Catalyst-Free Protocol for the Synthesis of Quinoxalines and Pyrazines in PEG

Tie-Qiang Huang,[a] Wen-Yan Qu,[a] Jin-Chang Ding,[a,b] Miao-Chang Liu,[a] Hua-Yue Wu,[a] and Jiu-Xi Chen[a]*

[a]College of Chemistry and Materials Engineering, Wenzhou University, Wenzhou 325035, People's Republic of China
[b]College of Light Industry, Wenzhou Vocational and Technical College, Wenzhou 325035, People's Republic of China
*E-mail: jiuxichen@wzu.edu.cn

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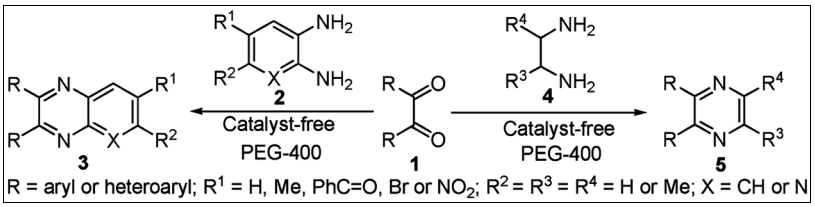

A facile and simple catalyst-free protocol has been developed for the condensation of 1,2-diketones with aromatic 1,2-diamines in polyethylene glycol (PEG), providing quinoxaline derivatives in good yields. The important features of the methodology are broad substrates scope, simple workup, catalyst free, environmentally benign, and no requirement for metal catalysts. It is noteworthy that the cyclization reaction of 1,2-diketones with aliphatic 1,2-diamines is also conducted smoothly to afford pyrazines in good yields under the standard conditions. In addition, PEG could be recovered easily and was reused without evident loss in activity

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INTRODUCTION

Quinoxaline derivatives have shown a broad spectrum of biological activities such as anticancer, anthelmintic, antifungal, and insecticidal agents [1]. Quinoxaline ring is a part of a number of antibiotics such as echinomycin, levomycin, and actinomycin, which are known to inhibit the growth of gram-positive bacteria and are also active against various transplantable tumors [2]. They have also found applications as dyes [3a], efficient electroluminescent materials [3b], organic semiconductors [3c], dehydroannulenes [3d], cavitands [3e], and chemically controllable switches [3f]. Consequently, a variety of synthetic strategies have been developed for the preparation of quinoxaline derivatives [4]. One of the most common methods is the condensation of 1,2-diamine with 1,2-dicarbonyl compounds in the presence of various catalysts, where water is the only theoretical by-product. The catalysts include Zn/L-proline [5], bismuth(III) triflate [6], SA (sulfamic acid)/MeOH [7], gallium(III) triflate [8], molecular iodine [9], Ni-nanoparticles [10], cerium (IV) ammonium nitrate [11], stannous chloride [12], zirconium tetrakis (dodecylsulfate) [13], amidosulfonic acid [14], montmorillonite K-10 [15], polyaniline-sulfate salt [16], niobium pentachloride [17], Wells-Dawson heteropoly acid [18], and ZrO_2/M_xO_y (M = Al, Ga, In, and La) mixed metal oxides supported on MCM-41 mesoporous molecular sieves [19]. The condensation has also been accomplished under catalyst-free conditions but needs for the microwave heating [20] in industry. In our previous work, we reported improved procedures for the preparation of quinoxaline derivatives by grinding under solvent-free conditions [21] and catalyst-free protocol under ultrasound irradiation [22]. Very recently, Srinivasan [23] and Meshram [24] reported an efficient synthesis of quinoxalines in the presence of imidazole-based hydrophilic ionic liquids such as 1-*n*-butylimidazolium tetrafluoroborate ([Hbim]BF_4) or 1-*n*-butyl-3-methylimidazolium tetrafluoroborate ([Bmim]BF_4). However, ionic liquids especially imidazolium-based systems containing BF_4 anion are toxic in nature as they liberate hazardous HF, and their high cost and disposability make their utility limited [25]. Although these methods are suitable for certain synthetic conditions, many of these procedures are associated with one or more disadvantages such as long reaction time, low yield, use of hazardous organic solvents, excess costly reagents or catalysts, and harsh reaction conditions, which leaves scope for further development of new environmentally clean syntheses.

In the recent years, development of environmentally friendly chemical synthesis is gaining considerable interest in both academia and industrial research [26]. Polyethylene glycol (PEG) is becoming prominent as alternative green reaction media with unique properties such as thermal stability, commercial availability,

nonvolatility, immiscibility with a number of organic solvents, and recyclability in synthetic chemistry. In general, PEG is nontoxic, being used in food products and cosmetics, is potentially recyclable and is water-miscible which facilitates its removal from reaction products [27]. On the other hand, PEG is inexpensive, completely nonhalogenated, and easily degradable. The use of PEG as a reaction solvent has received considerable attention in synthetic organic chemistry. Their potential as reaction media and promoters for organic reactions has attracted the attention of organic chemists in recent years [28]. However, it is surprised that the condensation of 1,2-diamines with 1,2-diketones in PEG is still unexplored so far.

