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An efficient method for the synthesis of quinoxaline derivatives catalyzed by titanium silicate-1

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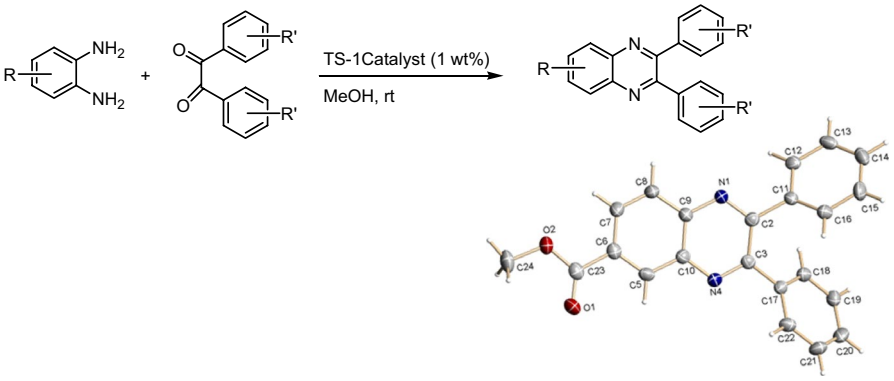
Abstract

A series of quinoxaline derivatives were efficiently synthesized by convenient and simple procedure in excellent yields using 1 wt.% of titanium silicate (TS-1) catalyzed reaction of 1,2-diamines and 1,2-diketones in methanol at room temperature. This reaction is scalable to multigram scale and the catalyst is recyclable.

Graphic abstract



Keywords Quinoxaline · Titanium silicate-1 · *o*-phenylenediamine · Aromatic 1,2-diketones · Room temperature

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Introduction

In recent years, *N*-containing heterocyclic compounds are the most valuable group of scaffolds in pharmaceuticals and bioactive natural products [1]. In this regard, quinoxalines [2, 3] are a versatile class of nitrogen-containing heterocyclic compounds of pharmacological importance due to its interesting biological activities (Fig. 1) such as antiviral, antibiotic, anti-inflammatory, antiprotozoal, antihelmintic, antimalarial, antitubercular, antidepressant, kinase inhibitors, anticancer and also active against AIDS. Besides the biological activities, quinoxaline derivatives have been reported for their applications in synthesis of organic semiconductors [4–6], dyes [7], and electroluminescent materials [8]. Due to their enormous applications in various fields, a number of synthetic strategies have been developed for the preparation of substituted quinoxalines. Most significant method for the synthesis of quinoxaline includes the condensation of an aryl 1,2-diamine with a 1,2-diketone in the presence of catalysts (or reagents) such as in refluxing ethanol or acetic acid [9], in MeOH=AcOH under microwave irradiation [10], in the presence of catalysts such as molecular iodine [11, 12], cerium ammonium nitrate [13–15], glycerine–cerium chloride [16], sulfamic acid [17], polymer-supported sulphanilic acid [18], $Yb(OTf)_3$ [19], oxalic acid [20], *o*-iodoxybenzoic acid (IBX) [21], $H_6P_2W_{18}O_{62}.24H_2O$ [22], $KHSO_4$ [23], Ni-nanoparticles [24], nano-Y-Fe_2O_3-SO_3H [25], nanostructured $Na_2PdP_2O_7$ [26], polyaniline-sulfate salt [27], $InCl_3$ [28], $MnCl_2$ [29], $CuSO_4.5H_2O$ [30], Zn=L-proline [31], tween 40 [32], $HClO_4.SiO_2$ [33], ZnO-loaded mesoporous silica (KIT-6) [34], graphite [35], montmorillonite K-10 [36], Yb-modified NaY zeolite

