Semester -I

19CHP111 ORGANIC CHEMISTRY PRACTICAL-I 4H 2C
(QUALITATIVE ANALYSIS AND SINGLE STAGE PREPARATIONS)

Instruction Hours/week:L:0 T:0 P:4 Marks: Internal:40External: 60 Total:100
External Semester Exam: 6 Hrs

Course Objectives

1. To provide the knowledge about the qualitative analysis by separation techniques of a two component mixture.
2. To develop skills for the preparation of organic compounds by name reactions.

Course outcomes

1. Familiarized the solubility nature of organic substances of different functional group.
2. Learned the pilot separation of bimixtures and familiarize the systematic procedure of organic substances analysis
3. Learned two stage preparation involving molecular rearrangement and well known organic reactions.
4. Familiarized the test involving identification of special elements
5. Learned the confirmatory test for various functional groups.
6. Understand the techniques involving drying and recrystalliation by various method
7. Expertise the various techniques of preparation and analysis of organic substances.
8. Calculated a limiting reagent, yield, and percent yield
9. Evaluated collected data to determine the identity, purity, and yield of products

Contents

Analysis of two – component mixtures: Separation and characterization of compounds.

Note: Each student should analyze a minimum of six organic mixtures.

Preparations involving one stage comprising of the following process: Nitration, acylation, halogenation, diazotisation, rearrangement, hydrolysis, reduction, alkylation and oxidation and one preparation illustrating the following: Benzoin condensation, Canizzaro reaction, Perkin reaction, Reimer-Tiemann reaction, Sandmayer reaction, Fries rearrangement, Skraup synthesis- single stage.

Note: Each student should do a minimum of six preparations involving single stage.

SUGGESTED READINGS

1. Thomas, A. O., (2003). Practical Chemistry. Cannanore: Scientific Book Center.
2. Bansal, R. K, (2008). Laboratory Manual of Organic Chemistry (IV Edition). New Delhi: New Age Publishers.

3. ArunSethi, (2003). Laboratory experiments in Organic Chemistry. New Delhi: New Age Publisher.
4. Furniss, B. S., Hannaford, A. J., Smith, P. W. G., &Tatchell, A. R., (2004). Vogel's Textbook of Practical Organic Chemistry (V Edition). Singapore: Pearson Education Ltd.
5. Lepse, P. A., & Lyle B. P., (1986). Lab Manual for Lingren's Essentials of Chemistry. New Delhi: Prentice Hall.

KARPAGAM ACADEMY OF HIGHER EDUCATION
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(Established Under Section 3 of UGC Act 1956)
COIMBATORE-21
DEPARTMENT OF CHEMISTRY

List of Experiments

Name of the Staff	:	**R. Kumar**
Department	:	**Chemistry**
Subject	:	**Organic Chemistry Practical -I**
Subject Code	:	**19CHP111**
Class	:	**I M.Sc-Chemistry**
Year and Semester	:	**I / I**

S.No	NAME OF THE EXPERIMENT
I	**Analysis of Organic Mixtures** **(5 Mixtures)**
II	**Organic preparations**
1	**Single stage preparations** Preparation of acetanilide from aniline
2	Preparation of p-bromoaniline from p-bromoacetanilide
3	Preparation of benzoic acid from benzaldehyde
4	Preparation of acetyl salicylic acid from salicylic acid

Lab Manual on

QUALITATIVE ANALYSIS AND SINGLE STAGE PREPARATIONS

DEPARTMENT OF CHEMISTRY

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Department : **Chemistry**
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CONTENTS

Analysis of Organic Mixtures

Pilot Test

S.No.	Experiment	Observation	Inference
1	**Ether Separation** A small portion of the given mixture is taken in a 100 ml dry beaker and about 25 ml of ether is added and the mixture is stirred well with a glass rod.	a. A part remained insoluble in ether. b. The whole mixtures gets dissolved.	Presence of benzamide, urea, thiourea and carbohydrates. Ether separation is not suitable.

If the whole mixture is completely soluble the following separations are done systematically.

2	**Sodium bicarbonate separation** The clear ether solution is taken in a test tube and is extracted with 10 % NaOH solution. Stirred well and the lower aqueous layer is removed and acidified with 1:1 HCl.	a. Precipitate is formed. b. No precipitate.	Presence of acidic component. Absence of acidic component.
3	**Sodium hydroxide separation** The clear ether solution is taken in a test tube and extracted with 10 ml of 10 % NaOH solution. Stirred well and the lower aqueous layer is removed and acidified with 1:1 HCl.	a. A precipitate or emlusion is formed. b. No precipitate.	Presence of phenolic component. Absence of phenol component.
4	**Acid separation** The clear ether solution is taken in a test tube and is extracted with 10 ml of 1:1 HCl. Sstirred well and the lower aqueous layer is removed and neutralized with 10 % NaOH solution.	a. A precipitate or emlusion is formed. b. No precipitate.	Presence of basic component. Absence of basic component.

