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L-proline as an efficient organocatalyst for the synthesis of quinoxaline derivatives under solvent-free conditions

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KEYWORDS
Quinoxaline derivatives; 1,2-diamines; 1,2-dicarbonyls; L-proline; Solvent-free conditions.

Abstract. L-Proline has been found to be an efficient organocatalyst for one-pot synthesis of quinoxaline derivatives under solvent-free conditions. The method provides advantages such as short reaction time, high yields and simple work-up.

1. Introduction

Synthesis of quinoxaline and its derivatives has occupied an important place in the realm of synthetic organic chemistry due to their wide range of biological activities, as in tranquilizers [1], antimycobacterials [2,3], cardiotonics [4], antidepressants [5], antitumors [6], anticancers [7], and activities as kinase inhibitors [8]. They have also found to have applications as dyes [9] and as key intermediates in the synthesis of organic semiconductors [10].

Due to their great importance, many synthetic strategies have been developed for the synthesis of quinoxalines. This includes the Bi-catalyzed oxidative coupling of epoxides and ene-1,2-diamines [11], microwave irradiation [12], ultrasound irradiation [13] with α-hydroxy ketones via a tandem oxidation process using $Pd(OAc)_2$ or $RuCl_2$-$(PPh_3)_3$-TEMPO [14] and MnO_2 [15], hetroannulation of nitroketene N,S-arylaminoacetals with $POCl_3$ [16], a solid-phase synthesis on SynPhase^{TM} lanterns [17], and cyclization of α-arylimino oximes of α-dicarbonyl compounds under reflux in acetic anhydride [18]. By far, the most common method is the condensation of 1,2-diamines with 1,2-dicarbonyl compounds. For this transformation, many catalysts and reagents have been reported, including I_2 [19,20], sulfamic acid [21], montmorillonite K-10 [22], polyaniline-sulfate salt [23], $H_6P_2W_{18}O_{62}.24H_2O$ [24], $InCl_3$ [25], $CuSO_45H_2O$ [26], Zn[(L)proline] [27], ceric ammonium nitrate [28], ρ-toluenesulfonic acid [29], $[Hbim]BF_4$ [30], silica gel [31], Ni-nanoparticles [32], zirconium(IV)-modified silica gel [33], sulfated titania [34], $Ga(OTf)_3$ [35], and LiBr [36]. However, several of these methodologies involve pollution, long reaction time and tedious work-up, leaving considerable scope for the development of a green, facile and efficient process for these important heterocyclic compounds.

Recently, L-proline has emerged as a powerful catalyst for various synthetic reactions, such as the synthesis of benzodiazepines [37], 1,8-dioxodecahydro acridines [38], imidazole derivatives [39], and polyfunctionalized pyrano[2,3-c]pyrazoles [40]. In addition, L-proline in combination with ionic liquids has proved to be an efficient system for direct asymmetric aldol reactions, cross-aldol reactions, Michael additions, α-aminations of aldehydes and ketones, Ullmann-type reactions, Mannich reactions, and di-

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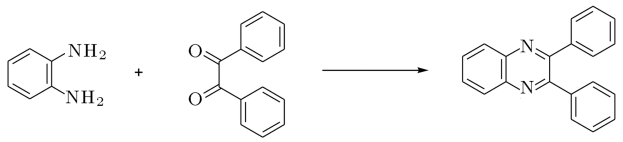
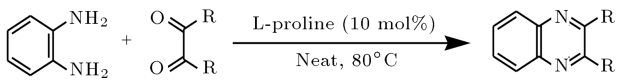
Table 1. The influence of solvent, temperature and amount of catalyst on the condensation reaction[a].



