

TECHNOCRATS

Lab Work Book of

**Pharmaceutical Organic
Chemistry-I**

(BP - 208P)

Department of Pharmacy

Lab Manual of
Pharaceutical Organic Chemistry-I
(BP - 208P)

Price : ₹ 100/-

Edition :

© Copyright Reserved

No part of this book can be reproduced or transmitted in any form or by any means, electronic or mechanical without the written permission of the publisher.

Disclaimer

Every possible effort has been made to bring out this book accurately to fullfill aspirations of all readers. The publisher and their associates do not make any warranty with respect to accuracy, completeness of the book and hence can not be held liable in any way for the loss or damage whatsoever.

Printed & Published by :



TECHNOCRATS
PUBLICATIONS

Arera Colony, Bhopal.

e-mail : technocratspublications@gmail.com



TECHNOCRATS
PUBLICATIONS

Lab Work Book
of
Pharmaceutical Organic Chemistry-I
(BP - 208P)

(Strictly According to RGPV Syllabus)

Name :

Enrollment No. :

Institute :

Academic Session :

Department of Pharmacy



TECHNOCRATS
PUBLICATIONS

Vision of the Institute

To grow as an institute of Excellence for Pharmacy Education and Research and to serve the humanity by sowing the seeds of intellectual, cultural, ethical, and humane sensitivities in the students to develop a scientific temper, and to promote professional and technological expertise.

Mission of the Institute

M 1: To inculcate ethical, moral, cultural and professional values in students

M 2: To provide state of art infrastructure facilities to the staff and students so as to enable them to learn latest technological advancements

M 3: State of art learning of professionalism by the faculty and students

M 4: To produce well learned, devoted and proficient pharmacists

M 5: To make the students competent to meet the professional challenges of future

M 6: To develop entrepreneurship qualities and abilities in the students

PROGRAM OUTCOMES (POs)

- 1. Pharmacy Knowledge:** Possess knowledge and comprehension of the core and basic knowledge associated with the profession of pharmacy, including biomedical sciences; pharmaceutical sciences; behavioral, social, and administrative pharmacy sciences; and manufacturing practices.
- 2. Planning Abilities:** Demonstrate effective planning abilities including time management, resource management, delegation skills and organizational skills. Develop and implement plans and organize work to meet deadlines.
- 3. Problem analysis:** Utilize the principles of scientific enquiry, thinking analytically, clearly and critically, while solving problems and making decisions during daily practice. Find, analyze, evaluate and apply information systematically and shall make defensible decisions.
- 4. Modern tool usage:** Learn, select, and apply appropriate methods and procedures, resources, and modern pharmacy-related computing tools with an understanding of the limitations.
- 5. Leadership skills:** Understand and consider the human reaction to change, motivation issues, leadership and team-building when planning changes required for fulfillment of practice, professional and societal responsibilities. Assume participatory roles as responsible citizens or leadership roles when appropriate to facilitate improvement in health and well-being.
- 6. Professional Identity:** Understand, analyze and communicate the value of their professional roles in society (e.g. health care professionals, promoters of health, educators, managers, employers, employees).
- 7. Pharmaceutical Ethics:** Honour personal values and apply ethical principles in professional and social contexts. Demonstrate behavior that recognizes cultural and personal variability in values, communication and lifestyles. Use ethical frameworks; apply ethical principles while making decisions and take responsibility for the outcomes associated with the decisions.
- 8. Communication:** Communicate effectively with the pharmacy community and with society at large, such as, being able to comprehend and write effective reports, make effective presentations and documentation, and give and receive clear instructions.
- 9. The Pharmacist and society:** Apply reasoning informed by the contextual knowledge to assess societal, health, safety and legal issues and the consequent responsibilities relevant to the professional pharmacy practice.
- 10. Environment and sustainability:** Understand the impact of the professional pharmacy solutions in societal and environmental contexts, and demonstrate the knowledge of, and need for sustainable development.
- 11. Life-long learning:** Recognize the need for, and have the preparation and ability to engage in independent and life-long learning in the broadest context of technological change. Self-assess and use feedback effectively from others to identify learning needs and to satisfy these needs on an ongoing basis.

