

KARPAGAM ACADEMY OF HIGHER EDUCATION (Deemed to be University Under Section 3 of UGC Act 1956) COIMBATORE-21 DEPARTMENT OF CHEMISTRY B.Sc Chemistry – Syllubus

Semester-III

4H 2C

16CHU313 NITROGEN CONTAINING FUNCTIONAL GROUPS, HETEROCYCLIC CHEMISTRY AND NATURAL PRODUCTS PRACTICAL

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

Marks: Internal: 40 External: 60 Total:100

Scope

The lab course involves the qualitative analysis of nitrogen containing functional groups. It also involves the analysis of alcohols, carboxylic acids, phenols, carbonyl compounds and esters.

Programme Outcome

This course enable the student to

- 1. Identify the presence of nitro, amine and amide groups
- 2. Identify functional groups like alcohols, carboxylic acids, phenols, carbonyl compounds and esters

Programme Learning Outcome

- 1. To develop and inculcate laboratory skills and techniques
- 2. To enable the student to understand the basic chemical concepts.
- 3. To develop basic competence of analysing and synthesising Functional compounds and mixtures

Methodology

Qualitative analysis of organic compounds

1. Functional group test for nitro, amine and amide groups.

2. Qualitative analysis of unknown organic compounds containing simple functional groups

(alcohols, carboxylic acids, phenols, carbonyl compounds and esters)

Suggested Readings

Text Books:

- 1. Mann, F.G. & Saunders, B.C. (2009). Practical Organic Chemistry. Pearson Education
- 2. Furniss, B.S., Hannaford, A.J., Smith, P.W.G. & Tatchell, A.R. (2012). *Practical Organic Chemistry*. 5th Ed. Pearson.
- 3. Ahluwalia, V.K. & Aggarwal, R. (2000). *Comprehensive Practical Organic Chemistry: Preparation and Quantitative Analysis*. University Press

Reference Books

1. Ahluwalia, V.K. & Dhingra, S. (2010). *Comprehensive Practical Organic Chemistry: Qualitative Analysis*. University Press.



KARPAGAM ACADEMY OF HIGHER EDUCATION Faculty of Arts, Science and Humanities

(Deemed to be University Under Section 3 of UGC Act 1956) Eachanari Post, COIMBATORE - 641 021, INDIA

DEPARTMENT OF CHEMISTRY

Name of the Faculty : Dr. M. Gopalakrishnan **Department** : Chemistry Title of the course : Nitrogen Containing Functional Groups Heterocyclic Chemistry and Course code : 16CHU313 natural products - Practical : II-B.Sc., Chemistry "A" Semester : III

Class

LIST OF EXPERIMENTS

S. No.	Duration Hours	Name of the Experiment	
1.	4	Writing experimental procedure and Demonstration for functional groups	
2.	4	Identification of functional group of amine (aniline)	
3.	4	Identification of functional group of di-amide (Urea)	
4.	4	Identification of functional group of amide (benzamide)	
5.	4	Identification of functional group of Nitro compounds (Nitro bezene)	
6.	4	Identification of functional group of amide (Glucose)	
7.	4	Identification of functional group of aldehydes (benzaldehyde)	
8.	4	Identification of functional group of alcohols (phenol)	
9.	4	Identification of functional group of Ketones (acetophenone)	
10.	4	Identification of functional group of monocarboxylic acid	
11.	4	Identification of functional group of dicarboxylic acid	
12.	4	Identification of functional group of Nitro compounds	
13.	4	Repetition Class	
14.	4	Viva-voice questions	
15.	4	Model practical examination	

Organic Lab Manual On

QUALITATIVE ANALYSIS OF ORGANIC COMPOUNDS CONTAINING SIMPLE FUNCTIONAL GROUPS



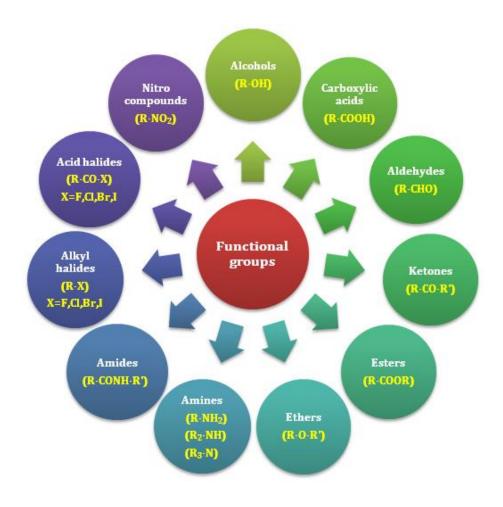
Enable | Enlighten | Enrich (Deemed to be University) (Under Section 3 of UGC Act 1956)

Dr. M. GOPALAKRISHNAN Assistant Professor, Chemistry

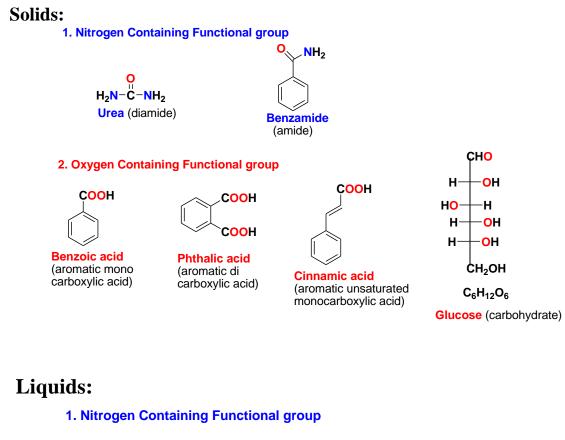
Karpagam Academy of Higher Education

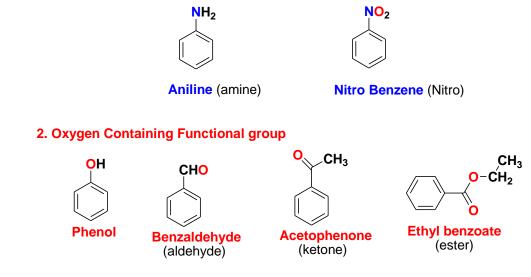
What are the functional Groups?

A functional group is a portion of a molecule that is a recognizable/classified group of bound atoms. In organic chemistry it is very common to see molecules comprised mainly of a carbon backbone with functional groups attached to the chain. The functional group gives the molecule its properties, regardless of what molecule contains it; they are centers of chemical reactivity. The functional groups within a molecule need to be identified when naming.



COMPOUNDS GIVEN FOR ORGANIC ANALYSIS





Department of Chemistry, KAHE.

QUALITATIVE ANALYSIS OF ORGANIC COMPOUNDS

Aim:

Systematically identify the functional groups in the given organic compound and perform the confirmatory tests after identifying the functional groups.

1. Reactions of amides

a. <u>Reaction of NaOH</u>

Amides are decomposed by NaOH to evolve ammonia. The gas can be tested by a moist red litmus paper which is then turned blue.

