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One-Pot, Simple and Efficient Synthesis of Triaryl-1*H*-imidazoles by $KMnO_4/CuSO_4$

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ABSTRACT

2,4,5-Triarylimidazoles have been obtained in excellent yields by the one-pot three-component condensation of bis-aryl α-hydroxyketones, aromatic aldehydes and ammonium acetate by the action of inexpensive, readily available and non-toxic $KMnO_4/CuSO_4$ under mild reaction conditions. The present method is simple, efficient, cost-effective and eco-friendly.

KEYWORDS

Triarylimidazoles, Three-component reaction, One-pot synthesis, Oxidation, bis-aryl α-hydroxyketone, $KMnO_4$, $CuSO_4$.

1. Introduction

The development of simple, efficient and general synthetic methods for widely used organic compounds from readily available reagents is one of the major challenges in organic synthesis. In 1858 Debus[1] reported the reaction of glyoxal and ammonia, a pioneering novel synthetic route to imidazole. The imidazole nucleus is a fertile source of biologically important molecules. They are well known as inhibitors of P38MAP kinase,[2] herbicides,[3] anti-inflammatory and antithrombotic agents,[4–5] plant growth regulators[6] and therapeutic agents.[7]

Redziszewski and Japp proposed the first synthesis of the imidazole core in 1882, starting from 1,2-dicarbonyl compounds, aldehydes and ammonia, to obtain 2,4,5-triarylimidazoles.[8] Trisubstituted imidazole derivatives are widely used as organic materials and photographic materials,[9–11] as well as in textile industry, e.g. to increase composition resistance and fluorescent whiteners on textile. It is also known that these compounds play key roles in many kinds of biological and pharmacological activities such as anti-HIV, anti-convulsant,[12] calcium antagonist and as inhibitors of thromboxane A_2 synthase,[13] anthihistaminic,[14] tranquillizing,[15] anti-parkinsonism[16] and MAO inhibiting activities.[17]

Since their discovery, imidazoles have attracted attention because of their chemical and biochemical properties. Even today, after more than 150 years, research in imidazole chemistry continues unabated.

Several procedures reported for the synthesis of triaryl-1*H*-imidazoles through condensation reactions of α-hydroxyketones with ammonium acetate and various aromatic aldehydes in the presence of H_2SO_4,[18] HOAc,[19] DMSO,[20] organocatalysts in HOAc,[21] a variety of solid catalysts,[22] ionic liquids[23] and different metal complexes.[24] Ceric ammonium nitrate,[25] iodine,[26] $NaHSO_3$,[27] zeolites HY/silica gel,[28] $ZrCl_4$,[29] $NiCl_4.6H_2O$,[30] TBAB (tetrabutyl ammonium bromide)[31] and microwave irradiation using Al_2O_3 or acetic acid[32] have also been reported for the preparation of imidazoles. However, some of the synthesis protocols reported above suffer from one or more disadvantages, such harsh reaction conditions, poor yields, prolonged reaction times, and the use of expensive equipments (microwave or ultrasonic), corrosive reagents, difficult work-up and often extensive acid catalysts. On the other hand, a literature survey showed that a $KMnO_4/CuSO_4$ mixture is a good choice for the oxidation of alcohols, oxidative coupling of primary aromatic amines as well as conversion of aryl and alkyl sulfides into their corresponding sulfones,[33] and the recently reported synthesis of quinoxalines.[34] Therefore, it was decided to use this mixture for the preparation of 2,4,5-triaryl imidazoles (Fig. 1).

2. Results and Discussion

As was mentioned above, the $KMnO_4/CuSO_4$ system could be used for the oxidation of bis-aryl α-hydroxyketones to bis-aryl diketones. In fact, control tests confirmed that the co-presence of $KMnO_4$ and $CuSO_4$ is necessary for the reaction to proceed. When use was made of this mixture in hot EtOH media, MnO_2 was generated *in situ* by the action of $KMnO_4$ on hot EtOH during the reaction. This freshly prepared active MnO_2 is a mild oxidant which can easily oxidize benzylic hydroxyl group into the corresponding carbonyl group, for which three routes have been proposed.[35]

In all previously reported procedures for the synthesis of 2,4,5-triarylimidazoles, starting from either bis-aryl α-hydroxyketones or bis-aryl diketones, the reaction time is higher when the starting material are bis-aryl α-hydroxyketones. However, in the case of $KMnO_4/CuSO_4$ in EtOH, the reverse is true, i.e. the reaction time for bis-aryl α-hydroxyketones is much less than that of bis-aryl diketones. In addition, the yields with bis-aryl α-hydroxyketones as starting materials are higher than those of bis-aryl diketones (see Table 1).