PEOs

PEO 1: To inculcate quality pharmacy education and training through innovative Teaching Learning Process.

PEO 2: To promote professionalism, team spirit, social and ethical commitment with effective interpersonal communication skills to boost leadership role assisting improvement in healthcare sector.

PEO 3: To enhance Industry-Institute-Interaction for industry oriented education and research, which will overcome healthcare problems of the society.

PEO 4: To adapt and implement best practices in the profession by enrichment of knowledge and skills in research and critical thinking

PEO 5: To generate potential knowledge pools with interpersonal and collaborative skills to identify, assess and formulate problems and execute the solution in closely related pharmaceutical industries and to nurture striving desire in students for higher education and career growth.

Course Outcomes (COs):

Student will be able to:

- CO1: Apply the qualitative analysis principles to analyse organic compound having different functional groups.
- CO2: Identify the unknown organic compound having different functional groups.
- CO3: Explain and understand the principals of qualitative analysis.
- CO4: Demonstrate the laboratory skills to prepare organic compounds.
- CO5: Explain the organic chemistry concepts by constructing the molecular models.

Index

S. NO.	EXPERIMENT	PAGE NO.
1	To purify the given compound by re-crystallisation.	1
2	To purify the given compound by decolorization.	4
3	To determine melting point of given compound.	7
4	To study the optical isomerism of given compounds by stereo-models.	10
5	To study the geometrical isomerism of given compounds by stereo-models	13
6	To study the structure isomerism of given compounds by stereo-models	16
7	To synthesize 2, 4, 6 – trinitro phenol (Picric acid) from phenol	19
8	To synthesize acetanilide from aniline	22
9	Microwave mediate synthesis of Mannich base.	25
10	To synthesize Schiff's base.	27
11	To determine solubility of given compound.	30

Experiment No. - 1

OBJECT:

To purify the given compound by re-crystallisation.

APPARATUS AND CHEMICAL REQUIREMENT:

Crude drug sample, Beaker, Funnel, Water bath, Glass rod, Filter paper, volatile solvent, watch glass.

THEORY:

Crystallization is used to purify a solid compound. The process requires a suitable solvent. A suitable solvent is one which readily dissolves the solid. The most common method of purifying solid organic compounds is by re-crystallization. In this technique, an impure solid compound is dissolved in a solvent and then allowed to slowly crystallize out as the solution cools. As the compound crystallizes from the solution, the molecules of the other compounds dissolved in solution are excluded from the growing crystal lattice, giving a pure solid. Crystallization of a solid is not the same as precipitation of a solid. In crystallization, there is a slow, selective formation of the crystal framework resulting in a pure compound. In precipitation, there is a rapid formation of a solid from a solution that usually produces an amorphous solid containing many trapped impurities within the solid's crystal framework. For this reason, experimental procedures that produce a solid product by precipitation always include a final re-crystallization step to give the pure compound. The process of re-crystallization relies on the property that for most compounds, as the temperature of a solvent increases, the solubility of the compound in that solvent also increases. re-crystallization is a widely-used technique to purify a solid mixture. The desired product is isolated from its impurities by differences in solubility.

PROCEDURE:

1. Take 2 gm of given crude drug sample.
2. Dissolve the sample of crude drug in 10 ml of solvent and warm if required.
3. The solution is filtered to remove insoluble impurities.
4. Evaporate the solvent.
5. Dry and collect the re-crystallized product.

RESULT AND DISCUSSION:

.....

.....

.....

.....

VIVA QUESTIONS

Q.-1. Which principle is involved in re-crystallisation process ?

.....

.....

.....

.....

Q.-2. How the solvent for re-crystallisation of compound is selected ?

