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Catalyst-Free One-Pot Synthesis of 2,4,6-Triaryl-1,4-dihydropyridines in Ionic Liquid and Their Catalyzed Activity on Two Simple Diels–Alder Reactions

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Abstract: A series of 2,4,6-triaryl-1,4-dihydropyridines bearing a hydroxyl group was synthesized greenly by the cascade aldol/Michael/addition reaction of aromatic aldehyde, acetophenone, and NH_4OAc (1:2:excessive) in ionic liquid [BmIm][BF_4] without any catalyst. The results of their catalyzed activity on two simple Diels–Alder reactions indicated that this kind of compound containing a poly aryl ring can be used as an effective catalyst on the Diels–Alder reaction. This method, because of its environmental friendliness, simplicity, mild conditions, effectiveness, and lower costs, is suitable for the synthesis of arrays of compounds.

Keywords: catalyzed activity, Diels–Alder reaction, synthesis, 2,4,6-triaryl-1,4-dihydropyridines

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INTRODUCTION

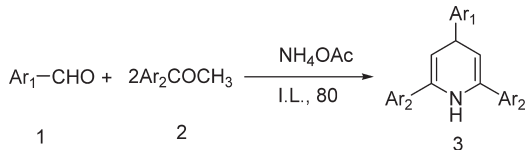
1,4-Dihydropyridines are important Ca^{2+}-channel regulators and are mainly used to treat cardiovascular disease, such as hypertension and congestive cardiopathy.[1–4] 2,4,6-Triaryl-1,4-dihydropyridines are useful intermediates in the synthesis of Kröhnke-type pyridines (2,4,6-triarylpyridines).[5] So, the development of new methods for the synthesis of this kind of compound is an ever-expanding area in organic chemistry and medicinal chemistry.

In general, they were synthesized by the Hantzsch reaction.[6–10] Eynde and coworkers developed a synthesis with higher yields than the one from pyridine, aldehydes, and $SOCl_2$ in CH_2Cl_2.[11] Evdokimov and coworkers[12] obtained target molecules through the reaction of aldehyde, malononitrile, and sulfur alcohol in ethanol. Breitenbucher and coworkers[13] developed a resin-support route to such compounds, but violent conditions (140°C, xylene, trifluoroacectic acid (TFA)) were necessary. These methods always used volatile and very toxic organic solvents and displayed only moderate to low final yields; at least four steps were necessary. To adopt the principles of green chemistry, Alajarin et al.[14] reported a microwave-assisted synthesis using ethanol as solvent in a few minutes, but it had poor recyclability and was difficult to industrialize. Most of products were symmetric, and there were few reports of 2,4,6-triaryl-1,4-dihydropyridine in these methods for asymmetric 1,4-dihydropyridine. Probably because of the deactivation and steric effect of the hydroxyl group on the carbonyl group, this kind of reaction, including 2′-hydroxyacetophenone, is difficult and has never been reported. As one hydroxyl group is added on the benzene ring, the yield will reduce 30–40%.[15] We have to conquer this difficulty because the hydroxyl group is a general group in the structure of most natural products and often displays biological activity.[16]

Green chemistry is the utilization of a set of principles that reduces or eliminates the use or products. Ionic liquid, which has unique properties of nonvolatility, nonflammability, recyclability, and ability to dissolve a wide range of materials, is useful for generating hazardous substances in the design, manufacture, and application of chemicals and has been the subject of considerable interest as environmentally benign reaction media in organic synthesis. Recently, many reports about the applications of ionic liquids have been published.[17–21]

Herein, we observe a new mild and simple protocol for the synthesis of 2,4,6-triaryl-1,4-dihydropyridines through the reaction of aromatic aldehydes with acetophenone and NH_4OAc in one pot (Scheme 1). Their catalytic functions on two simple Diels–Alder reactions were also studied.

At the onset of the research, an effort was made to establish the best condition for the model reaction of 4-cyanobenzaldehyde, 2′-hydroxyacetophenone, and ammonium acetate. Representative results are summarized in Table 1. [BmIm][BF_4] was selected as the most effective solvent to generated the desired product due to its biggest pK_a value of HBF_4.



