

East Point College of Pharmacy

East Point Campus, Jnana Prabha, Virgo Nagar Post
Bengaluru – 560049, Karnataka

Approved
by
Pharmacy Council of India, New Delhi



Affiliated
to
Rajiv Gandhi University of Health Sciences
Karnataka
Bengaluru – 560 041
India

LAB MANUAL

PHARMACEUTICAL ORGANIC CHEMISTRY

PHARM D

1st Year

PROGRAM OUTCOMES
DOCTOR OF PHARMACY
<p>PO1: Comprehensive Pharmaceutical Knowledge</p> <p>Acquire and demonstrate a deep understanding of fundamental concepts in pharmacotherapeutics, clinical pharmacy, pharmacoepidemiology, clinical pharmacokinetics, therapeutic drug monitoring, as well as behavioral, social, and administrative pharmacy. Showcase advanced proficiency by designing and executing effective medication therapy management plans.</p>
<p>PO2: Strategic Planning Proficiency</p> <p>Cultivate the capability to formulate and execute plans, proficiently organizing tasks to meet deadlines. Demonstrate adeptness in time management, resource allocation, delegation, and organizational skills. Exhibit competence in clinical decision-making by seamlessly integrating pharmaceutical knowledge with patient-specific factors to enhance healthcare outcomes.</p>
<p>PO3: Problem Solving Proficiency</p> <p>Apply the principles of scientific inquiry, engaging in analytical, clear, and critical thinking to address challenges and make informed decisions in routine clinical practice. Graduates will demonstrate adept communication skills, counseling patients on medication usage, potential side effects, and lifestyle adjustments, fostering patient comprehension and adherence.</p>
<p>PO4: Leadership and Entrepreneurship</p> <p>Demonstrate leadership skills and entrepreneurial spirit, contributing to the growth and development of the pharmaceutical profession and industry.</p>
<p>PO5: Professional Identity</p> <p>Exhibit a strong professional identity including a commitment to ethical practice, effective communication, and leadership in advocating for optimal patient care, continuous professional development, and active engagement with the broader healthcare professionals, promoters and stakeholders.</p>
<p>PO6: Adherence to Ethical Standards</p> <p>Uphold the highest ethical standards in pharmaceutical practice, adhering to the Pharmacy Council of India's code of ethics and promoting patient welfare.</p>
<p>PO7: Communication</p> <p>Demonstrate effective communication skills, sustaining clear and empathetic interactions with patients, healthcare professionals, and diverse stakeholders. They will proficiently convey pharmaceutical information, contributing to collaborative and patient-centered care. This emphasis on communication ensures graduates are well-equipped to navigate complex healthcare scenarios and advocate for optimal therapeutic outcomes.</p>
<p>PO8: Community Engagement</p> <p>Participate in community engagement activities, applying pharmaceutical knowledge to address healthcare needs and improve the overall well-being of the community.</p>
<p>PO9: Environment and Sustainability</p> <p>Demonstrate a profound understanding of environmental issues in the pharmaceutical domain, applying sustainable practices in research, development, and clinical settings. They will champion eco-friendly approaches, fostering a commitment to minimizing ecological impact and promoting responsible stewardship of natural resources.</p>
<p>PO10: Clinical Research Skills</p> <p>Proficient in conducting clinical research, applying ethical principles, and contributing to advancements in pharmaceutical sciences and healthcare.</p>
<p>PO11: Continuous Professional Development</p> <p>Embrace a commitment to lifelong learning, staying abreast of advancements in pharmaceutical sciences, healthcare policies, and technological innovations.</p>

PROGRAM SPECIFIC OUTCOMES	
DOCTOR OF PHARMACY	
PSO1	Acquire a thorough foundational knowledge in pharmaceutical sciences, including pharmacology, pharmaceutics, pharmaceutical chemistry, pharmacognosy and pharmaceutical analysis to excel in further academic pursuits
PSO2	Acquire and apply the pharmacotherapeutic concepts for better patient care enhancing employability across various sectors including clinical research organizations, academic and hospitals
PSO3	Equip with entrepreneurial skills and knowledge of pharmacoepidemiological studies and regulatory aspects to initiate and run successful ventures in the healthcare sector

Course:	Code: 1.4P Pharmaceutical Organic Chemistry
CO1	Understand principles and reactions used in the detection of the extra elements present in organic compounds and know the principle for the preparation of suitable solid derivatives from organic compounds. Can determine the boiling/melting point of organic compounds
CO2	Detect the extra elements present in organic compounds and identify unknown organic compounds by systematic qualitative analysis
CO3	Preparation of suitable solid derivatives of organic compounds
CO4	To answer principles and procedures

Table of Contents

Expt. No	EXPERIMENTS
Physical Constants	
1	Determination of Melting point
2	Determination of Boiling point
Preparation of Organic Compounds	
3	Preparation of Benzoic acid
4	Preparation of 1-Phenyl Azo 2-Naphthol
5	Preparation of Benzophenone oxime
6	Preparation of Dibenzylidene Acetone
7	Preparation of Cinnamic acid
8	Preparation of 2,4,6-tribromo aniline
9	Preparation of Benzanilide
10	Preparation of m-nitro benzene
Systematic General Procedure for Analysis of Unknown Organic Substance	
11	Preliminary Observation of the Compound
12	Detection of Extra elements
13	Detection of Solubility
14	Functional Group Analysis – Group – I
15	Functional Group Analysis – Group – II
16	Functional Group Analysis – Group -III A & B
17	Functional Group Analysis – Group – IV
18	Functional Group Analysis – Group – V
19	Functional Group Analysis – Group – VI
20	Functional Group Analysis - Group – VII

BASIC LABORATORY APPARATUS

Apparatus:

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

Observation:

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

I) Beaker :

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

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

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

II) Conical flask:

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

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

III) Burette :

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

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

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

Precautions: Before using any burette first it should be washed with water, and then it should be rinsed with the reagent which is going to be added in it.

IV) Funnel :

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

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

Disadvantage: The substances are not completely filtered out.

V) Separating Funnel:

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

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

VI) Buchner funnel:

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

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

VII) Volumetric flask:

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

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

VIII) Specific gravity bottle:

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

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

IX) Round Bottom flask (RBF):

It is a flask with round bottomed and short necked.

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

X) Reflux Condenser:

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

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

XI) Watch Glass:

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

XII) Measuring Cylinder:

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

XIII) China dish: It is used to take test solutions and powdered reagents. It is also used to melt or heat solid reagents.

Experiment: 01

DETERMINATION OF MELTING POINT

AIM:

To determine the melting point of given sample.

APPARATUS:

Thermometer, capillary tube, Burner, Stand, Thread

CHEMICALS REQUIRED:

Liquid paraffin wax, and sample substance

PRINCIPLE:

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

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

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

PROCEDURE:

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

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

The tube is filled with liquid paraffin. The thermometer with the capillary tube is immersed in liquid paraffin in such a way that the open end of the capillary tube and rubber band should be above the level of the liquid paraffin the side arm of the tube is heated at a uniform rate. The flame of the burner & adjusted in such a way that the temperature at which the last crystal is disappeared and this melting point is reported.

REPORT: The given sample melts at

Experiment: 02

DETERMINATION OF BOILING POINT

AIM:

To determine the boiling point of given sample.

APPARATUS:

Distillation flask, thermometer, stand, burner

PRINCIPLE:

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

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

PROCEDURE:

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

Record the temperature when it remains the constant. Collect the liquid and continue distillation until only a small volume of liquid remains in the flask. Observe the boiling point.

REPORT:

The given liquid boils at ...

Experiment: 03

PREPARATION OF BENZOIC ACID

AIM: To Synthesize Benzoic acid from Benzyl chloride.