.....

.....

.....

.....

Q.-3. What is meant by re-crystallisation ?

.....

.....

.....

.....

Q.-4. What are the uses of re-crystallisation in organic chemistry ?

.....

.....

.....

.....

Q.-5. What is meant by hot filtration in re-crystallisation process ?

.....

.....

.....

.....

Experiment No. – 2

OBJECT:

To purify the given compound by decolorization.

APPARATUS AND CHEMICAL REQUIREMENT:

Activated charcoal, Crude drug sample, Beaker, Funnel, Water bath, Glass rod, Filter paper.

THEORY :

Activated charcoal also called activated carbon is obtained by the destructive distillation of wood or from other plant and animal sources. The carbon residue obtained in this manner is activated by heating it with steam, oxygen or carbon dioxide. This process results in a finely divided solid with an extremely large surface area. The structure of activated charcoal is very porous and thus has a high affinity for many substances, especially organic compounds, chlorine, and many gases. Activated charcoal has been used since ancient times to remove undesirable contaminants from drinking water. Activated charcoal is an excellent adsorbent which is capable of attracting and binding the components of a mixture. Because of its high adsorption capacity, activated charcoal is a critical component in all modern water and air purification systems. It is used to decolorize, deodorize, and clarify water. The ability of activated charcoal to filter and remove impurities of organic compounds depends on the particle size, surface area, temperature and the types of impurities. The impurities are removed by adding activated charcoal to the hot solution, followed by a hot filtration to remove the charcoal. The activated charcoal adsorbs

organic compounds impurities on to its surface and purifies the organic compounds.

PROCEDURE:

1. Take 5 gm sample of crude drug.
2. Dissolve the crude drug sample in 100 ml water and warm the mixture if required.
3. Add 1 gm of activated charcoal.
4. Mix gently with a glass rod for 2 minutes.
5. Filter and collect clear, colourless filtrate, check all traces of colour removed by the charcoal.
6. Cool, filter and collect the final product.

OBSERVATION TABLE:

S. No.	Initial observation	Final observation
1	Red	
2	Yellow	

RESULT AND DISCUSSION:

.....

.....

.....

.....

.....

.....

VIVA QUESTIONS

Q.-1. What are the uses of activated charcoal ?

.....

.....

.....

.....

Q.-2. Which principle is involved in the decolorization process ?

.....

.....

.....

.....

Q.-3. What is the source of activated charcoal ?

.....

.....

.....

.....

Q.-4. What is meant by purification process ?

.....

.....

.....

.....

Q.-5. What are crude drugs ?

.....

.....

.....

.....

Experiment No. – 3

OBJECT:

To determine melting point of given compound.

APPARATUS AND CHEMICAL REQUIREMENT:

Salicylic acid, melting point apparatus, capillary tube, thermometer

THEORY:

The standard physical property of a solid is its melting point. The melting point is actually a melting point range. It is used to help determine the purity of a solid and to help verify the identity of the compound. A pure compound should melt over a narrow temperature range. Impurities usually cause the melting point range to widen and lower in value. To obtain the melting point range, record the temperature at which the first crystals begin to melt (solid to liquid phase) and the temperature at which the last crystal melts, eg; m.p. 124-126°C. The melting point of a solid is the temperature at which it changes state from solid to liquid at atmospheric pressure. At the melting point the solid and liquid phase exist in equilibrium. The melting point of a substance depends on pressure and is usually specified at standard pressure. Determining the melting point of a compound is one way to test if the substance is pure. A pure substance generally has a melting range (the difference between the temperature where the sample starts to melt and the temperature where melting is complete) of one or two degrees. Impurities tend to depress and broaden the melting range so the pure sample should have a higher or lower melting range than the original, impure sample.

