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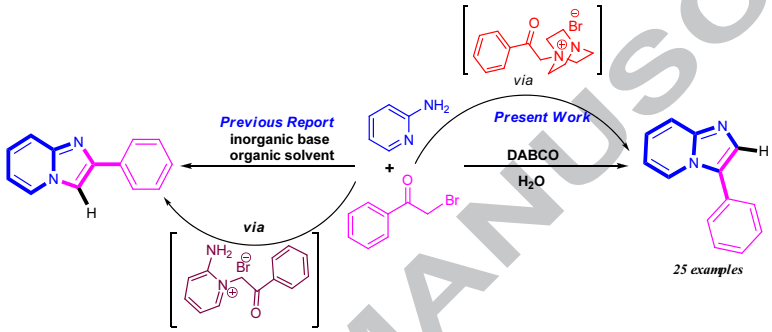

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DABCO catalyzed highly regioselective synthesis of fused imidazo-heterocycles in aqueous medium

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Abstract— An efficient regioselective synthesis of 3-aryl imidazo[1,2-a]pyridines, 5-aryl imidazo[2,1-b]thiazoles and 3-aryl benzo[*d*]imidazo[2,1-*b*][1,3]thiazoles is described by the reaction of phenacyl bromide with heterocyclic amine in presence of DABCO under aqueous medium. The method is applicable for a variety of substituted phenacyl bromides as well as 2-amino-heterocycles. An aqueous reaction medium, regioselectivity, mild reaction condition and high yield of products are the important features of this protocol.

Keywords: phenacyl bromide, 2-aminopyridine, regioselectivity, fused imidazo-heterocycles, DABCO, water.

Fused imidazo-heterocycles possesses significant biological activities like antiviral, antitumor, antimicrobial, herbicidal and immunosuppressive agents.[1] Additionally imidazo-heterocycles like imidazo-pyridines and imidazo-thiazoles constitute the core structure in some of the pharmaceutically active molecules.[2] Recently, imidazo-heterocycles has been reported as a ligand for detecting β-amyloid (Aβ) plaques in the brain, whose production is a pivotal event in the pathology of Alzheimer's disease.[3] In particular, the bioactive 3-aryl substituted imidazo-heterocycles, TP-003 (**I,** Figure 1) is an anxiolytic drug with anticonvulsant activity,[4] compound **II** is a ligand for GABA receptors used in treatment of adverse condition of the central nervous system including anxiety, convulsions and cognitive disorders.[5] Compound **III** also is a potent antimicrobial agent[6] (Figure 1)**.** Due to distinct pharmaceutical properties of imidazo-heterocycles, it is desirable to develop an efficient and convenient method for the synthesis of these molecules.

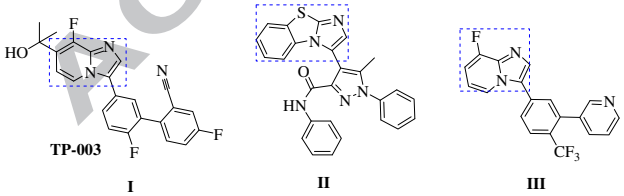


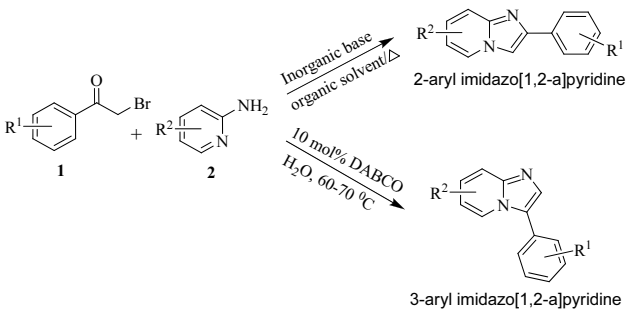
Figure 1. Representative examples of bioactive 3-aryl substituted imidazo-heterocyclic motifs.

