# Synthesis, Characterization of New 2-(Substituted Phenyl)-4, 5-Bis-(4-Methoxyphenyl) -1H - Imidazoles from P-Anisil

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***Abstract***- Substituted benzaldehydes are condensed with anisil and ammonium acetate to yield the corresponding 2-substitutedphenyl-4,5-bis-(4-methoxyphenyl)-1H-imidazoles . P-Anisil can be synthesized by the oxidation of P-Anisoin . All synthesized compounds were characterized on the basis of melting point, IR, NMR spectra and elemental analysis.

***Index Terms***- P-Anisil, ammonium acetate, glacial acetic acid, 2-(substitutedphenyl)-4,5-bis-(4-methoxyphenyl)-1H-imidazole.

## I. INTRODUCTION

This article guides a stepwise walkthrough by Experts for writing a successful journal or a research paper starting from inception of ideas till their publications. Research papers are highly recognized in scholar fraternity and form a core part of PhD curriculum. Research scholars publish their research work in leading journals to complete their grades. In addition, the published research work also provides a big weight-age to get admissions in reputed varsity. Now, here we enlist the proven steps to publish the research paper in a journal.

Substituted imidazoles, many of which play important roles in the biologically significant processes have been prepared by a variety of synthetic methods [1] . Imidazoles and their derivatives play important role as versatile building blocks for the synthesis of natural products and as therapeutic agents [2] .In particular 2,4,5- trisubstituted -1H- imidazoles are biologically active and occur in the structure of a large number of herbicides [3] , fungicides [4] , or as an inhibitor of P38MAP kinase [5-7] . There are several methods for the synthesis of 2,4,5 - triaryl-1H-imidazoles from benzoin / benzil , aromatic aldehdye and ammonium acetate using different catalyst such as Silica gel [8] ZrCl2 [9] , NiCl2.6H2O [10] , Iodine [11] , Sodium disulphide [12] , acetic acid [13] , and ammonium acetate [14] . **Keeping in view their biological activity , synthesis of some new 2-(Substituted phenyl) -4,5-bis -( 4-methoxyphenyl ) -1H-imidazole derivatives have been carried out without using catalyst .**The P-Anisoin was synthesized by benzoin condensation in presence of sodium cyanide catalyst which oxidized by using Conc. Nitric acid in presence of glacial acetic acid to obtained P-Anisil , The P-Anisil was reacted with substituted aromatic aldehyde , ammonium acetate in glacial acetic acid to obtained the 2-(Substituted phenyl)-4,5-bis-(4-methoxyphenyl)-1H-imidazoles .

## II. .EXPERIMENTAL

4- Methoxybenzaldehyde , Sodium cyanide , ethanol , Conc. Nitric acid , Substituted benzaldehyde , P-Anisil , ammonium acetate , glacial acetic acid is utilized chemicals . All the melting point was determined in open capillaries and are uncorrected. IR spectra were recorded in KBr using Perkin Elmer model 2000 spectrophotometer and reported wave number are given in $cm^{-1}$ , $^1H$ NMR spectra were recorded in DMSO on a Brucker Avance II 400MHz spectrophotometer using TMS as an internal standard . The purity of all the synthesized compound was tested by TLC on silica gel plate by using ethyl acetate and petroleum ether (80:20) .

*** General procedure for the synthesis of 2-hydroxy-1, 2-bis-(4-methoxyphenyl)-ethan-1-one-(B-1) --**

Mixture of 18.0 ml ( 0.15 mol ) 4- methoxybenzaldehyde in 60 ml ethanol ,add 4.9 gm ( 0.1 mol ) sodium cyanide in 20 ml water was heated for 3 hour under a water condenser , after which it was cooled under the cold water tap with continuous shaking for 15minutes . On allowing to stand over night, hard crusts of anisoin were formed , dried it and recrystallized from ethanol .

