A Dissertation thesis entitled

"TO STUDY THE SYNTHESIS OF SCHIFF-BASE DERIVATIVES"

Submitted in partial fulfilment of the requirements

For the award of the degree of

s

Master of Science

IN

INDUSTRIAL CHEMISTRY

Submitted By

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FACULTY OF SCIENCE (FOS)

DEPARTMENT OF INDUSTRIAL CHEMISTRY

ATMIYA UNIVERSITY

RAJKOT-360 005, GUJARAT, INDIA.

2022-2023

ATMIYA UNIVERSITY
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CERTIFICATE

This is to certify that the dissertation thesis entitled **"To Study the Synthesis of Schiff Base Derivatives"** is submitted by **Mr. Ramani Haresh Ashokbhai (Enroll. No: 210722037),** A Post Graduate student of Semester-IV, Department of Industrial Chemistry, Faculty of Science, Atmiya University, Rajkot,

He has undertaken and conducted this dissertation work as a part of the curriculum to earn credits for obtaining the degree of **Master of Science (M.Sc.)** in **Industrial Chemistry** during the **academic year 2022-2023**.

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Faculty of Science	Faculty of Science	Atmiya University
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Date: 14.04.2023
Place: Rajkot

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Dedicated to

My Beloved Family

Without their love, support and constant Encouragement,
This would not have been possible

DECLARATION

We undersigned, hereby declare that the work assimilated in the dissertation thesis entitled **"To Study on the synthesis of Schiff base Derivatives"** has been carried out by us at the Faculty of Science, Department of Industrial Chemistry, ATMIYA University, Rajkot, Gujarat, India, under the supervision and guidance of **Dr. Govind Vagadiya, Faculty of Science, Department of Industrial Chemistry, ATMIYA University, Rajkot, Gujarat, India.**

To the best of our knowledge and belief, the work included in this thesis is quite original and has not been submitted to any other Institution or University for the award of any degree either in this or any other form.

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We wish to express our sincere gratitude and honor to our Research supervisor **Dr. Govind Vagadiya, Assistant Professor, Faculty of Science, Department of Industrial Chemistry, Atmiya University, Rajkot,** for their inspiring, splendid, and authentic guidance, moral support, and constant encouragement throughout our research work. Their passion and dedication towards research have stimulated, provoked, and facilitated us to complete this endeavor. We could not have imagined having a better Research Supervisor and mentor for our M. Sc study. Their role will always remain fundamental in shaping our future.

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Contents:

1. ABSTRACT

Schiff base is a condensation product of primary amine and carbonyl compounds for gain the importance day by day in the present scenario. Schiff base is the compound containing azomethine or amine (-C=N-) functional group and it is found to be an inconstant pharmocophore for the design and development of some bioactive compounds. Schiff base are some of the most widely used organic compounds. They are used as pigments and dyes, catalysts, intermediates in organic synthesis, and as polymer stabilizer. Schiff base have also been shown to exhibit a broad range of biological activities, including antifungal, antibacterial, ant malarial, ant proliferative properties. Imines or azomethine groups are present in various natural, natural-derived, and non-natural compounds. The amine groups present in such compounds has been shown to be critical to their biological activities.

In this review we present the general approaches to the synthesis of Schiff base. We also highlight the most significant example of compounds belonging to this class, which exhibit anti-malarial, antibacterial, antifungal and antiviral activities to have been reported in the literature. The relationship between Schiff base and other pharmacological activities, such as ant proliferative activities, are not included in this review.

Keywords:

Carbonyl compounds, Schiff-base, Imines or azomethine, anti-conversant, anthelmintic, anti-microbial, anti-hypertensive. SDG-2030-Goal-3: Good Health & Wellbeing

2. LIST OF SYMBOLS, ABBREVIATIONS AND NOMENCLATURES

SN	Symbols/Abbreviations/ Nomenclature	Meaning
1	AR	Aryl
2	Ph	Phenyl
3	Me	Methyl
4	IR	Infrared
5	C=O	Carbonyl
6	C=N	Imine
7	Et	Ethyl
8	R-NO_2	Nitro compound
9	R-CHO	Aldehyde
10	R-OH	Hydroxyl
11	R-NH_2	Primary amine
12	R-COOH	Carboxyl
13	R-$CONH_2$	Amide
14	Me	Methyl

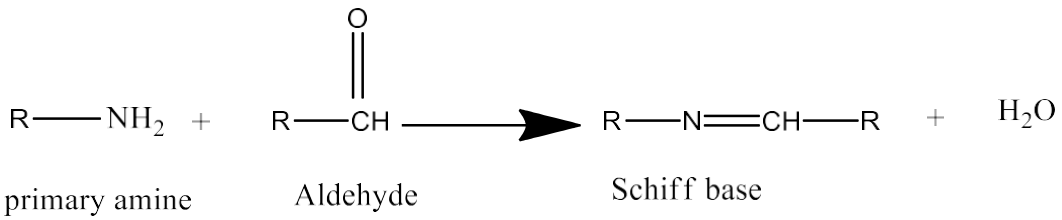
3. INTRODUCTION

3.1 SCHIFF BASE AND THEIR CHEMISTRY

Compounds containing an azomethine group (-CH-N-) are known as Schiff bases.

They are usually formed by condensation of a primary amine with a carbonyl compound

According to the following scheme:



Where R may be an aliphatic or an aromatic group. Schiff base of aliphatic aldehydes are relatively unstable and are readily polymerizable While those of aromatic aldehydes having an effective conjugation system, are more stable condensation of amines it aldehydes and ketones have numerous applications which include preparation use identification, detection and determination of aldehydes or ketones, purification of carbonyl or amino compounds or protection of these groups during complex or sensitive reaction.

