International Journal of Universal Print
ISSN: 2454-7263 ID: ACTRA 2018 029 Published Mar. 2018
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Title Key: Efficient Synthesis Of 2,4,5 -Triaryl imidazole …

Efficient Synthesis Of 2,4,5 -Triaryl imidazole : A Bioactive Molecule

Arshia Parveen*

***Department of Chemistry, B. Raghunath College, Parbhani-431401(MS)

arshiachemistry@yahoo.co.in , arshiairfanmalik@gmail.com

Abstract: - An efficient and rapid one-pot synthesis of 2, 4, 5-triaryl imidazole is carried out by reflux using aromatic aldehydes, benzil or benzoin in the presence of catalytic amount of CAN Ceric Ammonium Nitrate. Excellent yield in short reaction time is characterized by simple work up procedure and efficient recovery. The 2, 4, 5-triaryl imidazoles were synthesized and tested for antibacterial effects against ***Bacillus Subtilis***, ***Escherichia coli***, ***Staphylococcus aureus*** and ***Pseudomonas aeruginosa***. The antibacterial screening of the synthesized compounds was performed in vitro by the filter paper disc diffusion method and it is found that the 2, 4, 5-triaryl imidazoles show good antimicrobial activity.

Key words: ceric ammonium nitrate CAN, 2, 4, 5-triaryl imidazole, aromatic aldehydes, benzil, benzoin, antibacterial screening.

Introduction: The imidazole ring system is of particular interest as it is a component of histidine that produces histamine in metabolic process[1]. The potency and wide applicability of the imidazole pharmacophore can be attributed to its hydrogen bond donor-acceptor capability as well as its high affinity for metals which are present in many protein active sites.Triaryl imidazoles are used as a photosensitive materials in photography[2a] .In addition they are of interest because of their herbicidal[2b], analgesic[3], fungicidal[4], anti-inflammatory[5]and antithrombotic activities[6]. Recent advances in green chemistry and organo metallic chemistry have extended the boundary of imidazole to the synthesis and application of a large class of imidazoles as ionic liquids[23] and imidazole related N-heterocyclic Carbenes (NHC) [7] imidazoles also have COX-2 inhibitory activity[8]**.** Generally triaryl imidazoles are prepared by hetro-cope rearrangement[9]or by reaction of glyoxal, formaldehyde and ammonia[10-11]. Previous studies suggested the use of Zn-Al_2O_3[12], and PCl_5[13] diktones aldehyde and ammonium acetate in phosphoric acid [14] as well as in H_2SO_4[15] and DMSO[16].Micro wave assisted synthesis of imidazoles from 1,2-diketones in the presence of catalyst such as silica-gel[17],silica-gel/HY[18], MW/Al_2O_3[19], DMF[20] and MW/acetic acid[21].

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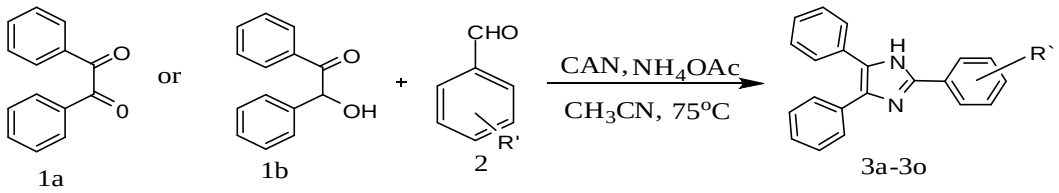
Reactant 1a,1b	Reactant 2	Product 3	Time(h)	Yield(%)



Schem-1

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Reported methods have one or the other limitations such as harsh reaction conditions, poor yields prolonged time period, use of hazardous and expensive catalysts and polar solvents. Recently ceric ammonium nitrate (CAN) received considerable attention as an inexpensive, nontoxic, readily available catalyst for various transformations, affording the corresponding products in excellent yield with high selectivity; In the proposed work we have synthesized trisubstituted imidazoles from benzil or benzoin with aldehyde at 75°C in the presence catalytic amount of CAN (schem-1). During the course of our studies toward the development of new routes to the synthesis of biologically active heterocycles[22].

economically expensive not available easily. When benzil ,benzoin **(1a,1b)** and aromatic.