Preliminary Report

The given mixture contains

1) Ether soluble components or

2) One ether soluble component and other ether insoluble component.

Bulk separation

After determining the separation procedures by doing pilot test, the major portion of the mixture is taken and the components are separated as follows.

1. Ether separation

The major portion of the mixture is stirred well with 25ml of ether in a 250 ml of dry beaker. The ether layer is decanted. The procedure is repeated three times. The ether soluble component is recovered by evaporation of ether.The ether insoluble component is dried.

2. Sodium bicarbonate separation

The ethereal solution in the beaker is transferred into a 250ml separating funnel. About 20ml of cold saturated solution of $NaHCO_3$ is added and shaken well releasing the pressure occasionally. Carboxylic acid if present will remain in the sodium bicarbonate layer as sodium salt and sodium bicarbonate insoluble component is present in the ethereal layer.

The aqueous layer is removed and transferred to a 100ml beaker. 1:1 HCl is added. Stirred well the solid is filtered on a Bucked funnel and washed thoroughly with water. The solid is dried.

3. Sodium hydroxide separation

About 25ml of 10% NaOH solution is added to the ethereal solution in a beaker and transferred into a 250ml separating funnel. Shaken well releasing the pressure occasionally. The ethereal layer and aqueous layer are found separated. The ethereal layer contains NaOH insoluble component and aqueous layer contains NaOH soluble component. The aqueous layer is removed and neutralized with 1:1 HCl. If a solid separates it is filtered on a Bucked funnel. If oily globules (emulsion) separates the following procdure is adopted.

The solution with oily globules is transferred into a separating funnel and about 25ml of dry ether is added. After shaking well the aqueous layer is separated and discovered. The ethereal layer is transferred into a clean beaker and ether is evaporated from it.

4. Acid separation

To the ethereal solution in the separating funnel 25ml of HCl is added and shaken well releasing the pressure occasionally. The ethereal layer and aqueous layer are found separated. The ethereal layer contains HCl insoluble component and aqueous layer contains HCl soluble component. The lower aqueous layer is removed and neutralized with 10% NaOH. If a solid separates it is filtered on a Bucked funnel. If oily globules or emulsion is formed the following procedure is adopted. The solution with oily globules is transferred into a separating funnel and about 25ml of dry ether is added. After shaking well the aqueous layer is separated and discarded. The ethereal layer is transferred into a clean beaker and ether is evaporated.

ANALYSIS OF THE COMPONENTS

PRELIMINARY TESTS

S.No.	Experiment	Observation	Inference
1.	**Colour and appearance** The colour of appearance of the substance is noted	a) Yellow liquid and solid	May be aromatic, nitro aromatic
		b) Dark brown liquid or solid	May be presence of phenol or aromatic amines
		c) colorless or white solid	May be presence of carbohydrates, carboxylic acids, amides or urea ketones like benzophenone.
		d) Colorless solid	May be presence of ester aldehydes or hydrocarbons
2.	**Odor** The odor of the compound is noted gently from a distance	a) Pleasant odor of fruity odor	Esters may be present
		b) Phenolic smell	Phenol may be present
		c) Fishy or aniline like smell	May be presence of aromatic amines
		d) Pungent or irritating smell	May be due to hydrocarbons
3.	**Solubility** i) A small quantity or a drop of the given substance is mixed with 1 ml of the H_2O taken in the test tube and shaken well	Soluble in cold water	May be due to carbohydrates, di-amide like urea or aliphatic amides
	ii) Contents of the above is heated in the test tube	a) Dissolves in hot condition	May be presence of aromatic acids

		b)Dissolves in hot condition and recrystallizes on cooling	May be presence of amides
	iii) To a small quantity of the substance 1 or 2 drops of dilute HCl is added shaken well and warmed	Soluble	Presence of amines
4.	**Litmus test** A little of the substance is shaken well with 2 ml of water. The solution is tested with Litmus paper.	a) Blue litmus turned red. b) Red litmus turned blue c)No colour change with blue litmus or red litmus paper	May be due to carboxylic acid or phenol May be due to amines
5.	To small quantity of the substance added 2 ml of 10% of NaOH and shaken well	If the substance is a solid a) Dissolves readily in the cold and when the solution is acidified with the drops of concentrated HCL and cooled the substance is regenerated. b) Dissolves readily the solution becomes yellow and the colour is removed by concentrated HCl c) Ammonia is evolved d) Solution turns yellow or brown when boiled e) oily globules obtained having aniline like smell If the substance is liquid f) Oily layer disappears on boiling and white precipitate is obtained on acidification with	Presence of acids or phenols Presence of amides or diamides Presence of nitrophenol or salicyladehyde Presence of carbohydrates Presence of anilines

