An Efficient Synthesis of Pyrazolo[3,4-*b*]quinolin-5(6*H*)-one Derivatives in Ionic Liquid

Da-Qing Shi[a]* and Fang Yang[b]

[a]Key Laboratory of Organic Synthesis of Jiangsu Province, College of Chemistry, Chemical Engineering and Materials Science, Soochow University, Suzhou 215123, People's Republic of China
[b]College of Chemistry and Chemical Engineering, Xuzhou Normal University, Xuzhou 221116, People's Republic of China
*E-mail: dqshi@suda.edu.cn

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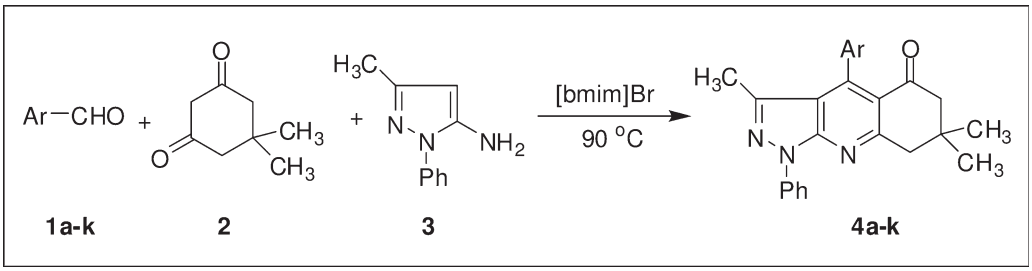

A series of 4-aryl-3,7,7-trimethyl-1-phenyl-7,8-dihydro-1*H*-pyrazolo[3,4-*b*]quinolin-5(6*H*)-ones were synthesized via the three-component reaction of aromatic aldehydes, 5,5-dimethyl-1,3-cyclohexandione and 5-amino-3-methyl-1-phenylpyrazole in ionic liquid without using any catalyst. This protocol has the advantages of easier work-up, milder reaction conditions, short reaction time, and environmentally benign procedure.

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INTRODUCTION

Pyrazole derivatives exhibit pharmacological activities such as hypotensive, antibacterial, antiinflammatory, and antitumor properties. In particular, condensed pyrazole are known for various biological activities, *e. g.* pyrazolo[3,4-*b*]quinolines have exhibited potential antiviral [1], antimalarial [2], and serum cholesterol lowering activities. A number of methods are available for the synthesis of pyrazolo[3,4-*b*]quinolines[3], the most efficient and commonly used method involves the reaction of aromatic aldehyde, 1,3-dicarbonyl compound and aminopyrazole in organic solvent such as EtOH [4]. All the reported methods of their synthesis have limitations of poor yields, difficult workups, and effluent pollution.

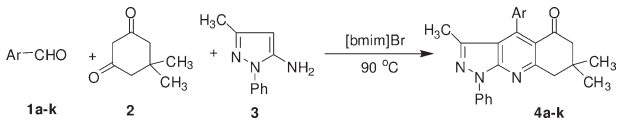
Multicomponent reactions (MCRs) in which multiple reactions are combined into one synthetic operation have been used extensively to form carbon–carbon bonds in the synthetic chemistry [5]. Such reactions offer a wide range of possibilities for the efficient construction of highly complex molecules in a single procedural step, thus avoiding the complicated purification operations and allowing savings of both solvents and reagents. In the past decade there has been tremendous development in three- and four-component reactions and great efforts continue to be made for developing new MCRs [6].