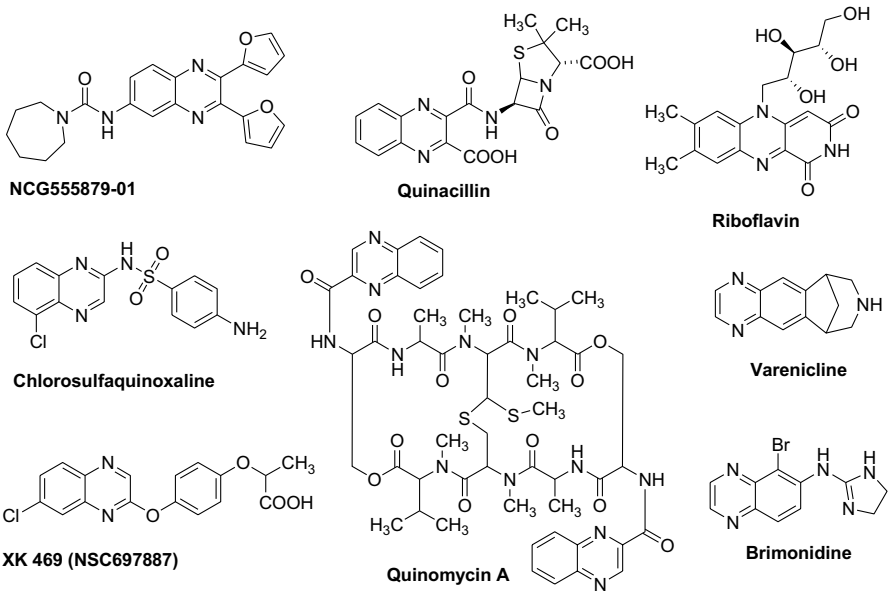


Fig.1 Biologically important quinoxaline derivatives

[37], NH_4X [38], *p*-toluenesulfonic acid [39] as catalyst and solid-phase synthesis [40–43]. Also, the synthesis of quinoxalines via the Ugi reaction is reported by Ayaz et al. [44] Numerous other methods have been reported for synthesis of quinoxaline derivatives including the $Pd(OAc)_2$ [45] or MnO_2 [46–48] catalyzed tandem oxidation of α-hydroxyl ketones, ruthenium catalyzed synthesis of quinoxalines from diols and ortho-diamines [49–52], Bi-catalyzed oxidative coupling of epoxides and ene-1,2-diamines [53], cyclization of *α*-arylimino oximes of *α*-dicarbonyl compounds under reflux in acetic anhydride [54], cyclization–oxidation of phenacyl bromides [55], heteroannulation of nitroketene *N*,*S*-aryliminoacetals with $POCl_3$ [56] and modified HY zeolite [57] catalyzed synthesis of 2-methylquinoxaline from 1,2-phenylenediamine and 1,2-propanediol at 350 °C. Recently, a copper-catalyzed tandem oxidative cycloamination of *N*-arylenamines or ketimines with sodium azide provided an efficient access to quinoxalines [58, 59]. Pardeshi et al. reported the one-pot reaction of styrenes with *o*-phenylenediamines catalyzed by *N*-bromosuccinimide for the synthesis of quinoxalines [60]. Further, oxalic acid was used as building block for the synthesis of quinoxalines from *o*-phenylenediamine [19, 61, 62].

Most of these methods for the preparation of quinoxaline derivatives are associated with one or more of the following drawbacks: (1) use of expensive catalysts, (2) need for anhydrous conditions, (3) unsatisfactory yields, (4) harsh reaction conditions and (5) inefficiency of the method with electron-withdrawing aryl 1,2-diamines substituents. Therefore, development of an efficient, simple, cheap, safe, and green method for the preparation of quinoxalines is desirable. Recently, zeolites as a catalyst in organic synthesis and transformations are becoming more popular due to their greener approach, catalytic loadings and high selectivity. In this regard, zeolite such as titanium silicate (TS-1) [63–74] is being extensively used as a heterogeneous catalyst in various organic transformations. It has several advantages, including being environmentally friendly, nontoxic, inexpensive, recoverable, and reusable. Therefore we decided to explore the catalytic suitability of TS-1 catalyst for the synthesis of quinoxalines. Herein we report, TS-1 (1 wt.%) catalyzed, a simple, efficient and practical method for the preparation of quinoxaline derivatives by the reaction of substituted *o*-phenylenediamine with aromatic 1,2-diketones.

