10minutes with occasional stirring.
- 2. Decant the solotion and keep it.
- 3. Repeat extraction twice with 25ml of boiling water each time.combine all the solutions(tea extract) and boil for 15 minutes.
- 4. Cool the solution to room temperature.
- 5. Add 2g of sodium carbonate so as to precipate most of the tannins. This will react with some of the coffe extract and make them extremely water soluble. Swirl the mixture until all the sodium carbonate dissolves.
- Add 25ml of methylene chloride(CH₂CL₂), and vigorously Swirl the mixture for 10minutes.Do not shake the mixture an emulsion will form.
- 7. Allow the mixture to stand and separate in to two layers, a dark aqueous top layer and

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clear organic bottom layer

- 8. Separate the organic layer(lower layer)using separating funnel and extract the aqueous layer with two additional 15 ml of dichloromethane.
- 9. Collect all the organic layers in 50ml Erlenmeyer flask. To this solution add a scoop of anhydrous Na₂SO₄ in order to remove the last traces of water.
- 10. Transfer the dried solution to a 50ml beaker and evaporate the dichloromethane on a warm hot plate kept in fume hood. When afraction of milliliter of liquid is left, remove the beaker from the hotplate. Allow the beaker to stand in the hood for aminute or two. The heat remaining in the glass will cause the last amount of ethylenechloride to evaporate and produce a solid residue of crude caffeine.
- 11. Weigh the crude caffeine and calculate the yield.

Report

Yield of the Product = -----

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ORGANIC QUALITATIVE ANALYSIS

Preliminary Tests : I.

- a) Physical characteristics
- b) Solubility test
- c) Heating on a nickel spatula
- d) Identification of the elements present
- e) Heating with soda lime
- f) Treatment with 10% NaOH (aqueous)solution
- g) Treatment with dil. HCl.
- h) Treatment with conc. H₂SO₄
- i) Treatment with saturated NaHCO₃ solution
- j) Test with Fehling's solution
- k) Test with Tollen's reagent
- 1) Reactions and coloration with Ferric chloride
- m) Treatment with 2, 4-dinitrophenyl Hydrazine.
- Test for Functional Groups present-Confirmatory tests : II.
- III. Derivative for the functional group present.

ORGANIC QUALITATIVE ANALYSIS SCHEME OF PROCEDURE FOR IDENTIFYING CHARACTERISTIC GROUPS I.

Preliminary	Tests
-------------	-------

e colour and appearance of	llow solid or liquid	esence of aromatic nitro-
the substance are noted.	Brown or dark	compounds, phenols,
	coloured solid or	aromatic amines etc.
	liquid.	esence of acids, esters,
	lourless solid or liquid	aldehydes ,ketones,
	1	hydrocarbons, anilides etc.
	asant smell	
e odour of the substance is		esence of esters, ketones.
noted.		nitro compounds.
	enolic smell	-
		enols present.
	hy smell	_
		omatic amines present.
	rosene-like smell	
		drocarbons, aromatic

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				compounds with halogen in
				the nucleus present.
		ngent o	r irritating smell	-
		0	0	esence of aromatic
				compounds with halogen in
		luble in	water and the	the side chain etc.
		solutio	is is	
	small amount of the	(i)	$\Delta cid to$	sence of aliphatic acids
	substance is shaken up with	(1)	litmus	polyhydric phonols nitro
	shout 1ml of water and the		nunus	porynyune phenois, into
	about This of water and the	(;;)	Noutral	phenois, annue saits etc.
	litmus	(11)	Incuttat	sonos of simple sloshols
	nunus.			esence of simple alcohols,
		а ·		sugars, urea etc.
		Sparin	gly soluble or	
		practic	cally insoluble	
		in wat	er and the	
		susper	ISION IS	
		(i)	Acidic to	
			litmus	
				omatic acids, simple
				phenols, nitro phenols, etc.
		(ii)	Basic to litmus	Present.
		(iii)	Neutral to	esence of Aromatic amines.
			litmus	resence of esters, aldehyde,
				ketones, hydrocarbons,
				amines, anilides.
		(i)	Blue litmus	Presence of acids and
			turned red	phenols.
	tmus Test			-
	A little of the substance is	(ii)	Red litmus	Presence of amines.
	brought in contact with a	È É	turned blue	
	moist ened litmus paper.			
		(iii)	No changes of	Presence of carbohydrates.
		· -/	colour takes	ketones, esters.
			place	hydrocarbons etc.
L		1	r-400	