As a continuing interest in developing novel synthetic routes for the formations of carbon–carbon and carbon–heteroatom bonds [21, 22, 29], we herein report an efficient, catalyst-free, and practical method for the synthesis of quinoxalines **3** [Scheme 1, eq. (1)] and pyrazines **5** [Scheme 1, eq. (2)] in PEG-400.

RESULTS AND DISCUSSION

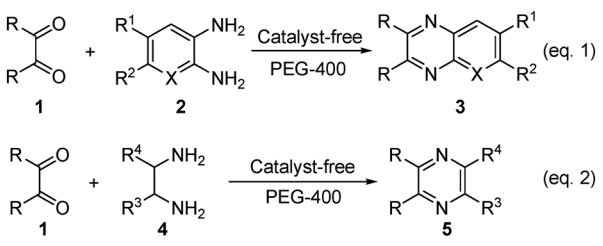
Initially, the model reaction of benzil (**1a**) with 1,2-diaminobenzene (**2a**) was conducted to screen the optimal reaction conditions, and the results were summarized in Table 1. The reaction (Scheme 2) was carried out in ethanol at room temperature under catalyst-free conditions, which resulted in poor yield after 3 h (Table 1, entry 1). However, the same reaction in boiling ethanol afforded the desired 2,3-diphenylquinoxaline (**3a**) in 47% yield within 40 min (Table 1, entry 1). Encouraged by the results, we next planned to determine the influence of solvent on the catalytic property of the model reaction. It was observed that the model reaction in various solvents (H_2O, toluene, xylene, 1,4-dioxane, DMSO, and DMF) gave comparatively lower conversions (Table 1, entries 2–7). Surprisingly, this reaction could be performed in PEG-400 for 30 min in the absence of catalyst in virtually quantitative yield (Table 1, entry 8). However, in the presence of these additional solvents (Table 1, entries 9–10), the yields of **3a** were found to be comparatively lower, probably due to the reduced efficiency of PEG because of dilution. Therefore, PEG-400 was chosen as the reaction media as the subsequent research.

With the optimized conditions in hand, the reactions of various 1,2-diketones with different aromatic 1,2-diamines were examined to explore the scope and generality of this present protocol for the synthesis of various quinoxalines **3** (Scheme 3), and the results were listed in Table 2.

As shown in Table 2, a series of aromatic 1,2-diamines bearing either electron-donating or electron-withdrawing groups on aromatic ring were investigated. It was observed that the electronic effect played important roles in this system. It was observed that the reaction of 1,2-diamine bearing electron-donating group (-Me) on the benzene ring, such as 4-methylbenzene-1,2-diamine (Table 2, entries 2, 8, 11, 16, and 19) and 4,5-dimethylbenzene-1,2-diamine (Table 2, entries 17 and 20) with various 1,2-diketones was examined, and the corresponding products were obtained in excellent yields. Whereas 1,2-diamines with electron-withdrawing group such as nitro (Table 2, entries 3 and 12) and benzoyl (Table 2, entries 5, 9, and 13) or less nucleophilic 1,2-diamine such as naphthalene-2,3-diamine (Table 2, entry 4) decreased slightly the product yields even longer time. Moreover, we have also subjected other than heterocyclic 1,2-diamine such as 5-bromopyridine-2,3-diamine and obtained the products **3f** and **3n** in good yields (Table 2, entries 6 and 14).

On the other hand, the substitution groups on the phenyl ring associated with 1,2-diketone had no obvious effects in terms of yields under optimal conditions (Table 2, entries 1, 7, 15, and 18). Furthermore, we also examined the condensation of heterocyclic 1,2-diketone,

Scheme 1



R = aryl or heteroaryl; R^1 = H, Me, PhC=O, Br or NO_2;
$R^2 = R^3 = R^4$ = H or Me; X = CH or N

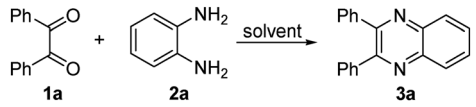
Table 1

Effect of various solvents on the reaction condensation of benzyl with benzene-1,2-diamine.[a]

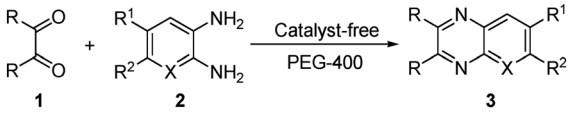
Entries	Solvents	Yield (%)[b]
1	EtOH	47[d], 22[c]
2	H_2O	38[d]
3	Toluene	69[d]
4	Xylene	68
5	1,4-Dioxane	71[d]
6	DMSO	61
7	DMF	62
8	PEG-400	99
9	PEG-400/toluene = 1:1	79
10	PEG-400/DMSO = 1:1	78

[a]All the reactions were performed using benzil **1a** (0.5 mmol), 1,2-diaminobenzene **2a** (0.7 mmol) in the solvent (5 mL) at 120°C for 40 min.
[b]Isolated yield.
[c]The reaction was carried out at room temperature for 3 h.
[d]Under reflux.