RCONH ₂ +	NaOH ———	RCOONa	⊦ NH ₃
Amide		Sodium salt of carboxylic acid	

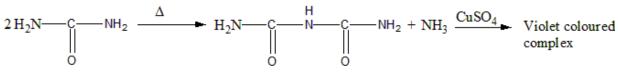
b. Alkaline hydrolysis of aromatic amides to aromatic acid

The soluble sodium salt of aromatic acid formed from aromatic amides upon hydrolysis is regenerated as white precipitate in acidic medium.

 $\begin{array}{cccc} C_{6}H_{5}CONH_{2} + NaOH & & RCOONa + NH_{3} & \stackrel{H^{+}}{\longrightarrow} & C_{6}H_{5}COOH \\ Aromatic amide & & Sodium salt & Ammonia & Aromatic acid \\ of aromatic acid & & Ammonia & Aromatic acid \end{array}$

c. Biuret Reaction for aliphatic diamide

When aliphatic diamide is heated at a temperature above its melting point, ammonia is evolved and crystalline biuret is formed. This biuret in alkaline medium gives a violet colour with a drop of copper sulphate solution.



Biuret reaction

d. Hydroxamic acid test for aromatic primary amides

Hydrogen peroxide reacts with aromatic primary amides to form the hydroxamic acid, which then reacts with ferric chloride to form ferric hydroxamate complex having a violet colour.

ArCONH₂ + H₂O₂ ArCONHOH + H₂O Aromatic amide 3ArCONHOH + FeCl₃ (ArCONHO)₃ Fe + 3HCl Ferric hydroxamate complex

2. Reactions of carboxylic acids

a. <u>Reaction with NaOH</u>

Carboxylic acids being acidic dissolves in NaOH to form sodium salt.

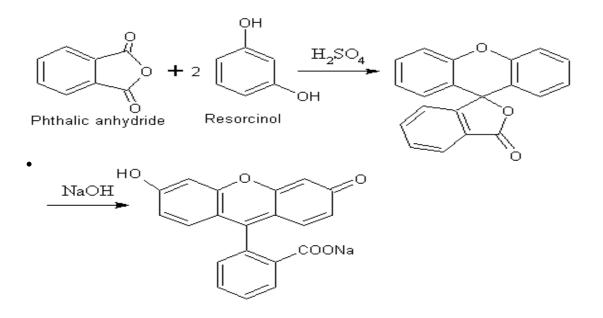
RCOOH + NaOH ------ RCOONa + H₂O Solid Soluble Sodium Salt

b. <u>Reaction of NaHCO3</u>

Forms salt with sodium bicarbonate solution with the evolution of carbon dioxide.

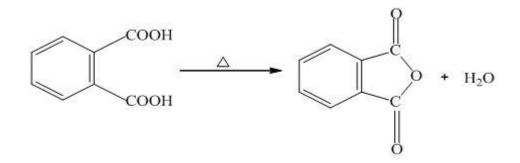
c. <u>Fluorescein Reaction:</u>

The anhydrides of aromatic 1,2-dicarboxylic acids on heating with resorcinol gives a dye fluorescein. This dye in NaOH solution gives a yellowish red solution with green fluorescence.



d. Anhydride formation

Aromatic 1,2-dicarboxylic acids decomposes to give its anhydride when heated at its melting point.



3. Reactions of phenols

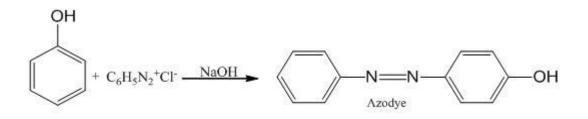
a. <u>Reaction of neutral ferric chloride solution</u>

Phenol form characteristic coloured iron complexes when treated with neutral ferric chloride solution. E.g. phenol and resorcinol - violet colour, catechol-green etc.

 $FeCl_3 + 6C_6H_5OH \longrightarrow [Fe(OC_6H_5)]^{3-} + 3HCl$

b. Azo dye formation

Aryldiazonium salts react with aromatic rings of phenols to form highly coloured azo compounds. These reactions are called coupling reactions.



c. **Benzoylation**

Phenols react with benzoyl chloride in presence of NaOH, to form esters.

$$ArOH + C_6H_5COC1 \xrightarrow{OH^-} C_6H_5COOAr + HC1$$

4. Reactions of primary amines:

a. Diazotisation

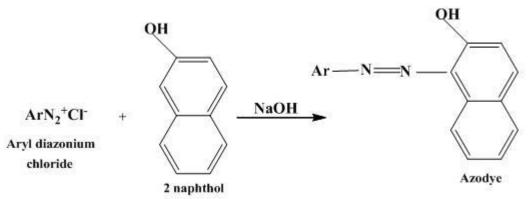
At low temperature $(0-5^{\circ}C)$ aromatic primary amines dissolved in strong acids (HCl and H₂SO₄) reacts with nitrous acid (NaNO₂+HCl) to form water soluble diazonium salts. Aliphatic primary amines do not form stable diazonium salts under similar condition. They react with nitrous acid to yield alcohols and nitrogen (causes rapid foaming).

$$\begin{array}{r} \text{ArNH}_2 \xrightarrow{\text{NaNO}_2} \text{ArN}_2^+\text{Cl}^- + \text{NaCl} + 2\text{H}_2\text{O} \\ \hline \text{Aryl diazonium} \\ \text{chloride} \end{array}$$

$$RNH_2 = \frac{NaNO_2}{HCI} = ROH + N_2$$

b. Azo dye formation for aromatic primary amines

Aryldiazonium salts react with aromatic rings of phenols to form highly coloured azo compounds. These reactions are calledcoupling reactions.



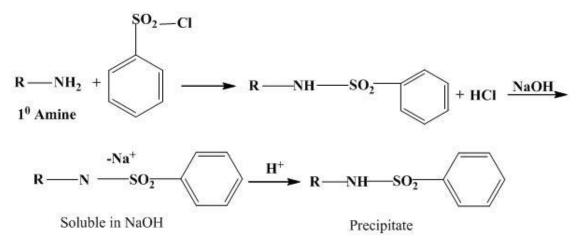
c. **Benzoylation**

Primary aromatic amines react with benzoyl chloride in presence of NaOH, replacing the H atom attached to the N atom with the benzoyl group to give anilides.

 $ArNH_2 + C_6H_5COCI \longrightarrow C_6H_5CONHAr + HCI$

d. Hinsberg reaction

Hinsberg reagent is called benzenesulfonyl chloride. Primary aliphatic amines on reaction with benzenesulfonyl chloride and NaOH gives N-alkylsulphonamide which contains an acidic hydrogen and hence dissolve in NaOH solution to form the soluble sodium salt. The solution thus obtained on acidification gives a precipitate of free sulfonamide which is insoluble in HCl.