Experimental

General procedure for the synthesis of quinoxalines

A mixture of 1,2-diamine **1** (1.05 mmol), 1,2-diketone **2** (1.00 mmol), and TS-1 (1 wt.%) in methanol (5 ml) was stirred at room temperature. The reaction progress was monitored by TLC. After the completion of reaction, solvent was evaporated under reduced pressure to provide crude product, which was further purified using silica gel column chromatography (hexane -ethyl acetate; 10:1) to obtain pure product **3**.

Spectral data for selected compounds

Methyl 2,3-diphenylquinoline-6-carboxylate (3f)

Yield = 98%; M. P. 144–146 °C; FTIR (KBr): 2935, 1726, 1546, 1498, 1442, 1261 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz): δ 4.02 (s, 3H), 7.28–7.41 (m, 6H), 7.50–7.58 (m, 4H), 8.20 (d, $J = 8$ Hz, 1H), 8.35 (dd, $J = 2$ and 8 Hz, 1H), 8.90 (d, $J = 2$ Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ52.4, 128.2, 128.99, 129.1, 129.3, 129.7, 131.0, 131.8, 138.5, 140.3, 142.99, 154.2,154.9, 166.1; MS: m/z = 339 $[M-1]^+$.

2,3-Bis-(4-fluorophenyl)-6-chloro-quinoxaline (3i)

Yield = 98%; M. P. 132–133 °C; FTIR (KBr): 2920, 1732, 1519, 1230, 839, 702, 678 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz): δ 7.07 (t, $J = 9.1$ Hz, 4H), 7.53 (dd, $J = 8.8$ & 5.6 Hz, 4H),7.72 (dd, $J = 2$ and 8.8 Hz, 1H), 8.10 (d, $J = 8.8$ Hz, 1H), 8.15 (d, $J = 2$ Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 115.6 ($^2J_{C\text{-}F} = 23.0$ Hz), 127.98, 130.3, 131.2, 131.8 ($^3J_{C\text{-}F} = 9.0$ Hz), 134.6 ($^4J_{C\text{-}F} = 3.0$ Hz),135.9, 139.6, 141.4, 152.3, 152.95, 163.4 ($^1J_{C\text{-}F} = 249.0$ Hz); MS: m/z = 353 $[M+1]^+$; HRMS: $m/z = 353.0657$, calcd. for $C_{20}H_{12}ClF_2N_2$, found 353.0652$[M+H]^+$.

Methyl 2,3-Bis-(4-fluorophenyl)quinoxaline-6-carboxylate (3 l)

Yield = 97%; M. P.187–189 °C; FTIR (KBr): 3645, 1742, 1558, 1512, 1230, 848, 457 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz): δ 4.03 (s, 3H), 7.06 (t, $J = 10$ Hz, 4H), 7.50–7.60 (m, 4H), 8.18 (d, $J = 8$ Hz, 1H), 8.3 (dd, $J = 2$ & 8 Hz, 1H), 8.88 (d, $J = 2$ Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 52.6, 115.6 ($^2J_{C\text{-}F} = 22.0$ Hz), 129.3, 129.7, 131.4, 131.8 ($^3J_{C\text{-}F} = 9.0$ Hz), 134.5 ($^4J_{C\text{-}F} = 3.0$ Hz),140.4, 143.1, 152.1, 153.8, 163.4 ($^1J_{C\text{-}F} = 256.0$ Hz), 166.2; MS: m/z = 377 $[M+1]^+$; HRMS: $m/z = 377.1102$, calcd. for $C_{22}H_{15}F_2N_2O_2$, found 377.1094 $[M+H]^+$.