Scheme 2



Scheme 3



such as 1,2-di(furan-2-yl)ethane-1,2-dione **1c** (Table 2, entries 10–14) with various 1,2-diamine, providing the desired products **3j**–**3n** in good to excellent yields.

To check the versatility of this method, we have also subjected other than aliphatic 1,2-diamine such as ethane-1,2-diamine and propane-1,2-diamine. As shown in Table 3, the reaction of aliphatic 1,2-diamine (Scheme 4) with various 1,2-diketones afford the desired products pyrazines **5a**–**5f** with yields ranging from moderate to good. It is worth mentioning that 1,2-diketones can react with aliphatic 1,2-diamines resulting in the formation of pyrazines, which can react in low yields or result in the formation of dihydropyrazines in the reported literatures [6–8, 13, 20b].

Table 2

Catalyst-free protocol for the synthesis of quinoxalines.[a]

		Diamine						
Entries	R (**1**)	R^1	R^2	X	Time (min)	Product	Yield (%)[b]	Mp (°C)
1	Ph **1a**	H	H	CH	40	**3a**	99	128–129
2	Ph **1a**	Me	H	CH	40	**3b**	96	117–118
3	Ph **1a**	NO_2	H	CH	45	**3c**	87	187–189
4	Ph **1a**	Naphthalene-2,3-diamine			40	**3d**	40	200–202
5	Ph **1a**	PhC=O	H	CH	40	**3e**	84	138–139
6	Ph **1a**	Br	H	N	60	**3f**	83	154–156
7	*p*-$(CH_3)C_6H_4$ **1b**	H	H	CH	40	**3g**	98	147–148
8	*p*-$(CH_3)C_6H_4$ **1b**	Me	H	CH	40	**3h**	95	135–137
9	*p*-$(CH_3)C_6H_4$ **1b**	PhC=O	H	CH	60	**3i**	81	188–191
10	2-furyl **1c**	H	H	CH	40	**3j**	99	132–134
11	2-furyl **1c**	Me	H	CH	40	**3k**	98	118–120
12	2-furyl **1c**	NO_2	H	CH	45	**3l**	84	164–166
13	2-furyl **1c**	PhC=O	H	CH	40	**3m**	87	135–136
14	2-furyl **1c**	Br	H	N	60	**3n**	80	134–136
15	*p*-$(OCH_3)C_6H_4$ **1d**	H	H	CH	40	**3o**	90	147–148
16	*p*-$(OCH_3)C_6H_4$ **1d**	Me	H	CH	40	**3p**	92	121–123
17	*p*-$(OCH_3)C_6H_4$ **1d**	Me	Me	CH	40	**3q**	91	126–127
18	*p*-$(Br)C_6H_4$ **1e**	H	H	CH	50	**3r**	89	189–191
19	*p*-$(Br)C_6H_4$ **1e**	Me	H	CH	50	**3s**	91	185–186
20	*p*-$(Br)C_6H_4$ **1e**	Me	Me	CH	50	**3t**	90	175–176

[a]All reactions were run with 1,2-diketone 1 (0.5 mmol) and 1,2-diamine 2 (0.7 mmol) in PEG-400 (5 mL) at 120°C.
[b]Isolated yields.

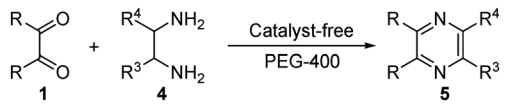
Table 3

Catalyst-free protocol for the synthesis of pyrazines.[a]

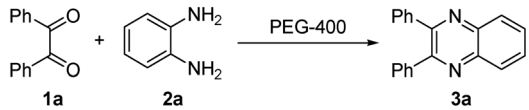
		Diamine				
Entries	R (**1**)	R^3	R^4	Product	Yield (%)[b]	Mp (°C)
1	Ph **1a**	H	H	**5a**	86	121–122
2	Ph **1a**	Me	H	**5b**	78	91–92
3	*p*-$(CH_3)C_6H_4$ **1b**	H	H	**5c**	88	121–122
4	*p*-$(CH_3)C_6H_4$ **1b**	Me	H	**5d**	71	121–123
5	2-Furyl **1c**	H	H	**5e**	75	78–80
6	2-Furyl **1c**	Me	H	**5f**	76	62–64

[a]All the reactions were performed using 1,2-diketone 1 (0.5 mmol), 1,2-diamine **4** (1.0 mmol) in PEG-400 (5 mL) at 120°C for 45 min.
[b]Isolated yield.