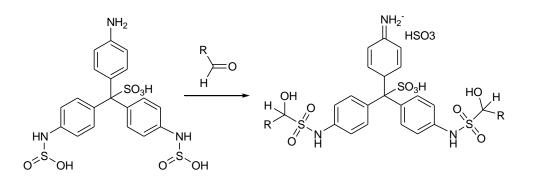


5. Reactions of aldehydes:

a. Reaction of Schiff's reagent

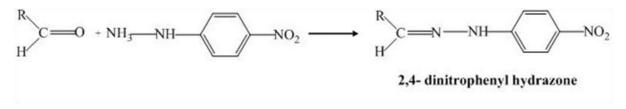
Schiff's reagent is a red solution of rosaniline hydrochloride dissolved in water which is decolourised by passing sulphur dioxide. Dilute solutions of aldehydes when added to Schiff's reagent restores its red colour slowly.

Dr. M. Gopalakrishnan, Department of Chemistry, KAHE.



b. <u>Reaction of Borsche's Reagent</u>

Aldehydes reacts with 2,4-dinitrophenyl hydrazine solution to give a orange/red precipitate of aldehyde 2,4-dintrophenylhydrazone derivative.



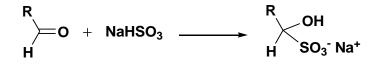
c. <u>Reaction of Tollen's Reagent:</u>

Aldehydes are oxidized to carboxylic acids accompanied by the reduction of silver ions to metallic silver which appears as a mirror under proper conditions.

RCHO +
$$2[Ag(NH_3)_2]OH$$
 _____ RCOONH₄ + $2NH_3$ + H_2O + $2Ag$

d. <u>Reaction of sodium bisulphite solution</u>:

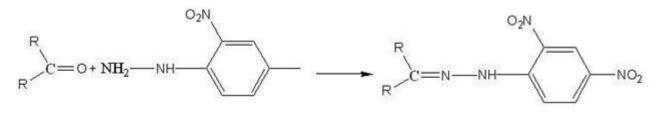
Saturated solution of sodium bisulphite in water, when mixed with aldehydes gives a white crystalline bisulphite addition compounds.



6. Reactions of ketones:

a. Reaction of Borsche's Reagent:

Ketones reacts with 2,4-dinitrophenyl hydrazine solution to give an orange/red precipitate of ketone 2,4 dintrophenylhydrazone derivative.



2,4-dinitrophenyl hydrazone

b. <u>Reaction of sodium nitroprusside solution:</u>

The nitroprusside ion, which may be regarded as a special carrier of the nitrosonium ion, forms a coloured complex with methyl ketones. In presence of alkali eg acetone is converted to CH_3COCH^{2-} ion which reacts with nitroprusside ion $[Fe(CN)_5NO]^{2-}$ to give highly coloured ion $[Fe(CN)_5NOCH_2COCH_3]^{2-}$.

$$CH_{3}COCH_{3} \xrightarrow{OH^{-}} CH_{2}COCH_{3} \xrightarrow{Na_{2}[Fe(CN)_{5}NO]} [Fe(CN)_{5}NO(CH_{2}COCH_{3})]^{2}$$

S. No.	Compound	Nature	Aromatic or Aliphatic	Functional Group
1.	Benzoic acid	Colourless solid	Aromatic	Monocarboxylic acid
2.	Phthalic acid	Colourless solid	Aromatic	Dicarboxylic acid
3.	Cinnamic acid	Colourless solid	Aromatic	Monocarboxylic acid
4.	Benzamide	Colourless solid	Aromatic	Amide
5.	Urea	Colourless solid	Aliphatic	Diamide
6.	Glucose	Colourless solid	Aliphatic	Carbohydrate
7.	Phenol	Colourless liquid	Aromatic	Phenol
8.	Aniline	Pale brown liquid	Aromatic	Amine
9.	Benzaldehyde	Colourless liquid	Aromatic	Aldehyde
10.	Acetophenone	Colourless liquid	Aromatic	Ketone

No	Experiment	Observation	Inference
1	Reaction of sodium hydroxide solution		
	i) A little of the substance is boiled with dil. NaOH.ii) Substance is boiled strongly with 20% NaOH solution. Then cooled and acidified with dil. HCl.	a. Ammonia is evolved.	Presence of amides.
		b. Substance dissolved.	Presence of acidic substances.
		c. White crystalline ppt.	Presence of aromatic amides.
2	Reaction of NaHCO₃ To a few ml of the saturated NaHCO ₃ solution taken in a test tube, a little of the substance is added.	Brisk effervescence with the liberation of CO ₂ .	Presence of acids.
3	Reaction of FeCl₃ solution To a little of the substance in water or alcohol a few drops of neutral FeCl ₃ solution is added.	Violet colour.	Presence of phenols.
4	Reaction of Schiff's reagent A little of the substance is added to 1ml of the Schiff's reagent.	Violet colour developed within 2 minutes.	Presence of aldehydes.
5	Reaction of Borsche's reagent A little of the substance in methanol is heated with few drops of Borsches reagent in a water bath.	A yellowish orange ppt.	Presence of aldehydes or ketones.

6	Diazotisation	Rapid	Presence of
	Dissolve a little of the sample in 2ml con HCl	foaming.	primary
	diluted with water and cool in ice. Dissolve		aliphatic
	sodium nitrite in water and add the solution		amines.
	dropwise to the cold solution nitrite in water and		
	add the solution drop wise to the cold solution.		

Tests for aldehydes

Reaction of Tollen's reagent A little of the substance is boiled with few drops of Tollen's reagent.	Black ppt	Presence of aldehydes
Reaction of sodium bisulphite solution Two drops of the aldehyde is shaken with saturated solution of NaHSO ₃	White crystalline ppt.	Presence of aldehydes.

Tests for ketones

Nitroprusside test:	Wine red colour.	Presence of methyl
Add a few drops of sodium nitroprusside solution to few drops of ketone. Then add NaOH solution in excess.		ketones.

Test for acids

Fluorescein Reaction: A little of the substance is heated with Conc.H ₂ SO ₄ and Resorcinol in a dry test tube. It is cooled and then	A red solution with intense green fluorescence.	Presence of dicarboxylic acid.
---	---	--------------------------------

poured into a beaker containing excess of NaOH.		
Anhydride formation: A little of the acid is heated in a dry china dish covered with an inverted funnel whose stem is closed. It is then cooled.	White shiny needles are deposited on the sides of the funnel.	Presence of dicarboxylic acid

Tests for amides:

Biuret reaction: A little of the substance is heated first gently in a dry test tube followed by strong heating. The solid residue is warmed with1 mL 10% NaOH then cooled and one drop of dil.CuSO ₄ added.	On heating smell of ammonia is evolved and violet colour on adding CuSO _{4.}	Presence of diamide.
Hydroxamic acid test: Place a little of the substance in 5mL water. Add few drops 3% hydrogen peroxide and 2 drops of 5% ferric chloride. Heat the solution.	Magenta colour	Presence of aromatic primary amide.