It seems that bis-aryl α-hydroxyketones are first oxidized to bis-aryl diketones, with the subsequent formation of Mn^{2+} which may act as an effective Lewis acid catalyst. In the case of bis-aryl diketones, practically no Mn^{2+} is formed, and Cu^{2+} is the only existing Lewis acid catalyzing the reaction. As a control test, running the reaction of 1,2-diphenyl-1,2-ethanedione in the presence of Mn^{2+} and Cu^{2+} reveals that the co-presence of Mn^{+2} may be the key point for the higher reaction rate of bis-aryl 1,2-diketones.

We have also studied the effect of different solvents on the synthesis of 2-(4-chlorophenyl)-4,5-diphenyl-1*H*-midazole, the results of which are summarized in Table 2. Among those examined, ethanol was found to be the most efficient solvent,

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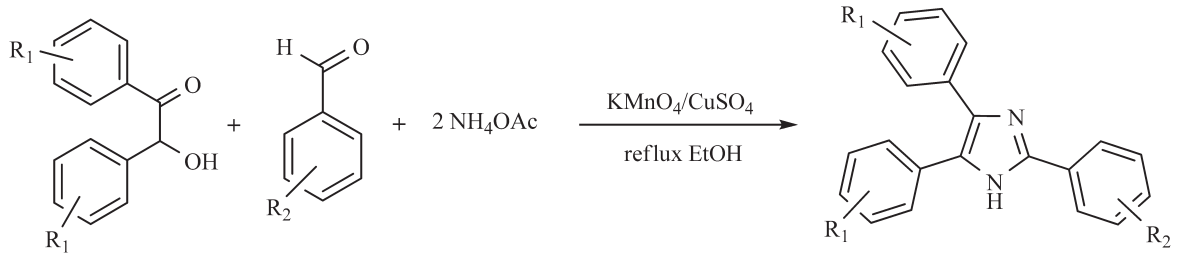


1: $R_1 = R_2 = H$
2: $R_1 = H$, $R_2 = 2\text{-Cl}$
3: $R_1 = H$, $R_2 = 4\text{-Cl}$
4: $R_1 = H$, $R_2 = 4\text{-CH}_3$
5: $R_1 = H$, $R_2 = 4\text{-CH}_3O$
6: $R_1 = H$, $R_2 = 4\text{-Br}$
7: $R_1 = H$, $R_2 = 2\text{-CH}_3O$
8: $R_1 = H$, $R_2 = 2,4\text{-diCl}$
9: $R_1 = H$, $R_2 = NO_2$
10: $R_1 = 4\text{-CH}_3$, $R_2 = H$
11: $R_1 = R_2 = 4\text{-CH}_3$

Figure 1 Synthesis of 2,4,5-triaryl imidazoles.

Table 1 Reaction times and yield for the two starting materials, bis-aryl α-hydroxyketones and bis-aryl diketones.

Entry	R_2	Time/min		Yield/%	
		bis-aryl α-hydroxyketone	bis-aryl diketone	bis-aryl α-hydroxyketone	bis-aryl diketone
1	H	65	90	89	78
2	4-Cl	70	100	85	77
3	4-MeO	110	160	75	68
4	4-Me	90	140	87	75

regarding both the shorter reaction times and higher yields. This is due to the role of ethanol acting both as a solvent and as a reducing agent for the *in situ* formation of active MnO_2.