An amino group is found in simple amines and Schiff base obtained from aromatic amines are known as anvils. Schiff base are generally bi- or tri-dentate lagends capable of forming very stable complex with transition metals. In chemistry , Schiff base find a versatile use ; some of them are the basic units in Schiff base appear to be important intermediates in a numbers of enzymatic reaction involvingof an enzyme with an amino or carbonyl group of the substrate. One of the most prevalent types of catalytic mechanism in biochemical processes involves condensation of a primary amine in an enzyme, usually that of a lysine reside, with carbonyl group of the substrate to from an imines or Schiff base.

Stereo chemical investigation carried out with the aid of molecular modes showed that Schiff base formed between in ethlglyoxal and the amino groups of the lysine side chains of proteins can bend

Back in such way towards the N atoms of peptide groups that a charge can occur between these groups and the oxygen atoms of the Schiff base. In this respect. Pyridoxal Schiff base deride from amino acids have been prepared and studied. Schiff base derived from pyridoxal and amino acids are considered very important legends from biological point of view. Transition metal complexes of such legends are important enzyme models. The rapid development of these legends resulted in an enhanced research activity in the field of coordination chemistry leading to very interesting conclusions certain dyes, whereas some are used as liquids crystals. In organic synthesis, Schiff base reaction are useful in making carbon-nitrogen bonds.

3.2 BIOLOGICAL IMPORTANCE OF SCHIFF BASE

Schiff to be important intermediates in several enzymatic reactions involving interaction of an enzyme with an amino or a carbonyl group of the substrate. One of the most prevalent types of catalytic mechanisms in biochemical processes involves condensation of a primary amine in an enzyme, usually that of a lysine residue, with a carbonyl group of substrate to form an amine, or Schiff base.

The biosynthesis of porphyry, for which glycine is a precursor, is another important pathway, which involves the intermediate formation of Schiff base between Kato group of one molecule of δ-amino levlinic acid and ε-amino group of lysine residue of an enzyme.

3.3 INFRARED SPECTROPHOTOMETER

Infrared spectroscopy is an extremely powerful analytical technique for both qualitative and quantitative analysis. The infra-red spectrum of any given substance is interpreted by the use of known group frequencies. It is one of the most widely used tools for the detection of functional group in pure compounds and mixtures, for compound comparison and for the identification of the substances.

Infrared spectroscopy involves twisting, bending, rotational and vibration motion of the atomic groups in a molecule, infrared rendition does not possess enough energy to cause the excitation of elicitation of electrons however it causes atoms and groups of atoms to vibrate faster about covalent bond or bond which connect them. The compounds absorb infrared energy in the particular region of spectrum. A highly complex absorption spectrum is obtained which is characteristic of the functional group comprising the molecule and overall configuration of the atom as well.

The infrared region constitutes three part.

1. Near infrared region

   In the near infrared region, which meets the visible region at about 12,500 cm^{-1} and extends to about 4000 cm^{-1} are found many absorption based resulting from harmonicovertones of fundamental bands and combination bands often associated with hydrogen atoms. Among these are the first overtones of the O-H and N-H stretching vibrations near 7140 cm^{-1} and 6667 cm^{-1}, respectively, combination bands resulting from C-H stretching, and deformation vibration of alkyl groups at 4548 cm^{-1} and 3850cm^{-1}.

2. Mid infrared region

   Middle infrared region is detected into the "Groups frequency" region, 4000-1300 cm^{-1} and the "finger print" region. 1300-650 cm^{-1}.

3. Far infrared region

   Far infrared region between 667 cm^{-1} and 10 cm^{-1} and 10 cm^{-1} contain the bending vibrations of carbon, nitrogen, oxygen and fluorine with atoms heavier than mass 19 and addition bending motion in cyclic or unsaturated systems.

   The infrared spectrum can give a perfect picture of the structure formula without a chemical investigation Schiff base appear to important intermediates in several enzymatic reactions involving interaction of an enzyme with an amino or a carbonyl groups of the substrate. One of the most prevalent types of catalytic mechanisms in biochemical processes involves condensation of a primary amine in an enzyme, usually that of a lysine residue, with a carbonyl group of the substrate to form an imines, or Schiff base.

   Stereo chemical instigations carried out with the aid of molecular models showed that Schiff base formed between Ethyl glyoxalin and the amino group of the lysine side chains of proteins can bend back in such a way towards the N atom of peptide groups that s charge transfer can occur between these groups and the oxygen atom of the Schiff base. In this respect, pyridoxal Schiff base derived from amino acids have been prepared and studied. Schiff base derived from pyridoxal point of view. Transition metal complexes of such legends are important enzyme models. The rapid development of these legends resulted in an enhanced research activity in the field of coordination chemistry leading to very interesting conclusions.

4. LITERATURE REVIEW

1. Synthesis of some new thiazole derivatives and their biological activity evaluation.

   Author name: Leyla Yorta's, Yusuf Özkay,HülyaKaracaGençer,andUlviyeAcar

   Vol. no.:-https://doi.org/10.1155/2015/464379

2. Synthesis and antibacterial activity of some new thiazole and thiophen derivatives.

   Author name:-A.M.Khalil, M.A.Berghot,M.A.Gouda
   Vol.no.:- https://doi.org/10.1016/j.ejmech.2009.06.002

3. Review of the synthesis and biological activity of thiazoles.

   Authorname:-SukinahH.Ali,AbdelwaheDRSayed
   Vol.no.-https://doi.org/10.1080/00397911.2020.1854787

4. Schiff base of short review of their antimicrobial activitie.

   Author name:-cleitonM.da silva, Daniel L. da silva

   Vol. no.doi:10.1016/j.jare.2010.05.004

5. Synthesis, spectroscopic, cytotoxic aspects and computational study of N-(pyridine-2-ylmethylene)benzo[d]thiazol-2-amine Schiff base and some of its transition metal complexes.