**Yield - 83 % . M.Pt - 108- 110 $^{O}$C , M.Wt - 272.30 , Formula – $C_{16}H_{16}O_4$**

**Anal Calculation for $C_{16}H_{16}O_4$ , Found** - C: 70.64 , H:5.94 , O: 23.65 **Calcd** - C:70.58 , H:5.88 , O:23.52 .

**IR - ( KBr $cm^{-1}$ )** 3465.09 ( O-H) ,3076.80 ( Ar C-H) , 2938.38 ( C-H Str ), 1668.78 ( C=O) 1597.80 ( Ar C=C) , 1265 .57 ( C-O Str ) .

**$^1$H NMR ( DMSO)** - 3.68 ( S ,3H , - $OCH_3$) , 3.76 ( S, 3H -$OCH_3$ ) , 5.41( S, 1H, C-H) 5.90 (S, 1H, OH) 6.7 to 7.9 (m, 8H aromatic).

*** Synthesis of P- Anisil [1,2-Bis - ( 4- methoxyphenyl - ethan-1,2-dione ) ] ( B-2) --**

Took 8.0 gm 2- hydroxy-1,2--bis- (4-methoxyphenyl ) -ethan-1-one ( B-1) dissolved it in 24 ml glacial acetic acid , then added 34 ml Conc. Nitric acid slowly to a reaction mixture ( Reaction mixture kept in an ice bath ) . Refluxed the reaction mixture for 2 hours untill the complete evolution of brown gas, stopped Cool reaction mixture and poured into crush icecold water with stirring Obtained a solid product , dried it and recrystallized from ethanol .

**Yield- 79%, M.Pt- 132-135$^O$C, M.Wt -270 , Formula - $C_{16}H_{14}O_4$.**

**Anal Calculation for $C_{16}H_{14}O_4$ Found -** C: 71.21 H: 5.26 , O : 23.76 **Calcd-** C:71.11 H: 5.18 O: 23.70

**IR ( KBr cm-1) :-** 3072( C-H Ar ) , 2979 ( C-H ali -OCH3 ) , 1690(C=O) , 1536 ( C=C ) , 1158 ( C-O)

**$^1$HNMR ( DMSO) :-** 4.0 ( S ,3H, -$OCH_3$ ) , 7.3 ( d, 2H ) , 7.4 ( d , 2H ) .

*** Synthesis of 2- (Substituted phenyl ) - 4,5- bis - ( 4-methoxyphenyl ) -1H- imidazoles –( 3a -3l)**

A mixture containing 1,2-bis-(4-methoxyphenyl) - ethan-1,2- dione ( 0.05mol) , benzaldehyde (0.05mol ) , ammonium acetate ( 0.1mol ) was taken in a 100ml round bottom flask was shaken in 15 ml glacial acetic acid , It was refluxed on a water condenser for 6 hours , cooled the reaction mixture and poured into crush ice-cold water , kept it for 10 to 20 minutes ,Obtained a solid product was filtered dried and recrystallized from ethanol .

**Colour – Colourless , Yield - 83 % , M.Pt- 198-200$^O$C**

*** Spectral Data - 2-Phenyl -4,5-bis-( 4-methoxyphenyl) -1H- imidazole ( 3a )**

**Solid , Colorless , M.Pt- 198-200$^O$C , Formula - $C_{23}H_{20}O_2N_2$ , M.Wt- 356 .**

**IR- ( KBr cm$^{-1}$)** 3450 ( N-H) , 3078.90 ( C-H aro ) 2977.03 (C-H aliph) 1618.12 ( C=N) 1461.56 ( C=C) .

**$^1$H NMR ( DMSO)** , 3.90 ( S , 1H ,-$OCH_3$) , 3.98 ( S ,1H ,-$OCH_3$) , 7.46( d,2H)

, 8.36(d,2H) 8.05( S,1H ) ,7.51-8.69 (m ,8H) , 8.6 ( S , N-H)