2,3-Bis-(4-bromophenyl)-6-bromo-quinoxaline (3p)

Yield = 95%; M. P.195–196 °C; FTIR (KBr): 3850, 3070, 1664, 1593, 1473, 1342, 820, 410 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz): δ 7.41 (d, $J = 8$ Hz, 4H), 7.51 (d, $J = 8$ Hz, 4H), 7.85 (dd, $J = 2.4$ & 8 Hz, 1H), 8.00 (d, $J = 8$ Hz, 1H), 8.33 (d, $J = 2.4$ Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 123.95, 124.1, 124.3, 130.4, 131.3, 131.4, 131.7, 133.9, 137.2, 137.3, 139.9, 141.7, 152.1, 152.6; MS: m/z = 520 $[M+1]^+$; HRMS: $m/z = 518.8530$, calcd. for $C_{20}H_{12}Br_3N_2$, found 518.8527 $[M+H]^+$.

Methyl 2,3-Bis-(4-bromophenyl)quinoxaline-6-carboxylate (3r)

Yield = 94%; M. P.191–192 °C; FTIR (KBr): 2922, 1730, 1448, 1257, 1217, 974, 833 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz): δ H), 4.03 (s, 3H), 7.40–7.47 (m, 4 H), 7.50–7.57 (m, 4 H), 8.18 (d, $J = 8$ Hz, 1 H), 8.37 (dd, $J = 2$ & 8 Hz, 1H), 8.86 (d,

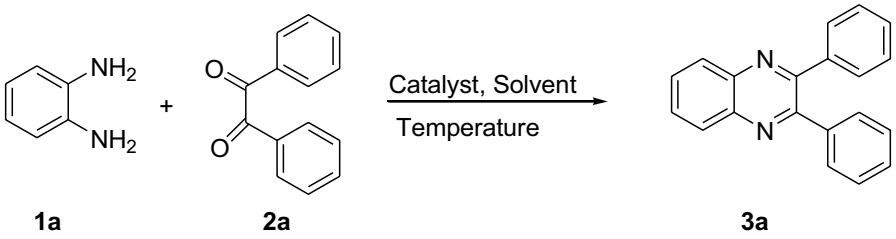
$J=2$ Hz, 1 H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 52.7, 124.1, 124.2, 129.4, 129.9, 131.4, 131.6, 131.7,131.8, 137.2,140.4,143.1, 152.8, 153.5, 166.1; MS: m/z = 499 $[M+1]^+$; HRMS: *m/z* = 498.9480, calcd. for $C_{22}H_{15}Br_2N_2O_2$, found 498.9477 $[M+H]^+$.

Results and discussion

Optimization of reaction conditions

We first studied the preparation of quinoxaline **3a** using *o*-phenylenediamine, **1a** and a slight excess of benzil **2a** as the model reaction (Scheme1, Table 1). When, the reaction of *o*-phenylenediamine, **1a** and benzil, **2a** was carried out using 10 wt.% of TS-1 under solvent-free conditions and in water, the reaction resulted in the formation of desired quinoxaline **3a** in 15% and 40% yields, respectively (Table 1, entries 1 and 2). Even after stirring the reaction mixture for longer time, no substantial increase in the yield was observed. We were delighted to observe the formation quinoxaline in excellent yields when the reaction was carried out using 10 wt.% of TS-1 in methanol (entry 3). Next, the efforts were directed towards the catalytic evaluation of TS-1 and solvent selection to improve the yield of reaction (entries 4–17). It is noteworthy that, the reaction was influenced by the amount of TS-1 and loading of 1 wt.% of TS-1 in methanol was found to be best condition for the synthesis of quinoxaline **3a** (entry 6).