   Author name:-Dina M. Abd El-Aziz ⇑, Safa Eldin H. Etaiw⇑ , Elham A. Ali

   Vol. no.Doi:10.1016/j.molstruc.2013.05.051

6. Schiff Bases: A Short Survey on an Evergreen Chemistry Tool.

   Author name:-Wenling Qin 1 , Sha Long 1 , Mauro Panunzio 2,* and Stefano Biondi 3,*

   Vol.no. doi:10.3390/molecules181012264

7. Synthesis, Characterization, and Antifungal Activity of Schiff Bases of Inulin Bearing Pyridine ring.

   Author name:-Lijie Wei 1,2, Wenqiang Tan 1 , Jingjing Zhang 1,2, Yingqi Mi 1,2, Fang Dong 1 , Qing Li 1,* and Zhanyong Guo 1,2,*

   Vol. no. DOI: 10.3390/POLYM11020371

8. Synthesis, Characterization, Antioxident And Anticancer HumanStudies Of New Metal Ion Complexes Of Poly Schiff Base Derived From 4-Aminoacetophenone With Salicylaldehyde And 4-Bromoaniline.

   Author name:-Tamara Q.M Al-Sahlanee*, Mohammed Ha Al-Amery

   Vol. no. DOI: 10.22159/ajpcr.2018.v11i11.29837

5. OBJECTIVES

- To synthesize and characterize the Schiff base.
- To observe anti-microbial properties of Schiff base.

PART: A

RAW MATERIAL, SYNTHESIS & CHARACTERIZATION OF

(N-BENZYLIDENEANILINE)

6. MATERIALS & METHODS (N-BENZYLIDENEANILINE)

- The Schiff base was synthesis by adding Benzaldehyde (10.0 ml) and Aniline (9.0ml) in MeOH.

- The reaction mixture was heated to reflux for 4-6 hrs.

- The product obtained was filtered off and was several times with small amount of MeOH then other the product was kept desiccators until used re-crystallization was carried out using EtOH.

- The progress of reaction was monitored by TLC.

- Physical data:-

Chemical formula: - $C_{13}H_{11}N$
Solubility : - Methanol
Color : - pale yellow
M.P :- 52-54 °C
M.W :- 181gm/mol
%Yield : - 82.40%

❖ **RAW MATERIAL :-**

1) Benzaldehyde = (10.0ml)
2) Aniline = (9.0ml)
3) MeOH
4) EtOH

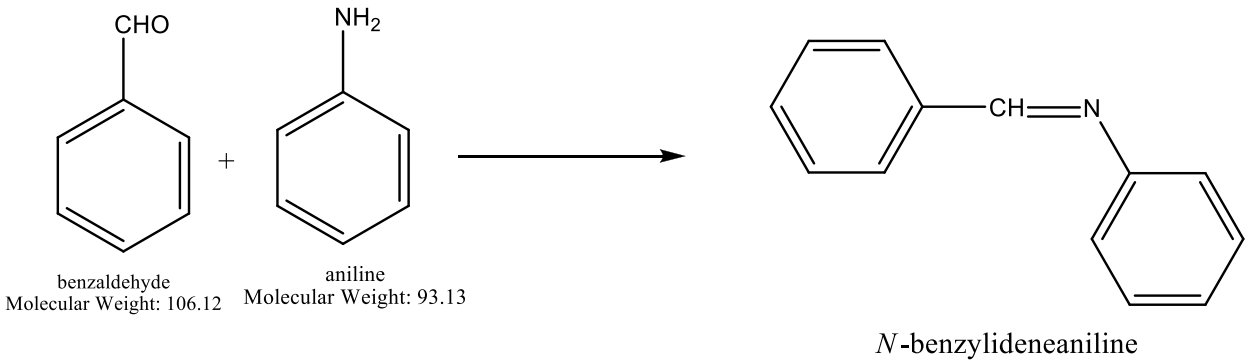
❖ TLC:-

- TLC PLATE:- Silica gel G-245
- TLC SOLENT SYSTE:-
- ETHYL ACETO ACETATE:- HEXANE (3 : 7)

