

## SYNTHESIS AND CHARACTERIZATIONS OF SOME NOVEL SCHIFF BASE DERIVED FROM DIBENZALACETONE

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### ABSTRACT

Dibenzalacetone reacts with Various aromatic Amine, .Finally the product were characterized by conventional and instrument methods.

**KEYWORDS:** Novel Schiff base, Dibenzal acetone, Amine.

### INTRODUCTION

Azomethines are generally known as Schiff bases to honour Hugo Schiff, who synthesized such compounds. These are the compounds containing characteristic  $-C=N-$  group. Several methods have been reported for the preparation of azomethines. Selvam *et.al*<sup>[1]</sup> have prepared sulfonamide and its derivatives as anti-HIV agents. More *et. al*<sup>[2]</sup> have marked the biological activity of Schiff bases synthesized from aminothiazoles. Ernst Bayer<sup>[3]</sup> has reported some metallocomplex Schiff bases derived from *o*-amino phenol. Schiff bases can be synthesized from an aromatic amine and a carbonyl compound by nucleophilic addition forming a hemiaminal, followed by a dehydration to generate an imine<sup>[4]</sup>. They are well known intermediates for the preparation of azetidiones, thiazolidinones, oxadiazolines and many other derivatives. Azomethines exhibit a wide range of pharmacological activities like antimicrobial, antiparasitic, anti-inflammatory, anticancer<sup>[5-13]</sup> *etc.*

### MATERIALS AND METHODS

Melting point were recorded in open capillaries and on Veergo melting point apparatus. The <sup>1</sup>H NMR Spectra were recorded on a Bruker 300 MHz using TMS as an internal standard. The IR spectra were recorded on a Perkin Elmer Spectrum 100 FTIR Spectrophotometer and

the Mass Spectra on a Waters Micromass Q-fit instrument. The chemical used are of A.R. grade.

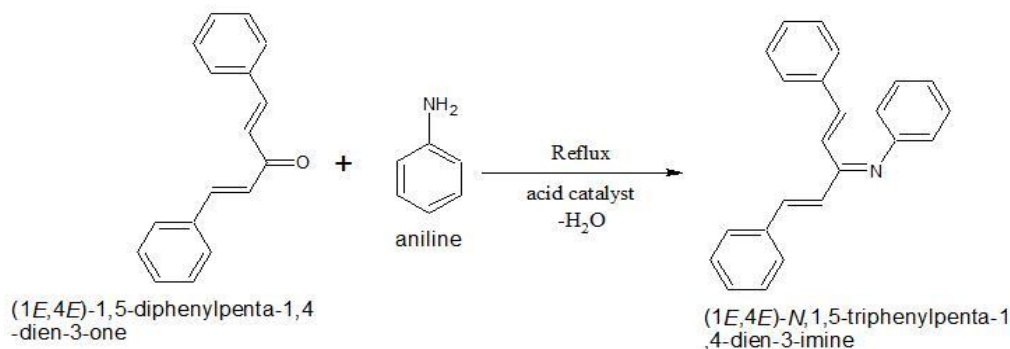
### Procedure for the synthesis of compound

#### Preparation of dibenzalacetone(1,5-diphenylpenta-1-4-dien-3-one)

In 250ml Erlenmeyer flask dissolve 7.5 gm. of sodium hydroxide in 75 ml DI water and 60 ml of ethanol. Maintain the temperature of the solution at 20-25 ° C. Add previously prepared mixture of 7.5 ml of Benzaldehyde and 3 ml Acetone shake well . A precipitate forms in 2-3 min. after 15 min add the remaining portion of the Benzaldehyde –Acetone mixture , shake for further 30 min now cool the Erelnmeyer flask and separate the solid precipitated was filtered, washed and recrystallized from hot Ethanol.