Scheme 1. Synthesis of 2,4,6,-triaryl-1,4-dihydropyridines 3.

The scope of the reaction was investigated, and the results are summarized in Table 2. It indicates that the steric effect of substituted group on the aryl group of the aldehyde strongly influenced the yields. When aromatic aldehydes has substitutes in the *o*-position reacted with 2′-hydroxyacetophenone, the reaction time were a little longer and the yields were much lower compared with *m*- or *p*-aromatic aldehydes because of steric hindrance. 3′-Nitroacetophenone and 4′-nitroacetophenone gave high yields (56.7–65.6%), and the reaction times were shorter than that of 2′-hydroxyacetophenone. The electronic nature of the substitutes had almost no influence on the yields. 4′-Chlorobenzaldehyde and 4′-bromobenzaldehyde reacted with 3′-nitroacetophenone to give higher yields than others.

Why did the Mannich reaction not take place in this experiment? We speculated the reason was that the ionic liquid weakened the nucleophilicity of NH_3 released from NH_4OAc. That proves that ionic liquid has good selectivity and is superior as a reaction medium.

By far, there were no reports about the Diels–Alder reaction catalyzed by 2,4,6-triaryl-1,4-dihydropyridines. Because 2,4,6-triarylpyranium (TP^+) had been used as a photoinduced electron-transfer reagent,[22] we used our products to catalyze two simple Diels–Alder reactions, and the results are shown in Table 3.

Table 3 indicated that all of products could increase the yields and shorten the reaction time of both Diels–Alder reactions. Theoretically, as the catalyst of a concerted reaction, their optimal catalytic activity should come from this kind of structure in which both electron-withdrawing group (EWG) and

Table 1. Effect of ionic liquid on the yield of **3a**

IL	pK_a^a	Isolated yield (%)
[BmIm[b]][Br]	−9	44.0
[BmIm][BF_4]	0.5	63.5
[BmIm][ClO_4]	−11	10.1
[bpy[c]][Br]	−9	27.6
[bpy][BF_4]	0.5	38.6
[bpy][ClO_4]	−11	9.1

[a]The pK_a value of the parent acid of the anions.
[b][BmIm] = 1-n-butyl-3-methylimidazolium.
[c][bpy]= 1-n-butylpyridinium.

Table 2. Synthesis of 2,4,6-triaryl-1,4-dihydropyridines

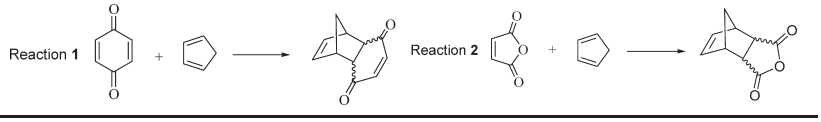
Compound	Ar_1	Ar_2	Mp(°C)	Yields (%)	Times (h)
3a	4-CNC_6H_4	2-OHC_6H_4	>300	63.5	24
3b	4-ClC_6H_4	2-OHC_6H_4	286.5–287.1	63.3	20
3c	4-BrC_6H_4	2-OHC_6H_4	253.7–254.8	68.5	23
3d	2-$OCH_3C_6H_4$	2-OHC_6H_4	235.9–236.7	54.4	24
3e	2-ClC_6H_4	4-$NO_2C_6H_4$	186.5–187.6	66.7	24
3f	4-ClC_6H_4	3-$NO_2C_6H_4$	189.5–190.2	78.2	23
3g	4-BrC_6H_4	3-$NO_2C_6H_4$	187.3–188.6	75.6	24

electron-donating group (EDG) were contained. This had been confirmed by correlative experiments, especially in reaction **1**: compound **3a–d** gave higher yields than others. If the electronic nature of two substitutents were uniform, the EWG one (**3e–g**) showed better catalytic activity than EDG (**3d**). From the comparability in structure between 2,4,6-triaryl-1,4-dihydropyridines and 2,4,6-triarylpyranium, we supposed that this reaction might also be rationalized as a photo-electron-transform (PET)-catalyzed a synchronous cation radical Diels–Alder reaction. The catalytic activity of compound **3** may be contribute to its long conjugated system and nonplanar framework, both of which are propitious to electron transfer from diene to dienophile.[23] Further experiments must be carried out to explain it.