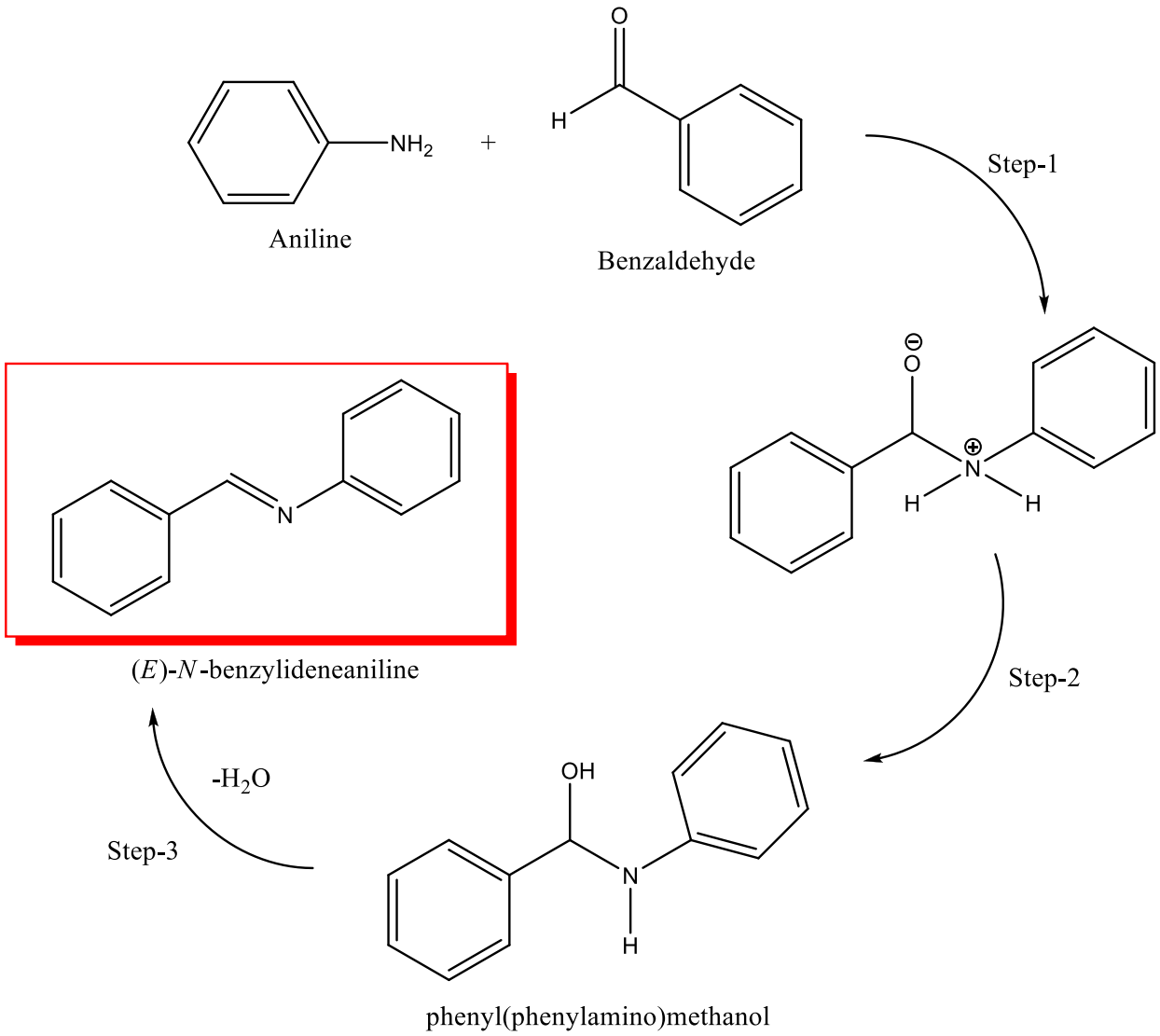
(1)Rf = 4.5/5.5
= 0.81

(2)Rf = 2.6/5.5
= 0.47

➢ Reaction:-



➢ Mechanism :-



❖ CALCULATION:-

- Theoretical yield:-

93gm Aniline = **181gm** N-benzylideneaniline

9.19gm Aniline = **(?gm)** N-benzylideneaniline

$$\text{Theoretical yield} = \frac{9.198 \times 181}{93}$$

$$= \mathbf{17.90gm}$$

- Practical yield = **14.75gm**

- % Yield $= \frac{14.75 \times 100}{17.90}$

$$= \mathbf{82.40\%}$$

PART - B

RAW MATERIAL, SYNTHESIS & CHERACTERIZATION OF

(1-BENZYLIDENE 2-PHENYLHYDRAZINE)

7. PROCESS OF SYNTHESIS:

(1-BENZYLIDENE-2-PHENYLHYDRAZINE)

- The Schiff base was synthesized by adding Benzaldehyde (10ml) dissolved in hot MeOH to PhenylHydrazine(5.75ml) in MeOH.

- The reaction mixture was heated to reflux for 6-8 hrs.

- The product obtained was filtered off and was several times with a small amount of MeOH then other the product was kept in desiccators until used recrystallization was carried out using EtOH.

- **Physical data:-**

Chemical formula: - $C_{13}H_{12}N_2$

Solubility : - Methanol

Color : - Light orange

M.P :- 156 °C

M.W :- 196.25gm/mol

%Yield : - 80.84%

❖ RAW MATERIAL :-

1) Benzaldehyde :- (10ml)
2) PhenylHydrazine :- (5.75ml)
3) MeOH
4) EtOH

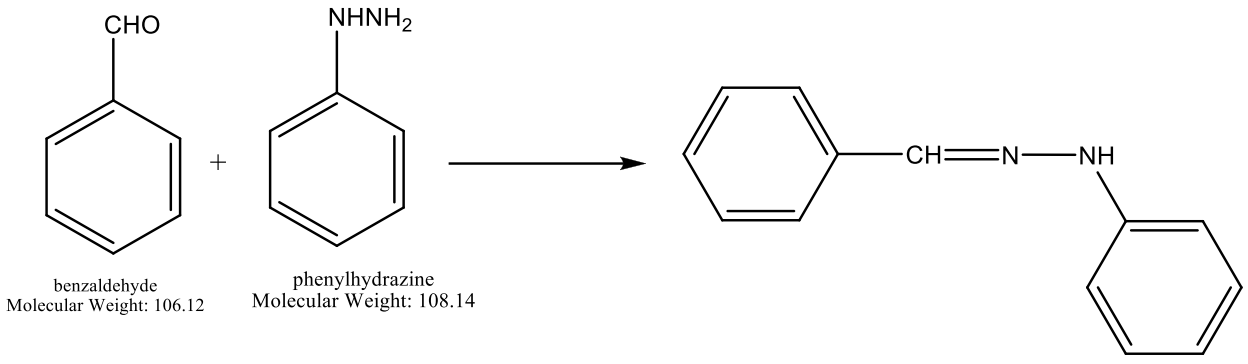
❖ TLC :-

- TLC PLATE :- SILICA GEL G-254
- TLC SOLVENT SYSTEM :-
- ETHYL ACETO ACETATE : HEXANE (3: 7)