In conclusion, this is the first report using a $KMnO_4/CuSO_4$ mixture for the synthesis of 2,4,5-triaryl-1*H*-imidazoles. The novelty of this procedure is that the reaction times for the synthesis of 2,4,5-triaryl-1*H*-imidazoles from bis-aryl α-hydroxyketones are less than those of reactions using bis-aryl diketones. Hence, it allows direct, one-pot synthesis of triarylimidazoles from bis-aryl α-hydroxyketones. Other advantages of this procedure are the use of ethanol as a green solvent, a cheap and readily available oxidizing agent, and high product yields when starting from bis-aryl α-hydroxyketones.

3. Experimental

Melting point apparatus, Stuart model SMP3, was used for measuring melting points. IR spectra were recorded on a PerkinElmer series II spectrum. ^{1}H NMR and ^{13}C NMR spectra were recorded in DMSO-d_6 using Bruker DRX-400 spectrometer at 400 and 100 MHz respectively. All chemicals and solvents were purchased from Aldrich, Merck and Fluka, and were used as received. Melting points and spectral data of all products are fully consistent with those of the reported ones.

Table 2 Synthesis of 1-(4-chlorophenyl)-2,4,5-triphenylimidazole using $KMnO_4/CuSO_4$ in different solvents.

Entry	Solvent	Reaction time/min	Yield/%
1	Water	120	–
2	Chloroform	120	–
3	Ethanol	70	85
4	Acetone	120	–
5	Acetonitrile	120	20
6	Toluene	120	–

3.1. Preparation of $KMnO_4/CuSO_4$ Mixture

The optimized amounts of $KMnO_4$ and $CuSO_4$, i.e. 5 mmol and 1 mmol, respectively, were ground together in a mortar to obtain a purple powder.

3.2. General Procedure for the Synthesis of 2,4,5-Triaryl Imidazoles

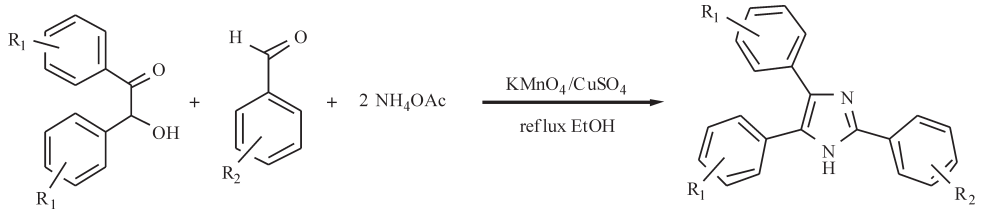
α-Hydroxy ketone (1.0 mmol), aromatic aldehyde (1.0 mmol), ammonium acetate (2.5 mmol) and the mixture of $KMnO_4/CuSO_4$ (0.4 mmol) were stirred and refluxed in ethanol. The progress of the reaction was monitored by TLC. After completion of the reaction, the mixture was poured in cold water (50 mL), the precipitated solid was filtered, washed several times with water, dried and recrystallized from EtOH or acetone:water (9:1) to get the corresponding 2,4,5-triaryl-1*H*-imidazoles. The experimental results are summarized in Table 3.

3.3. Spectral Data of Imidazole Derivatives

*2,4,5-Triphenyl-1*H*-imidazole* (**1**)

M.p.: 275–276 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ = 7.24 (t, 1H, J = 7.2 Hz), 7.32 (t, 2H, J = 7.2 Hz), 7.39 (t, 2H, J = 7.2 Hz), 7.44–7.53 (m, 6H), 7.57 (d, 2H, J = 7.6 Hz), 8.10 (d, 2H, J = 7.2 Hz),

Table 3. Synthesis of 2,4,5-triaryl-1*H*-imidazoles using bis-aryl α-hydroxyketones, ammonium acetate, aromatic aldehydes, and 40 mol% $KMnO_4/CuSO_4$ mixture.



Entry	R_1	R_2	Time/min	Yield/% [a]
1	H	H	65	89
2	H	2-Cl	80	91
3	H	4-Cl	70	85
4	H	4-Me	90	87
5	H	4-OMe	110	75
6	H	4-Br	80	89
7	H	2-OMe	80	88
8	H	2,4-DiCl	90	90
9	H	4-NO_2	150	82
10	4-Me	H	80	79
11	4-Me	4-Me	90	75

[a] Isolated yields

12.73 (1H, br) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ = 125.7, 127.0, 127.5, 128.3, 128.6, 128.7, 128.8, 128.9, 129.2, 129.2, 130.7, 131.5, 135.6, 137.6, 145.8 ppm; IR (KBr, cm^{-1}): 3434, 3031, 1601, 1503, 1455, 1202, 1125, 966, 765, 733, 695.