EXPERIMENTAL

Melting points were measured with a Fisher-Johns melting-point apparatus without correction. IR spectra were recorded on a Nicolet Nexus 670

Table 3. Catalyzed function of **3** on two simple Diels–Alder reactions



Catalyst	Time (min)	Yield (%)	Catalyst	Time (min)	Yield (%)
—	60	44 (44)[24]	—	156	75.1
3a	60	68.6	**3a**	154	88.7
3b	55	69.4	**3b**	153	85.5
3c	53	70.1	**3c**	149	85.2
3d	54	50.8	**3d**	149	80.2
3e	50	52.3	**3e**	152	83.3
3f	52	58.6	**3f**	150	81.6
3g	56	59.3	**3g**	150	82.1

spectrometer in KBr. The proton nuclear magnetic resonance (^{1}H NMR) spectra were measured on a Bruker AM-400 spectrometer with Me_4Si (TMS) as the internal reference and DMSO-d_6 as solvent. Elemental analyses were performed by a Carlo Erba 1106 elemental analysis meter and a Foss-Heraeus Vario EL instrument. Mass spectra were determined on a Finnigan 8230 mass spectrometer. Ionic liquids were synthesized according to the literature.[18,19]

General Procedure

A dry 50-mL flask was charged with aromatic aldehydes (**1**) (1 mmol), 2-hydroxylacetophenone (**2**) (2 mmol), NH_4OAc (5 mmol), and ionic liquid (2 mL). The mixture was stirred at 80°C for 20–24 h and then was poured into water (40 mL). The precipitate was washed with water two or three times and purified by column chromatography to give 2,4,6-triaryl-1,4-dihydropyridines **(3)**. Diels–Alder reactions were run according to the literature method.[24]

Data

3a: 4-(4-Cyanophenyl)-2,6-bi(2-hydroxyphenyl)-1,4-dihydropyridine

IR (KBr, ν, cm^{-1}): 3350, 3101, 2230, 1597, 1561, 1525, 1416, 1224, 847, 750, 636; ^{1}H NMR (400 MHz, DMSO-d_6) (δ, ppm): 8.81 (2H, s, pyridine-H), 8.60–8.65 (4H, m, Ar-H), 8.36 (1H, d, $J = 8.0$ Hz, Ar-H), 8.10 (4H, d, $J = 8.4$ Hz, Ar-H), 7.85 (1H, s, NH), 7.47 (1H, t, $J = 8.4$ Hz, Ar-H), 7.05–7.10 (2H, m, Ar-H), 6.25 (1H, s, NH), 4.41–4.44 (1H, m, CH). EI-MS (m/z): 366.1 $[M]^+$. $C_{24}H_{18}N_2O_2$ found (%): C, 78.58; H, 4.85; N, 7.60; calcd. (%): C, 78.67; H, 4.95; N, 7.65.

3b: 4-(4-Chlorophenyl)-2,6-bi(2-hydroxyphenyl)-1,4-dihydropyridine

IR (KBr, ν, cm^{-1}): 3390, 3028, 2680, 1606, 1541, 1494, 1486, 1411, 1295, 1213, 1093, 982, 831, 756; ^{1}H NMR (400 MHz, DMSO-d_6) (δ, ppm): 8.13 (2H, d, $J = 7.6$ Hz, Ar-H), 7.87 (2H, d, $J = 8.0$ Hz, Ar-H), 7.80 (2H, s, pyridine-H), 7.41 (2H, dd, $^3J = 8.4$ Hz, $^4J = 1.6$ Hz, Ar-H), 7.33 (1H, t, $J = 8.0$ Hz, Ar-H), 7.23 (1H, d, $J = 8.0$ Hz, Ar-H), 7.19 (1H, d, $J = 8.0$ Hz, Ar-H), 7.14 (1H, d, $J = 8.0$ Hz, Ar-H), 7.08 (1H, d, $J = 8.4$ Hz, Ar-H), 6.98 (1H, t, $J = 7.2$ Hz, Ar-H), 6.90 (1H, d, $J = 8.4$ Hz, Ar-H), 6.32 (1H, s, NH), 4.42–4.45 (1H, m, CH). EI-MS (m/z): 375.1 $[M]^+$. $C_{23}H_{18}ClNO_2$ found (%): C, 73.58; H, 4.85; N, 3.70; calcd. (%): C, 73.50; H, 4.83; N, 3.73.