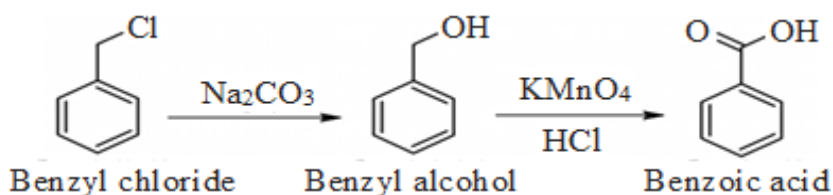
APPARATUS: Round Bottomed Flasks, Beakers, Conical Flasks, Water bath vessel, Reagentbottles, Funnel, Measuring cylinder, Pipette, Stirrer, etc.

INSTRUMENTS: Weighing balance, Melting point apparatus and Hot air oven.

CHEMICALS: Anhydrous sodium carbonate, Potassium permanganate, Benzyl chloride, Sodium sulphite, Hydrochloric acid.

PRINCIPLE: Oxidation reaction, If oxidation occur to an aromatic compound having an aliphatic side chain then, fission of side chain occurs between the first and second carbon atom from the benzene ring and the first carbon atom thus becoming part of carboxyl (COOH) group. The oxidation process is carried out with a mixture of potassium permanganate and sodium carbonate in aqueous solution, or with dilute nitric acid. The reaction is quite slow if the side chain is a simple alkyl chain group. The side chain containing chlorinated alkyl group of aromatic compounds are more susceptible to oxidation. Hence in comparison to toluene, benzyl chloride more rapidly oxidizes in presence of an aqueous oxidizing agent. Here benzyl chloride is first hydrolyzed to benzyl alcohol and then undergoes oxidation of primary alcohol to the corresponding carboxylic acid.

REACTION:



PROCEDURE: Take 200ml of water in a 500 ml flask, 5gm anhydrous sodium carbonate, 10gm of potassium permanganate and 5ml (5.5ml) of benzyl chloride are added one by one. The flask is fitted with a reflux water condenser and boiled gently for 1 to 1.5 hr to complete the reaction. During this boiling the permanganate is slowly reduced and manganese dioxide separates as a dark brown precipitate, then the flask is cooled and about 50ml con. HCl is added cautiously until the mixture is strongly acidic and all the benzoic acid is precipitated. Then about 100ml of 20% aqueous solution of sodium sulphite is added slowly with shaking

until the manganese dioxide is completely dissolved and only the white precipitate of benzoic acid remains. The product is filtered at the pump when cold and washed with water. It is recrystallized from boiling water.

$$\text{Percentage Yield} = \frac{\text{Practical Yield}}{\text{Theoretical yield}} \times 100$$

REPORT: Benzoic acid was prepared, submitted and reported the following

Particulars	Practical Value
Theoretical Yield (gm)	
Practical Yield	
Percentage Yield	

Experiment: 04

PREPARATION OF 1 PHENYL AZO- 2 NAPHTHOL

AIM:

To prepare and submit 1 phenyl azo 2-naphthol from Aniline.

CHEMICALS:

Aniline, Conc. Hydrochloric acid, Water, Sodium nitrite, β -Naphthol and 10% Sodium hydroxide solution

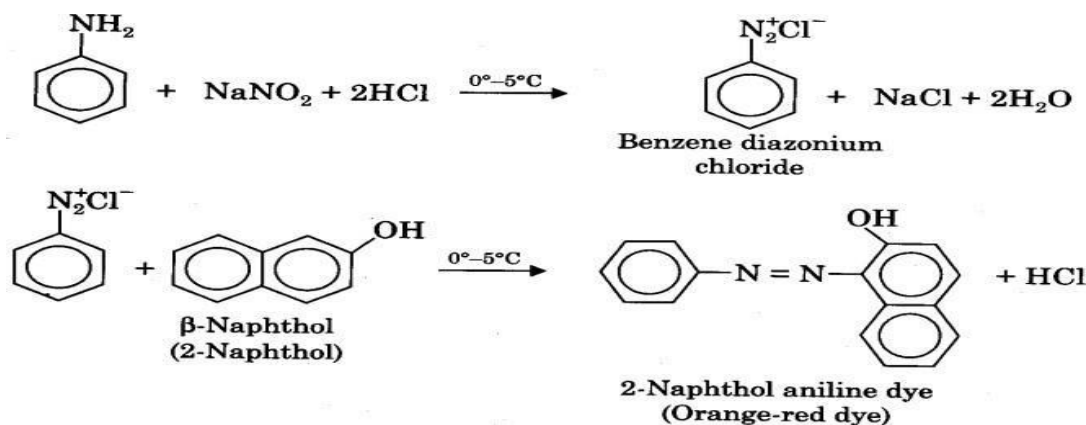
APPARATUS:

Conical flask, Thermometer, beakers, Stirrer, Glass rod, Ice bath, Buchner funnel.

PRINCIPLE:

Preparation of 1 phenyl azo – 2 – diazodization reaction and coupling reaction. Aniline is treated with Sodium nitrite and Conc. Hydrochloric acid at 0°C to form benzene diazonium chloride. This Benzene diazonium chloride reacts with β - Naphthol in presence of Sodium hydroxide to give 1 phenyl Azo – 2 naphthol.

REACTION:



PROCEDURE:

Place 2.5 ml of Aniline in a 100ml conical flask and 8.1 ml of conc. Hydro chloric acid And 8ml of water, place the thermometer in solution cool in a crushed ice till the temperature is in between $0-5^{\circ}\text{C}$. Dissolve 2.1 gm of Sodium nitrite in 10ml of water & cool in crushed ice and stir the solution. Then slowly add Sodium nitrite solution drop wise till the Diazodization is complete.

Dissolve 4gm of β -Naphthol in 22ml of 10% Sodium hydroxide in a beaker & Cool to 5°C in a crushed ice, stir the solution vigorously and add cold diazonium salt slowly. A bright red precipitate

is obtained. After complete addition of diazonium salt solution allow it to stand in the ice bath for about 30 minutes. Then filter the product and dry it and finally crystallize to find the percentage of the yield.

REPORT: 1-Phenyl Azo-2-naphthol was prepared, submitted and reported the following

Particulars	Practical Value
Theoretical Yield (gm)	
Practical Yield	
Percentage Yield	

Experiment: 05

PREPARATION OF BENZOPHENONE OXIME

AIM:

To prepare and submit Benzophenone Oxime.

APPARATUS:

Round Bottom flask, Reflux, Condenser, Tubes, Stand and Buchner funnel

CHEMICALS:

Benzophenone, Sodium hydroxide, Conc. Hydrochloric acid, rectified spirit.

PRINCIPLE:

Any aldehyde or ketone reacts with hydroxyl amine hydro chloride to give oximes. In this reaction Benzophenone reacts with hydroxyl amine hydro chloride to give Benzophenone oxime. When treated with some of the acidic reagents such as phosphorous penta chloride or thionyl chloride, it undergoes rearrangement to give n-substituted amines. This Beckmann's rearrangement is highly stereo specific reaction. The group which is Trans to the - OH group will undergo migration.

PROCEDURE:

In a 250 ml Round bottom flask, take 6gm of Benzophenone, 4gm of hydroxylamine hydrochloride, 2-3 ml of water and 1 ml of rectified spirit. To this mixture, add 7g of solid Sodium hydroxide slowly in proportions of about 0.5 gm with constant shaking and cooling.

Fit a reflux condenser in a flask and reflux for about 10 min. cool then pour the reacts mixture into solution of 20ml Conc. Hydrochloric acid in about 120 ml of water taken in a beaker. The Benzophenone oxime separates out as colorless crystals. Filter, wash & find percentage of yield.

REPORT: Benzophenone oxime was prepared, submitted and reported the following

Particulars	Practical Value
Theoretical Yield (gm)	
Practical Yield	
Percentage Yield	

Experiment: 06

PREPARATION OF DIBENZYLIDENE ACETONE

AIM:

To prepare and submit Dibenzylidene Acetone

APPARATUS:

Conical flask, funnel beaker, glass rod

CHEMICALS:

Benzaldehyde, Acetone, methylated spirit, 10%NaOH,

PRINCIPLE:

Benzaldehyde under the influence of sodium hydroxide will condense with acetone will form hydroxyl compound with an elimination of water unsaturated ketone. This reaction is known as Claisen condensation reaction.