*2-(2-Chlorophenyl)-4,5-diphenyl-1*H-*imidazole* (**2**)

M.p.: 198–200 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ = 7.24 (t, 1H, *J* = 7.1 Hz), 7.31–7.39 (m, 3H), 7.42–7.53 (m, 6H), 7.57 (d, 2H, *J* = 7.2 Hz) 7.61–7.64 (m, 1H), 7.81–7.83 (m, 1H), 12.69 (br, 1H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ = 127.1, 127.6, 127.7, 128.2, 128.4, 128.7, 129.2, 130.5, 130.6, 130.7, 131.3, 132.0, 132.1, 135.5, 137.4, 143.8 ppm; IR (KBr, cm^{-1}): 3448, 3059, 1601, 1503, 1478, 1320, 1201, 1070, 761, 693, 605.

*2-(4-Chlorophenyl)-4,5-diphenyl-1*H-*imidazole* (**3**)

M.p.: 262–264 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ = 7.22–7.57 (m, 12H), 8.11 (d, 2H, *J* = 8.4 Hz), 12.81 (br, 1H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ = 127.1, 127.3, 127.6, 128.4, 128.7, 128.9, 129.2, 129.3, 129.6, 131.3 133.2, 135.4, 137.8, 144.8 ppm; IR (KBr, cm$^-$): 3440, 3059, 1602, 1503, 1486, 1448, 1324, 1128, 1091, 969, 832, 766, 732, 697, 605.

*2-p-tolyl-4,5-diphenyl-1*H-*imidazole* (**4**)

M.p.: 236–237 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ = 2.36 (s, 3H), 7.23 (t, 1H, *J* = 7.4 Hz), 7.31 (t, 4H, *J* = 7.2 Hz), 7.38 (t, 1H, *J* = 7.2 Hz),7.45 (t, 2H, *J* = 7.4 Hz), 7.51 (d, 2H, *J* = 7.2 Hz), 7.56 (d, 2H, *J* = 7.6 Hz), 7.99 (d, 2H, *J* = 8.0 Hz) 12.63 (br, 1H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ = 21.3, 125.6, 127.0, 127.5, 128.1, 128.2, 128.3, 128.7, 128.9, 129.1, 129.7, 131.5, 135.7, 137.4, 138.2, 146.0 ppm; IR (KBr, cm^{-1}): 3420, 3030, 1600, 1494, 1450, 1128, 1070, 969, 823, 764, 731, 695, 672.

*2-(4-Methoxyphenyl)-4,5-diphenyl-1*H-*imidazole* (**5**)

M.p.: 232–235 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ = 3.82 (s, 3H), 7.06 (d, 2H, *J* = 6.8 Hz), 7.23–7.58 (m, 10H), 8.05 (d, 2H, *J* = 7.2 Hz), 12.56 (br, 1H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ = 55.7, 114.6, 123.5, 126.9, 127.2, 127.5, 128.0, 128.1, 128.6, 128.8, 129.1, 131.6, 131.7, 135.8, 137.2, 146.0, 146.1, 159.9 ppm; IR (KBr, cm^{-1}): 3431, 3060, 1614, 1493, 1293, 1249, 1177, 1030, 968, 829, 765, 695.

*2-(4-Bromophenyl)-4,5-diphenyl-1*H-*imidazole* (**6**)

M.p.: 264–266 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ = 7.25 (t, 1H, *J* = 7.4 Hz), 7.33 (t, 2H, *J* = 7.0 Hz), 7.40 (t, 1H, *J* = 7.8 Hz), 7.46 (t, 2H, *J* = 7.2 Hz), 7.51–7.58 (m, 6H), 8.12 (d, 2H, *J* = 7.2 Hz), 12.82 (br, 1H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ = 127.1, 127.4, 127.6, 128.4, 128.7, 128.9, 129.2, 129.3, 129.6, 131.3, 133.3, 135.4, 137.8, 144.8 ppm; IR (KBr, cm^{-1}): 3445, 3028, 1602, 1501, 1483, 1449, 1432, 1129, 1069, 826, 765, 729, 695, 500.

