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A mild and convenient synthesis of quinoxalines via cyclization–oxidation process using DABCO as catalyst

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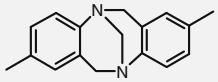
ABSTRACT

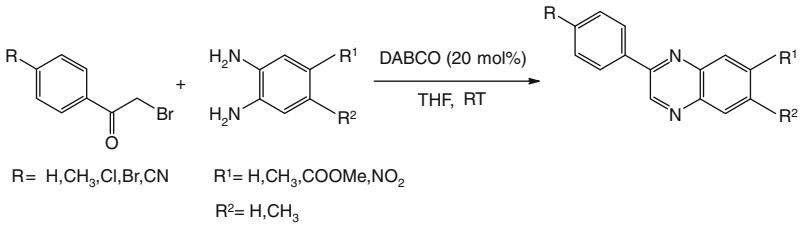
An efficient and general method has been described for the synthesis of quinoxalines by the reaction of 1,2-diamines with phenacyl bromides in the presence of DABCO. The method is suitable for the preparation of functionalized quinoxalines.

Quinoxaline is a core constituent of many pharmaceuticals as well as agrochemicals.[1] Quinoxaline ring is also found in antibiotics such as echinomycin, leromycin, and actinomycin.[2] In addition to this, quinoxaline ring is a part of several bioactive natural products. Some of the quinoxaline derivatives were found to exhibit broad spectrum of biological activity,[1c] while the other derivatives of quinoxaline have found wide application in dyes,[3a] efficient electroluminescent material,[3b] organic semiconductors,[3c] dehydroannulenes[3d], and chemically controllable switches.[3e] Therefore, these compounds have distinguished themselves as heterocycles with chemical and biological significance. As a result, synthesis of these molecules attracted considerable attention. A traditional method for the constitution of quinoxaline ring involves the condensation of aryl 1,2-diamines with 1,2-dicarbonyl compounds,[4] 1,4-addition of 1,2-diamines to diazenylbutens[5], and oxidation-trapping of α-hydroxy ketones with 1,2-diamines.[6] Other recently, reported methods accomplished the synthesis of quinoxaline by the reaction of 1,2-diamines with phenacyl bromides in solid-phase,[7] or using heterogeneous catalyst like $HClO_4$–SiO_2.[8] In addition to this β-cyclodextrin (β-CD)[9] has been employed for the synthesis of quinoxaline in heating condition. Though the reported

Table 1
Screened bases for the reaction of phenacyl bromides with diamines

Entry	Base[a]	Conversion[b] (%)	Yield[c] (%)
1		65	60
2		100	95
3		50	47
4		55	52

[a] In a typical experiment, the 1,2-diamine (1 equiv) was added to a mixture of the base (20 mol %) and the phenacyl bromide (1 equiv) in THF and stirred at rt.
[b] Conversion of quinoxaline evaluated by ^{1}H NMR.
[c] Isolated yields.



Scheme 1.

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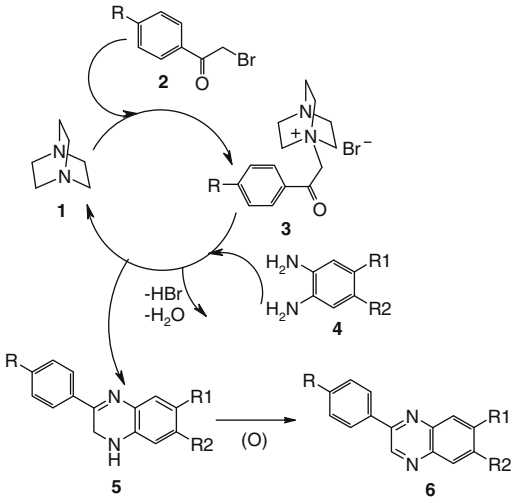
Table 2
Quinoxaline formation in different solvent conditions

Entry	Solvent	Yield[a] (%)
1	Benzene	65
2	Toluene	67
3	THF	95
4	CH_3CN	75
5	CH_2Cl_2	56

[a] All the reactions are carried at rt.