Test for aromatic amines (primary):

Confirmatory Tests for Functional Groups:

Add 2mL of cold diazonium solution to a solution of 0.1g 2 -naphthol in 2ml 10% NaOH. and 5 mL water.	Orange –red dye.	Presence of aromatic amines.
Benzoylation: Dissolved a little of the substance in 10mL 10% NaOH solution contained in a boiling tube. About 1mL of benzoyl chloride is added. The boiling tube is corked and shaken vigorously for about 15 minutes.	A white ppt is formed.	Presence of aromatic amines.

Test for aliphatic amines (primary):

To 0.3 mL or 300 mg of		
unknown substance in a test	Soluble in base.	Presence of aliphatic primary
tube add 5 mL of 10%		amines.
NaOH solution and 0.4 mL		
of benzenesulfonyl chloride.		
Close the test tube with a		
cork and shake the mixture		
vigorously. Test the solution		
to make sure that it is still		
alkaline using litmus paper.		
Cool the solution and add	A precipitate is formed.	Presence of aliphatic primary
10% HCl solution dropwise.		amines.

Tests for aromatic alcohol (Phenol):

Azo-dye formation: Dissolve two drops of aniline in 1 mL dil.HCl well cooled in ice. Few drops of saturated sodium nitrite solution are added. Then it is added to a well cooled solution of the phenolic compound in aqueous sodium hydroxide.	A red coloured substance is formed.	Presence of phenols.
Benzoylation: Dissolved a little of the substance in 5mL 20% NaOH solution contained in a boiling tube. About 1mL of benzoyl chloride is added. The boiling tube is corked and shaken vigorously for about 15 minutes.	A precipitate is formed.	Presence of phenols.

S. No.	Experiment	Observation	Inference
	I. Preliminary test		
1.	Colour and appearance Colour and appearance of the substance is noted.	a) Brown or dark coloured solid or liquid	a) May be aromatic amines or phenols
		b) Colourless liquid	b) May be aldehyde or ketone
		c) Colourless solid	c) May be acids or amides or carbohydrates
2.	Odour of the substance Odour of the given substance is noted.	a) Phenolic smell	a) May be phenols
		b) Aniline like smell	b) May be aromatic amine (aniline)
		c) Odour of almond	c) May be aldehyde
		d) Pleasant odour	d) May be ketone
		e) No characteristic odour	e) Absence of amine, phenols, aldehyde and ketone.
3.	Solubility test Solubility of the given organic substance is tested in the following solvents.		
	a) In water	a) Soluble in the cold condition	a) May be carbohydrates or diamide like urea
		b) Soluble in the hot condition	b) May be aromatic acids or amides
	b) In dilute hydrochloric acid	a) Soluble	a) May be aromatic amine (aniline)
		b) Insoluble	b) Absence of aromatic amine (aniline)
4.	Sodium carbonate test A little of the substance is added to 1 ml of a strong solution of sodium carbonate.	a) Vigorous effervescence takes place evolving carbon dioxide gas	a) Presence of acids
		b) No vigorous	b) Absence of acids

GENERAL PROCEDURE

		effervescence takes	
5.	Sodium hydroxide test: To a little of the substance about 2 ml of 10% sodium hydroxide solution is added and boiled gently.	place a) Dissolves readily in the cold condition and the substance is regenerated on adding dilute hydrochloric acid b) Solution turns yellow or brown on boiling	a) Presence of acidsb) May be carbohydrates
6.	Soda-Lime test A little of the substance is heated with powdered soda lime and heated strongly.	c) On heating ammonia gas is evolved. It gives dense white fumes with a glass rod dipped in conc. hydrochloric acid	c) May be amides
		a) Ammonia gas is evolved	a) May be amides
		b) No ammonia gas is evolved	b) Absence of amides
7.	Conc. sulphuric acid test A little of the substance is treated with 2 ml of conc. sulphuric acid and warmed.	a) The substance chars with smell of burnt sugar	a) May be carbohydrates
		b) No characteristic change	b) Absence of carbohydrates
8.	Neutral FeCl ₃ test To a little of the substance dissolved in water or alcohol, about 2 ml of neutral ferric chloride is added.	a) Violet or blue or green colour is obtained	a) Presence of phenol
	chioride is added.	b) No violet or blue or green colour is obtained	b) Absence of phenol
	II. Test for functional groups		•
1.	Test for acids a) Ester test A little of the substance is mixed with a few drops of alcohol and 2 drops of conc. sulphuric acid. The mixture is gently warmed and poured into a beaker containing dilute sodium carbonate solution.	a) A pleasant fruity odour is notedb) No fruity odour is noted	a) Presence of carboxylic acidb) Absence of carboxylic acid

	b) Phenolphthalein test		
	To about 2 ml of sodium hydroxide solution, 1 drop of dilute phenolphthalein indicator is added pink	a) Pink colour disappears	a) Presence of carboxylic acid
	colour appears. To this, the substance dissolved in water or alcohol is added drop by drop in excess.	b) Pink colour does not disappear	b) Absence of carboxylic acid
	c) Fluorescein test A small amount of the substance is mixed with few drops of resorcinol in a dry test tube. 3 drops of conc. sulphuric acid is added.	a) An intense greenish yellow fluorescence is produced	a) Presence of dicarboxylic acid
	Shake well, boil gently and then pour into 100 ml of cold water taken in a beaker. Stir well and then sodium hydroxide solution is added in drops.	b) No intense greenish yellow fluorescence is produced	b) Presence of monocarboxylic acid
2.	Test for carbohydrates a) Molisch's test To a little of the substance in water a few drops of an alcoholic solution of 1-naphthol are added. To this mixture, conc. sulphuric acid	a) Violet ring is obtained at the junction of the two layers and this spreads slowly	a) Presence of carbohydrate
	is added along the sides of the test tube without shaking.	b) No violet ring is obtained	b) Absence of carbohydrate
	b) Tollen's reagent test To a little of the substance add about 2 ml of Tollen's reagent. This is shaken well	a) Black precipitate or bright silver mirror is formed	a) Presence of carbohydrate
	and heated in a boiling water bath.	b) No black precipitate or bright silver mirror is formed	b) Absence of carbohydrate
	c) Fehling's test 1 ml of Fehling solution (A) is mixed with 1 ml of Fehling solution (B). The mixture is added to a little of the substance dissolved in	a) Red precipitate is obtained	a) Presence of reducing sugars
	water, shaken well and heated in a boiling water bath.	b) No red precipitate is obtained	b) Absence of reducing sugars