3c: 4-(4-Bromophenyl)-2,6-bi(2-hydroxyphenyl)-1,4-dihydropyridine

IR (KBr, ν, cm^{-1}): 3350, 3301, 3179, 3020, 1600, 1540, 1490, 1407, 1212, 982, 829, 756; ^{1}H NMR (400 MHz, DMSO-d_6) (δ, ppm): 8.04 (2H, s, pyridine-H), 7.94 (1H, dd, $^3J = 7.6$ Hz, $^4J = 1.2$ Hz, Ar-H), 7.54 (2H, d, $J = 8.4$ Hz, Ar-H), 7.49 (2H, d, $J = 8.4$ Hz, Ar-H), 7.45 (1H, d, $J = 8.0$ Hz, Ar-H), 7.35 (2H, d, $J = 8.4$ Hz, Ar-H), 7.14 (2H, d, $J = 8.4$ Hz, Ar-H), 7.02 (2H, t, $J = 8.0$ Hz, Ar-H), 6.95 (1H, d, $J = 8.4$ Hz, Ar-H), 6.52 (1H, s, NH), 4.40–4.43 (1H, m, CH). EI-MS (m/z): 419.1 $[M]^+$; $C_{23}H_{18}BrNO_2$ found (%): C, 65.58; H, 4.35; N, 3.40; calcd. (%): C, 65.73; H, 4.32; N, 3.33.

3d: 4-(2-Methoxylphenyl)-2,6-bi(2-hydroxyphenyl)-1,4-dihydropyridine

IR (KBr, ν, cm^{-1}): 3360, 3347, 2570, 1600, 1532, 1426, 1308, 1072, 967, 852, 773; ^{1}H NMR (400 MHz, DMSO-d_6) (δ, ppm): 11.09 (1H, s, OH), 7.96 (2H, s, pyridine-H), 7.88 (1H, d, $J = 8.0$ Hz, Ar-H), 7.65 (1H, d, $J = 8.0$ Hz, Ar-H), 7.12–7.16 (1H, m, Ar-H), 6.84–6.93 (2H, m, Ar-H), 6.71 (2H, t, $J = 8.0$ Hz, Ar-H), 6.02 (1H, s, NH), 3.99–4.02 (1H, m, CH), 3.48 (3H, s, OCH_3). EI-MS (m/z): 371.2 $[M]^+$ $C_{24}H_{21}NO_3$ found (%): C, 77.57; H, 5.80; N, 3.70; calcd. (%): C, 77.61; H, 5.70; N, 3.77.

3e: 4-(2-Chlorophenyl)-2,6-bi(4-nitrophenyl)-1,4-dihydropyridine

IR (KBr, ν, cm^{-1}): 3348, 3306, 3106, 2807, 1642, 1598, 1513, 1350, 1103, 863, 756, 693; ^{1}H NMR (400 MHz, DMSO-d_6) (δ, ppm): 8.34 (2H, s, pyridine-H), 8.29 (2H, d, $J = 8.8$ Hz, Ar-H), 8.16 (1H, d, $J = 8.4$ Hz, Ar-H), 8.10 (2H, d, $J = 9.2$ Hz, Ar-H), 7.63 (1H, dd, $^3J = 7.6$ Hz, $^4J = 1.6$ Hz, Ar-H), 7.53 (2H, dd, $^3J = 8.0$ Hz, $^4J = 2.0$ Hz, Ar-H), 7.44–7.47 (2H, m, Ar-H), 7.36 (2H, dd, $^3J = 8.8$ Hz, $^4J = 1.6$ Hz, Ar-H), 6.23 (1H, s, NH), 4.39–4.42 (1H, m, CH). EI-MS (m/z): 433.2 $[M]^+$. $C_{23}H_{16}ClN_3O_4$ found (%): C, 63.57; H, 3.80; N, 9.70; calcd. (%): C, 63.67; H, 3.72; N, 9.69.