*2-(2-Methoxyphenyl)-4,5-diphenyl-1*H-*imidazole* (**7**)

M.p.: 208–210 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ = 3.93 (s, 3H), 7.08 (t, 1H, *J* = 7.2 Hz), 7.17–7.24 (m, 2H), 7.30 (t, 2H, *J* = 7.2 Hz), 7.38–7.50 (m, 6H), 7.54 (d, 2H, *J* = 7.2 Hz), 8.06 (d, 1H, *J* = 7.6 Hz), 11.93 (br, 1H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ = 112.0, 119.3, 121.1, 126.9, 127.5, 127.8, 128.1, 128.6, 129.0, 129.1, 129.3, 130.3, 131.6, 135.7, 136.9, 143.5, 156.5 ppm; IR (KBr, cm^{-1}): 3450, 3063, 1601, 1585, 1481, 1470, 1446, 1250, 1099, 1018, 764, 747, 694, 613.

*2-(2,4-Dichlorophenyl)-4,5-diphenyl-1*H-*imidazole* (**8**)

M.p.: 178–180 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ = 7.26–7.59 (m, 11H), 7.79 (d, 1H, *J* = 7.6 Hz), 7.86 (d, 1H, *J* = 7.6 Hz), 12.76 (br, 1H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ = 127.1, 127.7, 127.9, 128.3, 128.7, 129.2, 129.3, 130.2, 131.2, 132.9, 133.1, 134.4, 135.4, 142.8 ppm; IR (KBr, cm^{-1}): 3446, 3069, 1594, 1552, 1501, 1476, 1426, 1322, 1135, 1101, 879, 768, 700, 497.

*2-(4-Nitrophenyl)-4,5-diphenyl-1*H-*imidazole* (**9**)

M.p.: 240–242 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ = 7.27–7.75 (m, 10H), 7.90–8.37 (m, 4H), 13.17 (br, 1H), ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ = 124.7, 126.2, 127.7, 129.0, 136.5, 143.8, 147.0 ppm; IR (KBr, cm^{-1}): 3393, 3081, 1600, 1582, 1512, 1442, 1336, 1245, 1109, 968, 853, 765, 695.

*2-Phenyl-4,5-di-p-tolyl-1*H-*imidazole* (**10**)

M.p.: 269–271 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ = 2.30 (s, 3H), 2.36 (s, 3H), 7.12 (d, 2H, *J* = 8.0 Hz), 7.25 (d, 2H, *J* = 8.0 Hz), 7.38–7.50 (m, 7H), 8.09 (d, 2H, *J* = 7.6 Hz), 12.61 (br, 1H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ = 21.2, 21.3, 125.6, 127.5, 128.2, 128.6, 128.7, 129.1, 129.2, 129.7, 130.8, 132.9, 136.0, 137.4, 137.5, 145.5 ppm; IR (KBr, cm^{-1}): 3435, 3032, 1520, 1497, 1460, 1411, 1322, 1126, 816, 721, 689.

*2,4,5-Tri-p-tolyl-1*H-*imidazole* (**11**)

M.p.: 238–240 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ = 2.30 (3H,

s), 2.36 (s, 6H), 7.12 (d, 2H, J = 8.0 Hz), 7.24 (d, 2H, J = 8.0 Hz), 7.29 (d, 2H, J = 8.0 Hz), 7.39 (d, 2H, J = 8.0 Hz), 7.45 (d, 2H, J = 8.0 Hz), 7.97 (d, 2H, J = 8.4 Hz), 12.51 (br, 1H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ = 21.2, 21.3, 21.4, 125.6, 127.5, 127.9, 128.2, 128.6, 128.7, 129.2, 129.6, 129.7, 132.9, 135.9, 137.2, 137.4, 138.0, 145.7 ppm; IR (KBr, cm^{-1}): 3442, 3021, 1520, 1500, 1431, 1319, 1125, 969, 818, 727, 503.

Acknowledgements

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