5. Test for the Elements

A dry small pellet of sodium is melted in an ignition tube by heating gently. A small quality of the solid substance or 2 drops of liquid substance is introduced and heated gently at first. When the vigour of the reaction has subsided, the tube is heated strongly till the bottom of the tube become red hot. The red hot end of the tube is quickly broken into about 10ml of distilled water in a china dish. The solution is well stirred, boiled and filtered. The following tests are carried out the filtrate:

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bout 3 ml of the filtrate are heated with an equal volume of a saturated aqueous FeSO ₄ solution boiled, cooled and then acidified with dilute sulphuric acid added drop by drop with constant shaking.	blue precipitate or a blue solution or at least a green solution is obtained.	trogen present.
out 3 ml of the filtrate are boiled with an equal volume of dilute nitric acid for 2 minutes, especially when nitrogen has been detected, cooled and then Silver nitrate solution is added.	A curdy white precipitate soluble in ammonium hydroxide. A pale yellow precipitate, sparingly soluble in NH4OH. A pale yellow precipitate insoluble in NH4OH	Chloride present Bromide present.
out 1 ml of the filtrate is treated with an equal volume of a freshly prepared solution of sodium nitro prusside.	A violet colour is obtained.	Sulphur present.

Test for Aliphatic or Aromatic:

small amount of the	(i)	The substance	e substance is aromatic.
substance is taken in a		burns with a	
nickel spatula and heated in		smoky, sooty	
the free flame of the Bunsen		flame.	
burner	(ii)	The substance	The substance is
		burns with a	aliphatic.
		non-luminous	
		flame.	
	(iii)	The substance	
		chars and burns	Presence of
		slowly.	carbohydrates,
			compounds like
			sulphanilic acid.
			The substance is
small quantity of the	A yellow	precipitate or a	aromatic.
substance is mixed with 1	yellow so	lution is	
ml of concentrated sulphuric	obtained.		

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	acid and 1 ml of concentrated nitric acid in a dry test tube, shaken well and heated for 10 to 15 minutes in a boiling water bath and then poured into about 20 ml of cold water.	No yellow precipitate and the solution are colourless.	The substance is aliphatic.	

small amount of the substance	omine water is decolourised	saturated compounds
is shaken up with 1 ml of	immediately.	present.
bromine water added drop		
by drop.	omine water is decolourised	Saturated compounds
	with the formation of a	like amines, phenols,
	white precipitate.	etc. which are readily
		brominated.
	e solution remains brown	
	without decolourisation on	Presence of saturated
	adding one or two drops of	compound.
	bromine water.	
	(a) Decolourisation	
A small amount of the	takes place readily	Presence of unsaturated
substance is shaken up with	and no fumes of	compound.
1 ml of carbon tetra chloride	hydrogen bromide	
in a dry test tube. A solution	are evolved.	
of bromine in CCl ₄ is added	(b) Decolourisation	~
in drops with shaking till a	takes place readily.	Readily brominated
brown colour is obtained and		saturated compounds
warmed if necessary.		like phenols, amines,
	(c) No decolourisation	etc. present
	after the addition	Durannes of softwarted
	of one drop of	Presence of saturated
	bromme.	compounds.
	(a) Decolourisation takes	
	(a) Decolourisation takes	
A small amount is shaken up	KMnO4 drops are used	Presence of unsaturated
with 1ml of water or acetone		compounds easily
and 2% solution of	чр.	oxidisable compounds
potassium permanganate is		like aldehydes phenols
added drop by drop with	(b) No decolourisation	amines etc.
constant shaking.	after the addition of 1	

Test for whether Saturated or Unsaturated:

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	or 2 drops of the solution.	Saturated compounds present

Action of sodium-bi-carbonate (NaHCO₃)

small amount of the substance	(a)Vigorous effervescence	esence of carboxylic
is added to 1 ml of a strong	takes place and the	acids
clear solution of NaHCO ₃	substance dissolves	·
without shaking; the changes		
are observed and then	(b)Slow effervescence	lyhydric phenols present
shaken well.	takes place	
		tro phenol present
	(c) Slow effervescence and	
	the solution turn yellow.	