		concentrated HCL and cooling g) No characteristics reaction	Presence of esters Absence of carboxylic acids, phenol, esters etc.
6.	**Action of con.H_2So_4** A little of the substance is treated with 1 ml of concentrated H_2SO_4	a) The substance becomes black with the smell of burnt sugar b) No Characteristic reaction	Presence of Carbohydrates Absence of Carbohydrates
7.	**Soda lime test** A small quantity of the substance is mixed with 1 g of soda lime and heated slowly.	a) Ammonia gas is evolved b) Kerosene like smell c) Aniline like smell d) Phenolic smell e) No characteristic smell	Presence of amides Presence of acids Presence of anilines Phenolic acid or ester Absence of carboxylic acids, amides phenols
8.	**Neutral $FeCl_3$ test** A small amount of the substance is shaken with about 1 ml of the H_2O Or OH and neutral FeCl3 is added drop by drop	a) A violet blue or green colour is obtained b) Blue or green precipitate is obtained c) Brown precipitate or yellow colour	Presence of phenols or phenolic aldehyde or acids or phenolic esters Presence of napthols Presence of carboxylic acids
9.	**Tollens reagent test** To a small amount of the	a) A bright silver mirrors or a	Presence of aldehydes,

	substance added 3 to 4 ml of the tollen's reagent and boiled in water bath	brownish black precipitate b) No characteristic reaction	glucose or fructose or polyhydric phenols Absence of aldehydes, glucose or fructose or polyhydric phenols
10.	**Bromine water Test** A small amount of the substance is shaken well with 1 ml of water and Br.H_2o is added drop by drop	a) Yellow colour is obtained b) Yellow colour persists	Presence of unsaturated compounds like cinnamic acid or cinnamaldehyde Presence of saturated compounds
11.	**Sodium bicarbonate Test** A small amount of the substance is treated with 1 ml of clear solution of $NaHCo_3$	a) Brisk effervescence b) No characteristic reaction	Presence of carboxylic acids, sulphonic acids and nitrophenol Absence of carboxylic acids
12.	**Bromine in Carbotetra chloride** A small amount of the substance is treated with 2 ml of Br in CCl_4	a) Yellow colour decolourised b) Yellow colour not decolourised c)Decolourised with white precipitate	Presence of unsaturated compounds like cinnamic acid or cinnamaldehyde Presence of unsaturated compounds Presence of Phenols
13.	**Bayer's Test** A small amount of the substance is treated with small quantity of Na_2Co_3 solution and dilute	a) Pink colour is decolourised	Presence of unsaturated compounds like cinnamic acid or cinnamaldehyde and

	solution of $KMno_4$	b) No decolourisation	substance like phenol Presence of saturated compounds
14.	**Test for aliphatic/aromatic ignition test** A little of the substance is ignited with nickel spatula	a) Burns with sooty flames b) Burns with non-sooty flames	Presence of aromatic compounds Presence of aliphatic compounds
15.	**Nitration** A small amount of the substance is mixed with 1 ml of concentrated sulphuric acid and 1 ml of concentrated $HN0_3$ in dry test tube shaken well and boiled for 15 minutes in water bath and poured into about 20 ml of cold water.	a) Yellow precipitate or yellow solution is obtained b) No yellow precipitate or solution is obtained	Presence of aromatic compounds Presence of aliphatic compounds

TEST FOR FUNCTIONAL GROUPS

S. No	Experiment	Observation	Inference
1.	**Sodium bicarbonate test** A small amount of the substance is added to 2ml of a strong solution of $NaHCO_3$.	a. Brisk effervescence with the evolution of CO_2 takes place. b. No characteristic reaction takes place.	Presence of carboxylic acids Absence of carboxylic acid
2.	**Esterification** About 0.5g of the substance is mixed with about 2ml of alcohol in a dry test tube, 4to5 drops of Con. H_2SO_4 are added, shaken well and gently heated for a minute. The mixture is then poured into about 30ml of a dilute solution of Na_2CO_3 , stirred well and then smell is noted.	A pleasant ester smell is obtained.	Presence of carboxylic acid

3.	**Fluorescein reaction** A small amount of the substance is mixed with crystals o resorcinol in a dry test tube, 3 drops of Con.H_2SO_4 are added. Shaken well, heated gently and then poured into about 100ml if cold water, stirred well and then an excess of NaOH solution is added.	A red solution gives an intense greenish yellow fluorescence on adding NaOH.	Presence of a dicarboxylic acid
4.	To about 2ml of NaOH one drop of a very dilute solution of phenolphthalein is added. Pink colour appears. To this, the substance dissolved in water is added in drop by drop and shaken well.	Pink colour disappears	Presence of carboxylic acids