Scheme 2. Plausible mechanism for the quinoxaline formation.

methods provide good yields, these methods suffer from tedious work-up, longer reaction time, use of metal catalyst, and narrow scope of substrates. Moreover, some of the methods have some drawbacks such as unsatisfactory yields, expensive and detrimental metal reagents. In view of this, there is still need to develop a general and efficient method for the synthesis of more functionalized quinoxaline derivatives.

Recently, use of non-metallic reagents is an area of growing interest because of environmental regulations. Among the various organic bases, 1,4-diazabicyclo[2,2,2]octane (DABCO) has been employed as a organic-hindered base to bring about various organic transformations like deprotection of peptides,[10] as a catalyst for the Baylis–Hillman reaction,[11] for isoxazoles preparation,[12] *o*-alkylations of phenols[13], and deprotection of benzylic trimethylsilyl ethers.[14] To the best of our knowledge, there is no report on the synthesis of quinoxalines using organic bases. In continuation of our work in the area of green chemistry[15] and particularly in the development of non-metallic reagents,[16] herein we wish to report a simple, efficient, and general method for the preparation of substituted quinoxalines by the reaction of *o*-phenylenediamines with phenacyl bromides in the presence of DABCO (Scheme 1).

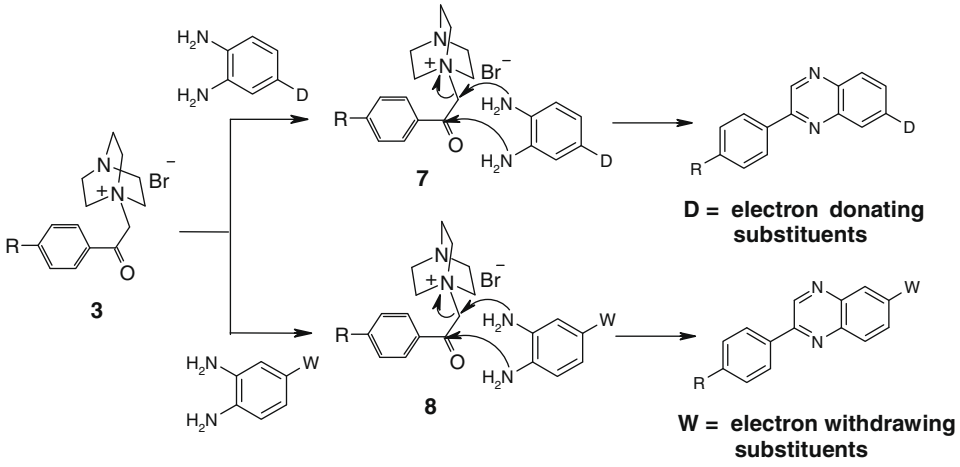
We have initiated our study by the reaction of phenacyl bromide with *o*-phenylenediamines in the presence of different organic bases such as quinuclidine, DABCO, urotropine, and Troger's base in THF (Table 1).

It was observed that the reaction proceeded efficiently using DABCO and resulted in high yield of desired product in short reaction time (30 min). However, with other bases the reaction was comparatively slow and gave less yield of the product even after stretching the reaction time (1 h). In addition to this we have screened acyclic tertiary amines such as TEA and N_1,N_1,N_2-triethyl-N_2-methylethane-1,2-diamine and found that the reaction was very sluggish with poor yield (10–20%) even with extended reaction time (4–5 h). We have attempted different ratios of DABCO (10, 15, 20, 25, 30, 40 mol %) and observed that 20 mol % was suitable for the optimum conversion. The increase in the mole ratio of DABCO also did not improve the yield.

Further to optimize the reaction conditions, the reaction was studied in different solvents such as benzene, toluene, dichloromethane, and acetonitrile. The reaction proceeded in all the solvents with different degrees of conversion (Table 2). However, the THF was the solvent of choice in terms of reaction time and yield.

The formation of product may be explained by the reaction of phenacyl bromide (**2**) with DABCO (**1**) which forms the quaternary salt **3**.[17] Later it reacts with diamine and subsequent cyclization and dehydrogenation result in expected product (**6**) (Scheme 2).