Action of NaOH solution

small amount of the substance	(a) Dissolves readily in esence of acids, phenols.
is added to 2 ml of a strong	the cold. When the
clear solution of NaOH,	solution is acidified
shaken well, the changes, if	with drops of
any are observed and then	concentrated HCl
heated to boiling.	and cooled, the
	substance is
	regenerated. Nitro phenols present.
	(b) Dissolved readily.
	The solution
	becomes yellow
	and the colour is
	removed on adding
	concentrated HCl. Polyhydric phenols
	(c) Dissolves readily present.
	and the solution
	turns yellow,
	brown and finally
	dark on shaking in
	cold condition. Esters present.
	(d) Substance dissolves
	gradually on
	warming. Presence of sugars,
	aliphatic aldehydes.

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		(e)	Solution turns yellow or brown when boiled.	Amides present
		(f)	Ammonia is evolved on continued boiling.	Anilides present.
	(a)If ammonia has been evolved on boiling or if the substance possesses the characteristic ester smell and has shown signs of gradual dissolution, about 2 gms of the substance are boiled under reflux with about 20 ml of 10% NaOH solution for about 30 minutes, cooled, acidified with concentrated HCl and again cooled.	(g) (h) A whit obtaine	On boiling, oily globules of a liquid are obtained possessing an aniline-like smell. The substance is unaffected in the cold and on boiling. e precipitate is ed.	Presence of nitro compounds, ketones, amines, hydrocarbons, aromatic halogen compounds, etc. Amide or ester of an aromatic acid present.

Action of Soda Lime

13.	A small amount of the	(a) Ammonia is evolved	Amides present.
	substance is mixed with 2 to	(b) Vapours with aniline	Anilides present.
	3 spatula full of powdered	like smell and	
	soda-lime and heated gently	condensing to oily	
	at first and then more	globules of liquid are	
	strongly.	evolved.	
		(c) Highly inflammable	Acids, certain esters
		vapours with	present.
		*	-

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14.	A small amount of the substance is dissolved in dry benzene in a dry test tube and a pellet of freshly cut sodium metal is added.	kerosene-like smell are evolved (d) Vapours with a phenolic smell and condensing to oily globules of a liquid are evolved. Distinct effervescence take place	Phenolic acids, Phenolic esters present. Presence of acids, Phenols.
15.	A small amount of the substance is shaken up with 1:1 Hydrochloric acid.	The substance readily dissolves and on adding 10% sodium hydroxide solution, the substance is regenerated.	Aromatic amines present.
16.	A small amount of the substance is added to about 2 ml of Conc.H ₂ SO ₄ , shaken well, the changes are observed and then gently heated.	 (a) A white precipitate is formed which dissolves when shaken up with water to form a clear solution. (b) A vigorous reaction takes place in the cold. Charing or blackening takes place with effervescence. 	Aromatic amines present. Presence of aromatic amines, carbohydrates, certain polyhydric phenols, hydroxyl acids etc.
17.	A small amount of the substance is shaken up with about 1 ml of water. Neutral ferric chloride solution is added drop by drop.	(a) A violet or blue or green colour is obtained.	Presence of phenols, and phenol derivatives like salicylic acid, phenolic aldehydes, nitrophenols etc.
	Note: This test is repeated with 1 ml of alcohol instead of water since some phenols give a characteristic colour reaction readily in alcoholic solution.	(b) A blue, bluish violet or green precipitate is obtained.	Certain amines like α- naphthyl amine, naphthols etc. Present Carboxylic acids present.

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		(c) A reddish –brown or	
		buff coloured	
18	A small amount of the	precipitate obtained.	Presence of aldehydes,
	substance is added to 3 or 4		reducing sugars like
	ml of Tollen's reagent and	A bright silver mirror or a	glucose, polyhydric
	heated in a boiling water-	brownish black precipitate of	phenols, and aromatic
	bath for 10 to 15 minutes.	silver is obtained.	primary amines.