3f: 4-(4-Chlorophenyl)-2,6-bi(3-nitrophenyl)-1,4-dihydropyridine

IR (KBr, ν, cm^{-1}): 3448, 3092, 2740, 2373, 1602, 1519, 1355, 883, 729, 674; ^{1}H NMR (400 MHz, DMSO-d_6) (δ, ppm): 9.03 (2H, s, pyridine-H), 8.64 (1H, d, $J = 8.0$ Hz, Ar-H), 8.49 (2H, dd, $^3J = 8.0$ Hz, $^4J = 1.6$ Hz, Ar-H), 8.38 (2H, d, $J = 8.0$ Hz, Ar-H), 7.76 (2H, t, $J = 8.0$ Hz, Ar-H), 7.65 (1H, d, $J = 8.4$ Hz, Ar-H), 6.24 (1H, s, NH), 4.52–6.57 (1H, m, CH). EI-MS (m/z): 433.2 $[M]^+$. $C_{23}H_{16}ClN_3O_4$ found (%): C, 63.52; H, 3.70; N, 9.73; calcd. (%): C, 63.67; H, 3.72; N, 9.69.

3g: 4-(4-Bromophenyl)-2,6-bi(3-nitrophenyl)-1,4-dihydropyridine

IR (KBr, ν, cm^{-1}): 3435, 3048, 2623, 2284, 1600, 1525, 926, 874, 693; ^1H NMR (400 MHz, DMSO-d_6) (δ, ppm): 9.03 (2H, s, pyridine-H), 8.61 (1H, d, $J = 8.0$ Hz, Ar-H), 8.48 (2H, d, $J = 8.0$ Hz, Ar-H), 8.38 (2H, d, $J = 7.6$ Hz, Ar-H), 7.75 (2H, t, $J = 8.0$ Hz, Ar-H), 7.69 (1H, d, $J = 8.0$ Hz, Ar-H), 7.62 (2H, dd, $^3J = 8.4$ Hz, $^4J = 1.6$ Hz, Ar-H), 6.25 (1H, s, NH), 4.54–6.57 (1H, m, CH). EI-MS (m/z): 477.2 $[M]^+$. $C_{23}H_{16}BrN_3O_4$ found (%): C, 57.66; H, 3.30; N, 8.75; calcd. (%): C, 57.76; H, 3.37; N, 8.79.

CONCLUSION

Overall, we have demonstrated a versatile new approach to synthesize a range of 1,4-dihydropyridines bearing aryl groups on the 2, 4, and 6 positions. The significance of our finding also relates to reducing the use of organic solvents as potentially toxic and hazardous materials, as well as its simplicity, mild conditions, and inherent lower costs. Moreover, the findings give further credence to the synthesis of activated natural products and analogous natural products. This kind of compound is first used as an effective catalyst on simple Diels–Alder reactions.