Room temperature ionic liquids, especially those based on 1-alkyl-3-methylimidazolium cations, have shown great promise as an attractive alterative to conventional organic solvents, and more attention has been currently focused on organic reactions promoted by ionic liquids [7]. They are nonvolatile, recyclable, nonexplosive, easily operable, and thermally robust [8]. There are many reports concerning the applications of ionic liquid in organic reactions, such as Friedel-Crafts reactions [9], Diels-Alder reactions [10], Heck reactions [11], Pechmann condensatios [12], Biginelli reactions [13], Beckmann rearrangements [14], and other reactions [15]. As part of our current studies on the developments of new routes to heterocyclic system in ionic liquid [16], we herein described a facile synthesis of pyrazolo[3,4-*b*]quinolin-5(6*H*)-one derivatives by the three-component reaction of aromatic aldehyde, 5,5-dimethyl-1,3-cyclohexandione and 5-amino-3-methyl-1-phenylpyrazole in ionic liquid without using any catalyst (Scheme 1).

RESULTS AND DISCUSSION

Choosing an appropriate solvent is of crucial importance for the successful organic synthesis. To search for the optimal reaction solvent, the reaction of 4-hydroxybenzaldehyde (**1a**) 5,5-dimethyl-1,3-cyclohexandione (**2**)

Scheme 1



and 5-amino-3-methyl-1-phenylpyrazole (**3**) was examined using a variety of ionic liquids 3-butyl-1-methylimidazolium bromide ([bmim]Br), [bmim]BF_4, 3-propyl-1-methylimidazolium bromide ([pmim]Br), and conventional reaction solvents; DMF, acetic acid, acetone, and ethanol, at different reaction temperature for the synthesis of the pyrazolo[3,4-b]quinolin-5(6*H*)-one (**4a**). The results are summarized in Table 1.

It can be seen from the Table 1 that the best result was obtained when the reaction was carried out in [bmim]Br at 90°C (Table 1, entry 1). [bmim]Br was chosen as the solvent for all further reactions as it is environmentally friendly and the toxic organic reagents can be avoided. Under these optimized reaction conditions, a series of pyrazolo[3,4-*b*]quinolin-5(6*H*)-one derivatives **4** were synthesized. The results are summarized in Table 2.

As shown in Table 2, this protocol could be applied to the aromatic aldehydes with either electron-withdrawing groups (such as halide groups) or electron-donating groups (such as alkyl and alkoxyl groups). The products were different from those literature reported. The reaction of 5-amino-3-methyl-1-phenylpyrazole with dimedone and aromatic aldehyde in ethanol afforded 4-aryl-7,7-dimethyl-4,7,8,9-tetrahydro-6H-pyrazolo[3,4-b] quinolin-5-one[4a]. The structures of the products were established on the spectroscopic data (IR, ^{1}H NMR, and HRMS).

Though the detailed mechanism of this reaction has not been clarified yet, the formation of **4** can be explained by the possible mechanism presented in Scheme 2. The reaction occurs via an initial formation of the α,β-unsaturated ketone, from the condensation of aldehyde and 5,5-dimethyl-1,3-cyclohexanedione as shown in Scheme 2, which suffers nucleophilic attack to give the Michael adduct [A]. The intermediate [A] then isomerizates, cyclizes, dehydrates and subsequently losses a hydrogen molecule to afford the fully aromatized compound. This type of hydrogen loss is well documented [17].

In summary, we have developed an efficient three-component reaction of aromatic aldehydes, 5,5-dimethyl-1,3-cyclohexanedione and 5-amino-3-methyl-1-phenylpyrazole for the synthesis of pyrazolo[3,4-*b*]quinolne-5(6*H*)-one derivatives using ionic liquid as solvent. Compared to the previous methods, this new protocol has the advantages of easier work-up, milder reaction conditions, short reaction time, and environmentally benign procedure.

Table 1

Solvent optimization for the synthesis of **4a**.