PROCEDURE:

Dissolve 2 ml of benzaldehyde and 1.6ml of pure acetone in 10ml methylated spirit contained in conical flask or wide mouth bottle. Dilute 4 ml of 10% aq. NaOH with 16 ml of water and add this dilute alkali solution to the former solution and shake mixture vigorously in the secured corked flask for about 10 minute and allow to stand for 30 minutes with occasional shaking.

Finally cool in ice water for few minutes. During shaking the Dibenzylidene acetone separates at first as fine emulsion which rapidly form Pale yellow crystals. Filter at the pump and wash well with water to eliminate traces of alkali and then drain thoroughly. Recrystallise from hot methylated spirit. Pale yellow crystals separates out. Melting point- 112°C.

REPORT: Dibenzylidene acetone was prepared, submitted and reported the following

Particulars	Practical Value
Theoretical Yield (gm)	
Practical Yield	
Percentage Yield	

Experiment: 07

PREPARATION OF CINNAMIC ACID

AIM: To Synthesize Cinnamic Acid from Benzaldehyde.

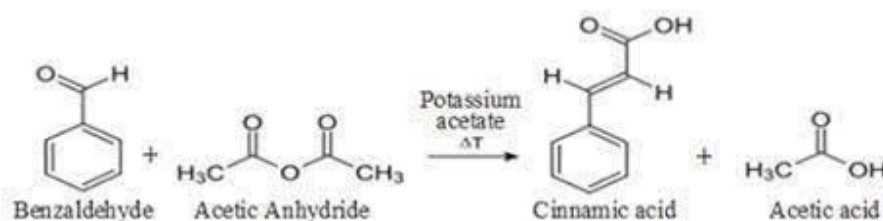
APPARATUS: 250ml Round Bottomed Flasks, Beakers, Reflux Condenser, Buchner flasks, Buchner funnel, thermometer, separatory funnel, etc.

INSTRUMENTS: Weighing balance, Melting point apparatus and Hot air oven.

CHEMICALS: Benzaldehyde, Acetic anhydride, Sodium Ammonium nitrate.

PRINCIPLE: Perkin reaction, When an aromatic aldehyde is heated with aliphatic acid (having at least two hydrogen atom) in presence of Na and K salt of corresponding acids, a condensation reaction leading to the formation of alpha, beta unsaturated acids, this reaction is called as Perkin reaction.

REACTION:



PROCEDURE: A mixture of pure Benzaldehyde (5gm), acetic anhydride (15gm) freshly fused and finely powdered sodium acetate 6gm is heated in a round bottom flask of 100 ml capacity fitted with air condenser at 160°C on oil bath for 1 hr. The temperature of the oil bath is raised to 170-180°C and heating continued for 3 hr. and more the hot mixture is poured into water extracted with ether about 30ml to remove the uncreated benzaldehyde remaining clear alkaline solution is acidified with HCl and cooled the separated cinnamic acid is filtered washed with cold water and recrystallized from hot water. The yield is 9gm and M.P. is 133°C. The Cinnamic acid obtained is more stable trans-isomer.

Notes:

- (1) The benzaldehyde must be free from benzoic acid ; it may be purified first.
- (2) An equivalent quantity of freshly fused sodium acetate may also be used, but the reaction is slower and a further 3-4 hours heating is necessary. Fused potassium acetate is prepared by melting the potassium acetate of commerce in a porcelain dish and heating gently, with occasional stirring, until no more vapour is evolved and the salt is completely fluid. When cold, the solid is finely

ground in a mortar and preserved in a tightly-stoppered bottle until required.

(3) Sodium hydroxide solution cannot be used at this stage since it may produce benzoic acid by the Cannizzaro reaction from any unchanged benzaldehyde. If, however, the reaction mixture is diluted with 3-4 volumes of water, steam distilled the unreacted benzaldehyde, the residue may then be rendered alkaline with sodium hydroxide solution. A few grams of decolorizing carbon are added, the mixture boiled for several minutes, and filtered through a fluted filter paper. Upon acidifying carefully with concentrated hydrochloric acid, cinnamic acid is precipitated. This is collected, washed and purified as above to remove

Percentage Yield = $\frac{\text{Practical Yield}}{\text{Theoretical Yield}} \times 100$

Theoretical yield

REPORT: Dibenzylidene acetone was prepared, submitted and reported the following

Particulars	Practical Value
Theoretical Yield (gm)	
Practical Yield	
Percentage Yield	

Experiment: 08

PREPARATION OF 2, 4, 6-TRIBROMO ANILINE

AIM: To Synthesize 2, 4, 6-Tribromo Aniline from Aniline.

APPARATUS REQUIRED: Round Bottomed Flasks, Beakers, Conical Flasks, Water bath vessel, Reagent bottles, Funnel, Measuring cylinder, Pipette, Stirrer, etc.

INSTRUMENTS REQUIRED: Weighing balance, Melting point apparatus, Hot air oven, Magnetic stirrer.

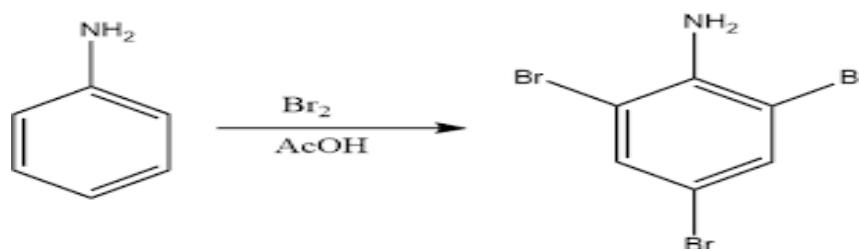
CHEMICALS REQUIRED: Aniline, Glacial acetic acid, Bromine solution, Ethanol, Activated charcoal.

PRINCIPLE: Bromination reaction, The bromination reaction is a very useful electrophillic aromatic substitution reaction because brominated arene rings can be used to undergo carbon-carbon bond-forming reactions to build up chemical structures (e.g., Grignard reaction). Traditionally, a solution of acetic acid and molecular bromine (Br_2) has been used to brominate aromatic systems. This is a hazardous mixture to handle, so a novel "green" way of performing the same reaction was invent. This novel method includes the reaction of sodium hypochlorite (Nacio) under acidic conditions with sodium bromide to generate molecular bromine. This is much safer than handling acidic solutions of Br_2 since most of the bromine made is consumed immediately by the electrophillic aromatic substitution reaction

The unsaturated organic compounds are readily converted in to saturated compounds when reacted with halogens (chlorine, bromine, iodine)

The amino group activates the nucleus to such an extent that substitution takes place at the two ortho and one para position. Thus reaction with bromine gives 2, 4, 6-tribromoaniline.

REACTION:



PROCEDURE: Place 4.66gm (0.05 mol) of aniline is dissolved in glacial acetic acid (50ml) in a 100 ml round bottom flask equipped with a magnetic stirrer. The solution is cooled on an ice/water bath before a solution of bromine 8.0ml (0.155mol) in glacial acetic acid (30ml) is added drop

wise from an addition funnel drop wise at such a rate that complete decolourization is achieved between each drop. Stirred the reaction mixture continuously with magnetic stirring during the addition. (Remove the flask from the ice/water bath if the acetic acid solidifies, momentarily). When all the bromine is added the reaction mixture is stirred 10min. (check TLC) before it is poured into water (250ml) containing a pinch of sodium bisulphite (NaHSO_3). The precipitate is filtered off and washed with water. The product is recrystallized from hot ethanol with a pinch of activated charcoal

Percentage Yield : $\frac{\text{Practical Yield}}{\text{Theoretical Yield}} \times 100$

Theoretical Yield

REPORT: 2, 4, 6-Tribromo Aniline was prepared, submitted and reported the following

Particulars	Practical Value
Theoretical Yield (gm)	
Practical Yield	
Percentage Yield	

Experiment: 09

PREPARATION OF BENZANILIDE

AIM: To Synthesize Benzanilide from Aniline by Benzoylation reaction

APPARATUS REQUIRED: 250ml Round Bottomed Flasks, Beakers, Reflux Condenser, Buchner flasks, Buchner funnel, thermometer, etc.