3.	Test for carbobydrates		
5.	Test for carbohydrates a) Molisch's test To a little of the substance in water a few drops of an alcoholic solution of 1-naphthol are added. To this mixture, conc. sulphuric acid	a) Violet ring is obtained at the junction of the two layers and this spreads slowly	a) Presence of carbohydrate
	is added along the sides of the test tube without shaking.	b) No violet ring is obtained	b) Absence of carbohydrate
	b) Tollen's reagent test To a little of the substance add about 2 ml of Tollen's reagent. This is shaken well	a) Black precipitate or bright silver mirror is formed	a) Presence of carbohydrate
	and heated in a boiling water bath.	 b) No black precipitate or bright silver mirror is formed 	b) Absence of carbohydrate
	c) Fehling's test 1 ml of Fehling solution (A) is mixed with 1 ml of Fehling solution (B). The mixture is added to a little of the substance dissolved in	a) Red precipitate is obtained	a) Presence of reducing sugars
	water, shaken well and heated in a boiling water bath.	b) No red precipitate is obtained	b) Absence of reducing sugars
4.	Test for Aldehyde and		
	Ketone a) Schiff's reagent test To a little of the substance Schiff's reagent is added and	a) Violet colour is formed	a) Presence of aromatic aldehyde
	shaken well.	b) No violet colour is formed	b) Absence of aromatic aldehyde
	b) Tollen's reagent test To a little of the substance a few drops of Tollen's reagent is added and kept it in a hot	a) Silver mirror is obtained	a) Presence of aldehyde
	water bath.	b) No silver mirror is obtained	b) Presence of ketone
	c) Legal's test To a little of the substance a few drops of freshly prepared sodium nitroprusside and a	a) Wine red or blue colour is formed	a) Presence of ketone like acetophenone
	few drops of 10% sodium hydroxide is added.	b) No wine red or blue colour is formed	b) Absence of ketone

	d) <i>m</i> -Dinitrobenzene test To a little of the substance a small amount of <i>m</i> -dinitrobenzene and a few drops of dilute sodium hydroxide is added.	a) Violet or red colour is formedb) No violet or red colour is formed	a) Presence of ketone like acetophenoneb) Absence of ketone
5.	Test for amides a) Sodium hydroxide test A little of the substance is heated with 5 ml of 10% sodium hydroxide solution till no more ammonia is evolved. It is then cooled and acidified with conc. hydrochloric acid.	a) White precipitate is obtainedb) No white precipitate is obtained	a) Presence of an aromatic amideb) Presence of an aliphatic amide (urea)
	b) Biuret test A little of the substance is heated in a dry test tube for a few minutes. It is cooled and the residue is dissolved in 2 ml of water. To this 2 drops of dilute copper sulphate solution are added and then 10% sodium hydroxide solution is added dropwise.	a) Violet colour is obtainedb) No violet colour is obtained	a) Presence of a diamide like ureab) Absence of a diamide like urea
	c) Oxalic acid test To a strong aqueous solution of the substance added oxalic acid solution, shaken well.	a) White precipitate is obtainedb) No white precipitate is obtained	a) Presence of a diamide like ureab) Absence of a diamide like urea
6.	Test for amines a) Reaction on nitrous acid A little of the substance is dissolved in about 3 ml of dilute hydrochloric acid. To this a strong solution of sodium nitrate is added drop wise cooling the mixture in ice cold water.	a) A clear solution is producedb) No clear solution is produced	a) Presence of aromatic primary amine (aniline)b) Absence of aromatic primary amine (aniline)
	To the clear solution, a solution of 2-naphthol in sodium hydroxide is added.	a) Scarlet red dye is obtainedb) No scarlet red dye is obtained	a) Presence of aromatic primary amine (aniline)b) Absence of aromatic primary amine (aniline)

Report:

- 1. Functional group present
- 2. The given organic compound is.....

MODEL ANALYSIS-1

Urea (Diamide)

S. No.	Experiment	Observation	Inference
5.110.	I. Preliminary test	Obset vation	Interence
1.	Colour and appearance		
1.	Colour and appearance	Colourless solid	May be acids or amides or
	of the substance is noted.	Colouriess solid	carbohydrates
2.	Odour of the substance		carbonyurates
۷.	Odour of the given substance	No characteristic odour	Absence of amine, phenols,
	is noted.	No characteristic odour	aldehyde and ketone.
3.	Solubility test		aldenyde and ketone.
5.	Solubility of the given		
	organic substance is tested in		
	the following solvents.		
	the following solvents.		
	In water	Soluble in the cold	May be carbohydrates or
	III water	condition	May be carbohydrates or diamide like urea
		condition	diamide like drea
4.	Sodium carbonate test		
	A little of the substance is	No vigorous	Absence of acids
	added to 1 ml of a strong	effervescence takes	
	solution of sodium	place	
	carbonate.	prace	
5.	Sodium hydroxide test:	On heating ammonia	
	To a little of the substance	gas is evolved. It gives	May be amides
	about 2 ml of 10% sodium	dense white fumes with	
	hydroxide solution is added	a glass rod dipped in	
	and boiled gently.	conc. hydrochloric acid	
6.	Soda-Lime test		
	A little of the substance is	Ammonia gas is	May be amides
	heated with powdered soda	evolved	5
	lime and heated strongly.		
7.	Conc. sulphuric acid test		
	A little of the substance is	No characteristic	Absence of carbohydrates
	treated with 2 ml of conc.	change	
	sulphuric acid and warmed.		
8.	Neutral FeCl ₃ test		
	To a little of the substance	No violet or blue or	Absence of phenol
	dissolved in water or alcohol,	green colour is	
	about 2 ml of neutral ferric	obtained	
	chloride is added.		
1	II. Test for functional groups		
1.	Test for acids		
	Ester test	No fruity adour is	Absonce of corboratio
	A little of the substance is	No fruity odour is	Absence of carboxylic

	mixed with a few drops of alcohol and 2 drops of conc. sulphuric acid. The mixture is gently warmed and poured into a beaker containing dilute sodium carbonate solution.	noted	acid
2.	Test for phenols Liebermann's reaction A little of the substance is mixed with a few crystals of sodium nitrate and 3 or 4 drops of conc. sulphuric acid. This is gently warmed and poured into water. To this sodium hydroxide is added and stirred well.	No red solution is obtained	Absence of phenol
3.	Test for carbohydrates Molisch's test To a little of the substance in water a few drops of an alcoholic solution of 1-naphthol are added. To this mixture, conc. sulphuric acid is added along the sides of the test tube without shaking.	No violet ring is obtained	Absence of carbohydrate
4.	Test for Aldehyde and Ketone a) Schiff's reagent test To a little of the substance Schiff's reagent is added and shaken well.	No violet colour is formed	Absence of aromatic aldehyde
	b) Legal's test To a little of the substance a few drops of freshly prepared sodium nitroprusside and a few drops of 10% sodium hydroxide is added.	No wine red or blue colour is formed	Absence of ketone