Entry	Solvent	Reaction temperature (°C)	Reaction time (h)	Isolated yield (%)
1	[bmim]Br	90	2	98
2	[bmim]BF_4	90	2	94
3	[pmim]Br	90	2	93
4	DMF	100	8	20
5	HOAc	100	8	52
6	acetone	Reflux	8	n. r.
7	ethanol	Reflux	8	39

Table 2

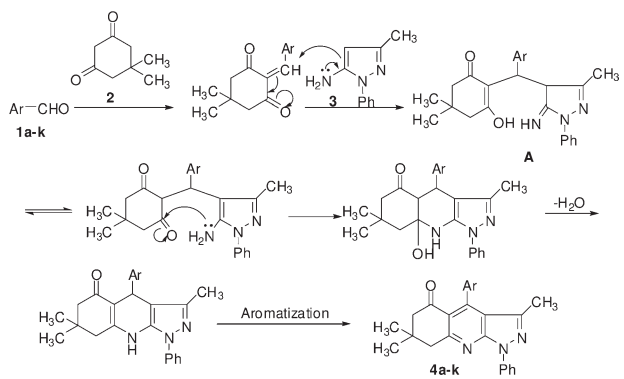
The synthesis of **4a-k** in ionic liquid [bmim]Br.

Entry	Ar	Time (h)	Yield (%)
4a	4-HOC_6H_4	2	98
4b	4-$CH_3C_6H_4$	1.5	95
4c	4-FC_6H_4	2	95
4d	4-ClC_6H_4	2	94
4e	4-BrC_6H_4	2.5	93
4f	2-NO_2-5-ClC_6H_3	3.5	90
4g	3,4-$Cl_2C_6H_3$	4	78
4h	2,4-$Cl_2C_6H_3$	4.5	79
4i	4-$CH_3OC_6H_4$	3	85
4j	3,4-$OCH_2OC_6H_3$	4	68
4k	4-$(CH_3)_2NC_6H_4$	3	86

EXPERIMENTAL

Commercial solvents and reagents were used as received. Melting points are uncorrected. IR spectra were recorded on a FTIR Temsor 27 spectrometer in KBr with absorptions in cm^{-1}. ^{1}H NMR was determined on Bruker DPX-400 MHz spectrometer in DMSO-d_6 solution. *J* values are in Hz. Chemical shifts are expressed in ppm downfield from internal standard TMS. HRMS data were obtained using Micromass TOF-

Scheme 2



MS instrument. Starting materials used were obtained from Alfa Aesar and used without further purification. Ionic liquids were prepared according to the stardard method [18].

General procedure for the synthesis of pyrazolo[3,4-*b*]quinolne-5-one derivatives 4. A dry 50 mL flask was charged with aromatic aldehyde **1** (1 mmol), 5,5-dimethyl-1,3-cyclohexandione **2** (1 mmol), 5-amino-3-methyl-1-phenyl-pyrazole **3** (1 mmol), and ionic liquid [bmim]Br (2 mL). The mixture was stirred at 90°C for 1.5–4.5 h to complete the reaction (monitored by TLC), then 5 mL water was added. The solid was filtered off and washed with water. The crude product was purified by recrystallization from the mixture of DMF and ethanol to give **4**.

4-(4-Hydroxyphenyl)-3,7,7-trimethyl-1-phenyl-7,8-dihydro-1H-pyrazolo[3,4-b]quinolin-5(6H)-one (4a). Mp: 277–279°C; IR (potassium bromide): 3304, 2957, 1666, 1613, 1594, 1571, 1557, 1512, 1472, 1455, 1433, 1385, 1265, 1124, 820, 758, 688 cm^{-1}; ^1H NMR (DMSO-d_6) δ: 1.07 (s, 6H, 2 × CH_3), 1.87 (s, 3H, CH_3), 2.51 (s, 2H, CH_2), 3.19 (s, 2H, CH_2), 6.84 (d, J = 8.4 Hz, 2H, ArH), 7.06 (d, J = 8.4 Hz, 2H, ArH), 7.35 (t, J = 7.6 Hz, 1H, ArH), 7.57 (t, J = 7.6 Hz, 2H, ArH), 8.24 (d, J = 8.4 Hz, 2H, ArH), 9.60 (s, 1H, OH); HRMS [Found: *m*/*z*: 397.1790 (M^+); Calcd for $C_{25}H_{23}N_3O_2$: M 397.1790].