PHENOL

S.NO	Experiment	Observation	Inference
1.	**Phthalein fusion test** A small amount of the substance is mixed with about 0.5g of phthalic anhydride in a test tube, 2drops of Con. H_2SO_4 are added, heated gently for a minute. The mixture is poured into about 10ml of water and then NaOH solution is added in slight excess.	a. A pink or red colour is obtained b. A yellowish-green fluorescence is got.	Presence of monohydric phenol. Presence of dihydric phenol.
2.	**Dye test** 1ml of aniline is dissolved in 4ml dil. HCl and the solution is cooled in ice. To the cold solution, $NaNO_2$ solution is added with constant shaking. To this 0.5g of substance dissolved in 5ml of dil. NaOH solution is added.	Orange or scarlet red dye is obtained	Presence of phenol
3.	**Libermann's test** A small amount of the substance s heated with a crystal of $NaNO_2$ in a dry tube. It is cooled and two	A red solution is obtained which turns blue or green on adding NaOH.	Presence of phenol.

	drops of Con. H_2SO_4 are added, shaken well and the mixture is poured into about 100ml of water and then NaOH solution is added in excess.		
4.	**o-Benzoylation Schotten-Baumann reaction** To 5ml of the aqueous suspension of the compound, added 1ml of benzoyl chloride and 10ml of 10% NaOH solution. The content is shaken well for 5 minutes, cooled and poured into water with stirring.	White precipitate is obtained.	Presence of phenol.

ESTERS

S.NO	Experiment	Observation	Inference
1.	**Hydroxamic acid test** To 5 drops of the liquid a pinch of hydroxylamine hydrochloride and 5ml of 10% NaOH solution are added. The contents are gently boiled for about 3 minutes, cooled, acidified with Con. HCl. About 5 drops of $FeCl_3$ solution is added and shaken.	A violet or deep red-brown colour obtained.	Presence of ester.

CARBOHYDRATES

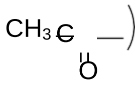
S.NO	Experiment	Observation	Inference
1.	**Molisch's test** A Small amount of the substance is disolved in 2 ml of water, a few drops of strong solution of α-naphthol in alcohol is added, along the side of the test tube.	Deep violet ring at the junction of the two liquids spreading throughout the solution.	Presence of carbohydrate

2.	**Phenylhydrazine test** The strong aqueous solution of the compound is added to 10 ml of a solution of phenyl hydrazine hydrochloride and sodium acetate in water in a boiling tube. It is heated on a water bath for 15 minutes with occasional shaking.	A Bright yellow pecipitate of osazone is obtained.	Presence of carbohydrates.
3.	**Tollen's test** The aqueous solution of the compound is added to Tollen's reagent in equal volume and heated on a water bath.	Black precipitate or bright silver mirror obtained.	Presence of reducing sugar.
4.	**Fehling's solution test** To an aqueous solution of the compound added 1 ml each of Fehling's solution 'A' and 'B' heated to boiling.	A red precipitate is obtained.	Presence of reducing sugar.
5.	**Barfoed's test** Added 2 ml of the aqueous solution of the compound to 2 ml of Barfoed's reagent and boiled gently.	A red precipitate is obtained.	Presence of reducing sugar (mono saccharide)

ALDEHYDES AND KETONES

S.NO	**Experiment**	**Observation**	**Inference**
1.	Borsche's test A Little of the substance is added to 3 ml of Borsche's reagent boiled and added con.HCL. The solution is cooled 3 ml of water.	a. Yellow or orange precipitate. b. No yellow or orange precipitate.	Presence of aldehyde or ketone.
2.	Sodium bisulphite test To small quantity of the substance 2ml of sodium bisulphate solution is added and shaken well.	a. White precipitate. b. No white precipitate.	Presence of aldehyde and ketone. Absence of aldehyde and ketone.
3.	Hydroxylamine test	a. White precipitate of the oxime is	Presence of aldehyde and

	A small quantity of the substance in alcohol is added to 5 ml of a solution of hydroxylamine hydrochloride and then a slight excess of $NaHCO_3$ solution. Heated on a water bath for 10 minutes, cooled and scratched the inner sides of the test tube with a glass rod.	obtained. b. No white precipitate.	ketone. Absence of aldehyde and ketone.
4.	**Semicarbazide test** About 0.5 g of semi carbazide hydrochloride is dissolved in 2 ml of water. To this 1 g of sodium acetate crystals are added. Shaken well, 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 precipitate of semicarbazone is obtained. No white precipitate.	Presence of aldehyde and ketone. Absence of aldehyde and ketone.
5.	**Phenyl hydrazine test** A little of the substance in alcohol is added to 5 ml of a solution of phenyl hydrazine hydrochloride and then a slight excess of sodium acetate solution. Heated ion a water bath for 15 minutes. Cooled and scratched the sides of the test tube with a glass rod.	Yellow precipitate of phenyl hydrozone. No yellow precipitate.	Presence of aldehyde and ketone. Absence of aldehyde and ketone.
6.	**Schiff's reagent test** A small amount of the substance is added to about 3 ml of schiff's reagent and shaken well.	A violet colour is produced quickly.	Presence of aldehyde. .
7.	**Fehling's solution test** A small amount of the substance is added to about 2 ml of each of the Fehling's solution A and B heated gently.	A red brown precipitate is obtained.	Presence of aliphatic aldehyde.
8.	**Tollen's reagent test** A small amount of the substance is added to about 4 ml of the tollen's reagent	A bright silver mirror or a brownish black precipitate of silver is obtained.	Presence of aldehyde.

