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Research Article

FE(ACAC)$_3$-CATALYZED ONE-POT SYNTHESIS OF SUBSTITUTED QUINOXALINE DERIVATIVES

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ABSTRACT

Objective: The study was to develop an efficient and practical method for the synthesis of substituted quinoxaline derivatives, which are known for their significant biological activities and structural versatility, and serve as important building blocks in organic synthesis.

Methods: A series of substituted quinoxaline derivatives were synthesized through a one-pot cyclocondensation reaction between substituted phenyl diamines and phenacyl bromide. The reaction was catalyzed by Fe(acac)$_3$ and carried out at 60 °C for a duration of 50–80 min.

Resuts: The reaction successfully afforded quinoxaline derivatives (3a–3m) in good to excellent yields ranging from 88–94%. The synthesized compounds exhibited high purity, demonstrating the efficiency of the developed protocol.

Conclusion: The present methodology offers several practical advantages, including a simple one-pot procedure, facile isolation of products, high yields, and an efficient cascading cyclocondensation process. This approach provides an effective and reliable route for the synthesis of substituted quinoxaline derivatives.

Keywords: Fe(acac)$_3$, Phenyldiamine, Cyclocondensation, Phenacyl bromide, Quinoxaline

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INTRODUCTION

Quinoxalines represent a significant class of nitrogen-containing heterocyclic compounds that have attracted considerable attention in recent decades due to their diverse biological and pharmaceutical potentials [1]. Although these compounds are rarely found in natural sources, they have been widely synthesized through a variety of chemical methodologies [2]. With the growing emphasis on environmental sustainability and green chemistry, significant efforts have been directed toward the development of eco-friendly and efficient synthetic strategies for quinoxaline derivatives. These approaches often utilize recyclable catalysts, non-toxic solvents, and mild reaction conditions, aligning with the principles of green chemistry [3].

In recent years, quinoxalines have emerged as highly promising scaffolds in medicinal chemistry owing to their broad spectrum of biological activities [4]. Numerous quinoxaline-based derivatives have demonstrated potent bioactivities, including antitubercular [5], anti-inflammatory, antioxidant [6], antifungal, anticancer, anti-HIV [7] and antiprotozoal properties [8]. Their structural versatility and ease of functionalization have made them valuable templates for drug design and development.

Moreover, many clinically approved drugs and investigational agents incorporate a quinoxalines moiety as a key pharmacophore unit within their molecular framework, which significantly contributes to their therapeutic efficacy. The incorporation of the quinoxaline nucleus often enhances target specificity, bioavailability and metabolic stability. Examples of such drugs are illustrated in fig. 1, showcasing the relevance of this heterocyclic core in modern pharmaceutical chemistry.

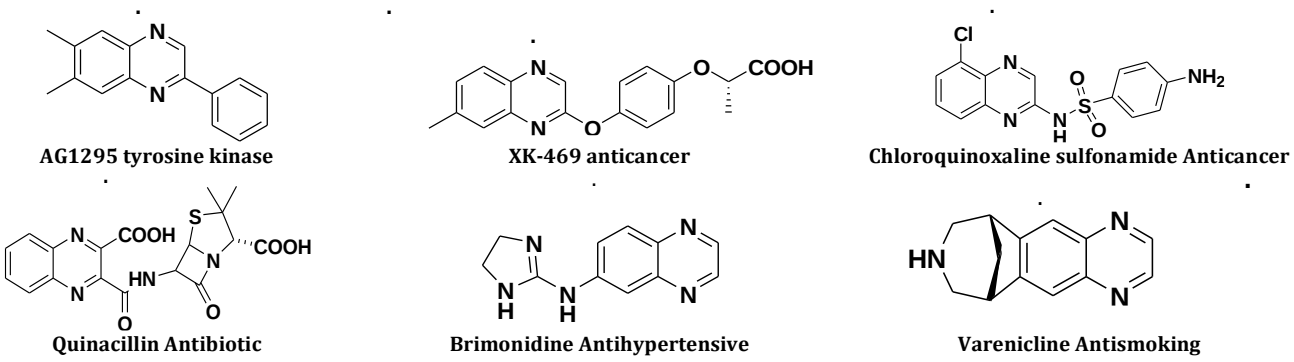


Fig. 1: Quinoxaline core with natural and bioactive compounds

Quinoxalines are nitrogen-containing heterocycles found in various natural and synthetic compounds. They serve as key building blocks in heterocyclic synthesis and play a crucial role in organic and medicinal chemistry. Recently, numerous methods have been developed to diverse