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REFERENCES

1. Liang, J. C.; Yeh, J. L.; Wang, C. S.; Liou, S. F.; Tsai, C. H.; Chen, I. J. *Bioorg. Med. Chem.* **2002**, *10* (3), 719–730.
2. Tasaka, S.; Ohmori, H.; Gomi, N.; Iion, M.; Machida, T.; Kiue, A.; Naito, S.; Kuwano, M. *Bioorg. Med. Chem. Lett.* **2001**, *11* (2), 275–277.
3. Mikhail, F. G.; Dinesh, V. P.; Bruce, P. E.; Supriya, J.; Jesse, D. C.; Eric, M. G. *Bioorg. Med. Chem. Lett.* **1998**, *6* (7), 883–889.
4. Tanabe, H.; Tasaka, S.; Ohmori, H.; Gomi, N.; Sasaki, Y.; Machida, T.; Iion, M.; Kiue, A.; Naito, S.; Kuwano, M. *Bioorg. Med. Chem.* **1998**, *6* (11), 2219–2227.
5. Kiselyov, A. S. *Tetrahedron Lett.* **1995**, *36* (51), 9297–9300.
6. Eisner, U.; Elvidge, J. A.; Linstead, R. P. *J. Chem. Soc.* **1953**, 1372–1379.
7. Kamal, A.; Qureshi, A. *Tetrahedron* **1963**, *19*, 513–520.
8. Wiley, R.; Ridgway, J. S. *J. Org. Chem.* **1961**, *26* (2), 595–597.
9. Biellmann, J. F.; Callot, H. J.; Goeldner, M. P. *Chem. Commun.* **1969**, 141–142.

10. (a) Dondoni, A.; Massi, A.; Minghini, E. *Synlett* **2002**, *2002* (1), 89–92; (b) Gevariya, H.; Desai, B.; Vora, V.; Shah, A. *Heterocyclic Commun.* **2001**, *7* (5), 481–484; (c) Sobolev, A.; Franssen, M. C. R.; Vigante, B. *Tetrahedron: Asymmetry* **2001**, *12* (23), 3251–3256; (d) Yamada, S.; Misono, T.; Ichikawa, M.; Morita, C. *Tetrahedron* **2001**, *57* (43), 8939–8949; (e) Obika, S.; Nishiyama, T.; Tatematsu, S. *Tetrahedron* **1997**, *53* (9), 3073–3082; (f) Caballero, E.; Puebla, P.; Sánchez, M. *Tetrahedron: Asymmetry* **1996**, *7* (7), 1985–1994.
11. Eynde, J. J. V.; Dorazio, P.; Mayence, A. *Tetrahedron* **1992**, *48* (7), 1263–1268.
12. Evdokimov, N. M.; Magedov, I. V.; Kireev, A. S. *Organic Letters* **2006**, *8* (5), 899–902.
13. Breitenbucher, J. G.; Figliozzi, G. *Tetrahedron Lett.* **2000**, *41* (22), 4311–4315.
14. Alajarin, R.; Jordan, P.; Vaquero, J. J.; Alvarez-Builla, J. *Synthesis* **1995**, *4*, 389–391.
15. Chen, W. M.; Guo, H. X. *Hecheng Huaxue* **1999**, *7*, 422–426.
16. Kai, T.; Nicole, D.; Jacques, P. R. *Org. Lett.* **2004**, *6*, 4595–4597.
17. Vincenzo, C.; Angelo, N.; Li, L. G. *Tetrahedron Lett.* **2000**, *41*, 8973–8976.
18. Lee, C. W. *Tetrahedron Lett.* **1999**, *40*, 2461–2464.
19. Hagiwara, H.; Sayuri, T.; Okabe, T.; et al. *Green Chem.* **2002**, *4* (5), 461–463.
20. Deng, Y. Q.; Shi, F.; Peng, J. J.; et al. *Mol. Catal. A: Chem.* **2001**, *165* (1), 33–36.
21. Wu, H.; Lu, L. L.; Shen, Y.; Wan, Y.; Yu, K. B. *Synth. Commun.* **2006**, *36*, 1193–1200.
22. Zhang, W.; Jia, X. D.; Yang, L.; Liu, Z. L. *Tetrahedron Lett.* **2002**, *43*, 9433–9436.
23. Singh, S.; Sharma, V. K.; Gill, S.; Sahoto, R. I. K. *J.Chem. Soc., Perkin Trans. 1* **1985**, 437.
24. Zeng, S. Q.; Zeng, H. P. *Experiment of Organic Chemistry*, 3rd Edn.; Publishing Company of Advance Education: Beijing, 2000, Chapter 3, pp. 140–142.