PROCEDURE:

1. Fuse the capillary tube at one end.
2. Take the drug sample as fine powder.
3. Fill the capillary tube with sample about 3 mm high through open side.
4. Tap the capillary tube so that sample touch the closed end.
5. Keep the capillary tube in melting point apparatus
6. Observe the melting process through the magnifying lens.
7. Note the temperature on thermometer at which the sample gets melted completely.
8. The temperature noted is the melting point of the given sample.

OBSERVATION:

MELTING POINT:

RESULT AND DISCUSSION:

.....

.....

.....

.....

.....

.....

VIVA QUESTIONS

Q-1. What is the correlation between purity of the compounds and melting point ?

.....

.....

.....

.....

Q-2. What is meant by Melting process ?

.....

.....

.....

.....

Q-3. Why melting point is determined for organic compounds ?

.....

.....

.....

.....

Q-4. Which principle is involve in melting process ?

.....

.....

.....

.....

Q-5. What is meant by narrow temperature range in melting point determination ?

.....

.....

.....

.....

Experiment No. – 4

OBJECT:

To study the optical isomerism of given compounds by stereo-models.

APPARATUS AND CHEMICAL REQUIREMENT: stereo-model kit

THEORY :

Isomers are molecules with the same chemical formula but different physical and chemical properties as well as different special arrangements of atoms. Models will help to develop the ability to visualize three-dimensional structures and will make the two-di-mensional pictures. Chiral molecules occur widely throughout all of nature.

For example, glucose, an important sugar and energy source, is chiral the enantiomer of naturally

occurring glucose cannot be utilized as a food source. All sugars, proteins, and nucleic acids are chiral and occur naturally in only one enantiomeric form. Chirality is important in medicine as well. Over half of the organic compounds used as drugs are chiral, and in most cases only one enantiomer has the desired physiological activity. In rare cases, the inactive enantiomer is toxic. The safety and effectiveness of synthetically prepared chiral drug molecules have become issues of increasing concern for both pharmaceutical manufacturers and the U.S. Food and Drug Administration (FDA). Stereoisomers are compounds that have the same atomic connectivity but a different arrangement of atoms in space.

PROCEDURE:

1. Select the optical active compound.
2. Note the chemical name and chemical formula of the given compound.
3. Draw the three dimensional structure of both isomers of given compound.
4. Recognized the functional groups, chemical bonds and valency of compound.
5. Make the model as per the drawn structure of the isomer of the compounds.
6. The stereo-model of the compound is made
7. Study its three dimentinal structure as its different arrangement in space.
8. Stereo models of other stereo compounds can be made and studied by the same process.

RESULT AND DISCUSSION:

.....

.....

.....

.....

.....

.....

VIVA QUESTIONS

Q.-1. What is asymmetrical carbon ?

.....

.....

.....

.....

Q.-2. What are optical isomers ?

.....

.....

.....

.....

Q.-3. What do you mean by *d* and *l* isomers ?

.....

.....

.....

.....

Q.-4. What is racemic mixture ?

.....

.....

.....

.....

Q.-5. What do you mean by optical activity ?

.....

.....

.....

.....

Experiment No. – 5

OBJECT:

To study the geometrical isomerism of given compounds by stereo-models..

APPARATUS AND CHEMICAL REQUIREMENT: stereo-model kit

THEORY:

Isomers are molecules with the same chemical formula but different physical and chemical properties as well as different special arrangements of atoms. Models will help to develop the ability to visualize three-dimensional structures and will make the two-di-mensional pictures. Chiral molecules occur widely throughout all of nature.

For example, glucose, an important sugar and energy source, is chiral the enantiomer of naturally

occurring glucose cannot be utilized as a food source. All sugars, proteins, and nucleic acids are chiral and occur naturally in only one enantiomeric form. Chirality is important in medicine as well. Over half of the organic compounds used as drugs are chiral, and in most cases only one enantiomer has the desired physiological activity. In rare cases, the inactive enantiomer is toxic. The safety and effectiveness of synthetically prepared chiral drug molecules have become issues of increasing concern for both pharmaceutical manufacturers and the U.S. Food and Drug Administration (FDA). Stereoisomers are compounds that have the same atomic connectivity but a different arrangement of atoms in space.