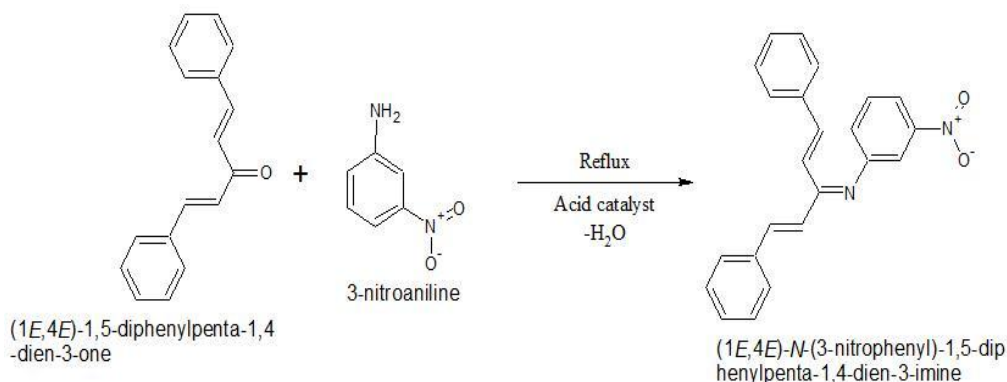
#### Preparation of (1E,4E)-N,1,5-triphenylpenta-1,4-dien-3-imine

A mixture of dibenzalacetone(2.34 g., 0.01mol.) and aromatic Amine, Aniline (0.913g., 0.01 mol.) was taken in absolute ethanol(20 ml.) and 1-2 drops of glacial acetic acid were added. Then the mixture was refluxed for 6-8 hours on water bath. The progress of the reaction was monitored by TLC. The excess solvent was distilled off and then remaining residue was poured in crushed ice. The separated solid was filtered, washed and recrystallized from ethanol. Their physical constant data given in Table 1.



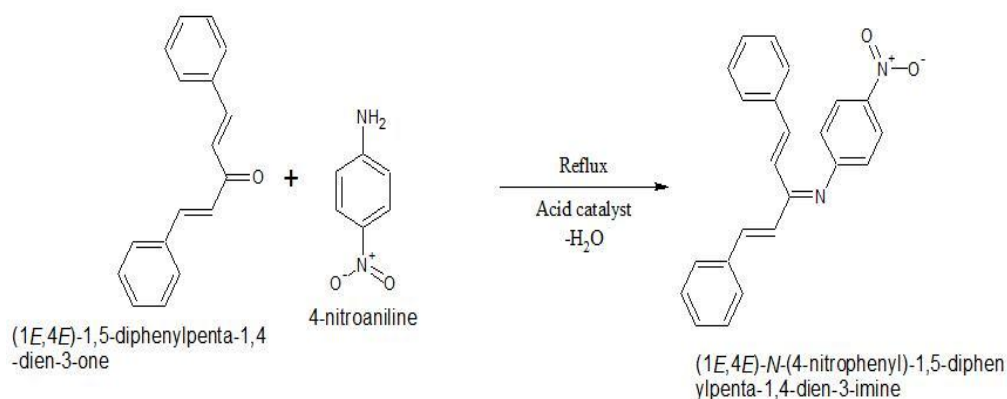
#### Preparation of (1E,4E)-N-(3-nitrophenyl)-1,5-diphenylpenta-1,4-dien-3-imine

A mixture of dibenzalacetone(2.34 g., 0.01mol.)and aromatic Amine, 3-nitro Aniline (1.38g., 0.01 mol.) was taken in absolute ethanol(20 ml.) and 1-2 drops of glacial acetic acid were added. Then the mixture was refluxed for 6-8 hours on water bath. The progress of the reaction was monitored by TLC. The excess solvent was distilled off and then remaining residue was poured in crushed ice. The separated solid was filtered, washed and recrystallized from ethanol. Their physical constant data given in Table 1.