TESTS FOR FUNCTIONAL GROUPS

I. Compounds in which carbon, Hydrogen and Oxygen are present

	I.1. Carboxylic Acids		
(a)	A pinch of the substance is shaken up with about 1 ml of water. One drop of phenolphthalein is added and then very dilute sodium hydroxide solution is added drop by drop with shaking.	A pink colour is produced only after adding a number of drops of sodium hydroxide. A pink colour is got even with the first drop of sodium hydroxide.	Presence of carboxylic acids. Acids absent
(b)	<i>Ester formation:</i> About 0.2 gm of the substance is mixed with about 2 ml of rectified spirit in a dry test tube; 2 to 3 drops of conc. H ₂ SO ₄ are added, shaken well and gently heated for a minute. The mixture is then poured into about 30 ml of a dilute solution of sodium carbonate, stirred well and the smell is noted.	A pleasant ester smell is obtained.	Carboxylic acid is present.
(c)	<i>Fluorescein reaction:</i> A small amount of the substance is mixed with a few crystals of resorcinol in a dry test tube. 2 drops of conc. H ₂ SO ₄ are added, shaken well, heated gently and then poured into about 100 ml of	The mixture turns deep-red on heating; forms a red solution with water; on adding sodium hydroxide, an intense greenish yellow fluorescein is obtained.	Dicarboxylic acids present.

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water, stirred well and then	
an excess of sodium	
hydroxide solution is added.	

	1		
(a)	Libermann's Reaction: A small amount of the substance is heated with a crystal of sodium nitrite in a dry test tube, cooled, two drops of conc. H_2SO_4 are added, shaken well and the mixture is poured into about 100 ml of water, stirred well and then an excess of sodium hydroxide is added.	A red solution is got which turns blue or green on adding sodium hydroxide.	Phenolic group present. Note: all phenols do not give this test.
(b)	Phthalien fusion Test : A small amount of the substance is mixed with about 0.5 gm of phthalic anhydride in a dry tube; 2 drops of conc.H ₂ SO ₄ are added, heated gently for a minute. The mixture is poured into about 100 ml of water and the sodium hydroxide is added in slight excess.	A pink or red colour is obtained. A yellowish green fluorescence is obtained.	Phenols like phenol, o- cresol, m-cresol, salicylic acid.ResorcinolNote: The colour depends on the nature of the phenol.
(c)	Azo-Dye Formation : About 5 drops of aniline are treated with 3 to 5 ml of dil. HCl, 10 to 15 drops of strong solution of sodium nitrite are added drop by drop with constant shaking and cooling, and the diazotised solution obtained is added to a solution of the substance in 2 to 3 ml of 10% NaOH solution in small quantities.	A scarlet red or brownish red or orange red precipitate of a dye is obtained.	Phenols like β-naphthol, α-naphthol, resorcinol etc.
(a)	About 1 mi of Fenling's	A red brown precipitate of	Polynyaric Phenol.

I.2. Phenolic Group

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	solution 'A' is mixed with 1 ml	cuprous oxide is obtained.	
	of Fehling's solution 'B'. The		
	mixture is treated with a small		
	amount of the substace and		
	boiled well.		

(a)	About 3 drops of the substance are added to a saturated solution of sodium bisulphite solution and shaken well.	A white crystalline precipitate is obtained.	Aldehydes and certain ketones present. [ketones with the keto group directly attached to benzene ring does not answer this test]
(b)	A small amount of the substance is added to about 3 ml of Borsche's reagent, a drop of conc.HCl added, gently heated for about 2 minutes and cooled well.	A yellow or red-brown crystalline precipitate is obtained.	Aldehydes and ketones present.
(c)	A mixture of 5 drops of Phenylhydrazine and 5 drops of glacial acetic acid are taken in dry test tube. A small amount of the substance is added, gently heated for a minute. Excess of cold water is added and shaken well.	 (a) A yellow or yellowish-white precipitate is obtained. (b) A bright yellow crystalline precipitate. 	Presence of Aldehydes and certain Ketones. Reducing sugars present.
(d)	About 0.3 gm of semicarbazide hydrochloride id dissolved in H_2O . About 0.5 gm of sodiumacetate crystals are added,shaken well to dissolve the solid and to this 2 ml of an alcoholic solution of the substance is added, heated in a water bath for 15 minutes and cooled.	A white crystalline precipitate of the semicarbazone is got.	Aldehydes and Ketones present.
(e)	A small amount of the substance is added to about	A violet colour is produced quickly.	Aldehydes confirmed.