INSTRUMENTS REQUIRED: Weighing balance, Melting point apparatus and Hot air oven.

CHEMICALS REQUIRED: Glycine, Sodium hydroxide, Benzoyl chloride, concentrated hydrochloric acid and Carbon tetrachloride.

PRINCIPLE: Benzanilide is prepared by Schotten – Baumann method of benzoylation, Electrophilic Aromatic Substitution Reactions

Electrophilic aromatic substitution reaction proceeded in three distinct stages:

- **Activation:** Since halogens (Cl_2 , Br_2) don't usually react with aromatic molecules at a reasonable rate, a Lewis acid catalyst (e.g. FeCl_3) is added to “activate” the electrophile toward attack.
- **Attack of Electrophile by aromatic ring:** The activated electrophile is attacked by the aromatic ring, resulting in a carbocation intermediate (this is the rate-determining step, and Step-1 in the generic mechanism of electrophilic aromatic substitution).
- **Deprotonation:** The carbocation intermediate is deprotonated by a weak base, restoring aromaticity (Step-2 in the generic mechanism of electrophilic aromatic substitution).

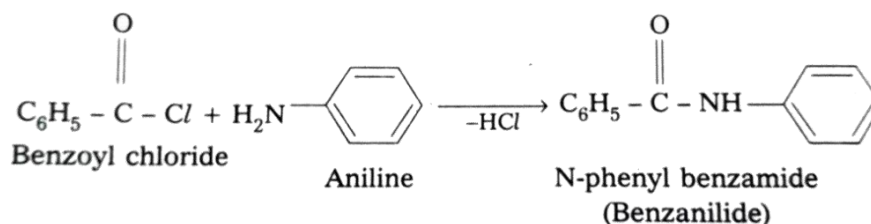
The substitution or the replacement of one or more active hydrogen atoms in an organic compound by one or more benzoyl groups ($-\text{COC}_6\text{H}_5$) is known as **benzoylation**. Benzoylating agents are those agents, which provide or supply the benzoyl moiety ($-\text{COC}_6\text{H}_5$). Benzanilide is prepared by the reaction between a primary amine, aniline and a Benzoylating agent, benzoylchloride in the presence of 10 % aqueous sodium hydroxide solution.

This particular reaction bears a close similarity to the concept of acetylation except that in this specific instance the reagent is (benzoyl chloride) which reacts in the presence of pyridine or 10% NaOH and not benzoic anhydride. The amines are more soluble in acid chloride than in sodium hydroxide, the reaction occurs preferably of Benzanilide; the liberated hydrochloric acid was neutralized by sodium hydroxide which also catalyze the reaction.

Uses of sodium hydroxide: 1. acting as a catalyst 2. As a neutralizing agent, it neutralizes the acid, HCL formed in the reaction to its Salt and water 3. It removes the un reacted benzyl chloride from the reaction mixture by converting in to sodium salt.

Uses of ethanol: 1. as a recrystallising agent. 2. it also removes the unreacted benzoyl chloride those escapes from the alkali Hydrolysis from the final product by converting in to its ester.

REACTION:



PROCEDURE: Place 2.6 gm (2.5 ml) of aniline and 25 ml of 10 % sodium hydroxide solution in a 100 ml conical flask, and then add 4.3 g (3.5 ml) of Benzoyl chloride to the above mixture. Stopper the flask and shake vigorously for 10-15 minutes. Heat is evolved in the reaction. The crude Benzoyl derivative separates as a white powder. Filter off the product with suction on a small Buchner funnel, break up the mass on the filter, wash well with water, and drain. Recrystallize from hot alcohol. Report the yield and melting point of the product.

$$\text{Percentage Yield} = \frac{\text{Practical Yield}}{\text{Theoretical Yield}} \times 100$$

REPORT: Benzanilide was prepared, submitted and reported the following

Particulars	Practical Value
Theoretical Yield (gm)	
Practical Yield	
Percentage Yield	

Experiment: 10

PREPARATION OF *m*-DINITROBENZENE

AIM: To Synthesize *m*-Dinitrobenzene from Nitrobenzene.

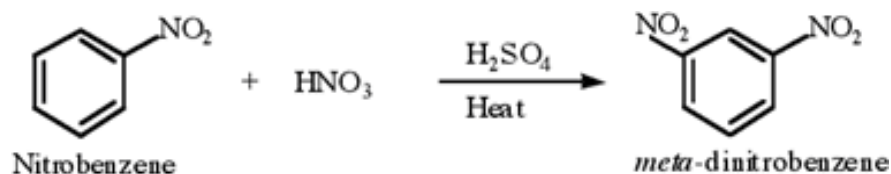
APPARATUS: Round Bottomed Flasks, Beakers, Conical Flask, Water bath vessel, Reagent bottles, Separating funnel, Funnel, Measuring Cylinder, Pipette, Stirrer, etc.

INSTRUMENTS: Weighing balance, Melting point apparatus and Hot air oven.

CHEMICALS: Nitrobenzene, Sulphuric Acid, Dinitrobenzene, Ethanol.

PRINCIPLE: Nitrobenzene is prepared by the reaction between nitrobenzene and nitrating mixture (conc. Nitric acid and conc. sulphuric acid). It is a nitration reaction and also an electrophilic aromatic substitution reaction. The substitution of one or more hydrogen atoms in an organic compound by one or more nitro groups is known as nitration. The nitro group already present on the aromatic ring is a deactivating group and also a meta director it directs the attacking groups or incoming electrophiles towards the meta position. If the rate of electrophilic substitution of monosubstituted benzene is less than that of benzene then the substituent present on the benzene ring is known as a deactivating group. Groups of any agents, that direct or cause the attack of the incoming species or attacking electrophiles mainly at the meta position, then the substituent or group is known as an *m*-director. Here sulphuric acid being a strong acid, is acting as a catalyst in the presence of sulphuric acid, and nitric acid generates the electrophile i.e., nitronium ion (NO_2^+).

REACTION:



PROCEDURE:

Place 7ml of conc sulphuric acid and 5ml of fluming nitric acid in a 100ml round bottom flask; add slowly 3 gms of nitrobenzene shake the flask to ensure thorough mixing. Attach a reflux condenser and heat the mixture on the boiling water bath for about 30 mints. Cool the mixture and pore it in a beaker containing ice water. Filter the precipitate, wash it thoroughly with water. Recrystallise the crude product from hot ethanol. The yield is 5.5gm and M.P. is 89-90°C.

$$\text{Percentage Yield} = \frac{\text{Practical Yield}}{\text{Theoretical Yield}} \times 100$$

REPORT: m-Dinitrobenzene was prepared, submitted and reported the following

Particulars	Practical Value
Theoretical Yield (gm)	
Practical Yield	
Percentage Yield	

SYSTEMATIC GENERAL PROCEDURE FOR ANALYSIS OF UNKNOWN ORGANIC SUBSTANCE

Experiment: 11

PRELIMINARY OBSERVATION OF THE COMPOUND

AIM: To carry out the systematic qualitative analysis of Organic sample.

TEST	OBSERVATION	INFERENCE
1. Physical state 2. Colour 3. Odour 4. Ignition test: Ignite a small quantity of the substance taken on a nickel spatula.	Solid. Liquid -Coloured compounds. -Colourless compounds. i. Odour of bitter almonds. ii. Fruity odour. iii. Odour of carbolic soap. iv. Fishy or Unpleasant odour. v. Pleasant odour. -Burn with sooty flame -No sooty flame	i. It may be benzaldehyde ii. nitrobenzene. ii. Ester. iii. Phenol iv. All Amines. v. Alcohols or Halogenated hydrocarbon. Aromatic compound. Aliphatic compound.
5. Saturation test Bromine test To 0.05 gm (or 2 drops) of the sample in 1ml of carbon tetrachloride or acetic acid. To this add drop wise a 2% soln. of Br ₂ in CCl ₄ . Bayer's test: Dissolve 0.05 gm or 2drops of the sample in acetone or water. Drop wise add a 2% aqueous soln. of KMnO ₄ (2-4 drops) and shake.	-Colour of Br ₂ remains same. -Colour of Br ₂ disappeared. -Colour of KMnO ₄ remains same. -Colour of KMnO ₄ disappeared.	-Compound is saturated. -Compound is unsaturated. -Compound is saturated. -Compound is unsaturated.