4-(4-Methylphenyl)-3,7,7-trimethyl-1-phenyl-7,8-dihydro-1H-pyrazolo[3,4-b]quinolin-5(6H)-one (4b). Mp: 184–186°C; IR (potassium bromide): 3021, 2953, 1677, 1591, 1560, 1499, 1470, 1455, 1438, 1415, 1386, 1367, 1355, 1305, 1263, 1232, 1180, 908, 803, 752, 690 cm^{-1}; ^1H NMR (DMSO-d_6) δ: 1.06 (s, 6H, 2 × CH_3), 1.83 (s, 3H, CH_3), 2.37 (s, 3H, CH_3), 2.43 (s, 2H, CH_2), 3.15 (s, 2H, CH_2), 7.04 (d, J = 8.0 Hz, 2H, ArH), 7.19–7.23 (m, 3H, ArH), 7.44 (t, J = 7.6 Hz, 2H, ArH), 8.18 (d, J = 8.0 Hz, 2H, ArH); HRMS [Found: *m*/*z*: 395.2012 (M^+); Calcd for $C_{26}H_{25}N_3O$: M 395.1998].

4-(4-Fluorophenyl)-3,7,7-trimethyl-1-phenyl-7,8-dihydro-1H-pyrazolo[3,4-b]quinolin-5(6H)-one (4c). Mp. 172–174°C; IR (potassium bromide): 3062, 2955, 1679, 1596, 1562, 1509, 1470, 1455, 1381, 1308, 1260, 1232, 1093, 912, 848, 814, 753, 690 cm^{-1}; ^1H NMR (DMSO-d_6) δ: 1.08 (s, 6H, 2 × CH_3), 1.83 (s, 3H, CH_3), 2.51 (s, 2H, CH_2), 3.21 (s, 2H, CH_2), 7.27–7.38 (m, 5H, ArH), 7.58 (t, J = 8.0 Hz, 2H, ArH), 8.24 (d, J = 7.6 Hz, 2H, ArH); HRMS [Found: *m*/*z*: 399.1735 (M^+); Calcd for $C_{25}H_{22}FN_3O$: M 399.1747].

4-(4-Chlorophenyl)-3,7,7-trimethyl-1-phenyl-7,8-dihydro-1H-pyrazolo[3,4-b]quinolin-5(6H)-one (4d). Mp: 180–183°C; IR (potassium bromide): 2955, 1680, 1595, 1558, 1509, 1489, 1455, 1386, 1264, 1234, 982, 910, 859, 844, 810, 791, 752, 690 cm^{-1}; ^1H NMR (DMSO-d_6) δ: 1.08 (s, 6H, 2 × CH_3), 1.84 (s, 3H, CH_3), 2.51 (s, 2H, CH_2), 3.21 (s, 2H, CH_2), 7.32–7.39 (m, 3H, ArH), 7.52–7.60 (m, 4H, ArH), 8.24 (d, J = 8.0 Hz, 2H, ArH); HRMS [Found: *m*/*z*: 415.1434 (M^+); Calcd for $C_{25}H_{22}^{35}ClN_3O$: M 415.1451].

4-(4-Bromophenyl)-3,7,7-trimethyl-1-phenyl-7,8-dihydro-1H-pyrazolo[3,4-b]quinolin-5(6H)-one (4e). Mp: 188–189°C; IR (potassium bromide): 2954, 1679, 1591, 1558, 1511, 1498, 1486, 1455, 1386, 1367, 1305, 1263, 981, 909, 858, 842, 806, 751, 690 cm^{-1}; ^1H NMR (DMSO-d_6) δ: 1.08 (s, 6H, 2 × CH_3), 1.84 (s, 3H, CH_3), 2.51 (s, 2H, CH_2), 3.21 (s, 2H, CH_2), 7.27 (d, J = 8.0 Hz, 2H, ArH), 7.37 (t, J = 7.2 Hz, 1H, ArH), 7.58 (t, J = 7.6 Hz, 2H, ArH), 7.67 (d, J = 8.0 Hz, 2H, ArH), 8.24 (d, J = 8.0 Hz, 2H, ArH); HRMS [Found: *m*/*z*: 459.0961 (M^+); Calcd for $C_{25}H_{22}^{79}BrN_3O$: M 459.0946].