5.	Test for amides a) Sodium hydroxide test A little of the substance is heated with 5 ml of 10% sodium hydroxide solution till no more ammonia is evolved. It is then cooled and acidified with conc. hydrochloric acid.	No white precipitate is obtained	Presence of an aliphatic amide (urea)
	b) Biuret test A little of the substance is heated in a dry test tube for a few minutes. It is cooled and the residue is dissolved in 2 ml of water. To this 2 drops of dilute copper sulphate solution are added and then 10% sodium hydroxide solution is added dropwise.	Violet colour is obtained	Presence of a diamide like urea
	c) Oxalic acid test To a strong aqueous solution of the substance added oxalic acid solution, shaken well.	White precipitate is obtained	Presence of a diamide like urea
6.	Test for amines Action on nitrous acid A little of the substance is dissolved in about 3 ml of dilute hydrochloric acid. To this a strong solution of sodium nitrate is added drop wise cooling the mixture in ice cold water.	No characteristic change	Absence of aromatic primary amine (aniline)

Report:

- 1. Functional group present Diamide
- 2. The given organic compound is **urea**

MODEL ANALYSIS-2

Aniline (Amine)

S. No.	Experiment	Observation	Inference
	I. Preliminary test	o sour fution	
1.	Colour and appearance Colour and appearance of the substance is noted.	Brown coloured liquid	May be aromatic amines
2.	Odour of the substance Odour of the given substance is noted.	Aniline like smell	May be aromatic amine (aniline)
3.	Solubility test Solubility of the given organic substance is tested in the following solvents.		
	In dilute hydrochloric acid	Soluble	May be aromatic amine (aniline)
4.	Sodium carbonate test A little of the substance is added to 1 ml of a strong solution of sodium carbonate.	No vigorous effervescence takes place	Absence of acids
5.	Sodium hydroxide test: To a little of the substance about 2 ml of 10% sodium hydroxide solution is added and boiled gently.	No characteristic change	Absence of acids, carbohydrates and amides
6.	Soda-Lime test A little of the substance is heated with powdered soda lime and heated strongly.	No ammonia gas is evolved	Absence of amides
7.	Conc. sulphuric acid test A little of the substance is treated with 2 ml of conc. sulphuric acid and warmed.	No characteristic change	Absence of carbohydrates
8.	Neutral FeCl ₃ test To a little of the substance dissolved in water or alcohol, about 2 ml of neutral ferric chloride is added.	No violet or blue or green colour is obtained	Absence of phenol
L			
1.			
1.	To a little of the substance dissolved in water or alcohol, about 2 ml of neutral ferric	green colour is obtained	Absence of phenol

	A little of the substance is mixed with a few drops of alcohol and 2 drops of conc. sulphuric acid. The mixture is gently warmed and poured into a beaker containing dilute sodium carbonate solution.	No fruity odour is noted	Absence of carboxylic acids
2.	Test for phenols Liebermann's reaction A little of the substance is mixed with a few crystals of sodium nitrate and 3 or 4 drops of conc. sulphuric acid. This is gently warmed and poured into water. To this sodium hydroxide is added and stirred well.	No red solution is obtained	Absence of phenol
3.	Test for carbohydrates Molisch's test To a little of the substance in water a few drops of an alcoholic solution of 1-naphthol are added. To this mixture, conc. sulphuric acid is added along the sides of the test tube without shaking.	No violet ring is obtained	Absence of carbohydrate
4.	Test for Aldehyde and Ketone a) Schiff's reagent test To a little of the substance Schiff's reagent is added and shaken well.	No violet colour is formed	Absence of aromatic aldehyde
	b) Legal's test To a little of the substance a few drops of freshly prepared sodium nitroprusside and a few drops of 10% sodium hydroxide is added.	No wine red or blue colour is formed	Absence of ketone

5.	Test for amides Sodium hydroxide test A little of the substance is heated with 5 ml of 10% sodium hydroxide solution till no more ammonia is evolved. It is then cooled and acidified with conc. hydrochloric acid.	No characteristic change	Absence of amides
6.	Test for amines Action on nitrous acid A little of the substance is dissolved in about 3 ml of dilute hydrochloric acid. To this a strong solution of sodium nitrate is added drop wise cooling the mixture in ice cold water.	A clear solution is produced	Presence of aromatic primary amine (aniline)
	To the clear solution, a solution of 2-naphthol in sodium hydroxide is added.	Scarlet red dye is obtained	Presence of aromatic primary amine (aniline)

Report:

- 1. Functional group present aromatic amine
- 2. The given organic compound is **aniline**

MODEL ANALYSIS-3

Carbohydrate (Glucose)

S. No.	Experiment	Observation	Inference
	I. Preliminary test		
1.	Colour and appearance Colour and appearance	Colourless solid	May be acids or amides or
2.	of the substance is noted. Odour of the substance		carbohydrates
Ζ.	Odour of the given substance is noted.	No characteristic odour	Absence of amine, phenols, aldehyde and ketone.
3.	Solubility test Solubility of the given organic substance is tested in the following solvents.	Soluble in the cold condition	May be carbobydrates
	m water	condition	May be carbohydrates or diamide like urea
4.	Sodium carbonate test A little of the substance is added to 1 ml of a strong solution of sodium carbonate.	No vigorous effervescence takes place	Absence of acids
5.	Sodium hydroxide test: To a little of the substance about 2 ml of 10% sodium hydroxide solution is added and boiled gently.	Solution turns yellow or brown on boiling	May be carbohydrates
6.	Soda-Lime test A little of the substance is heated with powdered soda lime and heated strongly.	No ammonia gas is evolved	Absence of amides
7.	Conc. sulphuric acid test A little of the substance is treated with 2 ml of conc. sulphuric acid and warmed.	The substance chars with smell of burnt sugar	May be carbohydrates
8.	Neutral FeCl₃ test To a little of the substance dissolved in water or alcohol, about 2 ml of neutral ferric chloride is added.	No violet or blue or green colour is obtained	Absence of phenol
	II. Test for functional groups	1	

Test for acids Ester test A little of the substance is mixed with a few drops of alcohol and 2 drops of conc. sulphuric acid. The mixture is gently warmed and poured into a beaker containing dilute sodium carbonate solution.	No fruity odour is noted	Absence of carboxylic acids
Test for phenols Liebermann's reaction A little of the substance is mixed with a few crystals of sodium nitrate and 3 or 4 drops of conc. sulphuric acid. This is gently warmed and poured into water. To this sodium hydroxide is added and stirred well.	No red solution is obtained	Absence of phenol
Test for carbohydrates a) Molisch's test To a little of the substance in water a few drops of an alcoholic solution of 1-naphthol are added. To this mixture, conc. sulphuric acid is added along the sides of the test tube without shaking.	Violet ring is obtained at the junction of the two layers and this spreads slowly	Presence of carbohydrate
b) Tollen's reagent test To a little of the substance add about 2 ml of Tollen's reagent. This is shaken well and heated in a boiling water bath.	Black precipitate or bright silver mirror is formed	Presence of carbohydrate
c) Fehling's test 1 ml of Fehling solution (A) is mixed with 1 ml of Fehling solution (B). The mixture is added to a little of the substance dissolved in water, shaken well and heated in a boiling water bath.	Red precipitate is obtained	Presence of reducing sugars
Test for Aldehyde and Ketone		
a) Schiff's reagent test		