Scheme 4



Scheme 5



Run 1, 98%; run 2, 96%; run 3, 94%; run 4, 93% and run 5, 93%

Finally, we investigated the recycling of PEG-400 in a subsequent reaction, for example, the synthesis of **3a** from the reaction of benzil **1a** with 1,2-diaminobenzene **2a** (Scheme 5). PEG-400 was reused for five runs without any appreciable loss of activity (with the yield of the corresponding product being 98, 96, 94, 93, and 93% yield, respectively).

In conclusion, we developed a highly efficient and eco-friendly synthesis of quinoxalines and pyrazines in PEG-400 under catalyst-free condition. Compared to previous reported methodologies, the present protocol features simple workup, broad substrates scope, no requirement of catalysts, and environmentally benign. Further investigations on the reaction mechanism, scope, limitations, and biological-activity evaluation of these new classes of compounds are under way.

EXPERIMENTAL

Chemicals and solvents were either purchased or purified by standard techniques. The melting points were uncorrected and were recorded on Digital Melting Point Apparatus WRS-1B. IR spectra were recorded on an AVATAR 370 FI infrared spectrophotometer. NMR spectroscopy was performed on a Bruker-300 spectrometer or Bruker-500 spectrometer using DMSO-d_6 or $CDCl_3$ as the solvent with tetramethylsilane (TMS) as an internal standard at room temperature. The mass-spectrometric identification of the products was performed on Thermo Finnigan LCQ-Advantage or SHIMADZU GCMS-QP2010 system. Elemental analysis was determined on a Carlo-Erba 1108 instrument.

General procedure for synthesis of quinoxalines (3) and pyrazines (5). To a mixture of 1,2-diketones **1** (0.5 mmol) and 1,2-diamine **2** or **4** (0.7 mmol), PEG-400 (5 mL) was added, and the mixture was stirred for the respective time at 120°C. The reaction was monitored by TLC. After completion of the reaction, water (10 mL) was added, and the mixture was extracted with ethyl acetate (3 × 10 mL), the organic layer washed with brine (3 × 10 mL), then dried over anhydrous Na_2SO_4 and concentrated. The crude product was separated and purified by column chromatography on silica gel (300–400 mesh) using an ethyl acetate/petrol mixture as the eluent to afford a pure product of **3** and **5**. Here, the selected characterization data is given for the representative known products **3a**, **5a**, and unknown product **3t**. For analytical data and spectra of other compounds, see Supporting Information.

2,3-Diphenylquinoxaline (3a). This compound was obtained as white solid, m.p. 128–129°C (Table 2, entry 1); ^{1}H-NMR (300 MHz, $CDCl_3$): δ 8.18–8.21 (2H, m), 7.74–7.78 (2H, m), 7.53–7.55 (4H, m), 7.34–7.36 (6H, m); ^{13}C-NMR (75 MHz, $CDCl_3$) δ 153.4, 141.2, 139.0, 123.0, 129.8, 129.2, 128.8, 128.3.

2,3-Bis(4-bromophenyl)-6,7-dimethylquinoxaline (3t). This compound was obtained as colorless needles solid, m.p. 175–176°C (Table 2, entry 20); IR: 1716, 1673, 1587, 1483.6, 1446, 1420, 1392, 1364, 1224, 1207, 1073, 1010, 971, 872, 831 cm^{-1}; ^{1}H-NMR (300 MHz, $CDCl_3$): δ 7.86 (s, 2H), 7.46 (d, J = 6.2 Hz, 4H), 7.34 (d, J = 8.4 Hz, 4H), 2.49 (s, 6H); ^{13}C-NMR (125 MHz, $CDCl_3$): δ 150.9, 141.1, 140.2, 138.0, 131.6, 131.5, 128.2, 123.4, 20.5; Anal. Calcd. for $C_{22}H_{16}Br_2N_2$: C, 56.44; H, 3.44; N, 5.98. Found: C, 56.35; H, 3.51; N, 6.06.

2,3-Diphenylpyrazine (5a). This compound was obtained as white solid, m.p. 121–122°C (Table 3, entry 1); ^{1}H-NMR (300 MHz, $CDCl_3$): δ 8.59 (s, 2H), 7.45–7.49 (m, 4H), 7.28–7.34 (m, 6H); ^{13}C-NMR (75 MHz, $CDCl_3$): δ 152.8, 142.1, 138.6, 129.6, 128.7, 128.3.

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