REPORT:

The given unknown organic sample contains -----.

Experiment: 12

DETECTION OF EXTRA ELEMENTS

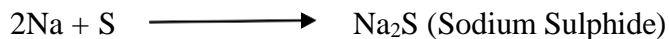
AIM: To carry out the detection of extra elements by the Lassaigne's test.

APPARATUS REQUIRED: Sodium fusion tubes (ignition tubes), china dish, pestle, test tubes, test tube holder, wire gauze and a pair of tongs.

CHEMICALS AND REAGENTS REQUIRED: Sodium metal, ferrous Sulphate, dilute sulphuric acid, dil. nitric acid, sodium nitroprusside, acetic acid, lead acetate, silver nitrate (AgNO_3).

PRINCIPLE OF LASSAIGNE'S TEST:

When an organic compound is fused with sodium, the nitrogen of the compound is converted into cyanide, sulphur into sulphide and halogens into sodium halides. These sodium salts being soluble in water come into aqueous solution. These salts are ionisable and hence the presence of cyanide, sulphide or halide ions is easily detected through the usual test.

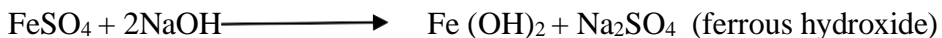


When nitrogen and sulphur both are present, Sodium thiocyanate is formed.



Nitrogen Test:

When ferrous sulphate is added to sodium fusion extract, it reacts with alkali and dirty green ppt. of ferrous hydroxide is produced. These react with sodium cyanide to give sodium ferrocyanide with which ferric ion produces bluish ferric ferrocyanide.



Sulphur Test:

1. When sodium fusion extract is treated with Lead acetate, a black precipitate of lead sulphide insoluble in acetic acid is produced

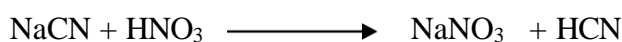


2. When sodium fusion extract is treated with freshly prepared sodium-nitroprusside, a violet coloured precipitate is obtained.

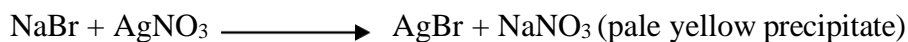


Test for Halogens:

Before adding silver nitrate, the sodium fusion extract is boiled with dil. HNO_3 to decompose NaCN , if NaCN is present it produces a white ppt. even though halogen is absent.



The halides react with silver nitrate giving the precipitate of the corresponding silver halides.



PROCEDURE:

Preparation of sodium fusion extract (LASSAIGNE'S TEST)

Place a freshly cut, clean and dry sodium metal into an ignition tube. The ignition is held in a vertical position with a pair of tongs. The lower part of the tube is heated gently till the sodium metal melts to a shining globule. The ignition tube is removed from the flame and a small amount of organic compound (about 0.05gm in case of solid and a drop in case of a liquid) is added which is under examination. The ignition tube is heated strongly in a non-luminous flame till it becomes red hot.

Now it is plunged into china dish containing distilled water and the china dish is immediately covered with clean wire gauze. After the completion of the reaction the china dish is heated to boil, and then filter. The resulting alkaline colourless filtrate is known as sodium fusion extract or Lassaigne's extract. The sodium fusion extract is divided into portions and can be utilized for the following test

DETECTION OF EXTRA ELEMENTS:

TEST	OBSERVATION	INFERENCE
<p>Test for Nitrogen To 2ml of sodium extract, add 0.2 gm of ferrous sulphate. Heat to boil and cool and add 1-2 drops of conc. H_2SO_4.</p>	<p>- Blue / Green colour appeared - No blue / Green colour appeared.</p>	<p>- Nitrogen is present - Nitrogen is absent.</p>
<p>2. Test for sulphur a) Sodium nitroprusside test: To 3 drops of SFE add one drop freshly prepared sodium nitroprusside solution. b) Lead acetate test: To 1 ml of SFE, add 1ml of dil. CH_3COOH and few drops of $(CH_3COO)_2Pb$ soln. And heat and shake.</p>	<p>-A black precipitate is obtained. -No black precipitate obtained. -A purple / violet precipitate is obtained.</p>	<p>-Sulphur is present -Sulphur is absent -Sulphur is present. -Chlorine is present. -Bromine is present.</p>
<p>3. Test for halogens To 2ml of SFE, add 1ml of dil. HNO_3, reduce the volume to half and add excess of $AgNO_3$.</p>	<p>-White ppt. soluble in NH_4OH solution. -A pale yellow ppt. sparingly soluble in NH_4OH solution. -Yellow ppt. obtained but insoluble in NH_4OH solution.</p>	<p>-Iodine is present.</p>

REPORT:

Experiment: 13

DETECTION OF SOLUBILITY

AIM: To determine the solubility of the given organic compound.

APPARATUS REQUIRED: Test tubes, TT holder.

PROCEDURE:

Shake 0.5ml (0.1g) of sample with 3-4 ml of the solvent and observe its solubility, tabulate the result in the given order.

NOTE:

1. Test the solubility of the substance in room temperature; do not heat.
2. Use dry test tube in finding the solubility in ether / in concentrated H₂SO₄.
3. Use the solvent in the order given below.
 - I. Water
 - II. Ether
 - III. 5% NaOH solution
 - IV. 5% HCl solution
 - V. Cold Concentrated. H₂SO₄
4. If the substance is soluble in earlier solvent, do not proceed further.
5. If the substance belongs to Group I & Group II, it may also show positive test for Group III, IV or V also.
6. If the substance is soluble in water, do not try solubility in ether.
7. If the substance contains N₂ or S and insoluble in water, 5% NaOH, 5% HCl, do not try its solubility in Concentrated H₂SO₄; regards this compound of Group VII.
8. If the substance reacts with Concentrated H₂SO₄ to form a solid, consider this compound to be soluble in Concentrated H₂SO₄. And it belongs to Group V.

SOLUBILITY CHART

Group	Water	Ether	5% NaOH	5% NaHCO ₃	5% HCl	Con. H ₂ SO ₄
I	+	+				
II	+	-				
IIIA	-	-	+	+		
IIIB	-		+	-		
IV	-		-		+	
V	-		-		-	+
VI*	-		-		-	-
VII	-		-		-	-

DETERMINATION OF THE SOLUBILITY GROUP:

Soluble in water	
Soluble in ether	Insoluble in ether
<i>Group-I</i>	<i>Group-II</i>
Lower molecular weights of alcohols, Aldehydes, ketones, carboxylic acids.	Amides & Carbohydrates

DETERMINATION OF THE SOLUBILITY GROUP:

Insoluble in Water					
Soluble in 5% NaOH (Group IV).		Soluble in 5% HCl.	Compounds not having N, S		Compounds having N, S, Halogen & not in Group I to IV
Soluble in NaHCO ₃ .	Insoluble in NaHCO ₃		Soluble in Conc. H ₂ SO ₄	Insoluble in Conc. H ₂ SO ₄	
Group-III A	Group-III B	Group-IV	Group-V	Group-VI	Group-VII
Aromatic Carboxylic acids	Phenols.	Amines	Higher molecular weights of Alcohols, Aldehydes, Ketones, Esters.	Hydro Carbons, Halogenated Hydrocarbons	Nitro Compounds , Amides, Anilides.

REPORT:

Experiment: 14

FUNCTIONAL GROUP ANALYSIS

AIM: To carry out the functional group analysis for the given sample.

APPARATUS REQUIRED: China dish, test tubes, test tubes holder, wire gauze and a pair of tongs.