4-(5-Chloro-2-nitrophenyl)-3,7,7-trimethyl-1-phenyl-7,8-dihydro-1H-pyrazolo[3,4-b]quinolin-5(6H)-one (4f). Mp: 193–194°C; IR (potassium bromide): 3077, 2959, 1677, 1599, 1573, 1519, 1479, 1458, 1389, 1378, 1296, 1261, 986, 926, 845, 762, 690 cm^{-1}; ^1H NMR (DMSO-d_6) δ: 1.02 (s, 3H, CH_3), 1.11 (s, 3H, CH_3), 1.85 (s, 3H, CH_3), 2.44 (d, J = 16.4 Hz, 1H, CH), 2.59 (d, J = 16.4 Hz, 1H, CH), 3.23 (s, 2H, CH_2), 7.39 (t, J = 7.6 Hz, 1H, ArH), 7.60 (t, J = 7.6 Hz, 2H, ArH), 7.72 (s, 1H, ArH), 7.88 (dd, J_1 = 1.2 Hz, J_2 = 8.8 Hz, 1H, ArH), 8.25 (d, J = 8.0 Hz, 2H, ArH), 8.38 (d, J = 8.8 Hz, 1H, ArH); HRMS [Found: *m*/*z*: 460.1327 (M^+); Calcd for $C_{25}H_{21}^{35}ClN_4O_3$: M 460.1302].

4-(3,4-Dichorophenyl)-3,7,7-trimethyl-1-phenyl-7,8-dihydro-1H-pyrazolo[3,4-b]quinolin-5(6H)-one (4g). Mp: 179–181°C; IR (potassium bromide): 3063, 2954, 1685, 1597, 1567, 1508, 1468, 1383, 1355, 1291, 1265, 985, 925, 884, 863, 809, 791, 755, 690 cm^{-1}. ^1H NMR (DMSO-d_6) δ: 1.09 (s, 6H, 2 × CH_3), 1.89 (s, 3H, CH_3), 2.53 (s, 2H, CH_2), 3.22 (s, 2H, CH_2), 7.32∼7.40 (m, 2H, ArH), 7.59 (t, J = 7.6 Hz, 2H, ArH), 7.68 (d, J = 1.6 Hz, 1H, ArH), 7.74 (d, J = 8.4 Hz, 1H, ArH), 8.23 (d, J = 8.0 Hz, 2H, ArH); HRMS [Found: *m*/*z*: 449.1093 (M^+); Calcd for $C_{25}H_{21}^{35}Cl_2N_3O$: M 449.1062].

4-(2,4-Dichorophenyl)-3,7,7-trimethyl-1-phenyl-7,8-dihydro-1H-pyrazolo[3,4-b]quinolin-5(6H)-one (4h). Mp: 127–129°C; IR (potassium bromide): 3052, 2957, 1677, 1592, 1562, 1508, 1484, 1383, 1291, 1264, 985, 910, 860, 846, 813, 778, 755, 693 cm^{-1}. ^1H NMR (DMSO-d_6) δ: 1.06 (s, 3H, CH_3), 1.10 (s, 3H, CH_3), 1.88 (s, 3H, CH_3), 2.55 (s, 2H, CH_2), 3.24 (s, 2H, CH_2), 7.36–7.43 (m, 2H, ArH), 7.56–7.61 (m, 3H, ArH), 7.81 (d, J = 1.6 Hz, 1H, ArH), 8.24 (d, J = 8.0 Hz, 2H, ArH); HRMS [Found: *m*/*z*: 449.1082 (M^+); Calcd for $C_{25}H_{21}^{35}Cl_2N_3O$: M 449.1062].