synthesis of quinoxaline derivatives via transition metals [9]. In recent years, focusing on several environmental issues and considering the pharmacological importance of quinoxaline scaffolds there are numerous synthetic methods have been reported, Some of them are the condensation of *o*-pheneylenediamine with 1,2-dicarbonyl compounds, Among the aforementioned protocols, condensation of *o*-pheneylenediamine with phenacyl bromides catalyzed by solid acid catalyst[10]and using transition metal catalysts as well [11] is also highly preferred which includes $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$ [12], $Na_2PdP_2O_7$ [13] and $Ga(ClO_4)_3$ [14], Synthesis of spiro-indeno[1,2-b]quinoxalines via a g-Fe_2O_3@Oxo-triazolidin-sultone Nano catalyst [15], $CuBr/O_2$ catalytic system [16]. Additionally, oxidative cyclization betweendeoxybenzoins and 1,2-pheneylenediaminein the presence of a catalytic amount of a Cu(II)-complex of a zwitterioniccalix [17], Ni(II)/1,10-phenanthroline-catalyzeddehydrogenativecoupling reaction for the synthesis of quinoxalines [18] and quinoxaline synthesis using heterogeneous solid zinc oxide nanoparticles loaded on mesoporous silica (ZnO-KIT-6) [19] have also been reported.

MATERIALS AND METHODS

All chemicals and solvents were dried before use and purchased commercially and used exactly as supplied. Chemicals and solvents were procured from Sigma Aldrich and Spectrochem. Melting points were determined in open capillary and are uncorrected. Reactions were monitored by thin layer chromatography (TLC) on silica gel plates (GF 254) using UV light to visualize the course of the reactions. ^{1}H NMR spectra and ^{13}C NMR spectra were recorded Bruker Avance 300-400 (FT-NMR) and Bruker DRX-300 instruments, respectively, using $CDCl_3$ and DMSO-d_6 as solvent. Chemical shifts are reported in δ ppm with TMS as internal standard. High-resolution mass spectra (HRMS) were obtained using the Agilent 6520 (Q-TOF) ESI-HRMS instrument. Routine monitoring of reaction was performed by TLC using 0.25 mm E. Merck precoated silica gel TLC plates (60 F254) hexane: ethyl acetate as eluent.

General experimental procedure for synthesis of quinoxalines

In a round-bottom flask, a mixture of phenacyl bromide (2a–2m, 0.001 mol) and $Fe(acac)_3$ catalyst (40 mg) was dissolved in ethanol (5 ml) and stirred at room temperature for 10 min. Subsequently, *o*-pheneylenediamine (1a–1b), 0.001 mol) was added slowly to the reaction mixture, and the resulting homogeneous mixture was stirred at 60 °C for the stipulated time. The progress of the reaction was monitored by thin-layer chromatography (TLC). The reaction mixture was diluted with ethyl acetate (20 ml) anddiluted HCl (5 ml, 0.1 N). The combined organic layers were dried using anhydrous $MgSO_4$, filtered, and the solvent was removed by evaporation. The crude product was purified by crystallization using ethanol to afford the pure 2-pheny quinoxalines (3a–3k). All desired product characterised by IR, H^1-NMR, C^{13}NMR and melting points of the desired products were found to be in good agreement with those reported in the literature.

2-Phenylquinoxaline (3a) [8]

Isolated as a Yellow Solid. Yield 90 %; Mp. 78 C(Lit[1, 4, 21]75-76 C). IR (KBr, cm^{-1}): ν_{max} 3447, 3059, 2921, 2852, 1631, 1544, 1487, 1314, 1224, 1028, 956, 766 cm^{-}[11]H NMR (500 MHz, Chloroform-d) δ 9.32 (s, 1H, =CH), 8.11-8.21 (m, 4H, Ar-H), 7.71-7.80 (m, 2H), 7.50-7.59 (m, 3H, Ar-H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 151.9, 143.5, 142.5, 141.7, 136.9, 130.3, 130.3, 129.8, 129.6, 129.2, 127.7 HRMS (ESI)$^+$m/z calcd. for $C_{14}H_{10}N_2$ $(M+H)^+$: 207.0923; found 207.0926.

2-(4-Fluorophenyl)Quinoxaline (3b) [4]

Isolated as a Yellow Solid, Yield 90%; Mp. 113-119 C (Lit[4, 21]112-118 C). IR (KBr, cm^{-1}): $\bar{\upsilon}_{max}$ 3421, 2927, 1633, 1583, 1534, 1475, 1418, 1101, 1073, 955, 830, 759 cm^{-1}^{1}H NMR (300 MHz, $CDCl_3$) δ 9.25 (s, 1H, =CH), 8.07-8.19 (m, 4H, Ar-H), 7.68-7.78 (m, 2H, Ar-H), 7.18-7.24 (m, 2H, Ar-H). ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 165.9, 162.6, 150.7, 142.9, 142.2, 141.5, 133.0, 132.9, 130.4, 130.2, 129.6, 129.6, 129.5, 129.2, 116.4, 116.1. HRMS (ESI)$^+$m/z calcd. for $C_{14}H_9FN_2$ $(M+H)^+$: 225.0823; found 225.0852