	and heated in a boiling water bath for 10 to 15 minutes.		
9.	**Legal's test** A small amount of the substance is added to 1ml of the freshly prepared sodium nitro prusside solution and then 2ml of the 10 % NaOH.	Orange colour changes to purple on adding glacial acetic acid.	Presence of methoxy ketone.  $CH_3\underset{\underset{O}{\|}}{C}—)$

If the compound is found to contain nitrogen it must be analysed for the following functional groups.

AMINES

1.	**Dye test** A small quantity of the substance is dissolved in 1 ml f the Conc. HCl, diluted to about 4ml, cooled in ice water and then treated to about 1ml of a strong solution of $NaNO_2$, added drop by drop with constant shaking.	a. A clear solution is obtained. b. An yellow or yellowish brown oily liquid separated. c. An yellowish brown precipitate is obtained.	Presence of aromatic primary amine. Presence of secondary amine. Presence of tertiary amine.
	(i) The clear diazotised solution is added to a solution of β- naphthol in NaOH solution.	A scarlet red dye is obtained.	Presence of aromatic primary amine.
	(ii) The yellow oily liquid is extracted with ether, and the ether is evaporated carefully in a water bat. The residual liquid is warmed with 2 drops of phenol and again with 2 drops of Conc. H_2SO_4 . (Libermann's reaction)	The mixture added to water gives a red solution which becomes green or blue on adding NaOH.	Presence of secondary amine.
	(iii) The precipitate is decanted, shaken up with water and treated with NaOH solution.	A green precipitate is obtained.	Presence of tertiary amine.
2.	**N - Benzoylation (Schotten – Baumann**	White precipitate is obtained.	Presence of primary or secondary amine.

	reaction) 1 ml of the benzoyl chloride and 10 ml of the 10 % NaOH are added to 5 ml of the compound. The contents are shaken well vigorously for 10 minutes, cooled and poured into water with stirring.		
3.	**Picric acid test** 2 ml of the alcoholic solution of the compound is mixed with 2 ml of an alcoholic solution of picric acid.	Yellow precipitate of picrate is obtained.	Presence of tertiary amine.

AMIDES

1.	**Biuret test** A small amount of the substance is gently heated in a dry test tube for a few minutes, cooled, the residues is shaken with 2 ml of copper sulphate solution and then 10% NaOH solution is added drop by drop.	Ammonia is evolved on heating. A violet or a violet red colour is obtained.	Presence of diamide like urea
2.	To the strong aqueous solution of the compound con.HNO_3 is added in equal volume, shaken well and cooled.	White precipitate (urea nitrate) is obtained	Presence of urea
3.	To a strong aqueous solution of the compound saturated solution of oxalic acid is added in equal volume and the solution is allowed to stand.	White precipitate (urea oxalate) is obtained.	Presence of urea

ANILIDES

1.	**Dye test** A small amount of the substance is boiled with 5 ml of dil. HCl for 3 minutes. The solution is cooled in ice and diazotized and the diazonium salt solution is coupled with β-naphthol in NaOH.	A scarlet red dye is obtained.	Presence of anilides
2.	**Bromination** A little of the substance is dissolved in 3 ml glacial acetic acid and to the solution added 1 ml of bromine in CH_3COOH. The solution is shaken well and poured into 25 ml water.	Pale yellow precipitate is obtained	Presence of anilide.

Reduction

A small amount of the substance is mixed with a few granules of tin and 2 ml of con. Hcl, gently heated and shaken well again heated and the process is continued for 5 minutes. The contents are filtered and the filtrate is tested for the presence of an amino group

S.No.	Experiment	Observation	Inference
1.	A little of the filtrate is diazotized with $NaNo_2$ solution and diazotized solution is added to the solution of β-napthol in NaOH	A scarlet red dye is obtained	Presence of nitro compound

Halogen compound

S.No.	Experiment	Observation	Inference
1.	**Alcoholic $AgNo_3$ Test** A small amount of the substance is shaken well with 2 ml of alcoholic $AgNo_3$	a) An immediate or a slow precipitate of AgX with a colour	Presence of aliphatic halides or aromatic halogen compound with halogen in

	And then gently warmed	b) No precipitate of AgX is obtained	side chain Aromatic halogen compound with halogen nucleus
2.	**Anilide test** A little of the substance added to 1 ml of aniline is shaken well poured into 20 ml water	White precipitate tis obtained	Presence of acid chloride

If Sulphur is found in sodium fusion test, the following test may be performed

S.No.	Experiment	Observation	Inference
1.	**Barium chloride test** A little of the substance is burnt in a nickel spatula the residue left is dissolved on dil.HCl and added $BaCl_2$ solution	White precipitate is obtained	Presence of sulphonic acid

PREPARATION OF SOLID DERIVATIVES

1. Carboxylic acid

(i) S – Benzylisothiuronium derivatives

Neutralised a solution of 1g of the compound in 1g of water with NaOH using phenolphthalein a indicator. A drop of HCl i added. Then the olution of 1 g of S – Benzyl isothiuronium chloride in 10 ml of water added and shaken well, white precipitate is obtained.