1 **2** **3a**

Entry	Amount of catalyst (mol%)	Solvent	Temperature (°C)	Time	Yield (%)[b]
1	10	C_2H_5OH	Reflux	2:10 h	90
2	10	CH_3COCH_3	Reflux	5:30 h	75
3	10	H_2O	Reflux	1 h	0
4	10	CH_3CN	Reflux	1 h	86
5	10	CH_2Cl_2	Reflux	3 h	93
6	10	$CH_3CO_2C_2H_5$	Reflux	6 h	83
7	10	None	80	1 min	100
8	10	None	60	1 h	90
9	10	None	40	2 h	79
10	10	None	rt	5 h	80
11	5	None	80	5 min	89

[a]: 1 mmol o-phenenylenediamine and 1 mmol benzil; [b]: Isolated yield.



Scheme 1. Synthesis of quinoxaline derivatives catalyzed by L-proline.

rect asymmetric α-aminoxylation of aldehydes and ketones [41]. Herein, we report a green and efficient method for the synthesis of quinoxaline derivatives in high yields and short reaction times by the condensation of 1,2-diamines with 1,2-dicarbonyl compounds catalyzed by L-proline under solvent-free conditions (Scheme 1).

2. Experimental

1,2-Diamine (1 mmol), 1,2-diketone (1 mmol), and L-proline (0.011 g, 10 mol%) were mixed. The mixture was stirred at 80°C and the progress of the reaction was monitored by thin-layer chromatography (TLC). Due to the solubility of L-proline in ethanol, it is not necessary to separate it from the reaction mixture. Therefore, after completion of the reaction, hot ethanol was added to the reaction mixture, and the pure product was crystallized from it. All products are known compounds, which were satisfactorily characterized by physical and spectra data.

3. Results and discussion

To evaluate the effect of the catalyst under different reaction conditions, the condensation of o-phenylenediamine **1** and benzil **2** was selected as a model reaction, and the results are shown in Table 1. First, we examined the solvents and solvent-free conditions (Table 1, entries 1-7). An excellent yield of product **3a** was obtained under solvent-free conditions. Second, the effect of catalyst loading and temperature on the reaction efficiency was examined (Table 1, entries 8-11). With 10 mol% of the catalyst, the yield of **3a** increased from 80% to 100% as the temperature increased from r.t to 80°C. Similar changes in yields were also observed with the catalyst amount at a constant temperature. Therefore, it is reasonable to conclude that the best result was achieved when the reaction was performed with 10 mol% of catalyst and at 80°C under solvent-free conditions.

In order to study the generality and scope of this protocol, a series of 1,2-diamines and 1,2-diketones was applied. The results in Table 2 show that all reactions proceeded efficiently and the products were obtained in good to excellent yields, in short reaction times. However, when the electron-donating group (-OMe) was placed in the phenyl group associated with 1,2-dicarbonyl compounds, the reaction needed more time for completion (Table 2, entries 2 and 8). It

Table 2. Synthesized quinoxaline derivatives.