Table 02: Synthesis of 2, 4, 5-triaryl imidazole

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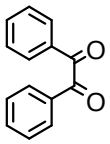
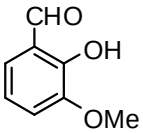
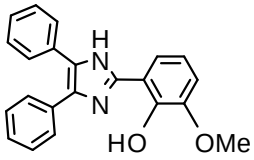
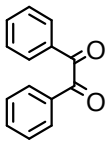
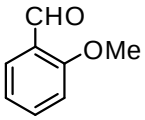
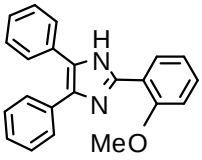
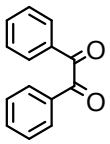
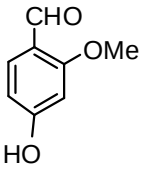
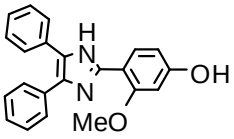
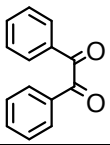
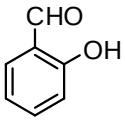
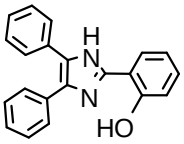
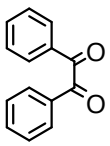
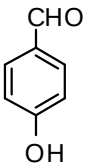
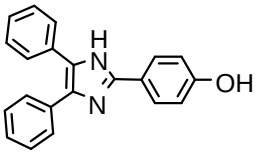
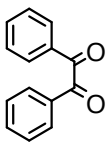
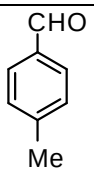
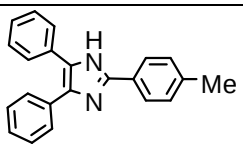
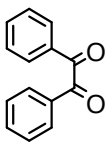
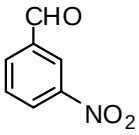
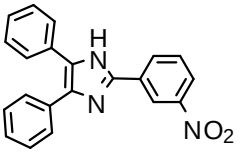
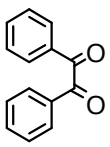
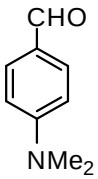
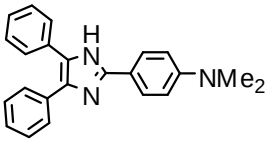
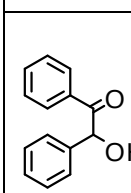
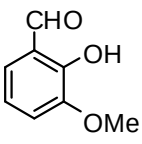
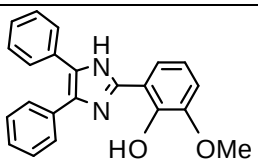
	2a 	3a 	3.5	91
	2b 	3b 	4	93
	2c 	3c 	3.2	93
	2d 	3d 	1.7	92
	2e 	3e 	3.7	94
	2f 	3f 	3.7	89
	2g 	3g 	3.5	92
	2h 	3h 	2.5	93
	2a 	3i 	3	90

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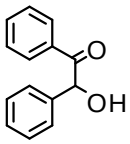
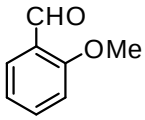
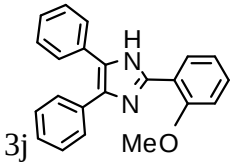
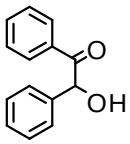
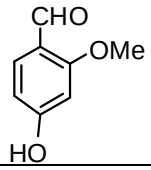
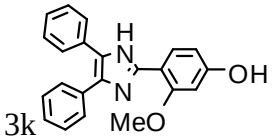
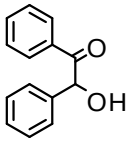
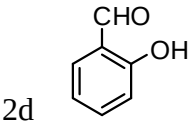
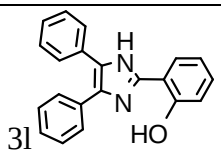
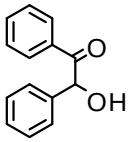
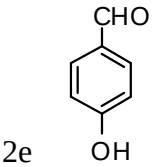
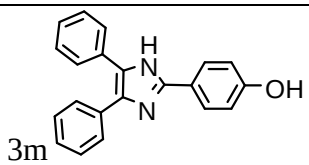
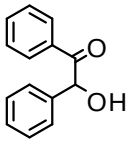
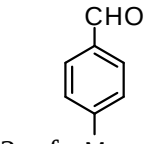
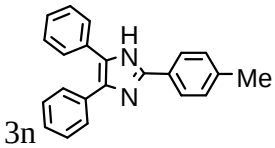
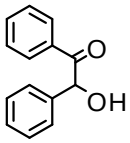
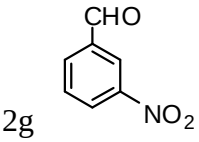
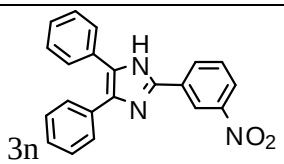
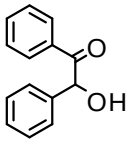
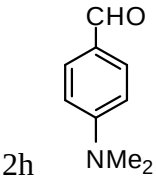
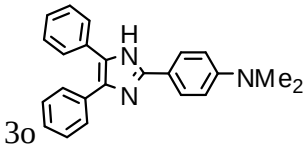
	2b 	 3j	4	90
	2c 	 3k	4	91
	 2d	 3l	4	90
	 2e	 3m	3	92
	 2 f	 3n	3	90
	 2g	 3n	5	89
	 2h	 3o	3.5	88