(1) RF = 2.9/4.8
= 0.60

(2) RF = 3.5/4.8
= 0.72

❖ Reaction:-

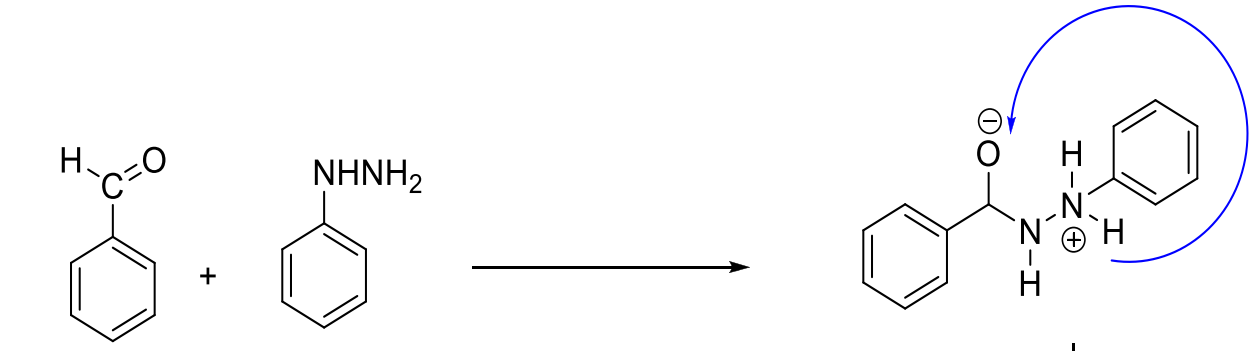


benzaldehyde
Molecular Weight: 106.12

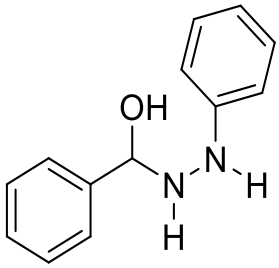
phenylhydrazine
Molecular Weight: 108.14

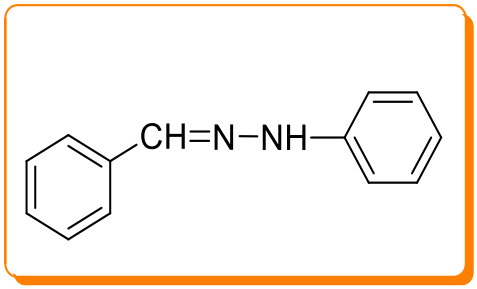
1-benzylidene-2-phenylhydrazine

➢ Mechanism :-



Benzaldehyde Phenylhydrazine





$-H_2O$

1-benzylidene-2-phenylhydrazine

❖ CALCULATION :-

- Theoretical yield :-

108.14gm Phenylhydrazine = 196.25gm 1-benzylidene-2-phenylhydrazine

6.325gm Phenylhydrazine = **(? gm)** 1-benzylidene-2-phenylhydrazine

$$\text{Theoretical yield} = \frac{196.25 \times 6.32}{108.14}$$

$$= \mathbf{11.47\ gm}$$

- Practical yield = **9.25gm**
- % Yield $= \frac{9.25 \times 100}{11.47}$

$$= \mathbf{80.84\ \%}$$

PART - C

RAW MATERIAL, SYNTHESIS & CHERACTERIZATION OF

(3-PHENYLALLYLIDEN ANILINE)

8. PROCESS OF SYNTHESIS (3-PHENYLALLYIDEN ANILINE)

- The Schiff base was synthesis by adding Cinnamaldehyde (10.91ml) dissolved in MeOH to aniline(9.13ml)in MeoH.

- The reaction mixture was heated to refax for 3-Phenylallyliden aniline.

- The product obtained was filtered off and wash several times small amount of MeoH then other the product was kept desiccators until used recrystallization was carried out using EtoH.

- The product of reaction was monitored by TLC.

- Physical data:-

  Name: 3-Phenylallyliden aniline
  Formula: $C_{17}H_{17}N$
  Molar Mass: 172.02gm/mol
  Boling Point: 21.7 C^{o}
  Density: 1.5gm/mol
  Solubility: Methenol

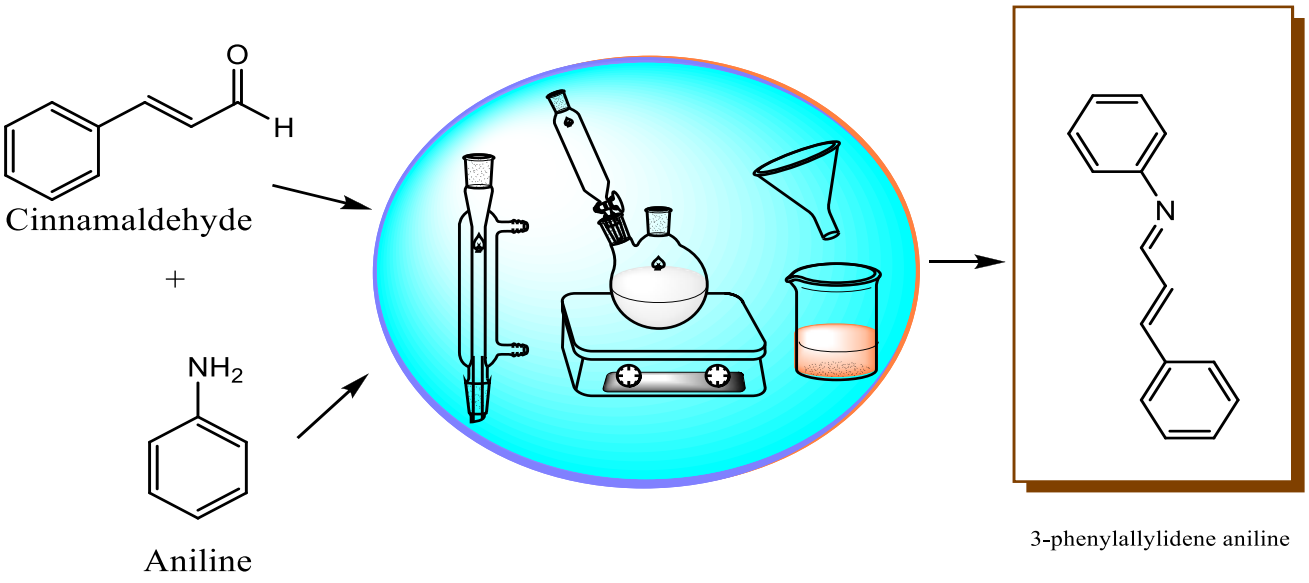
- TLC :-

  - TLC PLATE :- SILICA GEL G-254
  - TLC SOLVENT SYSTEM :-
  - ETHYL ACETO ACETATE : HEXANE (3: 7)