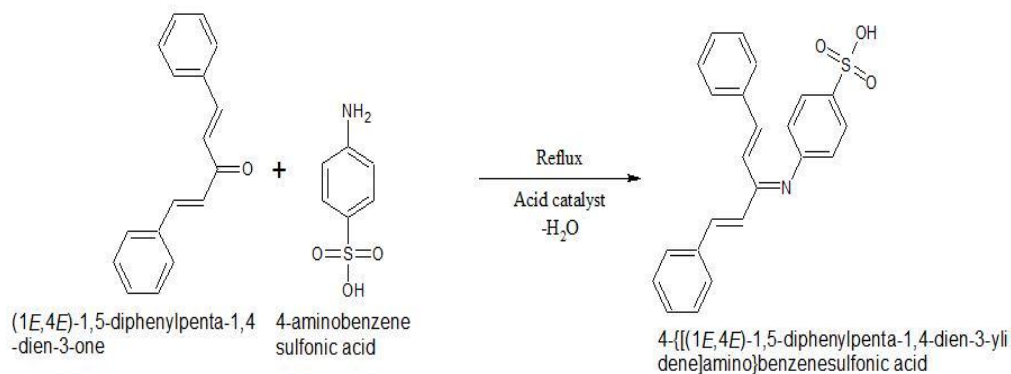
### Preparation of (1E,4E)-N-(4-nitrophenyl)-1,5-diphenylpenta-1,4-dien-3-imine

A mixture of dibenzalacetone (2.34 g., 0.01 mol.) and aromatic amine, 4-nitro aniline (1.38 g., 0.01 mol.) was taken in absolute ethanol (20 ml.) and 1-2 drops of glacial acetic acid were added. Then the mixture was refluxed for 6-8 hours on water bath. The progress of the reaction was monitored by TLC. The excess solvent was distilled off and then remaining residue was poured in crushed ice. The separated solid was filtered, washed and recrystallized from ethanol. Their physical constant data given in Table 1



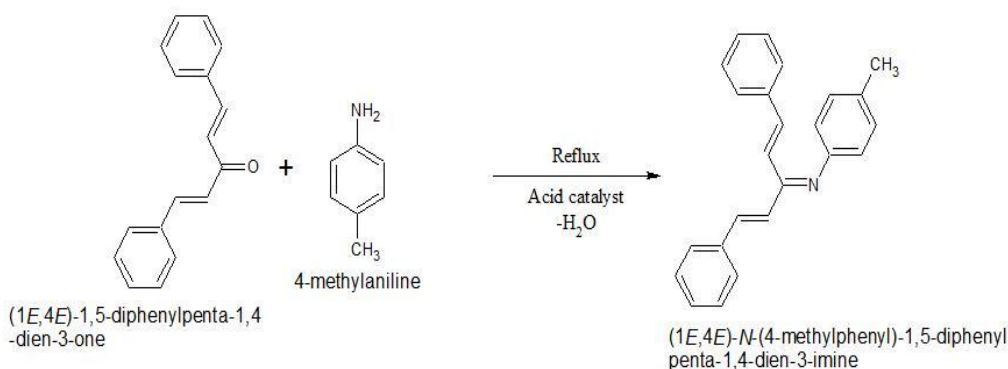
### Preparation of 4-[[1,5-diphenylpenta-1,4-dien-3-ylidene]amino]benzenesulfonic acid

A mixture of dibenzalacetone (2.34 g., 0.01 mol.) and aromatic amine, 4-aminobenzenesulfonic acid (1.73 g., 0.01 mol.) was taken in absolute ethanol (20 ml.) and 1-2 drops of glacial acetic acid were added. Then the mixture was refluxed for 6-8 hours on water bath. The progress of the reaction was monitored by TLC. The excess solvent was distilled off and then remaining residue was poured in crushed ice. The separated solid was filtered, washed and recrystallized from ethanol. Their physical constant data given in Table 1



### Preparation of (1E,4E)-N-(4-methylphenyl)-1,5-diphenylpenta 1,4-dien-3-imine

A mixture of dibenzalacetone (2.34 g., 0.01 mol.) and aromatic amine, 4-methylaniline (1.07 g., 0.01 mol.) was taken in absolute ethanol (20 ml.) and 1-2 drops of glacial acetic acid were added. Then the mixture was refluxed for 6-8 hours on water bath. The progress of the reaction was monitored by TLC. The excess solvent was distilled off and then remaining residue was poured in crushed ice. The separated solid was filtered, washed and recrystallized from ethanol. Their physical constant data given in Table 1.