GROUP I

The given organic compound is soluble in water and ether, it belongs to I group

- **Acidic compound:** It may be lower molecular weight **aliphatic acids** eg. Formic acid, acetic acid, poly hydrozyl phenol etc.
- **Neutral compounds:** It may be lower molecular weight **alcohols, aldehydes, ketone, esters etc.** These functional groups are identified by the subsequent tests:

1. QUALITATIVE TEST FOR ACIDS.

EXPERIMENT	OBSERVATION	INFERENCE
Test for Acids/ Neutral compounds:		
Litmus test: On a blue litmus paper place a drop of solution of substance.	-Litmus paper turns red colour -Litmus paper does not turn to red colour	-May be acid or Phenolic compound. -Compound is neutral compound.
Test For acidic compounds:		
1. Sodium bicarbonate test: Add the sample solution in to the freshly prepare Sodium bicarbonate solution.	- Formation of effervescence.	-Acid present. (Lower molecular weight aliphatic acid)
2. Esterification test: To the substances add 1 ml of ethyl alcohol and few drops of Conc. H ₂ SO ₄ .	-Fruity odor.	-Lower molecular weight aliphatic acid present.
3. Phenolphthalein Test: To a very dilute solution of NaOH, add one drop of Phenolphthalein then add the solution of the sample.	-The pink colour disappears.	-Lower molecular weight aliphatic acid present.
4. Confirmatory Test (Acetic Acid): Ferric chloride test: 1ml of sample solution and 1 ml of ammonium hydroxide, boil, cool add 2 drops of FeCl ₃ solution.	-A blood red colour obtained.	-The presence of acetic acid is confirmed.

2. QUALITATIVE TEST FOR ALDEHYDES.

EXPERIMENT	OBSERVATION	INFERENCE
1. 2,4-DNP Test 0.2g substance in 3ml dil. HCl + 2ml solution of 2,4-dinitrophenyl hydrazine in dil. HCl, shake well , allow for 5mins.	Yellow orange or red crystalline ppt. at once or on gentle warming on a water bath.	Aldehyde or Ketone present.
2. Sodium Bisulphite Test Equal amount substance and conc. Aq. solution of sodium bisulphate.	Pale yellow crystalline solid. (Exception Acetophenone).	Aldehyde or Ketone present.
3. Schiff's Reagent Test 2ml substance + 2ml schiff's reagent – shake for 2mins.	Immediate pink or red colour.	Aldehydes.
4. Fehling's solution Test Equal volumes of fehling's solution A & B + 0.2 g substance – boil.	Blue colour changes to reddish brown ppt.	Benzaldehyde present.
5. Tollen's Reagent Test To 2ml of tollen's reagent (1ml of AgNO ₃ soln. + 2 drops of NaOH + NH ₄ OH till brown ppt. just dissolves). Add 2-3 drops of substance – keep the test tube in hot water bath.	Shining silver mirror or black ppt.	Benzaldehyde present.

3. QUALITATIVE TEST FOR KETONES

EXPERIMENT	OBSERVATION	INFERENCE
1. 2,4-DNP Test 0.2g substance in 3ml dil. HCl + 2ml solution of 2,4-dinitrophenyl hydrazine in dil. HCl, shake well , allow for 5mins.	Yellow orange or red crystalline ppt. at once or on gentle warming on a water bath.	Aldehyde or Ketone present.
2. Sodium Bisulphite Test Equal amount substance and conc. Aq. solution of sodium bisulphate.	Pale yellow crystalline solid. (Exception Acetophenone).	Aldehyde or Ketone present.
3. Schiff's Reagent Test 2ml substance + 2ml schiff's reagent – shake for 2mins.	No Immediate pink or red colour.	Ketone present.
4. Legal's Test Substance + Sodium nitroprusside	Wine Red colour.	Acetophenone

solution + dil. NaOH. Shake well + glacial acetic acid.		present.
5. Zimmermann Test 1ml of sample + 1ml of alcohol + 0.1g of meta dinitrobenzene + NaOH soln – shake well.	Immediate violet colour.	Acetophenone present.
6. Iodoform Test Substance + Iodine in 20% KI soln. Warm, add more iodine soln till the colour persists + 1ml of 10% NaOH soln.	A yellow ppt. of iodoform.	Acetophenone confirmed.

4. QUALITATIVE TESTS FOR ALCOHOLS

EXPERIMENT	OBSERVATION	INFERENCE
1. Reaction with sodium Take 1ml of compound in a dry test tube, dissolve in dry benzene. Add a small piece of freshly cut sodium metal.	Effervescence of hydrogen gas evolved.	Alcohol group is present.
2. Reaction with acetic anhydride Take 1ml of compound in a dry test tube. Add equal amount of acetic anhydride and 2 drops of Conc. H ₂ SO ₄ . It is warmed and poured into a beaker containing 20ml of NaHCO ₃ soln.	Fruity Odour is obtained.	Alcohol group is present.
3. Lucas test: Take the sample in a test tube, add 5ml of Lucas reagent (Solution of ZnCl ₂ in conc. HCl) and shake vigorously for 5 min.	-Solution became turbid immediately. -Solution became turbid within 5 min. -Solution remain clear	-Tertiary alcohol. -Secondary alcohol. -Primary alcohol

5. QUALITATIVE TEST FOR ESTERS

EXPERIMENT	OBSERVATION	INFERENCE
<p>1. <u>Hydrolysis Reaction</u></p> <p>Take 1ml of compound in a test tube, add 2 drops of NaOH soln and a drop of Phenolphthalein indicator. Heat to this add 3ml of Conc.HCl, heat and cool.</p>	White ppt. is observed.	Aromatic Ester present.
<p>2. <u>Hydroxamine Acid Test</u></p> <p>Take 2 drops of compound in a test tube. Add 3ml of hydroxylamine HCl soln and a drop of Phenolphthalein indicator. To the resulting mixture, add alcoholic KOH till it gets pink colour. Boil & cool. Add 3ml of 2N HCl & 5 drops of FeCl₃ soln.</p>	Purple colour appeared.	Esters are present.

REPORT

The given unknown organic sample contains -----

Experiment: 15

FUNCTIONAL GROUP ANALYSIS

AIM: To carry out the functional group analysis for the given sample.

APPARATUS REQUIRED: China dish, test tubes, test tubes holder, wire gauze and a pair of tongs.

GROUP II

The given organic compound is soluble in water but insoluble in ether, it belongs to II group.

- **Nitrogenous compound:** It may be lower molecular weight **aliphatic amide or ureas**.
- **Non-nitrogenous compound:** It may be **carbohydrate**.

These functional groups are identified by the subsequent tests:

1. QUALITATIVE TEST FOR CARBOHYDRATES.

EXPERIMENT	OBSERVATION	INFERENCE
1. Substance + Conc. H ₂ SO ₄ warm.	Charring without evolution of gas.	Carbohydrates is present.
2. Molisch's Test: 0.1g substance in 3ml of water + 2-3 drops of Molisch's reagent (10% shake add 2ml of conc. H ₂ SO ₄ along the sides of the test tube without shaking.	Violet ring at the junction and deep violet colour on shaking.	Carbohydrates is present.
3. Fehling's Test Equal volumes of fehling's solution A and B + dilute solution of the substance, boil.	Red precipitate.	Reducing Sugar (Glucose, Fructose, Lactose, Maltose).
3. Barfoed's Test 1ml dilute solution of substance + 1ml fresh barfoed's reagent (0.3g neutral copper acetate in	Yellowish red ppt after prolonged heating .	Disaccharides.

5ml cold 1% acetic acid) – heat in a water bath.		
4. Osazone formation Substance + sodium acetate + phenyl hydrazine HCl - heat in a water bath.	Yellow crystalline ppt of osazone.	Carbohydrates.
5. Benedict's reagent test: 1ml of dilute soln of substance + benedict's reagent. Keep in a water bath, cool.	Orange or red ppt.	Carbohydrate is a reducing sugar.
6. Tollen's reagent test 1ml of dilute soln of substance + tollen's reagent. Keep in a water bath, cool.	Silver mirror or black ppt.	Carbohydrates is a reducing sugar.
7. Seliwanoff's Test: 1ml dilute soln of substance + seliwanoff reagent. Keep in a water bath, cool.	No Red colour.	Aldosugar.
8. Polysaccharides Test 1ml dilute soln of substance + Iodine solution.	No Blue colouration.	Polysaccharides absent.