The scope of this DABCO-catalyzed quinoxaline formation was explored under optimal condition[18] and the results are summarized in Table 3. We have studied the electronic effects of the substituents on the rate of the reaction and the mode of formation of quinoxalines. It was observed that electron-rich substituents on the diamine influence the reaction. For example, the presence of highly electron-rich substituents (entry 21) on the diamine ring activates the amino group at para position, which further attack on ion pair to form the single isomer through intermediate **7**. This phenomenon was not observed with weak electron-donating substituents like methyl. However, in the case of 4-methyl 1,2-phenylenediamine (entries 2, 11, 14 and 18) gave the two isomeric products depending on the course of cyclization. It was observed



Scheme 3. Detailed mechanism for the quinoxaline formation.

Table 3
Synthesis of quinoxalines in the presence of DABCO[a]

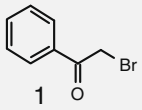
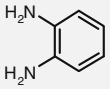
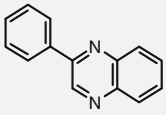
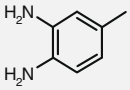
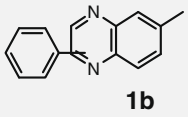
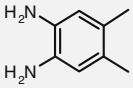
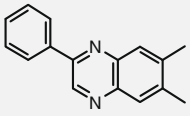
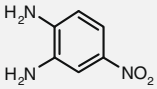
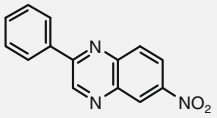
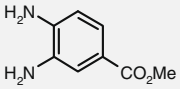
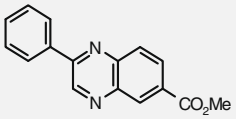
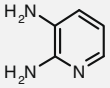
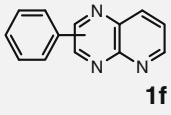
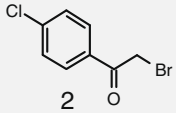
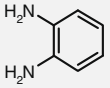
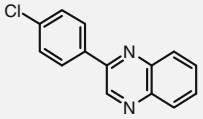
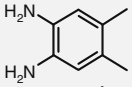
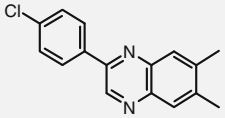
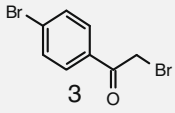
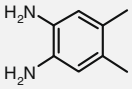
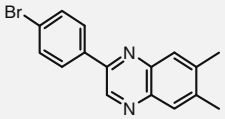
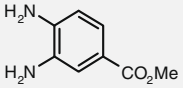
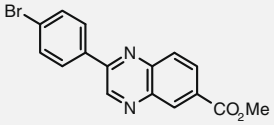
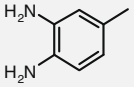
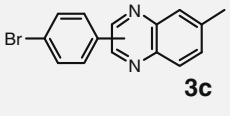
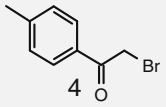
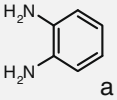
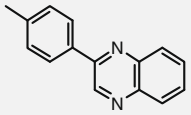
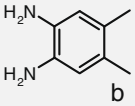
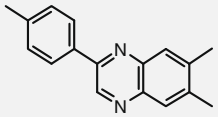
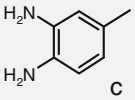
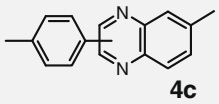
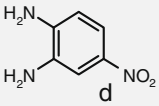
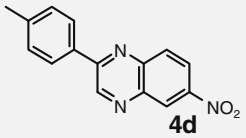
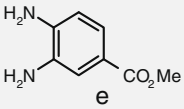
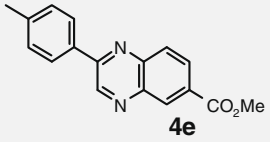
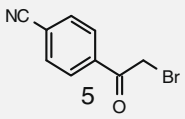
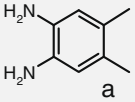
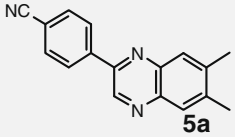
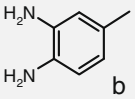
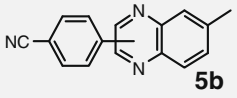
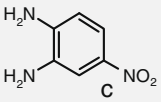
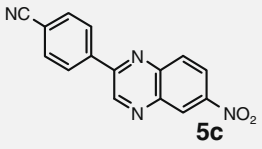
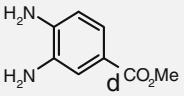
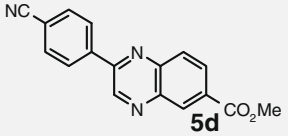
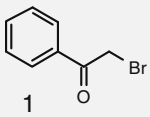
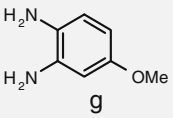
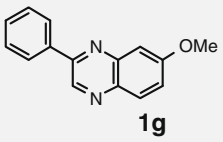
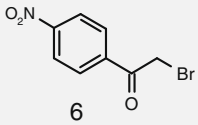
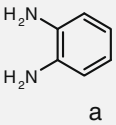
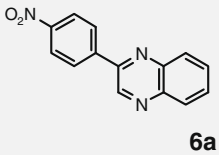
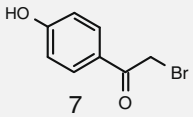
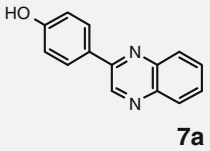
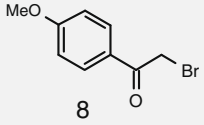
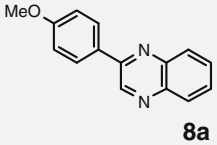
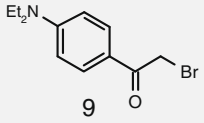
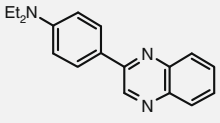
Entry	Phenacyl bromide	1,2-Diamine	Product	Time (min)	Yield[b] (%)
1	 1	 a	 **1a**	30	90
2		 b	 **1b**	26	92[c]
3		 c	 **1c**	25	93
4		 d	 **1d**	40	87
5		 e	 **1e**	37	89
6		 f	 **1f**	50	84[c]
7	 2	 a	 **2a**	32	90
8		 b	 **2b**	30	91
9	 3	 a	 **3a**	30	92
10		 b	 **3b**	40	86
11		 c	 **3c**	30	90[c]