4-(4-Methoxyphenyl)-3,7,7-trimethyl-1-phenyl-7,8-dihydro-1H-pyrazolo[3,4-b]quinolin-5(6H)-one (4i). Mp: 138–140°C; IR (potassium bromide): 3021, 2953, 1677, 1591, 1560, 1499, 1455, 1386, 1305, 1263, 982, 908, 803, 752, 690 cm^{-1}; ^1H NMR (DMSO-d_6) δ: 1.08 (s, 6H, 2 × CH_3), 1.81 (s, 3H, CH_3), 2.42 (s, 3H, CH_3), 2.51 (s, 2H, CH_2), 3.21 (s, 2H, CH_2), 7.16 (d, J = 8.0 Hz, 2H, ArH), 7.27 (d, J = 8.0 Hz, 2H, ArH), 7.36 (t, J = 7.6 Hz, 1H, ArH), 7.58 (t, J = 8.0 Hz, 2H, ArH), 8.24 (d, J = 8.0 Hz, 2H, ArH); HRMS [Found: *m*/*z*: 411.1926 (M^+); Calcd for $C_{26}H_{25}N_3O_2$: M 411.1947].

4-(3,4-Methylenedioxyphenyl)-3,7,7-trimethyl-1-phenyl-7,8-dihydro-1H-pyrazolo[3,4-b]quinolin-5(6H)-one (4j). Mp: 175–177°C; IR (potassium bromide): 3059, 2955, 1672, 1621, 1598, 1557, 1506, 1489, 1381, 1365, 1291, 1255, 987, 934, 907, 886, 876, 810, 788, 752 cm^{-1}; ^1H NMR (DMSO-d_6) δ: 1.07 (s, 6H, 2 × CH_3), 1.93 (s, 3H, CH_3), 2.51 (s, 2H, CH_2), 3.19 (s, 2H, CH_2), 6.12 (d, J = 8.0 Hz, 2H, OCH_2O), 6.72 (d, J = 7.6 Hz, 1H, ArH), 6.89 (s, 1H, ArH), 7.00 (d, J = 8.0 Hz, 1H, ArH), 7.36 (t, J = 7.2 Hz, 1H, ArH), 7.58 (t, J = 7.6 Hz, 2H, ArH), 8.24 (d, J = 8.0 Hz, 2H, ArH); HRMS [Found: *m*/*z*: 425.1727 (M^+); Calcd for $C_{26}H_{23}N_3O_3$: M 425.1739].

4-(4-Dimethylaminophenyl)-3,7,7-trimethyl-1-phenyl-7,8-dihydro-1H-pyrazolo[3,4-b]quinolin-5(6H)-one (4k). Mp: 194–196°C; IR (potassium bromide): 2954, 1687, 1612, 1598, 1567, 1514, 1506, 1490, 1473, 1385, 1364, 1283, 1260, 983, 947, 816, 804, 766, 692 cm^{-1}; ^1H NMR (DMSO-d_6) δ: 1.07 (s, 6H, 2 × CH_3), 1.92 (s, 3H, CH_3), 2.51 (s, 2H, CH_2), 2.99 (s, 6H, $(CH_3)_2N$), 3.18 (s, 2H, CH_2), 6.78 (d, J = 8.4 Hz, 2H, ArH), 7.09 (d, J = 8.4 Hz, 2H, ArH), 7.35 (t, J = 7.6 Hz, 1H,

ArH), 7.57 (t, $J = 7.6$ Hz, 2H, ArH), 8.25 (d, $J = 8.0$ Hz, 2H, ArH); HRMS [Found: *m*/*z*: 424.2246 (M^+); Calcd for $C_{27}H_{28}N_4O$: M 424.2263].

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