2. QUALITATIVE TEST FOR AMIDES/UREA.

EXPERIMENT	OBSERVATION	INFERENCE
1. Boil 0.3g of the substance + 3ml of 10% NaOH smell. Hold a moist red litmus paper or glass rod dipped in Conc. HCl near the mouth of test tube.	Smell of Ammonia. Red litmus turns to blue. Dense white fumes of NH ₄ Cl near the glass rod.	Amide or urea present.
2. Boil a small quantity of the substance with 3ml of 1:1 HCl.	Acetic acid evolved.	Amide or urea present.
3. Hoffmann reaction 1ml of NaOH + bromine	Decolourised with evolution	

water dropwise till yellow colour persists. Add 0.1g of the substance.	of Nitrogen.	Urea is present.
4 Nitrous acid Test: Dissolve 0.3g of the substance + 3ml of dil. HCl + 3ml of 10% NaNO ₂ (Sodium Nitrite).	Effervescence with evolution of nitrogen.	Urea is present.
5. Urea Nitrate Test Dissolve 0.3g of the substance + 3ml of water + 1ml Conc.HNO ₃ . Scratch the sides the test tube if necessary.	Crystals of Urea nitrate separates out.	Urea is present.
6. Urea Oxalate Test Dissolve 0.3g of the substance + 3ml of water + 1ml Conc. Oxalic acid solution. Scratch the sides the test tube if necessary.	Crystals of Urea oxalate separates out.	Urea is present.
7. Biuret Test Boil 0.3g of the substance in dry test tube + Dissolve in 3ml of dil. NaOH. Add solution of a very dil.CuSO ₄ dropwise.	Purple/Violet colour.	Urea is confirmed.
8. Test for Thiourea a. Boil 0.3g of the substance in 3ml of dil. NaOH. Heat, add solution of lead acetate. b. Boil 0.3g of the substance and NaOH in dry test tube + Dissolve in water. Add solution of aqueous FeCl ₃ solution.	Brown/Black colour. Blood red colour.	Thiourea present. Thiourea present.

REPORT

The given unknown organic sample contains -----

Experiment: 16

FUNCTIONAL GROUP ANALYSIS

AIM: To carry out the functional group analysis for the given sample.

APPARATUS REQUIRED: China dish, test tubes, test tubes holder, wire gauze and a pair of tongs.

GROUP III – A & B

The given organic compound is soluble in 5% NaOH and 5% NaHCO₃ solution and belongs to

III-A group.

The given organic compound is soluble in 5% NaOH and insoluble in 5% NaHCO₃ solution, belongs to **III-B group.**

III-A Compounds: It may be high molecular weight **aliphatic or aromatic acids.**

III-B Compounds: It may be high molecular weight **aliphatic or aromatic phenols.**

Test for III-A Compounds

1. Sample soln. and 5% Na OH soln. shake well.	-Compound dissolved.	-can be carboxylic acid or phenol.
2. Sample soln. and 5% NaOH soln. shake well.		-Carboxylic acid present
3. Esterification test: Boil the sample with 2 parts of absolute alcohol and 1 part of conc. H ₂ SO ₄ , cool and pour in aqueous Na ₂ CO ₃ soln. in a china dish.	-Compound dissolved. -Compound not dissolved.	-Phenol present.
	-sweet fruity smell obtained.	-Carboxylic acid present
Distinguishing test between Phenolic and non Phenolic carboxylic acid.		
4. Neutral ferric chloride test: Add few drops of neutral ferric chloride soln. in to the aqueous sample soln.	-Violet colour obtained. -No Violet colour obtained.	-Phenolic carboxylic acid -Non-phenolic carboxylic acid

Test for III-B Compounds

<p>1. Bromine test: Dissolved the sample in CCl_4 and add solution of bromine in CCl_4 drop wise until bromine colour present for 1 min.</p>	<p>-A white ppt. of HBr obtained.</p>	<p>-phenol is present.</p>
<p>2. Liebermann's Nitroso test: Fused little amount of sample with few crystals of NaNO_2 in a test tube. Cool and 1ml of conc. H_2SO_4.</p>	<p>-A deep green-blue colour appeared which is turns to pale red on adding excess cold water and again turns green-blue on adding excess NaOH soln.</p>	<p>-Phenol is present.</p>
<p>3. Azodye test: Dissolved few nl of aniline in dil. HCl, cool at 5°C and then add cold saturation soln. of NaNO_2. With shaking pour this soln. in the sample soln. prepared by dil. NaOH soln.</p>	<p>-Red/Orange colour appeared. -Red colour appeared.</p>	<p>-Phenol is present. -Phenol/Resorcinol.</p>
<p>4. Phthalein test: Heat little amount of sample with 1 gram of phthalic anhydride and few drops of conc. H_2SO_4 for 1 min. cool and make the soln. alkaline with dil. NaOH soln. pour few drops of resulting soln. to 10 ml of water.</p>	<p>-Red colour obtained. -blue colour obtained. -Fluorescent green colour obtained. -No colour.</p>	<p>-m-Cresol. -Resorcinol. -p-Cresol.</p>

REPORT

The given unknown organic sample contains -----.

Experiment: 17

FUNCTIONAL GROUP ANALYSIS

AIM: To carry out the functional group analysis for the given sample.

APPARATUS REQUIRED: China dish, test tubes, test tubes holder, wire gauze and a pair of tongs.

GROUP- IV

The given organic compound is soluble in 5% HCl, it belongs to **IV group**.

EXPERIMENT	OBSERVATION	INFERENCE
A. <u>Acetylation</u> Substance + acetyl chloride dropwise – shake.	Vigorous reaction, solid separates.	1 ⁰ or 2 ⁰ amines present.
B. <u>Carbylamine reaction</u> Substance + 2 drops of CHCl ₃ + 1ml alcoholic KOH warm.	Unpleasant odour.	1 ⁰ amine present.
C. Azodye test: Dissolve few ml of aniline in dil. HCl, cool at 5 ⁰ C and then add cold saturation soln. of NaNO ₂ . With shaking pour this soln. in the sample soln. prepared by dil. NaOH solution.	Orange Dye.	Aromatic 1 ⁰ amine present.
D. Sample + Conc. HCl + sodium nitrite.	Clear yellow solution Turns starch iodide paper blue.	Aromatic 1 ⁰ amine present.

IV group compound: It may be **Primary, Secondary and tertiary amines**. These functional groups are identified by the subsequent tests.

REPORT

The given unknown organic sample contains -----.

Experiment: 18

FUNCTIONAL GROUP ANALYSIS

AIM: To carry out the functional group analysis for the given sample.

APPARATUS REQUIRED: China dish, test tubes, test tubes holder, wire gauze and a pair of tongs.

GROUP V

The given organic compound is soluble in conc. H_2SO_4 , it belongs to **V group**.

- **Neutral compounds:** It high molecular weight **alcohols, aldehydes, ketone, esters** etc. These functional groups are identified by the subsequent tests:

1. QUALITATIVE TEST FOR ALDEHYDES.

EXPERIMENT	OBSERVATION	INFERENCE
1. 2,4-DNP Test 0.2g substance in 3ml dil. HCl + 2ml solution of 2,4-dinitrophenyl hydrazine in dil. HCl, shake well, allow for 5mins.	Yellow orange or red crystalline ppt. at once or on gentle warming on a water bath.	Aldehyde or Ketone present.
2. Sodium Bisulphite Test Equal amount substance and conc. Aq. solution of sodium bisulphate.	Pale yellow crystalline solid. (Exception Acetophenone).	Aldehyde or Ketone present.
3. Schiff's Reagent Test 2ml substance + 2ml schiff's reagent – shake for 2mins.	Immediate pink or red colour.	Aldehydes.
4. Fehling's solution Test Equal volumes of fehling's solution A & B + 0.2 g substance – boil.	Blue colour changes to reddish brown ppt.	Benzaldehyde present.
5. Tollen's Reagent Test To 2ml of tollen's reagent (1ml of $AgNO_3$ soln. + 2 drops of NaOH + NH_4OH till brown ppt. just dissolves). Add 2-3 drops of substance – keep the test tube in hot water bath.	Shining silver mirror or black ppt.	Benzaldehyde present.