There are scanty literature for the synthesis of imidazo-heterocycles.[7] However, the success of reported methods mostly depend on the use of organic solvents and inorganic bases.[8] Commonly, it is prepared by the condensation of heterocyclic amine with α-halo arylketone in ethanol at higher temperatures. Though the reported methods are satisfactory, they have some drawbacks such as use of expensive and harmful solvents, inorganic bases, higher temperature and longer reaction time (18-24 h). Moreover, the condensation reaction between heterocyclic amine with α-halo-arylketone in the presence of an inorganic base leads to the formation of 2-aryl imidazo[1,2-a]pyridines as a product (Scheme 1).[9]

The use of aqueous medium is a fascinating step in organic reactions which has attracted the increasing interest of chemist because of environmental and economical issue.[10] 1, 4-Diazabicyclo[2.2.2]octane (DABCO) has emerged as an efficient organic base which has been successfully used for various organic reactions like, Baylis–Hillman reaction,[11] cross-coupling reactions,[12] cyclization of *o*-alkynylaryl isocyanides.[13] However, to the best of our knowledge, present reaction have not yet been studied using organic base i.e. DABCO.

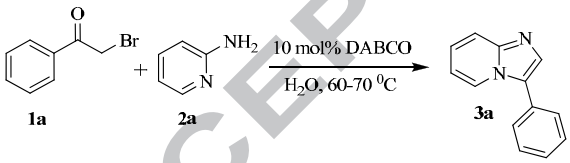
In continuation of our ongoing research on DABCO promoted reactions,[14] we observed an exciting result during our study which encouraged us to explore the effect of DABCO in order to understand the mechanistic aspects. Herein we wish to report an efficient and environmentally benign method for the regioselective synthesis of fused imidazo-heterocyclic system

by the reaction of phenacyl bromide with heterocyclic amine in presence of DABCO under aqueous medium.



Scheme 1. Regioselective synthesis of 3-aryl imidazo[1,2-a]pyridine derivatives

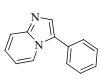
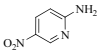
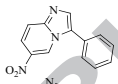
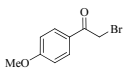
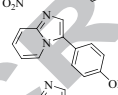
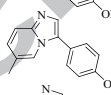
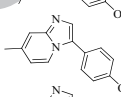
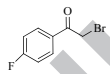
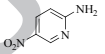
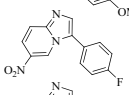
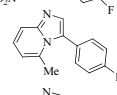
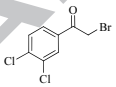
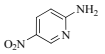
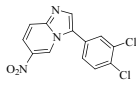
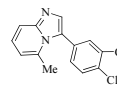
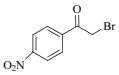
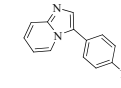
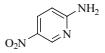
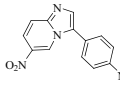
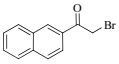
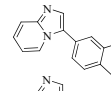
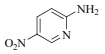
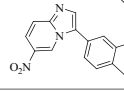
Initially, we attempted the reaction of phenacyl bromide (**1a**, 1mmol) with 2-aminopyridine (**2a**, 1mmol) in water (5 mL) in the presence of 10 mol % of DABCO. Though the reaction was sluggish at room temperature, it did improve at 60-70 °C affording 3-phenyl imidazo[1,2-a]pyridine (**3a)** as a sole product in 93% yield. The structure of product **3a** was confirmed by the spectral data.[15] Though the reaction of phenacyl bromide and 2-aminopyridine was reported[9] to give 2-phenyl imidazo[1,2-a]pyridine, the present protocol gave regioselectively 3-phenyl imidazo[1,2-a] pyridine. After extensive screening of different mole % of DABCO, we observed that 10 mol% of DABCO was sufficient for maximum conversion of product. On the basis of the above study, phenacyl bromide (1mmol), 2-aminopyridine (1mmol), DABCO (10 mol %) in water (5 mL) at 60-70 °C (Scheme 2, Table 1, entry a) were chosen as a set of optimized conditions[16] for the synthesis of 3-phenyl imidazo[1,2-a]pyridine.