(ii) Bromo derivative

About 1 g of the ubstance i mixed with 5 ml of bromine in glacial acetic acid slowly with constant stirring till yellow colour develop. The contents haken well for 10 – 15 minutes, poured into 50 ml water with stirring. Yellow crystals obtained.

2. Phenols

(i) o – Benzoyl derivative.

(ii) Bromo derivative.

3. Esters

Alkaline hydrolysis to carboxylic acid.

4. Aldehydes and ketones

(i) 2,4 – dinitro phenylhydrazone

(ii) semicarbazone

(iii) phenylhydrazone

5. Carbohydrates

Osazone

6. Amines

(i) N – Amylation

(ii) N – Benzoylation for primary and secondary amines

(iii) p – nitroso derivative for tertiary amine

(iv) Picrate derivative for tertiary amine

7. Amides

Alkaline hydrolysis to carboxylic acid

8. Diamides

(i) Urea oxalate

(ii) Urea nitrate

9. Anilides

(i) Bromo derivative

(ii) Azo dye

10. Nitro compounds

Reduction to amines – azo dye.

11. Halogen compounds

Oxidation of the side chain to carboxylic acid.

REPORT

The given organic mixtures contains

COMPOUND 1

1. Aliphatic/aromatic -
2. Saturated/unsaturated -
3. Elements present -
4. Functional group(s) present -
5. Derivative -

COMPOUND 2

1. Aliphatic/aromatic -
2. Saturated/unsaturated -
3. Elements present -

4.. Functional group(s) present -

5. Derivative -

ORGANIC PREPARATIONS

1. Preparation of acetanilide from aniline

Chemicals required

Aniline	-	2.5 ml
Acetic anhydride	-	3 ml
Glacial acetic acid	-	4 ml

Procedure

The glacial acetic acid was mixed with aniline in a dry conical flask and acetic anhydride is added slowly into the mixture with constant shaking. The mixture was shaken well from about 10 minutes. The reaction mixture was then poured into a beaker with stirring, containing crushed ice. The white solid that separated was filtered, washed with cold water, dried and the yield was noted.

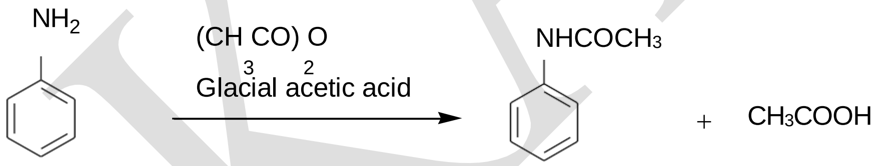
About 1 g the crude acetanilide was re-crystallised from hot water and the melting point was determined.

Result

Yield of the product =

Melting point of the re-crystallised sample =

Reaction



2. Preparation of p-bromoacetanilide from acetanilide

Chemicals required

Acetanilide	- 2.5 g
Glacial acetic acid	- 10 ml
Bromine	- 1 ml in 15 ml glacial acetic acid

Procedure

The powdered acetanilide was dissolved in 10 ml of cold glacial acetic acid contained in a round bottomed flask. The liquid bromine was measured from burette into 15 ml of glacial acetic acid contained in a separating funnel which was shaken gently and continuously to get a solution of uniform concentration. The bromine solution was added slowly to acetanilide with constant shaking to ensure through mixing. After the addition was over the contents of the flask were kept aside for about 15 minutes. The pale reddish brown solution was poured into 20 ml of water contained in a beaker and a few pieces of ice was added. The mixture was stirred and the precipitated p-bromoacetanilide was filtered, washed with cold water, dried and the yield was noted.

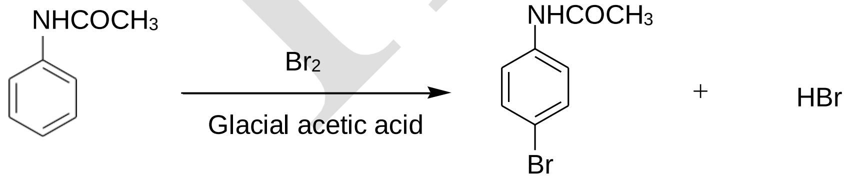
About 1 g of the sample was re-crystallised from alcohol and the melting point was determined.