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Results and discussion - In continuation to our endeavous to develop the biologically active compounds of substituted imidazole derivatives, we have developed the methodology for the synthesis of 2,4,5 trisubstituted imidazoles using neat reaction condition . The synthesis of trisubstituted imidazoles by aromatic aldehyde, benzil or benzoin and ammonium acetate in presence of ionic liquid [Hbim]BF_4 is a well established procedure[23].However,ionic liquid is aldehyde **2** were treated with a catalytic amount of **CAN** Cerric Ammonium Nitrate in acetonitrile for 2-6 hrs, then triaryl substituted imidazoles **3** were obtained in moderate to good yields (**Table 2**).

To examine the catalytic activity of CAN, we explored a modification of the reaction of (1a) and (1b) aromatic aldehydes in acetonitrile first without CAN then ,5mol%, 10mol%, 20mol%, 25mol% amount of CAN. The results are shown in **(Table.1).**According to observations in Table.1 (10mol%) of CAN was enough and efficient , as 90 % .91% yield **(entry 2)** for both (1a) ,(1b) respectively an excessive amount of the catalyst was check for the same reaction condition it is found that at the same reaction time, % yield did not increase . **(Table.1 entry 3-5)** .In the absence of CAN, no reaction was found (Table.1, entry 1) . To investigate the real catalyst species, CAN , $CeSO_4$, the experiment using $CeSO_4$ 20mol% in place of CAN has been tried. The product was obtained in both 1a,1b with yield of 60%, 58% at 75^oC (Table.1 entry 6) hence CAN should be the real catalyst species because its Lewis acidity.

Ammonium acetate is a solid source of ammonia which can be conveniently generated in situ through the dissociation of ammonium acetate. Usually, the amount of ammonium acetate used is loosely controlled. A large excess is often used for two reasons one is that it is water soluble and excess amount can be easily removed during a work up and secondly it is a neutral salt and not a significant active species other than as an ammonia source. .

Table:-1 Effect of catalytic amount of CAN[b]

[a]Entry	Catalyst	Amount (mol%)	Time(hrs)	Yield(%)[d] 1a	1b
1	NO	-	5	ND[c]	ND[c]
2	CAN	5	5	90	91
3	CAN	10	5	75	78
4	CAN	20	5	73	70
5	CAN	25	5	85	87
6	CeSO4	20	7	60	58