Scheme 2. Reaction of phenacyl bromide with 2-aminopyridine

Next, we studied the scope of the reaction with respect to a variety of functionlized phenacyl bromides and substituted 2-aminopyridines under optimized reaction condition and the results are summarized in Table 1. Interestingly, a range of phenacyl bromides and substituted 2-aminopyridines participated well in this conversion to afford corresponding 3-aryl imidazo[1,2-a]pyridines (**3**) in good to excellent yield (Table 1, entries b-k). From Table 1, it is evident that, electronic factors of aryl ring on phenacyl bromides had shown some effect on the conversion. In general, substituted phenacyl bromides bearing electron-withdrawing groups (Table 1, entries d, e, h, i) reacted faster and furnish high yields whereas phenacyl bromide bearing electron-donating groups (Table 1, entries c-e) reacted slowly and gave comparatively less yield of product.

Table 1: Synthesis of fused 3-aryl imidazo[1,2-a]pyridines.[a]

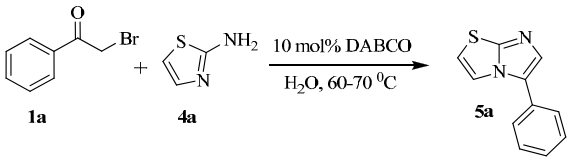
Entry	Phenacyl bromide (**1**)	Amines (**2**)	Product (**3**)	Time (h)	Yield %[b]
a			 **3a**[7a]	1	93
b	,,		 **3b**[8b]	2.5	86
c			 **3c**[15a]	3	82
d	,,		 **3d**	3	84
e	,,		 **3e**	4	83
f			 **3f**	2	92
g	,,		 **3g**	3	94
h			 **3h**	2.5	91
i	,,		 **3i**	3	95
j			 **3j**[21]	1	96
k	,,		 **3k**	4	92
l			 **3l**[21]	2.5	92
m	,,		 **3m**	3	83

[a] Reaction condition: phenacyl bromide (1 mmol), Amine (1 mmol) and DABCO (10 mol%) in water (5 ml) at 60 ^{O}C
[b] isolated Yields

Inspired by the results obtained from the reactions of phenacyl bromides with 2-aminopyridines, we extended our efforts to study the reaction of phenacyl bromides with 2-aminothiazoles and 2-aminobenzothiazoles under similar reaction condition. Accordingly, the reaction of phenacyl bromide (**1a)** with 2-aminothiazole (**4a)** in water (5 mL) in the presence of catalytic amount of DABCO (10 mol %) proceeded smoothly and resulted into corresponding 5-phenyl imidazo[2,1-b]thiazole (**5a**) in high yield (Scheme 3, Table 2, entry a).

Likewise, the reaction of phenacyl bromide (**1a**) with 6-methoxy benzo[d]thiazol-2-amine gave the corresponding 3-

phenyl benzo[*d*]imidazo[2,1-*b*][1,3]thiazole (**5b**) as a product (Table 2, entry b). The scope of the methodology is illustrated in Table 2 with respect to other phenacyl bromides and 2-aminothiazoles/2-aminobenzothiazoles. We found that, various substituted 2-aminothiazoles (Table 2, entries e, f, g, h, and j) and 2-aminobenzothiazoles (Table 2, entries c, d, i, k, and l) also reacted smoothly with phenacyl bromides resulting in the formation of respective 5-aryl imidazo[2,1-b]thiazole and 3-aryl benzo[*d*]imidazo[2,1-*b*][1,3]thiazole (**5**) derivatives with high yields.