With successful optimization results in hand, we moved on to investigate the scope of the process for reactions of variety of 1,2-diamines with substituted benzils (Scheme 2, Table 2). In the case of simple benzil **2a**, substitution at 1,2-diamine part is well tolerated and corresponding quinoxalines **3a**-**3f** were obtained in excellent yields (Table 2, entries 1–6). Next, when the substituted benzil **2b** and **2c** were used in the reaction under optimized conditions, the corresponding quinoxaline derivatives **3g**-**3r** were also obtained in excellent yields (entries 7–18). The compounds **3k-l** and **3q-r** required longer reaction time as well as loading of 2 wt.% of the TS-1 catalyst (entries 7–10 and 13–16, respectively). A variety of functional groups were well tolerated under the present reaction conditions. The structure of the quinoxaline derivatives **3** were confirmed by the studies of IR, ^{1}H-, ^{13}C-NMR and Mass spectroscopy.



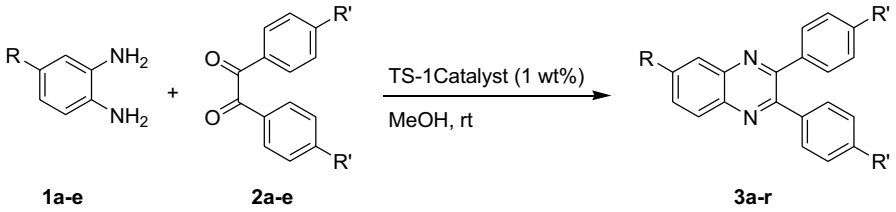
Scheme 1 Synthesis of Quinoxaline **3a** by the reaction of **1a** with **2a** using TS-1 catalyst

Table 1 The reaction of **1a** with **2a** using TS-1 catalyst under different conditions[a]

Entry	Solvent	TS-1 catalyst (wt.%) (%)	Time (min)	% Yield[b]
1	–	10	6 h	15
2	Water	10	6 h	40
3	Methanol	10	30	99
4	Methanol	5	40	99
5	Methanol	2	60	99
6	Methanol	1	60	99
7	Ethanol	1	60	91
8	Dioxane	1	60	89
9	Acetonitrile	1	60	80
10	DCM	1	60	83
11	Chloroform	1	60	92
12	Water	1	12 h	31
13	THF	1	60	87
14	DMF	1	12 h	29
15	DMSO	1	12 h	18
16	Methanol	–	24 h	86
17	Ethanol	–	24 h	42

[a]All the reactions were performed on 1.05 mmol of **1**, 1.00 mmol of **2** and 1 wt.% of TS-1 catalyst in methanol at room temperature unless and otherwise stated

[b]Isolated yields after column chromatography



Scheme 2 Synthesis of Quinoxaline **3** by the reaction of **1** with **2** using TS-1 catalyst

In order to confirm the structure of methyl 2,3-diphenylquinoxaline-6-carboxylate (**3f**), we carried out the X-ray analysis of this compound. Single crystals of the compound **3f** were grown by slow evaporation of the solution mixture of hexane and ethyl acetate. An ORTEP drawing, depicted in Fig. 2, shows the crystal structure of the **3f** [84]. CCDC reference: 973,448, mp 144–146 °C. Pale yellow colored crystal of approximate size $0.23 \times 0.13 \times 0.04$ mm^3, was used for data collection on *Bruker SMART APEX* CCD diffractometer using Mo K_α radiation. Crystal, exposure/frame = 5.0 s/frame, θ range = 2.33°–24.49°, completeness to θ of 24.49° is 99.9%. SADABS correction applied, $C_{22}H_{16}N_2O_2$, $M = 340.37$. Crystals belong to triclinic, space group P-1, $a = 7.8345(4)$,

Table 2 Synthesis of Quinoxaline derivatives **3a–3r** with 1 wt.% of TS-1 catalyst[a]