Anal Calculation for $C_{23}H_{20}O_2N_2$ , **Calcd** C: 77.52 H: 5.61 O: 8.98 , N:7.86

**Found** C: 77.59 H : 5.59 O : 8.91 ,N :7.90

*** Spectral data - 2- ( 4-Chlorophenyl) -4,5-bis - ( 4-Methoxyphenyl) -1H- imidazole ( 3b ) -**

**Solid , Colour- Yellow , Yield - 62 % , M.Pt 120-123$^{O}$C Formula $C_{23}H_{19}O_2N_2Cl$ , M.Wt - 390.5**

**IR ( KBr $cm^{-1}$)** 3448$cm^{-1}$(N-H) ,3078$Cm^{-1}$ (C-H arom) , 2846$cm^{-1}$( C-H ,-$OCH_3$) ,1613 ( C = N ), 1536 ( C = C ), 1012 ( C-O str) aryl , -$OCH_3$ , 829$cm^{-1}$ ( 4-substituted benzene ring ) .

**$^1$HNMR (DMSO)** 3.82 Singlet (6H) , $(CH_3O)$ x 2 ,6.9-7.5 q ( 8H ) , $(C_6H_4OCH_3)$x2

6.9 (m 2H) , 8.4( m ,2H ) , 9.9 broad singlet (1H ,N-H ) .

Anal .Calculation for $C_{23}H_{19}O_2N_2.Cl$ **Calcd** C: 70.76 H: 4.86 O:8.19 N:7.17 Cl:9.09

**Found** C: 70.79 H:4.81 O:8.16 N:7.20 Cl:9.12

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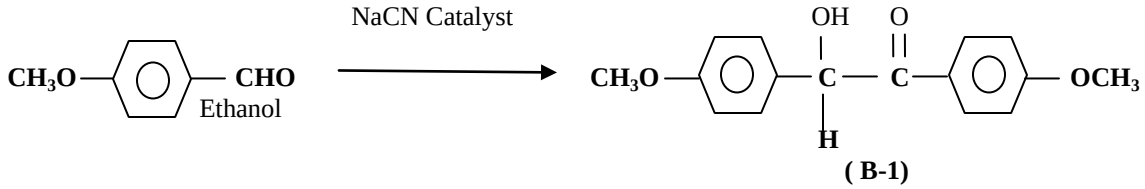
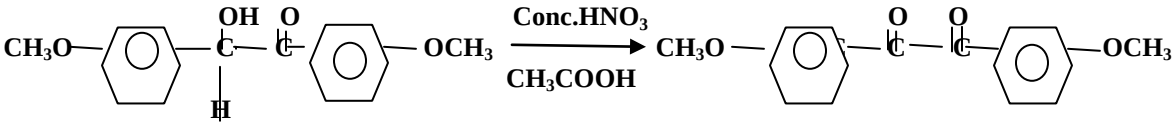
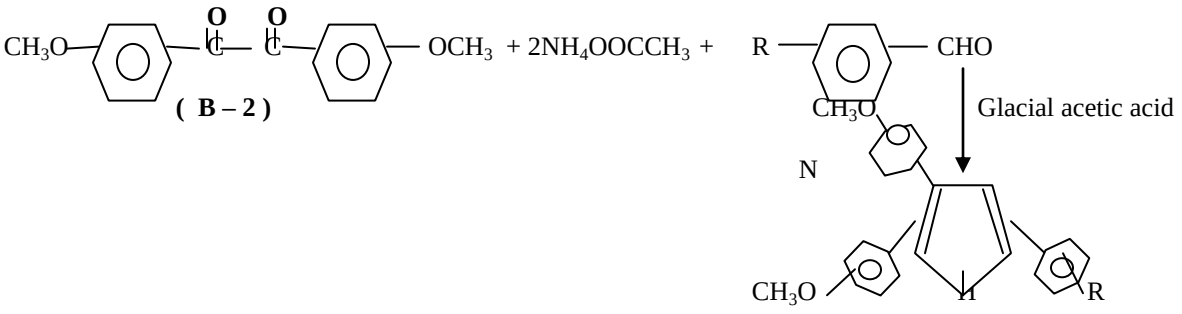