Table 3 (*continued*)

Entry	Phenacyl bromide	1,2-Diamine	Product	Time (min)	Yield[b] (%)
12	 4	 a	 **4a**	35	90
13		 b	 **4b**	30	92
14		 c	 **4c**	33	90[c]
15		 d	 **4d**	40	88
16		 e	 **4e**	37	89
17	 5	 a	 **5a**	27	93
18		 b	 **5b**	28	92[c]
19		 c	 **5c**	30	90
20		 d	 **5d**	30	90
21	 1	 g	 **1g**	30	90
22	 6	 a	 **6a**	45	85
23	 7		 **7a**	35	91

(*continued on next page*)

Table 3 (*continued*)

Entry	Phenacyl bromide	1,2-Diamine	Product	Time (min)	Yield[b] (%)
24	 8		 **8a**	25	92
25	 9		 **9a**	32	90

[a] Reaction conditions: phenacyl bromide (1 equiv), 1,2-diamines (1 equiv), DABCO (20 mol %), in THF (4 ml).
[b] Isolated products.
[c] Mixture of isomers (8:2).

that the corresponding 2-aryl-7-methyl quinoxaline was the major product, whereas the other isomer 2-aryl-6-methyl quinoxaline was obtained as a minor product. While the symmetric diamines like 4,5-dimethyl-1-2-phenylenediamine (entries 3, 8, 9, 13 and 17) with phenacyl bromides resulted in the corresponding quinoxaline as a sole product in high yields. Further it is observed that the presence of electron-withdrawing substituents (entries 4, 5, 10, 15, 16, 19, and 20) on the diamine ring deactivates the para amino group. Due to this, another amino group first participates in the reaction and leads to intermediate **8** which on cyclization and oxidation gives the corresponding product (Scheme 3).