2. QUALITATIVE TEST FOR KETONES.

EXPERIMENT	OBSERVATION	INFERENCE
1. 2,4-DNP Test 0.2g substance in 3ml dil. HCl + 2ml solution of 2,4-dinitrophenyl hydrazine in dil. HCl, shake well , allow for 5mins.	Yellow orange or red crystalline ppt. at once or on gentle warming on a water bath.	Aldehyde or Ketone present.
2. Sodium Bisulphite Test Equal amount substance and conc. Aq. solution of sodium bisulphate.	Pale yellow crystalline solid. (Exception Acetophenone).	Aldehyde or Ketone present.
3. Schiff's Reagent Test 2ml substance + 2ml schiff's reagent – shake for 2mins.	No Immediate pink or red colour.	Ketone present.
4. Legal's Test Substance + Sodium nitroprusside solution + dil. NaOH. Shake well + glacial acetic acid.	Wine Red colour.	Acetophenone present.
5. Zimmermann Test 1ml of sample + 1ml of alcohol + 0.1g of meta dinitrobenzene + NaOH soln – shake well.	Immediate violet colour.	Acetophenone present.
6. Iodoform Test Substance + Iodine in 20% KI soln. Warm, add more iodine soln till the colour persists + 1ml of 10% NaOH soln.	A yellow ppt. of iodoform.	Acetophenone confirmed.

3. QUALITATIVE TESTS FOR ALCOHOLS.

EXPERIMENT	OBSERVATION	INFERENCE
1. Reaction with sodium Take 1ml of compound in a dry test tube, dissolve in dry benzene. Add a small piece of freshly cut sodium metal.	Effervescence of hydrogen gas evolved.	Alcohol group is present.
2. Reaction with acetic anhydride Take 1ml of compound in a dry test tube. Add equal amount of acetic anhydride and 2 drops of Conc. H ₂ SO ₄ . It is warmed and poured into a beaker containing 20ml of NaHCO ₃ soln.	Fruity Odour is obtained.	Alcohol group is present.
3. Lucas test: Take the sample in a test tube, add 5ml of Lucas reagent (Solution of ZnCl ₂ in conc. HCl) and shake vigorously for 5 min.	-Solution became turbid immediately. -Solution became turbid within 5 min. -Solution remain clear	-Tertiary alcohol. -Secondary alcohol -Primary alcohol

4. QUALITATIVE TEST FOR ESTERS.

EXPERIMENT	OBSERVATION	INFERENCE
1. Hydrolysis Reaction Take 1ml of compound in a test tube, add 2 drops of NaOH soln and a drop of Phenolphthalein indicator. Heat to this add 3ml of Conc.HCl, heat and cool.	White ppt. is observed.	Aromatic Ester present.
2. Hydroxamine Acid Test Take 2 drops of compound in a test tube. Add 3ml of hydroxylamine HCl soln and a drop of Phenolphthalein indicator. To the resulting mixture, add alcoholic KOH till it gets pink colour. Boil & cool. Add 3ml of 2N HCl & 5 drops of FeCl ₃ soln.	Purple colour appeared.	Esters are present.

REPORT: The given unknown organic sample contains -----.

Experiment: 19

FUNCTIONAL GROUP ANALYSIS

AIM: To carry out the functional group analysis for the given sample.

APPARATUS REQUIRED: China dish, test tubes, test tubes holder, wire gauze and a pair of tongs.

GROUP VI

The given organic compound is insoluble in conc. H_2SO_4 , not containing nitrogen or sulphur, it belongs to the **VI group**.

Compounds: It maybe **aromatic hydrocarbon or halogenated hydrocarbon** etc.

These functional groups are identified by the subsequent tests

TEST	OBSERVATION	INFERENCE
1. Sulphonation: To sample in dry test tube, add 1 ml of conc. H_2SO_4 heat the mixture till it becomes clear, cool and pour into 10 ml of cold water taken in a breaker. Separate the solids in a test tube add zinc granule. Conc. HCl then heat for 2min. transfer the clear liquid to the another test tube and cool and add cold saturated solution of $NaNO_2$ followed by alkaline solution of β -naphthol.	-Pale yellow solids Separated out. -Red/orange dye formed.	-Aromatic hydrocarbon or halogenated hydrocarbon present.

Confirmatory test for Aromatic hydrocarbons		
<p>1. Dimethyl sulphate test: Little amount of sample and 2ml of dimethyl sulphate solution, shake well.</p> <p>2. Aluminium chloride test: 0.5 gram of sample dissolved in 1ml of CCl₄ then add a pinch of anhydrous Aluminum chloride.</p>	<p>-Substance dissolve with the liberation of heat.</p> <p>-A red or green colour obtained.</p>	<p>-Aromatic hydrocarbon present.</p> <p>-Aromatic hydrocarbon confirmed.</p>
Confirmatory test for Halogenated hydrocarbons:		
<p>1. Beilstein's test: Heat a copper wire in the flame till it gives no colour and then cools, deep the wire in the sample and again heat on the flame.</p> <p>2. Silver nitrate test: 0.5 ml of sample solution and alcoholic KOH, boil, cool acidity with dil. HNO₃ and then add alcoholic AgNO₃ solution.</p>	<p>Green or greenish blue flame obtained.</p> <p>-gray / Pale yellow ppt. obtained.</p> <p>-No ppt. obtained.</p>	<p>-Halogenate hydrocarbon present.</p> <p>-Halogen attached to side chain.</p> <p>-Halogen attached to the ring directly.</p>

REPORT

The given unknown organic sample contains -----.

Experiment: 20

FUNCTIONAL GROUP ANALYSIS

AIM: To carry out the functional group analysis for the given sample.

APPARATUS REQUIRED: China dish, test tubes, test tubes holder, wire gauze and a pair of tongs.

GROUP-VII

The given organic compound having nitrogen or sulphur which are not soluble in water, 5% NaOH, 5% HCl it belongs to VII group.

Compound present: It may be **nitro compound or substituted amides.**

These functional groups are identified by the subsequent tests:

EXPERIMENT	OBSERVATION	INFERENCE
1. Mulliken's and Barker's Test Substance + 2ml ethanol + 1ml CaCl ₂ solution + pinch of Zn dust or tin boil for 5min cool and filter into 2ml of tollen's reagent. Heat on water bath if necessary.	Black ppt.	Nitrocompound present.
2. Acid Reduction Test Substance + conc. HCl + pinch of Zn dust boil, cool perform dye test. Add 0.1g of NaNO ₂ soln, cool at 0-5 ⁰ c and add B-Naphthol soln. in NaOH.	Red colour Dye.	Nitrocompound present.
3. Janowsky Reaction Substance + 5ml acetone + 2ml of 5% NaOH. Shake well.	Faint yellow colour.	Mono Nitrocompounds present.
4. Substance + FeSO₄ crystals + dil. H₂SO₄ + ethanolic KOH, shake well.	Brown ppt.	Mono Nitrocompounds present.

REPORT: The given unknown organic sample contains -----.



Vision and Mission of the Institution

Vision

The East Point College of Pharmacy aspires to be a globally acclaimed institution, **recognized for excellence in** pharmaceutical education, research and nurturing students for **holistic development**.

Mission

- M1** Create pharmacy graduates through **quality education**
- M2** Promote innovation, **creativity**, and excellence **in teaching**, learning, and **research**
- M3** **Inspire** integrity, teamwork, critical thinking, **personal** development, and ethics in **students** and lay **the** foundation for lifelong learning
- M4** **Serve** the **healthcare, technological, scientific**, and **economic** needs of then **society**.