	To a little of the substance Schiff's reagent is added and shaken well.	No violet colour is formed	Absence of aromatic aldehyde
	b) Legal's test To a little of the substance a few drops of freshly prepared sodium nitroprusside and a few drops of 10% sodium hydroxide is added.	No wine red or blue colour is formed	Absence of ketone
5.	Test for amides Sodium hydroxide test A little of the substance is heated with 5 ml of 10% sodium hydroxide solution till no more ammonia is evolved. It is then cooled and acidified with conc. hydrochloric acid.	No characteristic change	Absence of amides
6.	Test for amines Action on nitrous acid A little of the substance is dissolved in about 3 ml of dilute hydrochloric acid. To this a strong solution of sodium nitrate is added drop wise cooling the mixture in ice cold water.	No characteristic change	Absence of aromatic primary amine (aniline)

Report:

- 1. Functional group present aliphatic aldehyde
- 2. The given organic compound is **carbohydrate**

MODEL ANALYSIS-4

Benzoic acid (Monocarboxylic acid)

S. No.	Experiment	Observation	Inference
	I. Preliminary test		
1.	Colour and appearance Colour and appearance of the substance is noted.	Colourless solid	May be acids or amides or carbohydrates
2.	Odour of the substance Odour of the given substance is noted.	No characteristic odour	Absence of amine, phenols, aldehyde and ketone.
3.	Solubility test Solubility of the given organic substance is tested in the following solvents.		
	In water	Soluble in the hot condition	May be aromatic acids or amides
4.	Sodium carbonate test A little of the substance is added to 1 ml of a strong solution of sodium carbonate.	Vigorous effervescence takes place evolving carbon dioxide gas	Presence of acids
5.	Sodium hydroxide test: To a little of the substance about 2 ml of 10% sodium hydroxide solution is added and boiled gently.	Dissolves readily in the cold condition and the substance is regenerated on adding dilute hydrochloric acid	Presence of acids
6.	Soda-Lime test A little of the substance is heated with powdered soda lime and heated strongly.	No ammonia gas is evolved	Absence of amides
7.	Conc. sulphuric acid test A little of the substance is treated with 2 ml of conc. sulphuric acid and warmed.	No characteristic change	Absence of carbohydrates
8.	Neutral FeCl₃ test To a little of the substance dissolved in water or alcohol, about 2 ml of neutral ferric chloride is added.	No violet or blue or green colour is obtained	Absence of phenol
	II. Test for functional groups	5	

1	Tost for asida		
1.	Test for acids		
	a) Ester test		
	A little of the substance is mixed with a few drops of	A placent fruity adaur	Presence of carboxylic
	mixed with a few drops of	is noted	Presence of carboxylic acid
	alcohol and 2 drops of	is noted	aciu
	conc. sulphuric acid. The mixture is gently warmed		
	and poured into a beaker		
	containing dilute sodium		
	carbonate solution.		
	b) Phenolphthalein test		
	To about 2 ml of sodium	Pink colour disappears	Presence of carboxylic
	hydroxide solution, 1 drop of		acid
	dilute phenolphthalein		
	indicator is added pink		
	colour appears. To this, the substance dissolved in water		
	or alcohol is added drop by		
	drop in excess.		
	drop in excess.		
	c) Fluorescein test		
	A small amount of the	No intense greenish	
	substance is mixed with few	yellow fluorescence is	monocarboxylic acid
	drops of resorcinol in a dry	produced	
	test tube. 3 drops of conc.		
	sulphuric acid is added.		
	Shake well, boil gently and then pour into 100 ml of cold		
	water taken in a beaker. Stir		
	well and then sodium		
	hydroxide solution is added		
	in drops.		
2.	Test for phenols		
	Liebermann's reaction		
	A little of the substance is		
	mixed with a few crystals of	No red solution is	Absence of phenol
	sodium nitrate and 3 or 4	obtained	
	drops of conc. sulphuric		
	acid. This is gently warmed		
	and poured into water. To this sodium hydroxide is		
	added and stirred well.		

3.	Test for carbohydratesMolisch's testTo a little of the substance in water a few drops of an alcoholic solution of 1-naphthol are added. To this mixture, conc. sulphuric acid is added along the sides of the test tube without shaking.	No violet ring is obtained	Absence of carbohydrate
4.	Test for Aldehyde and Ketone a) Schiff's reagent test To a little of the substance Schiff's reagent is added and shaken well.	No violet colour is formed	Absence of aromatic aldehyde
	 b) Legal's test To a little of the substance a few drops of freshly prepared sodium nitroprusside and a few drops of 10% sodium hydroxide is added. 	No wine red or blue colour is formed	Absence of ketone
5.	Test for amides Sodium hydroxide test A little of the substance is heated with 5 ml of 10% sodium hydroxide solution till no more ammonia is evolved. It is then cooled and acidified with conc. hydrochloric acid.	No characteristic change	Absence of amides
6.	Test for aminesAction on nitrous acidA little of the substance isdissolved in about 3 ml ofdilute hydrochloric acid. Tothis a strong solution ofsodium nitrate is added dropwise cooling the mixture inice cold water.	No characteristic change	Absence of aromatic primary amine (aniline)

Report:

- 1. Functional group present Aromatic monocarboxylic acid
- 2. The given organic compound is **Benzoic acid**.

VIVA-VOCE QUESTIONS

Detection of functional group present in the organic compound.

Q.1. What are the functional group?

Q.2. What do you understand by the term nitrogen containing functional group? Give a few examples.

Q.3. What do you understand by the term oxygen containing functional group? Give a few examples.?

Q. 4. Write the Biuret reaction? This test is used for which class of Functional group?

Q.5. What is different between the amide and amine? Give examples?

Q.6. List out the carboxylic acid with examples.

Q.7. Why monocarboxylic acid should not answer the fluorescein test?

Q.8. Give the formulae of the complex responsible for violet colour in test for phenol.

Q.9. How to prepare the aryldiazonium salts? Explain the coupling reactions

Q.10. What is primary amine? Give a few examples

Q.11. What is color of Schiff base solution?

Q.12. Schiff's test is used for which class of compounds?

Q.13. Name a reagent used to detect carbonyl group in a compound?

Q.14. What is nitrous acid?

Q.15. Aldehyde when treated with Fehling's solution A and Fehling's solution B gives a red coloured precipitate. What is that precipitate?

Q.16. Which one is more acidic alcohol or phenol?

Q.17. Name a test by which you can distinguish between hexylamine and aniline?

Q.18. White precipitate of silver chloride dissolves in ammonia solution. Why?

Q.19. Name two tests which distinguish aldehydes and ketones?

Q.20. Name of the reagent used to detect the aldehydes group in a compound?

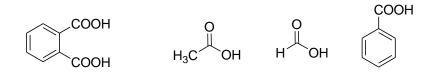
Q.21. What is Tollen's reagent?

Q.22. What is the application of carbylamines test?