Entry	R	R´	Time (min)	Product	Yield (%)[c]	Mp (°C) (observed)	Mp (°C) (reported)	References
1	–H	–H	60	**3a**	99	128–129	126–127	[11]
2	$–CH_3$	–H	45	**3b**	98	104–106	115–116	[75]
3	–Cl	–H	60	**3c**	97	104–106	122–123	[76]
4	–Br	–H	60	**3d**	98	122–123	123.5–123.6	[77]
5	$–NO_2$	–H	24 h	**3e**	97	193–194	193–194	[30]
6	$–CO_2$**Me**	–H	60	**3f**	98	144–146	144–145	[78]
7	–H	–F	45	**3 g**	99	135–136	135–137	[21]
8	$–CH_3$	–F	45	**3 h**	98	165–166	165–167	[21]
9	–Cl	–F	60	**3i**	98	132–133	133–134	[79]
10	–Br	–F	60	**3j**	98	168–169	168–170	[80]
11	$–NO_2$	–F	24 h	**3 k**	96	175–176	176–177	[31]
12	$–CO_2$**Me**	–F	3 h	**3 l**	97[b]	187–189	188–189	[78]
13	–H	–Br	60	**3 m**	98	138–141	194–195	[81]
14	$–CH_3$	–Br	60	**3n**	98	175–176	185–186	[81]
15	–Cl	–Br	60	**3o**	95	166–168	167–168	[82]
16	–Br	–Br	60	**3p**	95[b]	195–196	–	[83]
17	$–NO_2$	–Br	24 h	**3q**	89[b]	166–167	188–190	[77]
18	$–CO_2$**Me**	–Br	6 h	**3r**	94	191–192	193–194	[78]

[a]All the reactions were performed on 1.05 mmol of **1**, 1.00 mmol of **2** and 1 wt.% of TS-1 catalyst in methanol at room temperature unless and otherwise stated

[b]Reactions were carried out using 1.05 mmol of **1**, 1.00 mmol of **2** and 2 wt.% of TS-1 catalyst in methanol at room temperature

[c]Isolated yields after column chromatography

$b = 9.5425(5)$, $c = 12.5293(6)$ Å, $\alpha = 91.285(1)°$, $\beta = 99.154(1)°$, $\gamma = 112.820(1)°$, $V = 848.74(7)$ Å^3, $Z = 2$, $D_c = 1.332$ mg m^{-3}, $T = 170(2)$ K, 7632reflections measured, 2814unique [$I > 2\sigma(I)$], R value 0.0683, wR2 = 0.1707. All the data were corrected for Lorentzian, polarization and absorption effects. SHELX-97 (ShelxTL) [85] was used for structure solution and full matrix least squares refinement on F^2. Hydrogen atoms were included in the refinement as per the riding model.

The four bond length of N(1)-C(2), N(4)-C(3),N(1)-C(9) and N(4)-C(10) in **3f** were found to be 1.313(3) Å, 1.311(3) Å, 1.358(3) Å and 1.366(3) Å, respectively, and clearly shows that nitrogen atoms are the part of aromatic ring. The carbon–oxygen bond lengths in the ester groups were found to be 1.211(3)Å for O(1)-C(23) double bond and 1.440(3)Å for O(2)-C(24) single bond. The torsion angles for N(1)-C(9)-C(10)-N(4), N(4)-C(3)-C(2)-N(1) and C(17)-C(3)-C(2)-C(11) were -6.2(4)°, -9.7(4)° and -13.8(4)°, respectively.