**( B-1)**




**( B-1)** **( B-2)**




**( B – 2 )** ( 3a – 3 l )

R = H , 4-Cl , 4-$OCH_3$ , 4- $NO_2$ , 4- N( $CH_3)_2$ , 2-(OH) , 4-( OH) -3-($OCH_3$) 3,4,5- ($OCH_3$) , 2-Cl , 4-OH .

The other compound of this series (3a – 3l ) were prepared similarly and are recorded in table -1

Tab**le-1 , Physicochemical data of the synthesized imidazole derivatives ( 3a -3l) -**

| , Sr.No. | Code | R | M.Pt($^{O}C$) | Yield | M.Wt | Formula |
|---|---|---|---|---|---|---|
| 1 | 3a | -H | 198-200 | 83 | 356 | $C_{23}H_{20}O_2N_2$ |
| 2 | 3b | -4Cl | 120-123 | 78 | 390.5 | $C_{23}H_{19}O_2N_2Cl$ |
| 3 | 3c | -4$OCH_3$ | 170-172 | 92 | 386 | $C_{24}H_{22}O_3N_2$ |
| 4 | 3d | -4$NO_2$ | 170-175 | 77 | 401 | $C_{23}H_{19}O_4N_3$ |
| 5 | 3e | -2$NO_2$ | 210-212 | 65 | 401 | $C_{23}H_{19}O_4N_3$ |
| 6 | 3f | -4$N(CH_3)_2$ | 155-158 | 68 | 399 | $C_{25}H_{25}O_2N_3$ |
| 7 | 3g | -2.OH | 201-204 | 73 | 372 | $C_{23}H_{20}O_3N_2$ |
| 8 | 3h | -4(OH)-3-($OCH_3$) | 227-230 | 74 | 402 | $C_{24}H_{22}O_4N_2$ |
| 9 | 3i | 3,4,5-($OCH_3$) | 233-235 | 91 | 466 | $C_{26}H_{26}O_5N_2$ |
| 10 | 3j | 2-Cl | 183-185 | 75 | 390.5 | $C_{23}H_{19}O_2ClN_2$ |
| 11 | 3k | 4-OH | 170-173 | 71 | 372 | $C_{23}H_{20}O_3N_2$ |
| 12 | 3l | 3-$NO_2$ | 240-242 | 75 | 401 | $C_{23}H_{19}O_4N_3$ |

## III. RESULT AND DISCUSSION

2-Substitutedphenyl -4,5-bis-( 4-methoxyphenyl) -1H-imidazoles (3a-3l)was synthesized by condensation reaction involving the reagent such as P-anisil , Substituted benzaldehyde and ammonium acetate in glacial acetic acid . The physical data of compound were collected and presented under compound name and spectral data .The yield of the compounds in the range 65-85 % , most of them are colourless crystalline solid. The IR spectrum of compound

2a show the characteristic band at 3450 cm-1 due to the N-H . The IR spectrum of compound 3a,3b ,3c shows the characteristic band at 1500- 1600cm-1 due to - C=N group. The 1HNMR spectrum of compound 3a, 3b, 3d shows single of N-H at 8.6 ppm, 9.9ppm, 8.7ppm which confirmed the presence of N-H bond of imidazole .We have presented efficient synthesis of 2-substituted phenyl -4,5-bis -( 4-methoxyphenyl ) -1H - imidazole in absence of catalyst .

## IV. CONCLUSION

This report illustrate, new finding on the synthesis of some 2-substituted phenyl-4,5-bis -(4-methoxyphenyl) -1H- imidazole . in absence of catalyst .

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