## RESULT AND DISCUSSION

Table. 1

Comp. Name	Molecular formula	Molecular weight	M.P °C	Yield %	% of C	% of H	% of N
Kpp-1	C <sub>23</sub> H <sub>19</sub> N	309.40	150	35	89.28	6.19	4.53
Kpp-2	C <sub>23</sub> H <sub>18</sub> N <sub>2</sub> O <sub>2</sub>	354.40	141	28.8	77.95	5.12	7.90
Kpp-3	C <sub>23</sub> H <sub>18</sub> N <sub>2</sub> O <sub>2</sub>	354.40	88	82	77.95	5.12	7.90
Kpp-4	C <sub>23</sub> H <sub>18</sub> NO <sub>3</sub> S	389.46	99	53.5	70.93	4.92	3.60
Kpp-5	C <sub>24</sub> H <sub>21</sub> N	323.43	101	42	89.12	6.54	4.33

Various Schiff's base derivatives kpp-1-5 were prepared using of dibenzalacetone (1,5-diphenylpenta-1-4-dien-3-one) with aromatic amine in presence 1ml. of glacial acetic acid

gave Schiff base derivatives series kpp1-5. All the compounds synthesized were adequately characterized by their elemental analyses and spectral IR, <sup>1</sup>H-NMR and Mass Spectra.

#### **Spectra study of (1E,4E)-N,1,5-triphenylpenta-1,4-dien-3-imine**

IR(KBr. cm<sup>-1</sup>):1622 cm<sup>-1</sup>(C=N), 1235 cm<sup>-1</sup>(N=CH str), 1036 cm<sup>-1</sup>, 1010(CH=N),3420 cm<sup>-1</sup> (N-H),1438 cm<sup>-1</sup> (C=N, Ar), <sup>1</sup>H NMR(ppm) (CDCl<sub>3</sub>):5.56(s,1H,Ar-H),6.66-6.68(m,4H,Ar-H),2.59(s,3H,CH<sub>3</sub>), MS:310.40[M+1].

#### **Spectra study of (1E,4E)-N-(3-nitrophenyl)-1,5-diphenylpenta 1,4-dien-3-imine**

IR(KBr. cm<sup>-1</sup>):1637 cm<sup>-1</sup>(C=N), 1235 cm<sup>-1</sup>(N=CH str),1023(CH=N),3520 cm<sup>-1</sup>(N-H),1330,1550(-NO<sub>2</sub>Ar),898,760(M-Sub)<sup>1</sup>HNMR(ppm) (CDCl<sub>3</sub>):5.26(s,1H,Ar-H),6.59-6.62(m,4H,Ar-H)5.79(s,N-H) MS:355.40[M+1].

#### **Spectra study of (1E,4E)-N-(4-nitrophenyl)-1,5-diphenylpenta-1,4-dien-3-imine**

IR(KBr. cm<sup>-1</sup>):1645 cm<sup>-1</sup>(C=N), 1256 cm<sup>-1</sup>(N=CH str),1017(CH=N),3560 cm<sup>-1</sup>(N-H),1359,1550(-NO<sub>2</sub> Ar), 770,850(P-Sub)<sup>1</sup>HNMR(ppm) (CDCl<sub>3</sub>):7.2(s,1H,Ar-H),6-7(m,4H,Ar-H)5.06(s,N-H) MS:355.40[M+1].

#### **Spectra study of (1E,4E)-N-(4-methylphenyl)-1,5-diphenylpenta 1,4-dien-3-imine**

IR(KBr. cm<sup>-1</sup>):1636 cm<sup>-1</sup>(C=N), 1200cm<sup>-1</sup>(N=CH str),1015(CH=N),3480 cm<sup>-1</sup>(N-H),<sup>1</sup>HNMR(ppm) (CDCl<sub>3</sub>):5.46(s,1H,Ar-H),6.62-6.64 (m,4H,Ar-H)5.89(s,N-H) MS:324.43[M+1].

#### **Spectra study of 4-[(1E,4E)-1,5-diphenylpenta-1,4-dien-3-ylidene]amino} benzenesulfonic acid**

IR(KBr. cm<sup>-1</sup>):1644 cm<sup>-1</sup>(C=N), 1234cm<sup>-1</sup>(N=CH str),1046(CH=N),3451 cm<sup>-1</sup>(N-H),<sup>1</sup>HNMR(ppm) (CDCl<sub>3</sub>):5.46(s,1H,Ar-H),6-7(m,4H,Ar-H) 5.89(s,N-H) MS:390.46[M+1].

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