Next we have extended this protocol for the heterocyclic diamines. Thus 1,2-pyridine diamine (entry 6) reacted with phenacyl bromide and led to isomers of products in good yields. From Table 3 it can be seen that different substituted phenacyl bromides reacted with substituted 1,2-phenylenediamines and provided functionalized quinoxalines. We have also examined phenacyl bromides having different substituents such as NO_2, CN, Cl, Br, OH, OMe, *N,N*-dialkyl, and Me. The electron-rich functionalities (entries 7–9, 10–16, and 23–25) influence the reaction and furnish the corresponding quinoxalines in high yield. Whereas the electron-withdrawing substituents (entries 17–20 and 22) on phenacyl bromide gave comparatively low yield of quinoxaline under identical conditions. It is worthy to mention that the present method provides access for the synthesis of new functionalized quinoxalines (entry 5, 10, 16–19, and 20) which have not been synthesized earlier. These quinoxalines bearing the nitrile and ester functionalities may provide scope for further extension to build up pharmaceutically important molecules.

In conclusion, we have demonstrated an efficient and mild method for the synthesis of functionalized quinoxalines using DABCO as a catalyst. This method is applicable for a variety of phenacyl bromides and *o*-phenylenediamines. Moreover, this protocol provides access for the synthesis of functionalized quinoxalines.

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18. *General procedure*: A mixture of phenacyl bromide (1 equiv) and DABCO (20 mol %) was stirred at rt for 5 min. Then *0*-phenylenediamine (1 equiv) was added slowly and the resultant mixture was stirred at rt for stipulated time (see Table 3). After completion of the reaction, as indicated by TLC, the mixture was poured into water. It was extracted with ethylacetate (3 × 15 ml), dried over Na_2SO_4, and evaporated under reduced pressure. The crude product was purified by passing through small pad of silica gel to give pure product (ethyl acetate:hexane). All the compounds were characterized by ^{1}H NMR, ^{13}C NMR, mass, and IR spectral data.[19]
19. *Spectral data for new compounds*:
Compound ***1e***: mp 152–154 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 4.0 (s, 3H), 7.58 (m, 3H), 8.16 (d, 1H, J = 8.309 Hz), 8.24 (d, 2H, J = 8.309 Hz), 8.35 (d, 1H, J = 2.26 Hz), 8.78 (s, 1H), 9.38 (s, 1H). ^{13}C NMR (75 MHz, $CDCl_3$): 52.5, 127.6, 129.1, 129.6, 129.7, 130.7, 131.5, 135.9, 140.4, 144.2, 153.1, 166.1. IR (KBr) ν = 2923, 2852, 1714, 1542, 1444, 1291, 1251, 1172, 1089, 770, 686 cm^{-1}. MS (ESI) m/z 265 (M^+1). *Compound* ***3b***: mp 134–136 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 4.05 (s, 3H), 7.7 (d, 2H, J = 1.88 Hz), 8.16 (dd, 3H, J = 2.83, J = 3.02 Hz), 8.38 (d,