[a] Entry 1-6

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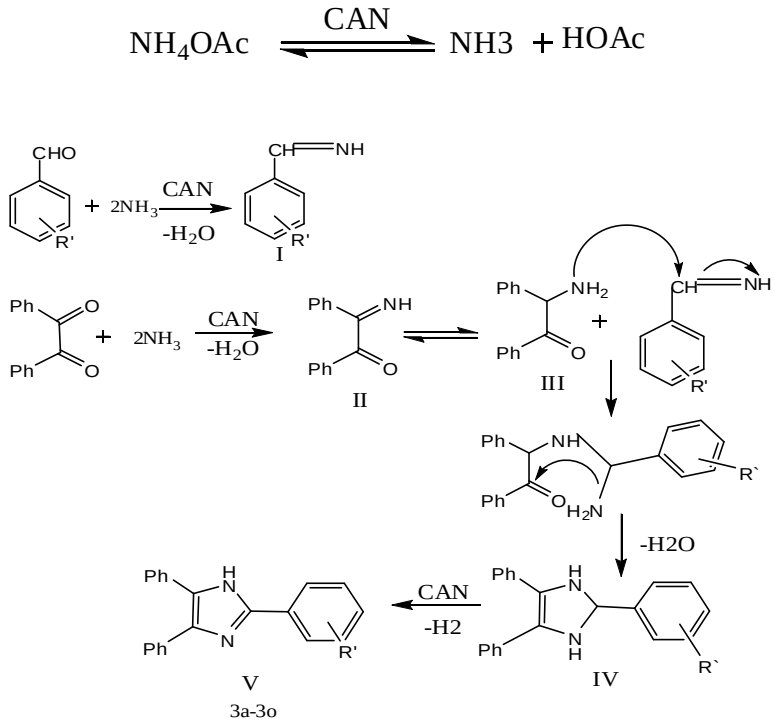
[b]CAN Ceric ammonium nitrate $[(NH_4)Ce(NO_3)]$, ND[c] No product formation, [d] isolated yield
1a(benzil) 1b(benzoin) obtained by column chromatography.

Schem 2 plausible mechanism for the formation of triarylsubstituted imidazole 3a-3o:



According to the literature survey it was reported that Balalai et.al[18] and Qing Xiang Guo[24] ,synthesized 2,4,5 trisubstituted imidazole by using benzoin(1b), zeolite HY and Sio_2 respectively in microwave irradiation in our methodology we were reported the formation of imidazole by using directly benzoin(1b)with the same reaction condition scheme 1.The benzoin(1b) reflux with acetic acid and the product was not found even after 24 hrs. When we used CAN a powerful oxidizing reagent (scheme 1) we found very good results summarized in Table 2. The CAN has promoted this heterocyclization reaction by virtue of its inherent bronsted acidity which makes it capable of bonding with the carbonyl oxygen increasing the relativities of the parent carbonyl compounds. The CAN promotes the splitting of ammonia required for the initial condensation.

For the postulated mechanism starting from 1,2-diketone schem.3.The CAN may facilitate the formation of a amine intermediate I, which under Bronsted acid catalysis of the CAN condenses with the carbonyl carbons of the 1,2-diketone followed by dehydration to afford the imino intermediate II ,which rearranges to the required the tri-aryl imidazole III . A probable mechanism for the synthesis involving benzoin schem 3 .It

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is highly probable that the Bronsted acidity of the CAN may have promoted the formation of α-amino ketone II , aryl aldimine I their subsequent condensation and intermolecular cyclization to the imidazole III, which dehydrogenates to the triaryl imidazoles V. It was thought that the dissolved oxygen in the CAN may have brought about the formal oxidation of the imidazole.

Antimicrobial Activity:

The antibacterial activities of the synthesized compounds (g) and (h) were studied against four bacteria, viz. ***Bacillus subtilis*** **(G+),** ***Escherichia coli*** **(G–),*Staphylococcus aureus*** **(G+)** and ***Pseudomonas aeruginosa*** **(G–).** For the detection of antibacterial activities, the filter paper discs diffusion method was used[25]. Streptomycin sulphate was used as positive control. Nutrient agar (NA) was used as basal medium for test bacteria. The discs were prepared by impregnating them in methanol solution of each sample (1 mg/1 mL). Each culture was prepared to a turbidity equivalent to McFarland and spread on the test tube. The paper disc containing the compound was placed on the agar surface previously inoculated with suspension of each microbe to be tested. All determinations were made in duplicate. Inhibition diameter was determined after incubation at 37°C ± 1 for 24 h. The antimicrobial activity was indicated by the presence of the clear inhibition zones around each disc.