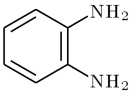
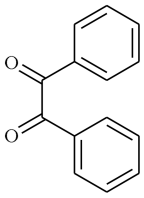
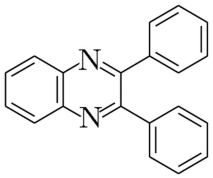
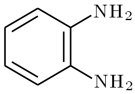
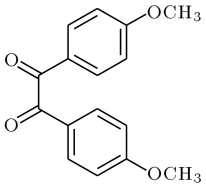
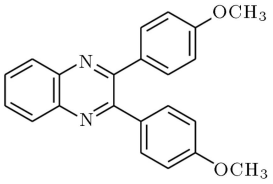
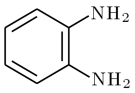
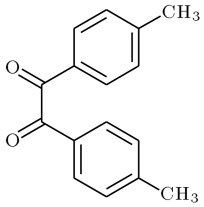
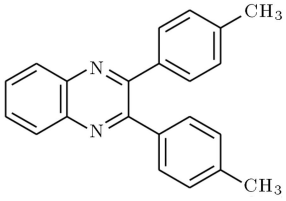
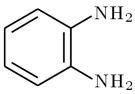
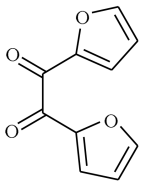
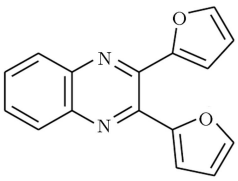
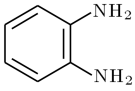
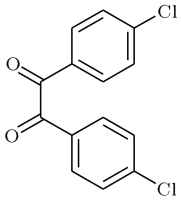
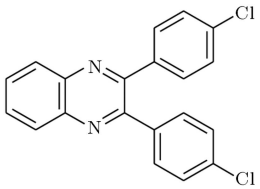
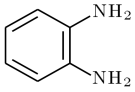
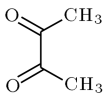
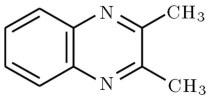
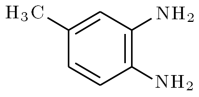
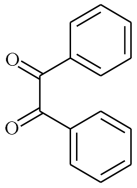
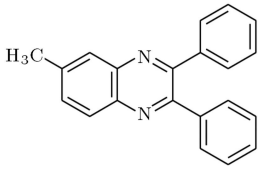
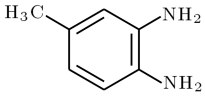
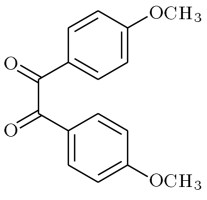
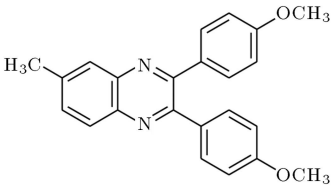
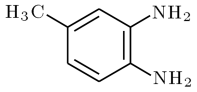
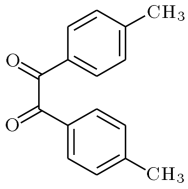
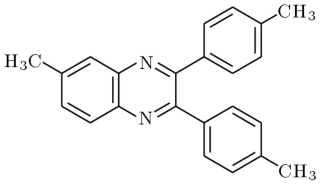
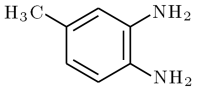
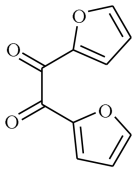
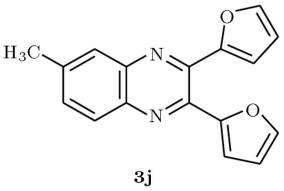
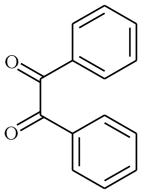
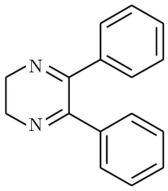
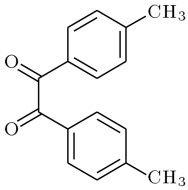
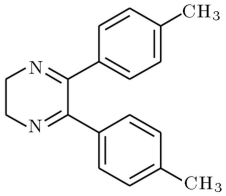
Entry	1,2-diamine	1,2-diketone	Product[a]	Time (min)	Yield[b] (%)	M.p. (°C) [Ref.]
1			 **3a**	1	100	120-122 [13]
2			 **3b**	60	67	140-141 [13]
3			 **3c**	15	90	147-148 [13]
4			 **3d**	35	85	128-130 [13]
5			 **3e**	3	86	180-182 [35]
6			 **3f**	2	98	104-106 [21]
7			 **3g**	2	100	109-110 [42]
8			 **3h**	60	100	120-121 [13]

Table 2. Continued.