1H, J = 1.88 Hz), 8.79 (d, 1H, J = 1.88 Hz), 9.37 (s, 1H). ^{13}C NMR (75 MHz, $CDCl_3$): 52.3, 127.2, 127.6, 128.1, 130.7, 131.9, 132.8, 134.3, 135.2, 137.9, 141.5, 149.9, 165.8. IR (KBr) ν = 2925, 2854, 1726, 1584, 1458, 1291, 1170, 1088, 1005, 828, 756, 558 cm^{-1}. MS (ESI) m/z 341 (M^+2). *Compound* ***3c***: mp 118–120 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.62 (s, 3H), 7.58 (d, 1H, J = 8.49 Hz), 7.68 (d, 3H, J = 8.49 Hz), 7.88 (d, 1H, J = 7.74 Hz), 7.98 (d, 1H, J = 8.49 Hz), 8.10 (d, 2H, J = 8.49 Hz), 9.2 (s, 1H). ^{13}C NMR (75 MHz, $CDCl_3$): 21.8, 124.8, 126.1, 127.6, 129.0, 132.2, 132.7, 135.5, 140.7, 141.2, 141.5, 142.1, 150.5. IR (KBr) ν = 2925, 2854, 1584, 1458, 1086, 959, 757, 709 cm^{-1}. MS (ESI) m/z 299 (M^+). *Compound* ***4e***: mp 149–151 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.47 (s, 3H), 4.03 (s, 3H), 7.35 (d, 2H, J = 7.554 Hz), 8.13 (d, 3H, J = 8.309 Hz), 8.35 (dd, 1H, J = 1,51 Hz), 8.77 (d, 1H, J = 1.51 Hz), 9.66 (s, 1H). ^{13}C NMR (75 MHz, $CDCl_3$): 21.4, 52.5, 127.5, 128.7, 129.1, 129.9, 131.6, 133.2, 140.4, 141.2, 144.7, 166.2. IR (KBr) ν = 2923, 2854, 1721, 1606, 1541, 1431, 1293, 1172, 1088, 760 cm^{-1}. MS (ESI) m/z 279 (M^+1). *Compound* ***5a***: mp 157–160 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.55 (s, 6H), 7.8 (s, 1H), 7.85 (s, 1H), 8.05 (d, 2H, J = 8.309 Hz), 8.33 (d, 2H, J = 8.309 Hz), 9.2 (s, 1H). ^{13}C NMR (75 MHz, $CDCl_3$): 20.3, 113.1, 117.7, 127.6, 132.3, 132.6, 139.7, 140.6, 140.9, 141.5, 148.4. IR (KBr) ν = 2924, 2854, 2221, 1688, 1456, 1260, 1108, 1021, 842, 546 cm^{-1}. MS (ESI) m/z 259 (M^+). *Compound* ***5b***: mp 159–161 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.62 (s, 3H), 7.6 (t, 1H, J = 8.309 Hz), 7.82 (d, 2H, J = 8.309 Hz), 7.86 (d, 1H, J = 4.34 Hz), 8.0 (d, 1H, J = 2.63 Hz), 8.34 (d, 2H, J = 8.309 Hz), 9.25 (s, 1H). ^{13}C NMR (75 MHz, $CDCl_3$): 21.6, 113.3, 117.4, 126.5, 132.0, 133.6, 139.5, 140.4, 140.8, 142.5, 148.2. IR (KBr) ν = 2923, 2853, 2224, 1606, 1492, 1440, 1308, 1196, 1049, 959, 843, 718, 539 cm^{-1}. MS (ESI) m/z 246 (M^+1). *Compound* ***5c***: mp 209–211 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.9 (d, 2H, J = 8.39 Hz), 8.32 (d, 1H, J = 8.39 Hz), 8.5 (d, 2H, J = 8.39 Hz), 8.6 (d, 1H, J = 8.39 Hz), 9.0 (s, 1H), 9.66 (s, 1H). MS (ESI) m/z 276 (M^+). ^{13}C NMR (75 MHz, $CDCl_3$): 97.9, 112.6, 117.8, 124.1, 124.5, 126.4, 133.9, 135.2, 139.2, 142.2, 143.4, 150.2, 150.5. IR (KBr) ν = 2928, 2843, 2236, 1570, 1459, 1263, 1112, 830 cm^{-1}. *Compound* ***5d***: mp 212–214 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 4.0 (s, 3H), 7.84 (d, 2H, J = 7.55 Hz), 8.2 (d, 1H, J = 9.06 Hz), 8.4 (d, 3H, J = 7.554 Hz), 8.83 (d, 1H, J = 9.06 Hz), 9.42 (s, 1H). ^{13}C NMR (75 MHz, $CDCl_3$): 51.1, 112.4, 116.7, 124.2, 127.9, 129.2, 131.2, 132.6, 133.9, 134.8, 138.2, 141.4, 142.2, 148.4, 165.9. IR (KBr) ν = 2924, 2853, 2221, 1725, 1535, 1458, 1247, 1089, 955, 759 cm^{-1}. MS (ESI) m/z 290 (M^+1).