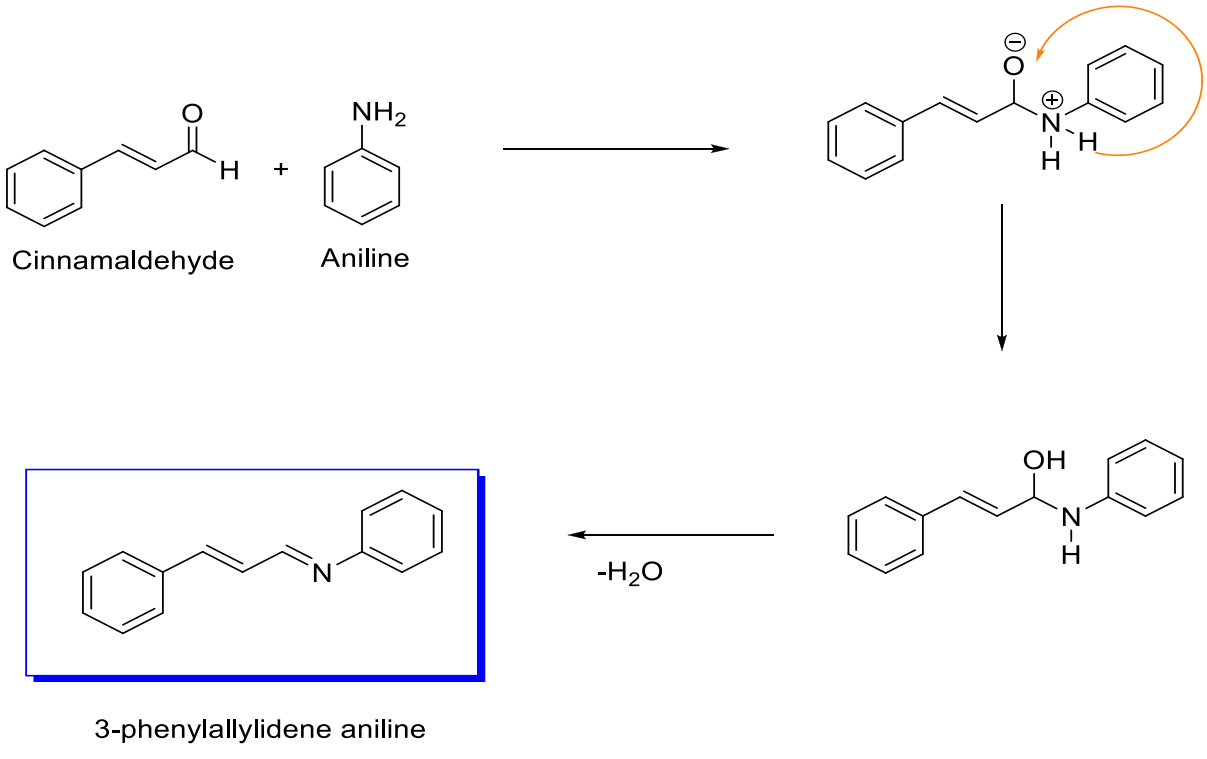
(1) RF = 3.5/5
= 0.70

(2) RF = 3.9/5
= 0.78

❖ **Reaction:-**



➢ **Mechanism:-**



❖ CALCULATION:-

➢ Theoretical yield:-

93gm aniline = **207.27**gm (3-phenylallyiden aniline)

7.50gm aniline = **(?)** gm (3-phenylallyiden aniline)

➢ Theoretical yield = $((207.27 \times 7.50)/93)$

$= \mathbf{16.71\ gm}$

➢ Practical yield = **13.67 gm**

➢ % yield $= \frac{13.67 \times 100}{16.71}$

= **81.80%**

PART- D

RAW MATERIAL, SYNTHESIS & CHERACTERIZATION OF

(3-PHENYL-2-STYRYLTHIAZOLIDIN-4-ONE)

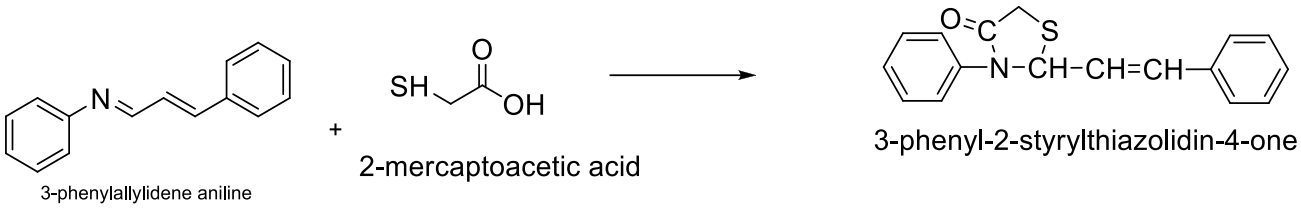
9. PROCESS OF SYNTHESIS:

(3-PHENYL-2- STYRYLTHIAZOLIDIN-4-ONE)

- The Schiff base was synthesis by adding 3-phenylallyliden aniline and adding MeOH to 2- Mercaptoacetic acid.
- The Product obtained was filtered off and wash several times with small amount of MeOH then The product was kept desiccation until used recrystallization was carried out.
- The progress of reaction was monitored by TLC.
- Physical data:

Name: 3-phenyl-2-styrylthiazolidin-4-one
Formula: $C_{16}H_{15}NSO$
Molar Mass: 269.4 gm/mol
Boiling Point: 132 to 135 C^{O}
Density: 1.05 gm/mol
Solubility: Methenol

❖ Reaction :-



❖ calculation :-

- Theoretical yield:-

  207.27gm 3-phenylallylidene aniline = **269.4gm** 3-phenyl-2-styrylthiazolidin-4-one