Result

Yield of the product =

Melting point of the recrystallised sample =

Reaction



3. Preparation of p-bromoaniline from p-bromoacetanilide

Chemicals required

p-bromoacetanilide	- 5 g
Concentrated hydrochloric acid	- 25 ml

Procedure

p-Bromoacetanilide was boiled with con HCl in a round bottom flask fitted with a refluxed condenser and allowed it to reflux for 1.5 hours till the test portion of the liquid remained clear on dilutionwith water. The p-bromoaniline was present as hydrochloride. After hydrolysis, 20 ml of water was added to the contents of the flask and the free base was liberated by adding liquid ammonia till the mixture gave a distinct smell of ammonia. A colourless oily substance separates which goes to a solid after adding few pieces of ice and stirring. The solid was filtered, washed with water, dried and yield was noted.

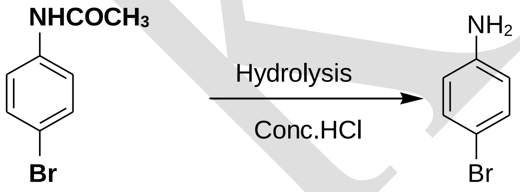
About 1 g of a sample was re-crystallised from dilute alcohol and melting point was determined.

Result

Yield of the product =

Melting point of the recrystallissed sample =

Reaction



4. Preparation of diphenyl hydantion from benzil

Chemicals required

Benzil	-	1 g
Urea	-	0.5 g
Rectified spirit	-	15 ml
30% NaOH	-	4 ml

Procedure

In a round bottom flask 1 g of benzil, 0.5 g of urea and 15 ml of ethanol and 4 ml of 30% aqueous sodium hydroxide was taken, attached to a reflux condenser and the mixture was boiled for 2 hours. The reaction mixture was cooled and added 25 ml of water.Filtered the solution to remove the sparingly soluble product. Filtrate was acidified with hydrochloric acid.

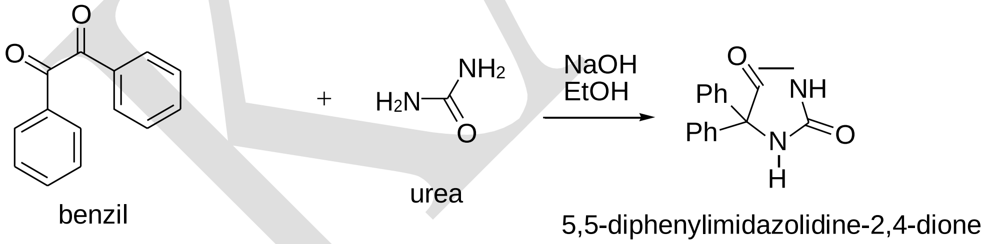
The precipitate was filtered and washed with water, dried and yield was noted. Melting point was determined.

Result

Yield of the product =

Melting point of the re-crystallised sample =

Reaction



5. Preparation of m-dinitrobenzene from nitrobenzene

Chemicals required

Nitro benzene -5ml

Fuming nitric acid -5ml

Concentrated H_2SO_4 -10ml

Procedure

5 ml of fuming nitric acid is taken in a round bottom flask and 10 ml of con.H_2SO_4 is added in a little of time, cooling the flask during the time. 5 ml of nitrobenzene was taken and added to the nitrating mixture with small intervals of time. It is shaken well after each addition. Finally it was heated for about 45 minutes by immersing the round bottom flask in a boiling water bath, till a small quantity of the reaction mixture is added into water which gives solid immediately. The contents of the flask are then poured into water, while hot. The mixture was shaken continuously and vigorously, during addition. m-dinitrobenzene was separated as a solid. It was filtered, washed several times with water, dried and yield was noted.

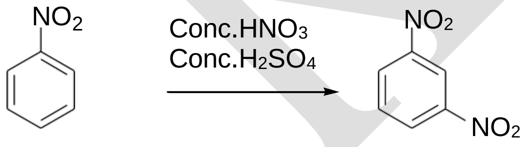
A Small portion was re-crytallised with alcohol and the melting point was noted.

Result

Yield of the product =

Melting point of the re-crytallissed sample =

Reaction



6. Preparation of benzoic acid from benzaldehyde

Chemicals required

Benzaldehyde	-3 ml
Aqueous Na_2CO_3	-10% 30 ml
Potassium permanganate	-5 g
Sodium sulphide	-5 g
Dilute sulphuric acid	-20 ml
Concentrated hydrochloric acid	-20 ml

Procedure

Benzaldehyde mixed with aqueous sodium carbonate solution was taken in a round bottom flask. The round bottom flask was fitted with a condenser and the content was gently boiled and an aqueous solution of potassium permanganate was added to the flask from the top of the condenser in a small quantity until the solution remained slightly pink. The flask was shaken gently during the addition of potassium permanganate. Heating is continued for 30 minutes. Benzaldehyde was oxidized to benzoic acid and manganese precipitated.