PROCEDURE:

1. Select the stereoisomers.
2. Note the chemical name and chemical formula of the given compound.
3. Draw the three dimensional structure of both isomers of given compound.
4. Recognize the functional groups, chemical bonds and valency of compound.
5. Make the model as per the drawn structure of the isomer of the compounds.
6. The stereo-model of the compound is made
7. Study its three dimentinal structure as its different arrangement in space.
8. Stereo models of other stereo compounds can be made and studied by the same process.

RESULT AND DISCUSSION:

.....

.....

.....

.....

.....

.....

VIVA QUESTIONS

Q.-1. What are isomers ?

.....

.....

.....

.....

Q.-2. What are stereo properties of compounds ?

.....

.....

.....

.....

Q.-3. What do you mean by *cis* and *trans* isomerism ?

.....

.....

.....

.....

Q.-4. What are the use stereo compounds in pharmaceutical science ?

.....

.....

.....

.....

Q.-5. What do you mean by *Z* and *E* isomerism ?

.....

.....

.....

.....

Experiment No. – 6

OBJECT:

To study the structure isomerism of given compounds by stereo-models..

APPARATUS AND CHEMICAL REQUIREMENT: stereo-model kit

THEORY:

Isomers are molecules with the same chemical formula but different physical and chemical properties as well as different special arrangements of atoms. Models will help to develop the ability to visualize three-dimensional structures and will make the two-di-mensional pictures. Chiral molecules occur widely throughout all of nature.

For example, glucose, an important sugar and energy source, is chiral the enantiomer of naturally

occurring glucose cannot be utilized as a food source. All sugars, proteins, and nucleic acids are chiral and occur naturally in only one enantiomeric form. Chirality is important in medicine as well. Over half of the organic compounds used as drugs are chiral, and in most cases only one enantiomer has the desired physiological activity. In rare cases, the inactive enantiomer is toxic. The safety and effectiveness of synthetically prepared chiral drug molecules have become issues of increasing concern for both pharmaceutical manufacturers and the U.S. Food and Drug Administration (FDA). Stereoisomers are compounds that have the same atomic connectivity but a different arrangement of atoms in space.

PROCEDURE:

1. Select the structure isomers.
2. Note the chemical name and chemical formula of the given compound.
3. Draw the three dimensional structure of both structure isomers of given compound.
4. Recognize the functional groups, chemical bonds and valency of compound.
5. Make the model as per the drawn structure of the isomer of the compounds.
6. The structure isomer model of the compound is made
7. Study structure isomer as its different arrangement.
8. Structure isomer models of other compounds can be made and studied by the same process.

RESULT AND DISCUSSION:

.....

.....

.....

.....

.....

.....

VIVA QUESTIONS

Q.-1. What is isomerism ?

.....

.....

.....

.....

Q.-2. What do you mean by structure isomer ?

.....

.....

.....

.....

Q.-3. What are different types of structure isomers ?

.....

.....

.....

.....

Q.-4. What do you mean by chain and position isomerism ?

.....

.....

.....

.....

Q.-5. What do you mean by functional isomerism ?

.....

.....

.....

.....

Experiment No. – 7

OBJECT :

To synthesize 2, 4, 6 – trinitro phenol (Picric acid) from phenol.