  9.5gm 3-phenylallylidene aniline = **(?gm)** 3-phenyl-2-styrylthiazolidin-4-one

- Theoretical yield = $\frac{9.5 \times 269.4}{207.27}$

  = **12.34gm**

- Practical yield = **10.27gm**

- % Yield = $\frac{10.27 \times 100}{12.34}$

  = **83.22%**

PART – E

RAW MATERAL, SYNTHESIS & CHERACTRIZATION OF

(2, 3 – DIPHENYLTHIAZOLIDIN – 4 – ONE)

10. PROCESS OF SYNTHESIS:

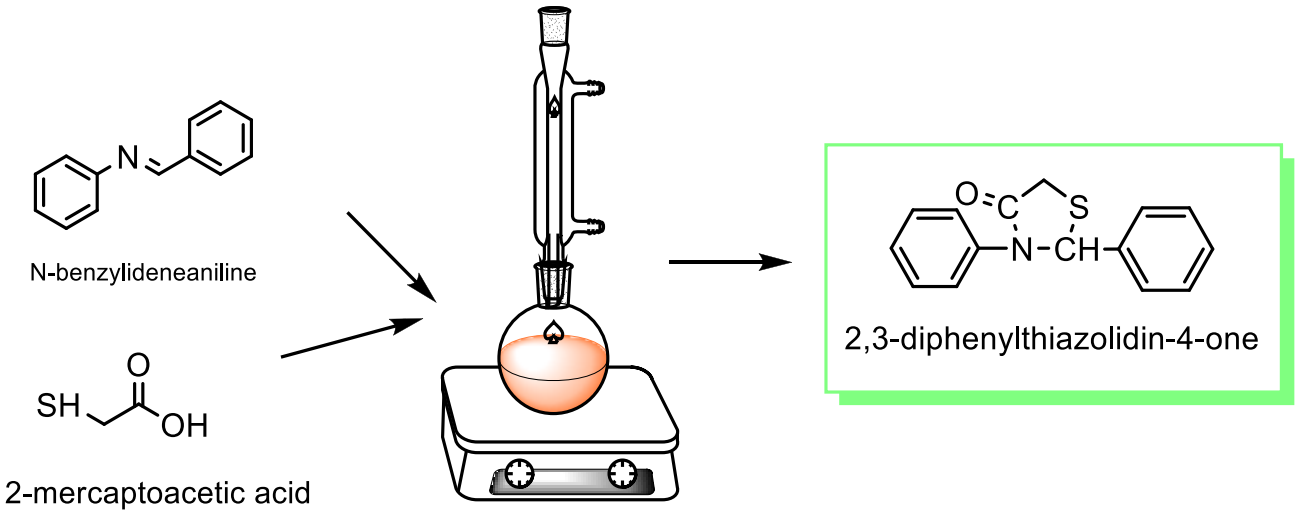
(2, 3 DIPHENYLTHIOZOLIDIN 4-ONE)

- The Schiff base was synthesis by adding N- benzylideneaniline and adding MeOH to 2- mercaptoacetic acid.
- The Product obtained was filtered off and wash several times with small amount of MeOH. The product was kept desiccation until used recrystallization was carried using out.
- The progress of reaction was monitored by TLC.

- **Physical data:-**

Name: 2, 3- diphenylthiazolidin -4-one
Formula: $C_{15}H_{13}NOS$
Molar Mass: 255.3 gm/mol
Boiling Point: 131 to 133C^{o}
Density: 1.04gm/mol
Solubility: methanol