I.3. Aldehydes and Ketones

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	3ml of Schiff's reagent and shaken well.		
(f)	A small amount of the substance is added to 2 ml of Fehling's solution A and B and gently heated.	A reddish brown precipitate is got.	Presence of Aliphatic aldehydes (reducing sugars, and polyhydric phenols also answer).

1.4.Carbohydrates

(a)	Molisch test:		
	A small amount of the	A violet, purple or red ring	Carbohydrates
	substance is dissolved in 2 ml	is formed at the junction of	confirmed.
	of water, a few drops of a	the two layers and the	
	strong solution of (10%) of α -	colour slowly spreads	
	naphthol in pure alcohol is	throughout the liquid.	
	added, shaken well, and 2 ml		
	of conc.H ₂ SO ₄ are carefully		
	added along the side of the		
	tube so that the acid forms a		
	separate layer at the bottom.		
(b)	A small amount of the	A red brown precipitate is	Reducing sugar present.
	substance is shaken up with	obtained.	
	one ml of water. The solution		
	is added to about 2 ml of		
	Fenling's A and B and heated		
	on a water bath for 15 minutes.		
(a)	About 0.3 gm of the substance	A bright vollow grustelling	Paducing sugars like
(C)	About 0.5 gill of the substance	A blight yellow crystalline	duces fructose and
	water, a mixture of 2 ml of	precipitate is obtained.	lactose present
	phenylbydrazine and 2 ml of		lactose present.
	glacial acetic acid is added the		
	contents heated in a boiling		
	water bath for about 15		
	minutes with occasional		
	shaking and then cooled		

1.5.Esters:

About 3 drops of the liquid and a pinch A violet or deep red Esters present	

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	of hydroxylamine hydrochloride are	brown colour	
	added to about 5 ml of 10% NaOH	obtained.	
	solution. The contents are gently boiled		
	for 3 minutes cooled, acidified with		
	conc.HCl, added drop by drop and then		
	about 5 drops of FeCl ₃ solution are		
	added and shaken well.		
(b)	The liquid is refluxed with 10% NaOH	A white precipitate is	Ester of an aromatic
	solution. The residue is acidified with	obtained.	acid is present.
	con.HCl and cooled.		

II. COMPOUNDS IN WHICH NITROGEN IS PRESENT II. 1. Nitro compounds

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sodium nitrite solution and the diazotised solution of β- naphthol in NaOH.	A scarlet-red dye is obtained.	An aromatic nitro group present.

(a)	A small quantity of the	A clear solution is obtained.	Aromatic primary amine
	substance is dissolved in		present.
	about 1 cc of conc.HCl,	A yellow or yellowish-	Secondary amines present.
	diluted to about 4 cc, cooled	brown oily liquid separates.	
	in ice- cold water and then		
	treated with about 1 cc of a	A yellowish brown	Presence of tertiary amine.
	strong solution sodium nitrite,	crystalline precipitate is	
	added drop by drop with	obtained. The precipitate is	
	constant shaking.	removed, dissolved in water	
		and when treated with	
		sodium hydroxide gives a	
	<u>^</u>	green solid.	
	(i) The clear diagotized	A coordat rad due is	Aromatia primary amina
	(1) The clear diazonsed	A scallet leu uye is	prosent
	added to a solution of B	precipitated.	present.
	naphthol in NaOH solution		
	haphthof in NaOH solution.		
	(ii) The yellow oily liquid in	The mixture added to water	Presence of secondary
	above is mixed with 2 drops	gives a red solution which	amines.
	of phenol, heated gently, 2	becomes green or blue on	
	drops of conc.H ₂ SO ₄ acid	adding NaOH.	
	added, again warmed, added		
	to 20cc of water and then		
	NaOH solution added in		
	excess (Libermann's		
	reaction)		
(b)	A small amount of the	A characteristic offensive	Primary amines present.
	substance is mixed with a few	smell of iso-cyanide is	
	drops of chloroform .2cc of	obtained.	
	alcoholic potash added and		
	warmed gently.		
		A	Deine number 1
(C)	About 0.5 gm of the	A white crystalline	Primary or secondary
	substance is snaken up with	precipitate of the acetyl	annnes present.