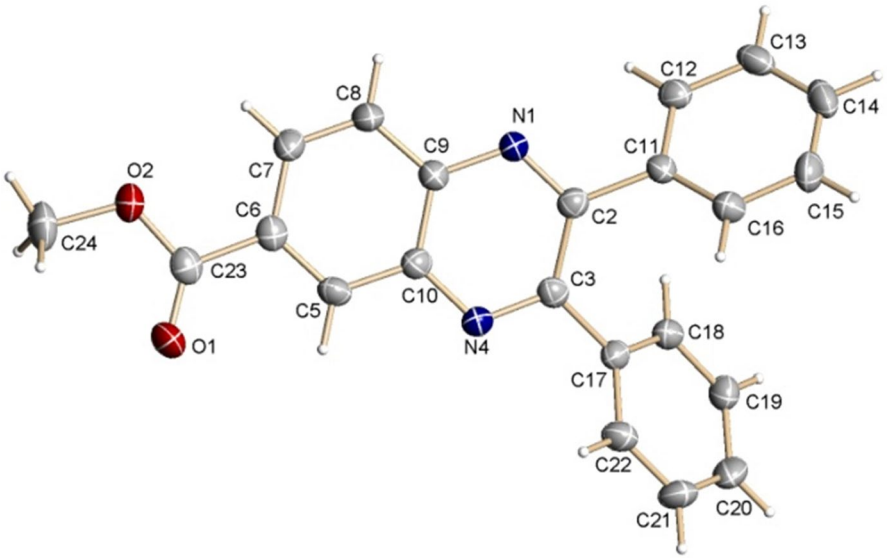


Fig.2 Crystal structure x-ray ORTEP diagram of compound **3f**. Ellipsoids are drawn at 50% probability

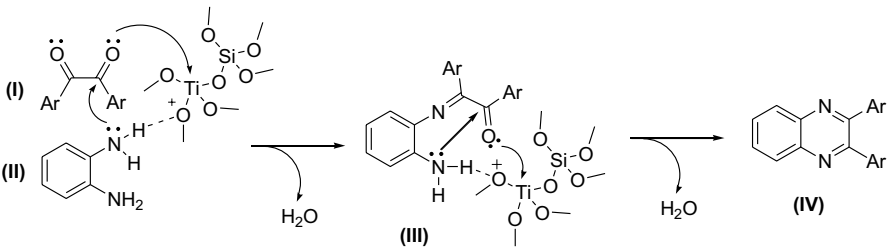


Fig. 3 Plausible reaction mechanism for quinoxaline formation in the presence of TS-1 catalyst

Proposed mechanism for the formation of quinoxaline derivatives

Figure 3 shows a plausible reaction mechanism for the formation of quinoxalines. Firstly, the lone pair of one of the carbonyl oxygen of 1,2-diketone (**I**) interacts with TS-1 catalyst to enhance electrophilicity and nucleophilic attack of nitrogen of *o*-phenylenediamine (**II**) takes place immediately. At the same time, the TS-1 catalyst acts as a Bronsted base and accelerates the reaction by elimination of water molecule to form a carbon–nitrogen double bond (C=N) (**III**). Respectively, one more elimination of water molecule results in formation of second carbon–nitrogen double bond (C=N) giving rise to the required target molecule quinoxaline (**IV**).

Table 3 Reusability of TS-1 catalyst[a]

Reaction turn	Time (min)	% Yield (3a)[b]
1st	60	99
2nd	60	98
3rd	60	98
4th	60	97

[a]Reaction details: **1a** (1.135 g, 10.5 mmol), **2a** (2.102 g, 10.0 mmol), TS-1 (2 wt.%), methanol, room temperature

[b]Isolated yields after column chromatography

Catalyst reusability

Recyclability of catalyst is the most important and salient feature of present reaction. As shown in Table 3, the reusability of catalyst is checked at gram scale by reaction of *o*-phenylenediamine, **1a** (1.135 g, 10.5 mmol) with benzil, **2a** (2.102 g, 10.0 mmol) using 2 wt% of TS-1 catalyst in methanol. The reactions resulted in formation of required quinoxaline compound **3a** in excellent yields after column chromatographic purification. The TS-1 catalyst could be reused at least five times. Side products were not observed in these reactions. Furthermore, the reaction can be scaled up to a multigram scale.

Conclusion

In conclusion, we have developed a convenient and simple methodology for the synthesis of quinoxaline derivatives using the reaction of 1,2-diamines and 1,2-diketones with 1 wt.% of titanium silicate (TS-1) in methanol at room temperature. Structure of the compound **3f** was confirmed by x-ray analysis. This reaction is scalable to multigram scale and the catalyst is recyclable.

Supporting information

Full Experimental details with ^{1}H-NMR and ^{13}C-NMR spectra can be found via the "Supplementary Content" section of this articles webpage.

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