❖ Reaction:-



❖ CALCULATION :-

- Theoretical yield:-

181gm N-benzylideneaniline = **255.3gm** 2, 3-diphenylthiazolidin-4-one

9.5gm N-benzylideneaniline = **(?gm)** 2, 3-diphenylthiazolidin-4-one

Theoretical yield = $\frac{9.5\times255.3}{181}$

=**13.29gm**

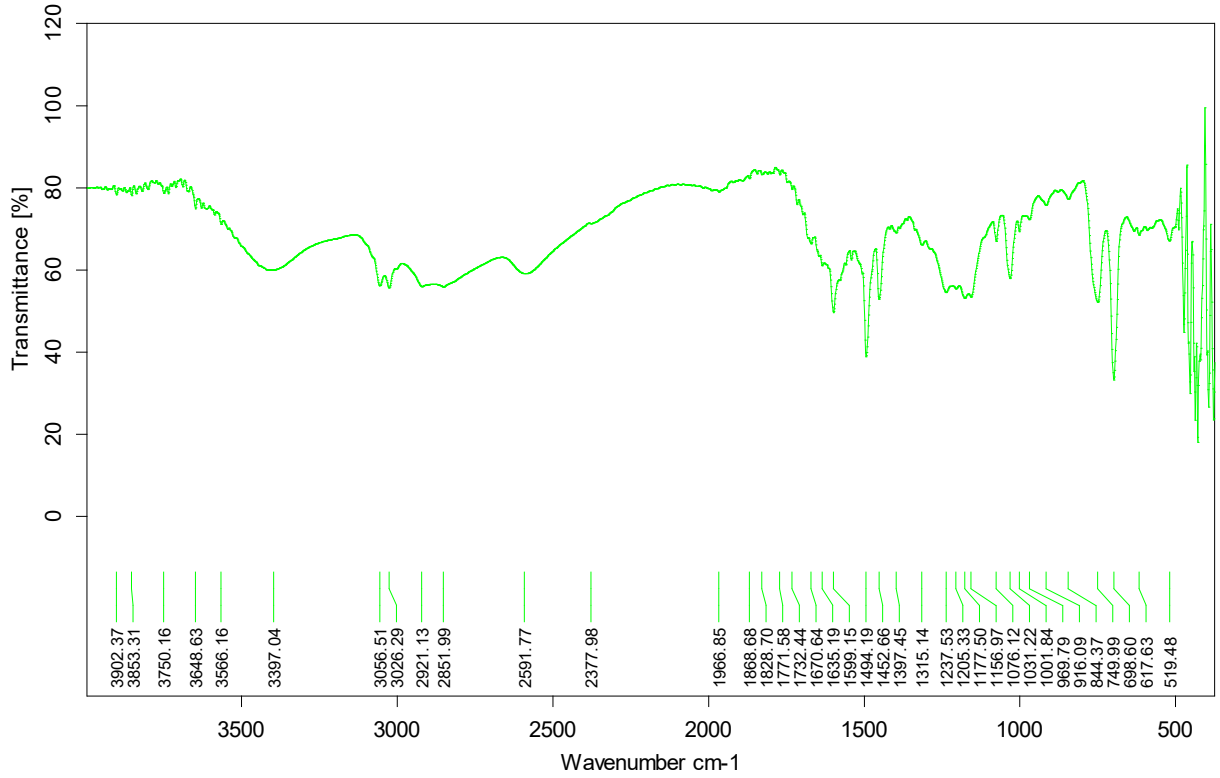
- Practical yield = **10.24gm**

- % Yield = $\frac{10.24\times100}{13.29}$

= **77.05%**

11. RESULT AND DISCUSSION

- Schiff base has been characterized by Infrared (IR) method and their result are as discussed below.



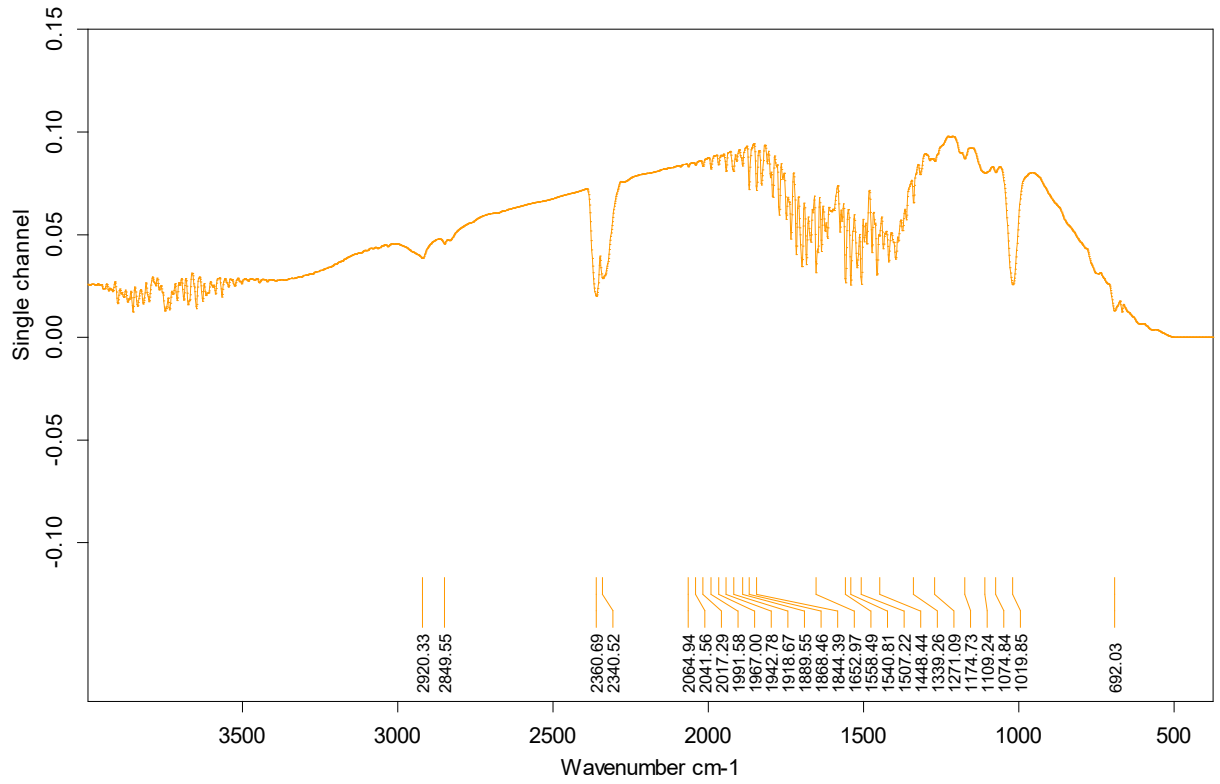
Graph 1: IR of 3-Phenylallyidene Aniline

- Above mentioned graph is the reaction of Cinnamaldehyde with Aniline which are sustain base. Hare, various bends can be seen which can be identified by the below table.

Table 1: IR data of 3-phenylallyidene aniline

Vibration Mode	Reported Frequency (cm^{-1})	Observed Frequency (cm^{-1})
C=C	1585-1480	1541.41
C-H	3060-3020	3026.29
C=N	1600-1570	1590.17
C-CH_2	3040-3010	-

➢ **Graph:**



Graph 2: IR of 3-phenyl-2-styrylthiazolidin-4-one

➢ Above mentioned graph is the reaction of 3-phenylallylidene aniline with 2- mercaptoacetic acid Which are sustain base. Hare, various bends can be seen which can be identified by the below table.

Table 2: IR data of 3-phenyl-2-styrylthiazolidin-4-one

Vibration Mode	Reported Frequency (cm^{-1})	Observed Frequency (cm^{-1})
C=C	1585-1480	1540.81
C-H	3060-3020	3040.65
C-N	1250-1350	1339.26
C=O	1675-1750	1693.51
C-S	600-700	692.03

12. CONCLUSION

In conclusion, Schiff bases have proven to be a versatile class of organic compounds with a wide range of applications in coordination chemistry, catalysis, sensing, medicinal chemistry, and more. Their ability to coordinate to metal ions and their tunable properties make them useful for a variety of purposes. As research in this field continues to progress, it is likely that even more applications of Schiff bases will be discovered. Overall, the study of Schiff base chemistry is an exciting and rapidly evolving field that holds great promise for the future.

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