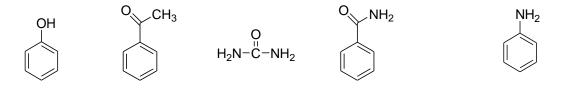
- Q.23. Why aniline is weaker base than ammonia?
- Q.24. How can you distinguish methanol and Ethanol?
- Q.25. Whencompound treated with Br₂ water gives white precipitate?
- Q.26. Explain the bromination test for phenol and aniline?
- Q.27. Why Tollen's reagent give block precipitate for aromatic aldehyde?
- Q.28. Which compound give silver mirror with Tollen's reagent? Why.
- Q.29. Carboxylic acid react with alcohol give which compound?
- Q.30. In fruity odour test what is role for sulfuric acid?
- Q.31. What type of reaction involve in the Phenolphthalein test?
- Q.32. Which reaction evolves the CO₂ gas?
- Q.33. Which reaction evolves the NH₃ gas?
- Q.34. What is functional group present in alkene and alkyne?
- Q.35. What is lime water?
- Q.36. Explain two test for identification of carboxylic acid?
- Q.37. Write chemical reaction for "naturalization"?
- Q.38. Identify the organic class to which the functional group belongs.

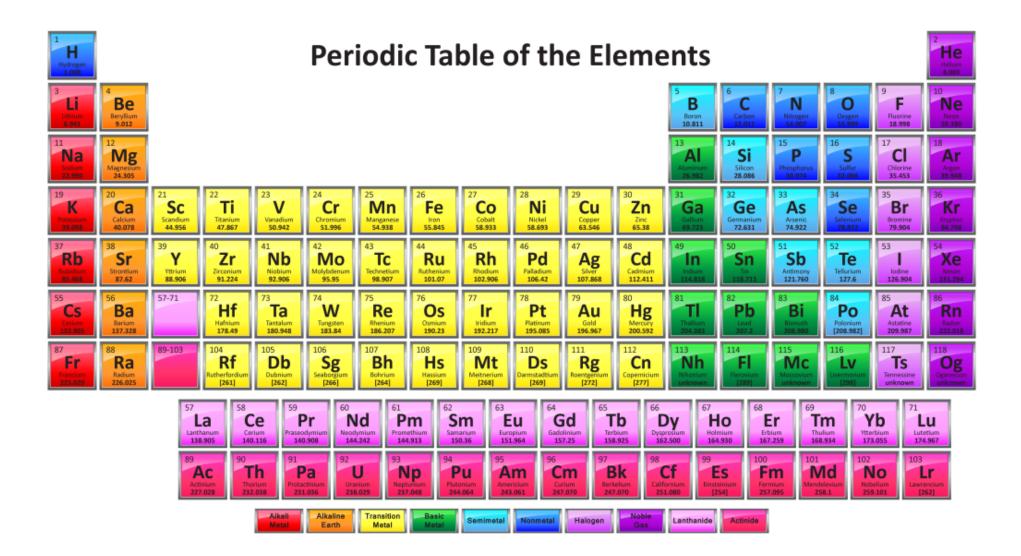


Q.39. Identify the solid carboxylic acids



Q.40. Identify the liquid organic compounds.





VIVA-VOCE QUESTIONS

Detection of functional group present in the organic compound.

Q.1. What are the functional group?

Q.2. What do you understand by the term nitrogen containing functional group? Give a few examples.

Q.3. What do you understand by the term oxygen containing functional group? Give a few examples.?

Q. 4. Write the Biuret reaction? This test is used for which class of Functional group?

Q.5. What is different between the amide and amine? Give examples?

Q.6. List out the carboxylic acid with examples.

Q.7. Why monocarboxylic acid should not answer the fluorescein test?

Q.8. Give the formulae of the complex responsible for violet colour in test for phenol.

Q.9. How to prepare the aryldiazonium salts? Explain the coupling reactions

Q.10. What is primary amine? Give a few examples

Q.11. What is color of Schiff base solution?

Q.12. Schiff's test is used for which class of compounds?

Q.13. Name a reagent used to detect carbonyl group in a compound?

Q.14. What is nitrous acid?

Q.15. Aldehyde when treated with Fehling's solution A and Fehling's solution B gives a red coloured precipitate. What is that precipitate?

Q.16. Which one is more acidic alcohol or phenol?

Q.17. Name a test by which you can distinguish between hexylamine and aniline?

Q.18. White precipitate of silver chloride dissolves in ammonia solution. Why?

Q.19. Name two tests which distinguish aldehydes and ketones?

Q.20. Name of the reagent used to detect the carbonyl group in a compound?

Q.21. What is Tollen's reagent?

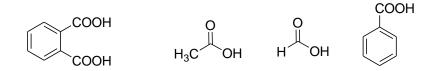
Q.22. What is the application of carbylamines test?

Q.23. Why aniline is is weaker base than ammonia?

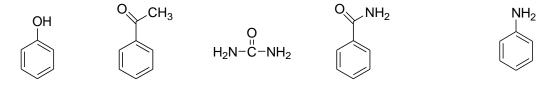
- Q.24. How can you distinguish methanol and Ethanol?
- Q.25. Whencompound treated with Br₂ water gives white precipitate?
- Q.26. Explain the bromination test for phenol and aniline?
- Q.27. Why Tollen's reagent give block precipitate for aromatic aldehyde?
- Q.28. Which compound give silver mirror with Tollen's reagent? Why.
- Q.29. Carboxylic acid react with alcohol give which compound?
- Q.30. In fruity odour test what is role for sulfuric acid?
- Q.31. What type of reaction involve in the Phenolphthalein test?
- Q.32. Which reaction evolves the CO₂ gas?
- Q.33. Which reaction evolves the NH₃ gas?
- Q.34. What is functional group present in alkene and alkyne?
- Q.35. What is lime water?
- Q.36. Explain two test for identification of carboxylic acid?
- Q.37. Write chemical reaction for "naturalization"?
- Q.38. Identify the organic class to which the functional group belongs.



Q.39. Identify the solid carboxylic acids



Q.40. Identify the liquid organic compounds.





KARPAGAM ACADEMY OF HIGHER EDUCATION (Deemed to be University Under Section 3 of UGC Act 1956) COIMBATORE-21 DEPARTMENT OF CHEMISTRY B.Sc Chemistry – Syllubus

DEPARTMENT OF CHEMISTRY <u>Model Practical Examination-September-2017</u>

Date	: 24-09-2017 (FN)	Time: 09.30 – 12.30 (3 hrs)
Title of the Exam	: Nitrogen Containing Functional Groups Heterocyclic	
	Chemistry and natural products - Practical	
Subject Code	: 16CHU313	Class: II-B.Sc.Chemistry "A"

QUESTION PAPER

1. Identity the functional group and the organic compound present in the unknown sample

Group-1:	16CHU002-007
Group-2:	16CHU008-014
Group-3:	16CHU015-022
Group-4:	16CHU023-027
Group-5:	16CHU028-034
Group-6:	16CHU035-040

Internal Examiner