Minimum inhibition concentration:

The determinations of the minimum inhibitory concentration (MIC), the serial dilution technique were followed using nutrient broth medium. The MIC was defined as the lowest concentration of samples that had restricted the growth of microbial. The MIC value of compound (g) was determined against *Escherichia coli* (G–)

Experimental:

All reported yields are isolated yields. Melting points are uncorrected and were recorded by open capillary.Infra red spectra were recorded with ATI MATT-SON RS-1 FTIR spectrometer in (KBr) .`HNMR spectra were recorded on a Bruker AC-200 (MHz) spectrometerin $CDCl_3$/DMSO-d6, with TMS as an internal standard.

General procedure for synthesis of 2,4,5-triaryl imidazoles from 1,2-diketones (1a)or a-hydroxyketone(1b)

A mixture of 1,2-diketones (la) or the a-hydroxyketone (1b) (1 mmol), substituted aldehydes (2a-h,1mmol), ammonium acetate (10 equiv) and CAN (10mol%) was reflux at 75°C for the appropriate time mentioned in Tables 2. The completion of reaction was monitored by TLC using ethyl acetate: petroleum ether (1:9). After completion of reaction, the reaction mixture was diluted with water. The solid imidazole products, which separated out, were filtered, washed with sodium bisulphate and dried. The crude products, thus isolated, were pure (single spot on TLC). They were subjected to further purification by column chromatography 10% EtOAc in petroleum ether used as eluent to yield the desired substituted imidazoles in excellent yields of 86-92% The IR spectrum of **86k** showed absorption at 1565, 1638 and 3438 cm^{-1} corresponds to C=C, C=N, –NH respectively. The ^1H NMR spectrum of **86k** showed singlet of three protons at δ 3.85 corresponds to methoxy group, multiplet for ten protons in the region δ 7.25-7.59 for aromatic proton and 12.52 (brs, 1H) corresponds to –NH. The ^{13}C NMR spectrum showed peaks at δ 54.6 and 145.7 corresponding to methoxy

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carbon and C=N respectively, elemental data confirms with the structure of **86k** and corresponds to the known data (**Scheme 1.1**).

Organism	**2,4,5-triaryl imidazoles**	Streptomycin sulphate
Bacillus subtilis	–	22·0 ± 0·3
Staphylococcus aureus	–	22·5 ± 0·7
Escherichia coli	12·5 ± 0·3	22·0 ± 0·0
Pseudomonas aeruginosa	–	22·0 ± 0·0

Antimicrobial Screening:

The antibacterial activity of compounds (g) and (h) has been assayed at the concentration 1000 µg/mL against four human pathogenic bacteria. Among them two were gram-positive and the other two were gram negative. The inhibitory effect of compounds (g) and (h) against these organisms are given in Table 3.The screening results indicate that only compound (h) was active against a gram-negative bacteria, Escherichia coli with a mean zone of inhibition 12·5 ± 0·3 mm (Table 3).

Determination of the minimum inhibitory concentration (MIC) :

The active sample in the disc diffusion method was then tested for its activity by the serial dilution method to determine the minimum inhibition concentration (MIC-value). The MIC value obtained for flavanone (f) was 1000 µg/mL against Escherichia coli.

Table 3. Antibacterial screening for the compounds (g) and (h)

In conclusion, we have developed an efficient , convenient and one-pot protocol for the synthesis of biologically potent 2,4,5-triaryl imidazoles via the condensation of aromatic aldehyde and benzil or benzoin with ammonium acetate using ceric ammonium nitrate. The process give rise to excellent isolated yield of triaryl imidazole .The study of antimicrobial activity is under progress.