II.2. Amines

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	2cc of acetic acid. About 5	derivative is obtained.	
	drops of acetic anhydride are		
	added, shaken well for some		
	time and then diluted with		
	water.		

(a)	The substance is refluxed with 10% NaOH solution, acidified with conc. HCl and	Ammonia is evolved while refluxing	Aliphatic or aromatic Amide present.
	cooled again.	On adding HCl, a white precipitate is obtained.	Aromatic amide present
		On adding HCl, no precipitate is obtained.	Presence of amides of lower aliphatic acids and urea.
(b)	<i>Biuret Test:</i> A small amount of the substance is gently heated in a dry test tube for a few minutes, cooled, the residue is shaken up with 2 ml water, a few drops of copper sulphate are added and then 10% NaOH solution is added drop by drop.	Ammonia is evolved on heating. A violet or red colouration is obtained.	Diamides like urea present.

II.3. Amides

II.4 .	Anilides

	II.T. Minues				
(a)	A few crystals of the solid are	Offensive smell of	Presence of anilides.		
	treated with a few drops of chloroform and 2cc of alcoholic potash and gently heated.	isocyanide is obtained.			
(b)	About 1 gm of the substance is boiled under reflux with 2cc of conc. HCl. The solution is cooled, diazotised with sodium nitrite and coupled with β - naphthol	A scarlet red dye is formed.	Anilides, toluidides present.		

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 dissolved in sodium
 hydroxide.

(a)	A small amount of the	A characteristic colour	Aliphatic halide,
	substance is refluxed with 10	precipitate of silver halide is	aromatic halogen
	ml of strong, chloride free	obtained.	compound with halogen
	NaOH solution, cooled and		in the side chain.
	the aqueous solution is		
	acidified with drops of conc.	Oily drops do not disappear	Aromatic halogen
	HNO ₃ , cooled and then silver	and silver halide is not	compound with halogen
	nitrate solution is added.	formed.	in the nucleus.
(b)	A small amount of the	An immediate or slow	Aliphatic halide,
	substance is shaken well with	precipitate of silver halide	aromatic halogen
	2 to 3 ml of alcoholic silver	with a characteristic colour.	compound with halogen
	nitrate and then gently		in the side chain.
	warmed.		
		No precipitate of silver	Aromatic halogen
		halide is obtained.	compound with halogen
			in the nucleus.

III. Halogen Substituted Hydrocarbons

IV. Compounds in which Sulphur is present

(a)	A little of the substance is mixed with 2 ml of absolute alcohol and then with a few cc of sodium hydroxide solution.	Ammonia is evolved.	Presence of thiourea or sulphonamide.
(b)	A small amount of the substance is treated with concentrated hydrochloric acid.	Pungent smell.	Presence of substituted thiourea.
(c)	A little of the substance is fused with NaOH, dissolved in water and dilute HCl is	(i)Hydrogen sulphide is evolved.	Presence of thiourea.
	added.	(ii) SO_2 is evolved and a phenol is formed shown by blue or violet colour with neutral ferric chloride.	Presence of Sulphonic acid.

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		(iii) Ammonia is evolved during fusion. No phenol is formed. SO ₂ is evolved only on adding acid.	Presence of sulphonamide.

V.Hydrocarbons

(a)	A small amount of the	The substance remains	Aliphatic Hydrocarbon.
	substance is added to 2 cc of	unaffected by the acid.	
	conc. H ₂ SO ₄ (preferably		
	fuming sulphuric acid) and	The substance slowly goes	Aromatic Hydrocarbon.
	shaken well for some time.	into solution in the acid and	
	The mixture is then added to	is not regenerated when	
	about 25 cc of cold water.	added to cold water.	
(b)	Picrate formation:		
	About 0.5 gm of the	Yellow precipitate.	Aromatic polynuclear
	substance dissolved in hot		hydrocarbon present.
	benzene is added to picric		
	acid dissolved in benzene and		
	mixed well.		

RESULT:

The given compound is = Aromatic (or) aliphatic

The given compound is = Saturated (or) Unsaturated

Nitrogen, Sulphur and oxygen is = present (or) Absent

The given functional group is