APPARATUS AND CHEMICAL REQUIREMENT:

Phenol, Sulphuric acid, Nitric acid, Ethanol, Round bottom flask, Beaker, Funnel, Filter paper, Condenser, Water bath, Conical flask, Glass rod, Hot air oven,

THEORY :

Picric acid is the chemical compound formally called 2,4,6-trinitrophenol (TNP). Picric acid is an explosive. Its name comes from Greek pik'ros meaning bitter, reflecting its bitter taste. Its primary use, now outdated is as an explosive. It has also been used in medicine as an antiseptic and burn treatments, dyes and as a chemistry agent. It has found some use in organic chemistry for the preparation of crystalline salts of organic bases (picrates) for the purpose of identification and characterization. Clinical chemistry lab testing utilizes picric acid for the Jaffe reaction to test for creatinine. It forms a colored complex that can be measured using spectroscopy. Much less commonly, wet picric acid has been used as a skin dye, temporary branding agent. It reacts with proteins in the skin to give a dark brown color that may last as long as a month. In the early 20th century, picric acid was stocked in pharmacies as an antiseptic and as a treatment for burns, malaria, herpes, and smallpox. Picric acid also commonly stocked in first aid kits from that period as a burn treatment.

PROCEDURE:

1. Take 8 gm of phenol into clean and dry 500 ml flask
2. Add 10 ml of conc. H_2SO_4 and shake the mixture.
3. Heat the reaction mixture on water bath for 30 minutes to complete the formation of phenol sulphonic acid.
- Cool the flask thoroughly in ice water mixture
5. Place the flask on a wooden surface of the fuming cupboard
6. Add 30 ml of conc nitric acid and mix well.
7. The reddish fumes come out from the flask
8. Heat the mixture on water bath for 1 hr

9. Add 100 ml of cold water and mix well
10. Cool, filter and collect the yellow crystals of picric acid.
11. Wash the product with water to eliminate excess acid.
12. Dry, recrystallize and collect pure picric acid crystals.

OBSERVATION:

Theoretical yield: 13 gm

Practical yield:

% yield:

Melting Point :

RESULT AND DISCUSSION:

.....

.....

.....

.....

.....

.....

VIVA QUESTIONS

Q.-1. Which principle is involved in the synthesis of Picric acid ?

.....

.....

.....

.....

Q.-2. How can be recrystallized Picric acid ?

.....

.....

.....

.....

Q.-3. What are the medicinal uses of Picric acid ?

.....

.....

.....

.....

Q.-4. What is the colour of Picric acid ?

.....

.....

.....

.....

Q.-5. What is the use of H_2SO_4 in synthesis of Picric acid ?

.....

.....

.....

.....

Experiment No. –8

OBJECT:

To synthesize acetanilide from aniline.

APPARATUS AND CHEMICAL REQUIREMENT:

Aniline, Acetic acid, Acetic anhydride Round bottom flask, Beaker, Funnel, Filter paper, Condenser, Heating mantle, Glass rod, Hot air oven

THEORY:

Acetanilide is used as an inhibitor of hydrogen peroxide decomposition and is used to stabilize cellulose ester varnishes. It has also found uses in the intermediation in rubber accelerator synthesis, dyes and dye intermediate synthesis, and camphor synthesis. Acetanilide is used for the production of 4-acetamidobenzenesulfonyl chloride, a key intermediate for the manufacture of the sulfa drugs. It is also a precursor in the synthesis of penicillin and other pharmaceuticals. Acetanilide was the first aniline derivative serendipitously found to possess analgesic as well as antipyretic properties and was quickly introduced into medical practice under the name of Antifebrin by A. Cahn and P. Hepp in 1886. But its unacceptable toxic effects, the most alarming being cyanosis due to methemoglobinemia, prompted the search for supposedly less toxic aniline derivatives such as phenacetin. After several conflicting results over the ensuing fifty years, it was established in 1948 that acetanilide was mostly metabolized to paracetamol in the human body and that it was the paracetamol that was responsible for the analgesic and antipyretic properties. The observed methemoglobinemia after acetanilide administration was ascribed to the small proportion of acetanilide that is hydrolyzed to aniline in the body. Acetanilide is no longer used as a drug in its own right, although the success of its metabolite paracetamol is well known.