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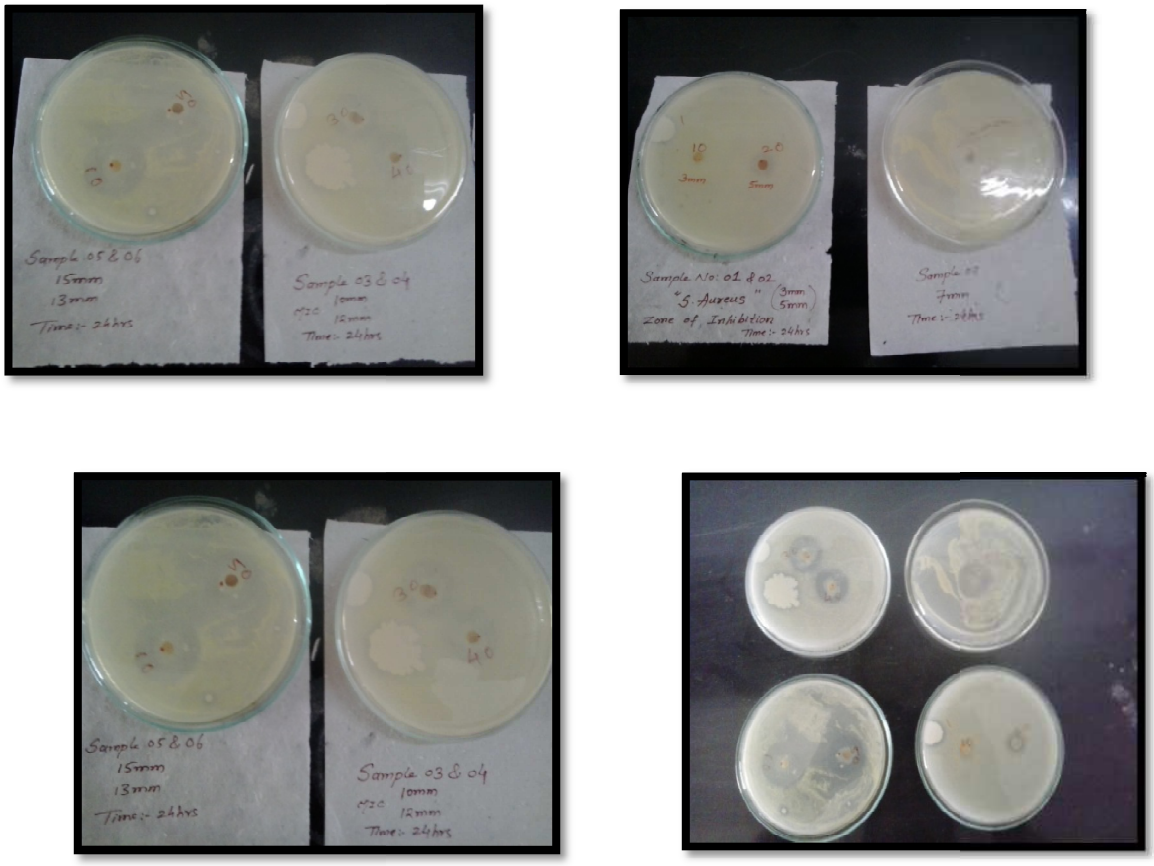

Fig:- Diameter of the zone of inhibition of Sample 3g-3h

Spectral Data:

2-(4,5-Diphenyl-1*H*-imidazol-2-yl)-6-methoxy phenol(3a).

MP 168^{o}C; IR ((cm^{-1})) .730,1234,1210,1654, 2920,3512,3600; 'HNMR (($CDCl_3$/DMSO-d_6, 200 MHz) δ: 3.86 (s, 3H), 6.32-5.45 (m, 3H), 7.22-7.12 (m, 5H), 7.40-6.95 (m, 5H), 12.4 (brs, IH); ^{13}C NMR (CDCl,/DMSO-d_6, 200 MHz) 544.3, 110.9, 112.1, 155.6, 117.1, 126.3, 125.6, 122.1, 126, 1

2-(4-Methoxy-phenyl)-4,5-diphenyl-l*H*-imidazole (3b).

Mp 220 ^{o}C; IR ((cm^{-1})) 1212, 1600, 2260, 2893. 3420; 'H NMR ($CDCl_3$/DMSO-d_6, 200 MHz) δ: 3.85 (s, 3H), 6.93-6.60 (d, 7 = 8.8 Hz. 2H), 7.10-7.02 (m, 10H), 8.16-8.12 (d, ,J=8.7 Hz, 2H). 12.39 (brs, IH); ^{13}C NMR (CDCl d_6/DMSO-d_6, 200MHz) *5* 48.7, 111.2, 120.7, 126.3, 126.5, 127.3, 127.4, 132.8, 145.7,

2-(4,5-Diphenyi-l *H* -imidazol-2-yl)-2-methoxy phenol (3c).