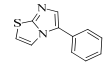
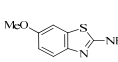
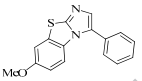
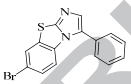
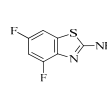
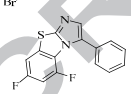
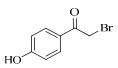
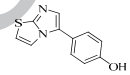
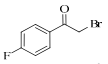
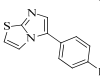
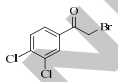
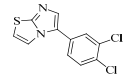
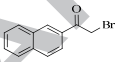
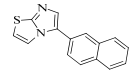
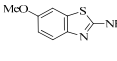
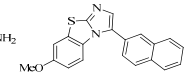
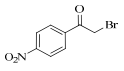
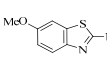
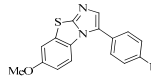
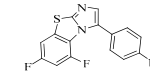
Scheme 3. Reaction of phenacyl bromide with 2-aminothiazole.

The efficiency of the protocol is demonstrated by the successful reaction of bicyclic phenacyl bromide. For example 2-bromo-1-(naphthalen-2-yl)ethanone also reacted smoothly with 2-aminothiazole and 6-methoxy-2-aminobenzothiazole (Table 2, entries h and i respectively) under optimized condition and gave corresponding product in good yield. This method provides the access for the synthesis of novel compounds (**3e-3g, 3i, 3k,** and **5c-5l**) which are not prepared earlier.

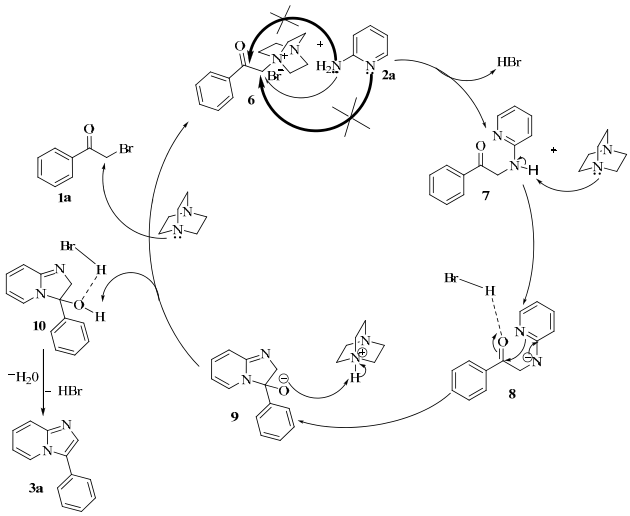
Generally the condensation of 2-aminopyridine and phenacyl bromide is sustained by the most favorable attack of ring nitrogen of pyridine due to the formation of highly stable pyridinium ion intermediate.[17] But technically the formation of stable pyridinium ion has been inhibited, which is a key step for the formation of 2-phenyl imidzo[1,2-a]pyridine by inserting DABCO as a catalyst. This catalyst forms a stable water soluble quaternary salt (**6**) (scheme 3) with phenacyl bromide.[18] The tentative mechanistic pathway for the synthesis of 3-phenyl imidazo[1,2-a]pyridine (**3a**) is proposed and shown in Scheme 4. We reasoned that initially phenacyl bromide reacts with DABCO to form quaternary salt (**6**). Further it reacts with amine to furnish intermediate (**7**)[19] which on cyclization followed by dehydration[20] leads to the desired product (**3a**). This regioselective formation of product (**3a**) may be attributed to DABCO which forms quaternary salt (**6**) and diverts the attack of primary amine on carbon near to quaternary nitrogen instead of carbonyl carbon (Scheme 4).