PROCEDURE:

1. Add 20 ml of acetic acid and 10 ml of aniline in round bottom flask.
2. Reflux the reaction mixture for 10 min at 70°C on water bath.
3. Pour the hot reaction mixture into 200 ml of cold water, stirring the contents well.
4. Filter and wash it thoroughly with water.
5. Recrystallized the product with acetic acid and water.

OBSERVATION:

Theoretical yield: 10 gm

Practical yield:

% yield:

Melting Point:

RESULT AND DISCUSSION:

.....

.....

.....

.....

.....

.....

VIVA QUESTIONS

Q.-1. What are the uses Acetanilide ?

.....

.....

.....

.....

Q.-2. What is the chemical name of Acetanilide ?

.....

.....

.....

.....

Q.-3. What are the temperature and time use for the synthesis of Acetanilide ?

.....

.....

.....

.....

Q.-4. How can purified Acetanilide ?

.....

.....

.....

.....

Q.-5. What is the role of acetanilide in synthesis of paracetamol ?

.....

.....

.....

.....

Experiment No. –9

OBJECT:

Microwave mediate synthesis of Mannich base.

APPARATUS AND CHEMICAL REQUIREMENT:

Benzaldehyde, Diethylamine, Acetone, Microwave, Beaker, Funnel, Filter paper, Glass rod, Hot air oven.

THEORY :

The mannich reaction is an organic reaction which consists of an amino alkylation of an acidic proton placed next to a carbonyl functional group by formaldehyde and a primary or secondary amine or ammonia. The final product is a β -amino-carbonyl compound also known as mannich base. Reactions between aldimines and α -methylene carbonyls are also considered mannich reactions because these imines form between amines and aldehydes. The reaction is named after chemist Carl Mannich. The mannich reaction is an organic reaction used to convert a primary or secondary amine and two carbonyl compounds (one non-enolizable and one enolizable) to a β -amino carbonyl compound, also known as a mannich base, using an acid or base catalyst. Mannich reaction is a carbon-carbon bond forming nucleophilic addition reaction and is a key step in synthesis of a wide variety of natural products, pharmaceuticals and so forth.

PROCEDURE:

10.6 gm benzaldehyde, 7.3 gm diethylamine and acetone 5.8 gm were taken in beaker (1:1:1).

Kept the reaction mixture under microwave for 1-2 min.

Poured the reaction mixture into a 125 ml of water.

Allowed to stand for 20 min, filtered and collect the product.

OBSERVATION:

Theoretical yield: 8 gm

Practical yield:

% yield:

Melting Point:

RESULT AND DISCUSSION:

.....

.....

.....

.....

.....

.....

VIVA QUESTIONS

Q.-1. What is mannich base ?

.....

.....

.....

.....

Q.-2. Give the reaction mechanism of mannich base synthesis ?

.....

.....

.....

.....

Q.-3. What are the uses of mannich base ?

.....

.....

.....

.....

Q.-4. What are the advantage of microwave mediate synthesis ?

.....

.....

.....

.....

Q.-5. What are the temperature and time use for the synthesis of mannich base in microwave ?

.....

.....

.....

.....

Experiment No. – 10

OBJECT:

To synthesize Schiff's base.

APPARATUS AND CHEMICAL REQUIREMENT:

Aniline, Methanol, Benzaldehyde, Zinc chloride, Round bottom flask, Beaker, Funnel, Filter paper, Condenser, Heating mantel, Glass rod, Hot air oven

THEORY:

Schiff bases can be synthesized from an aliphatic or aromatic amine and the carbonyl compound by the nucleophilic addition reaction. The Schiff bases are common ligands in coordination chemistry. The imine nitrogen is basic and exhibits pi-acceptor properties. The ligands are typically derived from alkyl diamines and aromatic aldehydes.

The term schiff base is normally applied to these compounds when they are being used as ligands to form coordination complexes with metal ions. Such complexes do occur naturally but the majority of schiff bases are artificial and used to form many important catalysts. Schiff bases are common enzymatic intermediates where an amine, such as the terminal group of a lysine residue reversibly reacts with an aldehyde or ketone of a cofactor or substrate.