Mp 195 °C; IR ((cm^{-1})) 1240, 1470, 1620, 2910. 3510, 3614; 'H NMR ($CDCl_3$/DMSO-d_6, 200 MHz) δ: 3.80 (s, 3H), 6.75-6.69 (d, 7 = 8.22 Hz, IH], 7.11-7.19 (m, 5H), 7.22-7.23 (d. 7 = 8.1 Hz, IH), 7.40-7.45 (m, 5H), 7.55-7.56 (d, 7= 8 Hz, IH), 12.52 (brs, IH); ^{13}C NMR (CDCl /DMSO- d_6 200 MHz) *d* 55.1, 108.5, 114.6, 118.1, 121.1, 126.2. 127.3, 127.5, 132.3, 146.3, 146.8.

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2-(4,5-Diphenyl-l *H* -imidazol-2-yl)-phenol (3d).
Mp 203-205 ^{o}C; IR ((cm^{-1})) 1211, 1608, 2500. 2953. 3475, 3696; 'H NMR ($CDCl_3$/DMSO-d_6, 200 MHz) δ : 6.87-6.95 (d, J=7.5 Hz, 2H), 6.96-7.01 (d, ,J = 8.06 Hz, 2H), 7.17-7.23 (m, 10H). 12.74 (brs, IH): ^{I3}C NMR ($CDC1_3$/DMSO- d_6, 200MHz) *d* 112.7, 116.4, 118.1. 124.8, 127.4, 127.8, 129.1, 145.7.

4-(4,5-Diphenyl-l *H* -imidazol-2-yl)-phenol (3e).
Mp 230-231°C; IR (cm ^{1}) 1216. 1638, 2465, 2998, 3432, 3596; 'H NMR ($CDCl_3$/DMSO-d_6, 200 MHz) δ:
6.97 (d, *J*= 8Hz, 2H), 7.52-7.87 (m, 10H), 7.88-7.92 (d, J = 8.5 Hz. 2H), 12.58 (brs, IH); ^{I3}C NMR ($CDCl_3$/DMSO-d_6, 200MHz) *d* 111, 117.6, 124, 123.3, 120.1, 122, 140.

2-(4-Methyl-phenyl)-4,5-diphenyl-l*H* -imidazole (3f).
Mp 158-161 ^{o}C IR(cm^{-1}) 1215, 1453, 1486, 1496, 1601, 2926, 'H NMR ($CDCl_3$/DMSO-d_6, 200 MHz) δ:2.30 (s, 3H), 7.41-7.51 (d,.10H), 7.29-8.52 (d, 4H), 13.58 (s,1H) ^{13}C NMR (CD Cl_3 /DMSO-d_6, 200MHz) 48.8, 126.5, 127.1, 128.3, 128.8, 129.5, 130.7, 134.4, 138.2, 147.3.

2-(3-Nitrophenyl)-4,5-diphenyl-1*H*-imidazole (3g).
Mp 198-200 ^{o}C IR(cm^{-1}) 1446.3, 1533.8, 1540.7, 1602.6, 3058. 'H NMR ($CDCl_3$/DMSO-d_6, 200 MHz) δ:13.11 (s,1H), 8.98 (s, 1H), 8.53 (d, j=9Hz 1H), 8.22 (d, j=9Hz, 1H), 7.76 (t, 1H), 7.25-7.5 (m, 10H) ^{13}C NMR (CD Cl_3 /DMSO-d_6, 200MHz) 122.8, 123.9, 127.5, 127.6, 128.7, 129.2, 129.3, 130.1, 131.5, 133.6, 138.2, 148.4, 177.1.

2-(4-Dimethylaminophenyl)-4,5-diphenyl -1*H*-imidazole (3h).
Mp 237-240 ^{o}C IR(cm^{-1}) 1445.7, 1508, 1551, 1661, 2919.1, 3057,8 . , 'H NMR ($CDCl_3$/DMSO-d_6, 200 MHz) δ: 2.98 (s, 6H), 6.78-7.92 (m,14H), 13.56 (s,1H). ^{13}C NMR (CD Cl_3 /DMSO-d_6, 200MHz) 112, 121.3, 126.9, 127.1, 127.3, 127.8, 127.9, 128.2, 128.3, 129.4, 129.5, 134.5, 145.1, 150.1

Acknowledgment:- Author Thankful to the Department of Chemistry, B.Raghunath ACS College, Parbhani for providing facilities.

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ISSN: 2454-7263 ID: ACTRA 2018 029 Published Mar. 2018
Volume No. 04, Issue No.03, Copyright © Universal Print
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