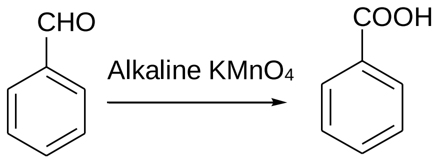
The flask was cooled and contents were transferred into a 250 ml beaker. A saturated solution of sodium sulphide was added with constant stirring until the precipitated manganese dioxide was dissolved. To liberate free acid, concentrated hydrochloric acid was added till precipitation was complete. The precipitated acid was filtered off, washed with water, dried and yield was noted. About 1 g of crude benzoic acid was re-crystallised from minimum amount of hot water.

Result

Yield of the product =

Melting point of the re-crystallised sample =

Reaction



7. Preparation of acetyl salicylic acid from salicylic acid

Chemicals required:

Salicylic acid - 1.5g

Acetic anhydride - 2.5ml

Concentrated sulphuric acid - 2drops

Ethanol - 6ml

Procedure

Salicylic acid was mixed with acetic anhydride in a 100ml conical flask and 2 drops of Con.H_2SO_4 and was added. The mixture was shaken well and heated on a water bath at 50^o to 60^oC, for about 10 to 15 minutes with constant shaking. Then the conical flask was removed, the contents were cooled under tap and then poured into 60ml if water in a beaker. The acetyl salicylic acid that separates out was filtered, washed with water and dried. The yield was noted.

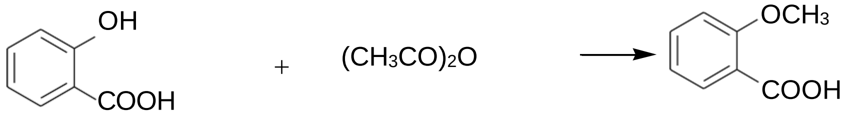
About 1g of the sample was dissolved in minimum amount of hot ethanol and poured in 40ml of water in a 250ml beaker. The turbid solution was then warmed to get a clear solution. The resulting solution was allowed to cool slowly then needles of acetyl salicylic acid separated out, it was filtered, dried and melting point was noted.

Result

Yield of the product =

Melting point of the re-crystallised sample =

Reaction



8. Preparation of anthraquinone from anthracene

Chemicals required

Anthracene	- 5g
Glacial acetic acid	- 60ml
Chromium trioxide	- 10g

Procedure

5g of powdered anthracene is dissolved in 60ml of glacial acetic acid and the solution was taken in a 250ml round bottom flask.

10g of chromium trioxide was dissolved in 8ml of water and to it 35ml of glacial acetic acid was added. The solution was taken in a separating funnel.

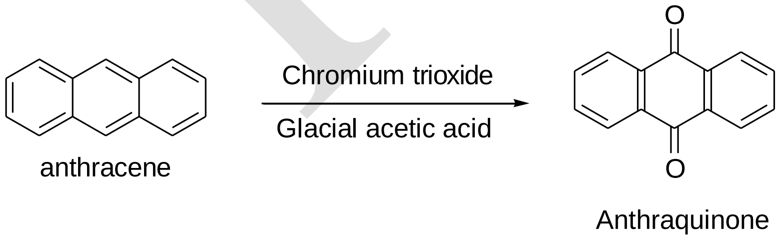
The anthracene solution was heated to boiling and to the boiling solution, chromium trioxide solution was added slowly. The content was boiled for 15minutes and the resulting deep green coloured solution was added to 250ml of cold water. The separated crude anthraquionoine was filtered and washed with hot water, hot dilute sodium hydroxide solution and finally with water. The solid was re-crystallised by sublimation.

Result

Yield of the product =

Melting point of the re-crystallised sample =

Reaction



9. Preparation of s-benzyl isothiouronium chloride

Chemicals required

Benzyl chloride	- 6.4ml
Thiourea	- 3g
Rectified spirit	- 10ml

Procedure

A mixture of benzyl chloride, thiourea and rectified spirit were placed in a 250ml round bottom flask fitted with a reflux condenser and heated on a water bath. A sudden exothermic reaction takes place and all the thiourea dissolved in the solution. The yellow solution was allowed to reflux for 30minutes and then cooled in ice. The white crystals were filtered and dried in air. The filtrate was concentrated to half of its original volume and cooled to give further small crystals. Both the products were collected and yield was noted. A small portion of the sample was re-crystallised from 0.2 N HCl and melting point was noted.

Result

Yield of the product =

Melting point of the re-crystallised sample =

10. Preparation of β-naphthol methyl ether

Chemicals required

β-naphthol - 6g

Sodium hydroxide - 2g Di-

methyl sulphate - 4ml

Procedure

A mixture of 6g of β-naphthol and 2g of sodium hydroxide was taken in a 250ml round bottomed flask. After cooling the flask in a ice water, 4ml of dimethyl sulphate was added. The contents were warmed at 70°-80°C for one hour. The methylation product was filtered at the pump. Washed with sodium hydroxide solution followed by water, dried and re-crystallised from benzene.

Result

Yield of the product =

Melting point of the re-crystallised sample =