PROCEDURE:

1. Dissolve 9.3 gm aniline in 30 ml methanol.
2. Benzaldehyde 10.6 gm(0.1 mol) and zinc chloride (1 gm) was added to it.
3. The mixture was refluxed for 2 hr.
4. Volume of methanol was reduced to half by distillation under reduced pressure.
5. The resulting solution was poured on crushed ice.
6. White precipitate obtained was separated by filtration, dried and recrystallized by ethanol.

OBSERVATION:

Theoretical yield: 8.4 gm

Practical yield:

% yield:

Melting Point:

RESULT AND DISCUSSION:

.....

.....

.....,

.....

.....,

.....

VIVA QUESTIONS

Q.-1. What is Schiff's base ?

.....

.....

.....

.....

Q.-2. What are the uses of Schiff's base ?

.....

.....

.....

.....

Q.-3. What are the principal involve in synthesis of Schiff's base ?

.....

.....

.....

.....

Q.-4. What is the use of methanol and zinc chloride in the synthesis Schiff's base ?

.....

.....

.....

.....

Q.-5. Give the example of different amine and aldehyde which are also use for the synthesis of Schiff's base ?

.....

.....

.....

.....

Experiment No. – 11

OBJECT:

To determine solubility of given compound.

APPARATUS AND CHEMICAL REQUIREMENT:

THEORY:

Solubility is the property of a solid, liquid, or gaseous chemical substance called solute to dissolve in a solid, liquid, or gaseous solvent. The solubility of a substance fundamentally depends on the physical and chemical properties of the solute and solvent as well as on temperature, pressure and the pH of the solution. The extent of the solubility of a substance in a specific solvent is measured as the saturation concentration, where adding more solute does not increase the concentration of the solution and begins to precipitate the excess amount of solute. The solubility of a substance is an entirely different property from the rate of solution. At the molecular level, solubility is controlled by the energy balance of intermolecular forces between solute-solute, solvent-solvent and solute-solvent molecules. Recall from general chemistry that intermolecular forces come in different strengths ranging from very weak induced dipole – induced dipole interactions to much stronger dipole-dipole forces (including the important special case, hydrogen bonding). However there is a simple, very useful and practical empirical rule that is quite reliable. That simple rule is “like dissolves like” and it is based on the polarity of the systems i.e. polar molecules dissolve in polar solvents (e.g. water, alcohols) and non-polar molecules in non-polar solvents (e.g. the hydrocarbon hexane). This is why ionic compounds like table salt (sodium chloride) or compounds like sugar, dissolve in water but do not dissolve to any great extent in most organic solvents. It also applies to the separation of oil and water (e.g. in salad dressings). The polarity of organic molecules is determined by the presence of polar bonds due to electronegative atoms (e.g. N, O) in polar functional groups such as amines (-NH_2) and alcohols (-OH).

PROCEDURE:

Take small (approximately 10 mg of a solid) amounts of material to use for determine a solubility test compound.

Add 1-2 ml of a solvent.

Shake the tube or stir with a glass stirring rod.

Make the soluble a homogeneous solution.

Add additional solvent, up to 1 ml, if compound does not completely dissolve with the smaller amount.

OBSERVATION:

Solubility profile:

S No	Solvent	Solubility
1	Water	
2	Ethanol	
3	Acetone	
4	Chloroform	

RESULT AND DISCUSSION:

.....

.....

.....

.....

.....

.....

VIVA QUESTIONS

Q.-1. What is solubility ?

.....

.....

.....

.....

Q.-2. What is the co-solvent ?

.....

.....

.....

.....

Q.-3. What are the different types solvents use for solubility determine ?

.....

.....

.....

.....

Q.-4. What is the importance of solubility ?

.....

.....

.....

.....

Q.-5. What is the effect of temperature on solubility ?

.....

.....

.....

.....