Entry	1,2-diamine	1,2-diketone	Product[a]	Time (min)	Yield[b] (%)	M.p. (°C) [Ref.]
9			 **3i**	35	86	135-136 [13]
10			 **3j**	40	99	113-114 [13]
11			 **3k**	30	98	160-161 [21]
12			 **3l**	55	98	164-166 [43]

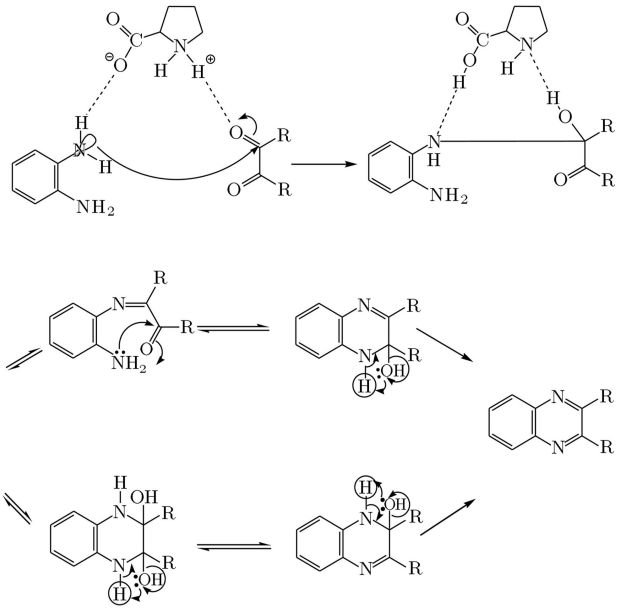
[a]: 1 mmol o-phenenylenediamine and 1 mmol benzil;

[b]: Isolated yield.

is noteworthy to mention that all reactions proceeded very cleanly and no side-products were observed.

In order to show the merit of this method, in Table 3, we have compared our result with results obtained by some other reported methods for synthesis of **3a**. The data presented in this table show the promising feature of our method in terms of reaction rate and the yield of product compared with those reported in the literature.

A plausible reaction mechanism and the role of L-proline catalyst are depicted in Scheme 2.

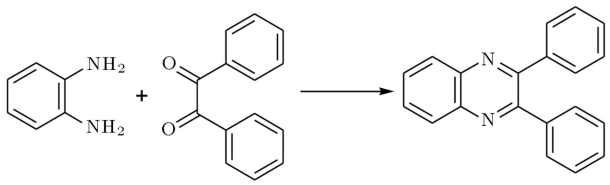


Scheme 2. Proposed mechanism.

4. Conclusion

In conclusion, we have demonstrated that L-proline is a powerful organocatalyst for the synthesis of various quinoxaline derivatives under solvent-free conditions. The features introduced by this protocol, such as using a cheap and available organocatalyst, avoidance of hazardous organic solvents and a very simple work-up (employing only ethanol), are in accordance with the

Table 3. Comparison of protocols for the synthesis of quinoxaline derivatives.



1 **2** **3a**

Entry	Catalyst	Solvent	Temperature	Time	Yield (%)	Ref.
1	Silica gel, grinding	Neat	100°C	45 min	**94**	[31]
2	AcOH	AcOH	Reflux	1 h	**83**	[44]
3	Acidic alumina	Neat	Microwave 900 W	3 min	**85**	[42]
4	$H_4SiW_{12}O_{40}$	H_2O	r.t	1 h	**96**	[45]
5	Ultrasound irradiation	Ethanol	r.t	1 h	**98**	[13]
6	Zirconium(IV)-modified silica gel	Ethanol	r.t	1.5 h	**92**	[33]
7	Zn[(L)proline]	HOAc	r.t	10 min	**95**	[27]
8	I_2	DMSO	r.t	35 min	**95**	[19]
9	Amidosulfonic Acid	CH_2Cl_2	r.t	2 h	**92**	[46]
10	L-proline	Neat	80	1 min	**100**	This work

principles of green chemistry. Therefore, this protocol can be regarded as a great improvement in the synthesis of quinoxaline derivatives.

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