In conclusion, we have demonstrated DABCO catalyzed convenient and efficient method exclusively for the synthesis of 3-phenyl imidazo[1,2-a]pyridine in high yield. The aqueous medium makes the procedure more eco-friendly. This procedure is suitable for substituted heterocyclic amines and substituted phenacyl bromides. Moreover the method provides an access for the regioselective synthesis of 3-aryl imidazo[1,2-a]pyridine as a single isomeric product. We believe that the present method may be an attractive alternative over the earlier methods.

Table 2: Synthesis of fused 5-aryl imidazo[2,1-b]thiazole and 3-aryl benzo[*d*]imidazo[2,1-*b*][1,3]thiazole derivatives.[a]

Entry	Phenacyl bromide (**1**)	Amines (**4**)	Product (**5**)	Time (h)	Yield %[b]
a			 **5a**[7b]	2	91
b	"		 **5b**[22]	3	93
c	"		 **5c**	3.5	90
d	"		 **5d**	3	92
e			 **5e**	4	83
f			 **5f**	2	90
g			 **5g**	2	91
h			 **5h**	2.5	90
i	"		 **5i**	3	90
j			 **5j**	1	95
k	"		 **5k**	3	93
l	"		 **5l**	3	90

[a] Reaction condition: phenacyl bromide (1 mmol), Amine (1 mmol) and DABCO (10 mol%) in water (5 ml) at 60 ^{0}C
[b] isolated Yields



Scheme 4: Plausible reaction pathway.

Acknowledgments

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16. **General procedure:** The mixture of phenacyl bromide (1 mmol), 2-aminopyridine (1 mmol) and DABCO (10 mol %) was stirred in water (5 mL) at 60-70 °C for 1 h. The reaction was monitored by TLC. After the completion of the reaction, it was extracted with ethyl acetate (3x5 mL). The combined organic layer washed with brine solution, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The crude products were purified by silica gel column chromatography (ethyl acetate/hexane, 1:10). All compounds were characterized by M.P., NMR, Mass, HRMS and IR spectral data.
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Spectral data for representative compounds:

Compound 3d: White solid. mp 152–154 °C; ^{1}H NMR (300 MHz, $CDCl_3$+DMSO-d_6): δ = 7.92 (s, 1H), 7.56-7.49 (m, 3H), 7.45 (s, 1H), 7.19 (d, *J* = 8.31 Hz, 1H), 7.12 (d, *J* = 7.36 Hz, 2H), 3.84 (s, 3H), 2.28 (s, 3H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$+DMSO d_6): δ = 159.82, 146.02, 143.93, 137.67, 132.04, 127.28, 125.35, 125.02, 117.14, 113.65, 55.28, 20.05 ppm. IR (KBr) ν = 3123, 1562, 1245, 1423, 715 cm^{-1}; MS (ESI) *m/z* 261(M+Na)$^{+}$. HRMS (ESI): *m/z* calcd. For $C_{15}H_{14}N_2NaO[M+Na]^{+}$ = 261.09983, found 261.09960.

Compound 3e: White solid. mp 145–147 °C; ^{1}H NMR (300 MHz, $CDCl_3$+DMSO-d_6): δ = 7.98 (d, *J* = 8.86 Hz, 1H), 7.50 (s, 1H), 7.48-7.29 (m, 3H), 7.15 (d, *J* = 7.17 Hz, 2H), 6.54 (d, *J* = 8.31 Hz, 1H), 3.86 (s, 3H), 2.28 (s, 3H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$+DMSO d_6): δ = 159.90, 145.95, 138.67, 136.23, 134.19, 127.26, 125.24, 124.13, 119.14, 113.47, 55.28, 20.05 ppm. IR (KBr) ν = 3119, 1570, 1435, 1259, 722 cm^{-1}; MS (ESI) *m/z* 261(M+Na)$^{+}$. HRMS (ESI): *m/z* calcd. For $C_{15}H_{14}N_2NaO[M+Na]^{+}$ = 261.09983, found 261.09960.

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DABCO catalyzed highly regioselective synthesis of fused imidazo-heterocycles in aqueous medium

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