2-(4-Chloro phenyl)-Quinoxaline (3c)lit [1, 21]

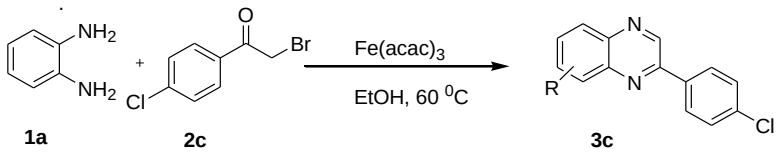
Isolated as a Yellow solid, Yield 94%; Mp: 128-132 °C; IR (KBr, cm^{-1}): ν_{max}2924, 1591, 1537, 1479, 1310, 1122, 1043, 958, 832, 753 cm^{-1}. ^{1}H NMR (400 MHz, CDCl3) δ 9.28 (s, 1H, =CH), 8.09-8.15 (m, 4H), 7.72-7.80 (m, 2H), 7.51 (d, J = 8.5 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 150.7, 142.9, 142.3, 141.8, 136.7, 135.3, 130.6, 129.9, 129.7, 129.5, 129.3, 128.9. (CH_3). MS (ESI):m/z calcd for $C_{14}H_9ClN_2$ $[M+H]^+$: 241.3113, found 241.0512

2-(4-Bromo phenyl)-Quinoxaline (3e)lit [4, 21]

Isolated as a pale yellow solid, Yield 92%; Mp. 128-134 °CIR (KBr, cm^{-1}): ν_{max} 3443, 2925, 1634, 1583, 1536, 1481, 1421, 1307, 1121, 1070, 954, 827, 710 cm-[11]H NMR (400MHz, CDCl3) δ 9.26 (s, 1H =CH), 8.04-8.13 (m, 4H, Ar), 7.65-7.79 (m, 4H, Ar). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 150.7, 142.8, 142.3, 141.8, 135.7, 132.4, 130.6, 129.9, 129.7, 129.3, 129.1, 125.1. MS (ESI): m/z calcd for $C_{14}H_9BrN_2$ $[M+H]^+$: 287.21, found 287.07

RESULTS AND DISCUSSION

The synthesis of quinoxaline were the model reaction of *o*-pheneylenediamine (1a) and phenacyl bromide (2c) was carried out in presence iron acetyl acetonate ($Fe(acac)_3$) stirred at 60 °C for 60 min, with good yield 92 % with high purity productsas shown in scheme 1.



Scheme 1: Synthesis of quinoxalines using $Fe(acac)_3$ catalyst

The optimization of the reaction was performed by varying the reaction parameters, such as reaction time, solvent, and temperature. It was observed that the quinoxaline formation in ethanol solvent proceeds with an excellent yield at 60 °C for 50 min (table 1, entry 3).

The IR spectrum of compound quinaxoline indicates presence of two bands at 3447 and 3059 cm^{-1}. (C_{Ar}), 1631, 1544, (C=N), which corresponds to Imine and Aromatic region respectively. 2-Phenyl quinoxalines 3a characterized by ^{1}H-NMR spectrum, the Aromatic peak-appears as a doublet peak at δ 8.21 – 8.11 due to Ar-N and Ar-H of ten proton and The very important signal is obtained at 9.32 δ, which is singlet of Aromatic ring HC=N. The

2-Phenyl quinoxalines 3a compound charecretised by ^{13}C NMR 151.9(C=N), (C_{Ar}), 143.5 (C, S-C=N), 142.5 indicates that formation of Quinoline ringlit [1-4, 21].

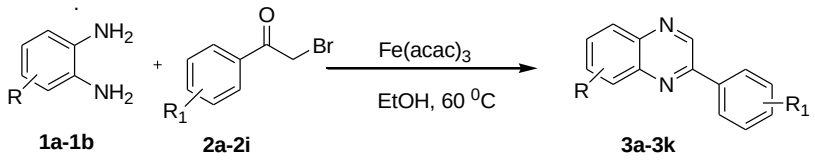
First, we optimized the solvent for synthesizing the target molecule. The model reaction initially water was used as the solventyielded low product. To improve the yield of this, we Screen different solvents, including protic (water, methanol, ethanol) solvents and aprotic (dioxane, Chloroform, DCM, acetonitrile). The results are summarized in table 1. The highest yield was obtained with ethanol (table 1, entry 3), while protic solvents resulted in higher yields than aprotic ones. Among them, ethanol proved most effective for Quinoxalinesynthesis, making it the optimal solvent table 1.

Table 1: Screening of reaction condition with respect to solvent 3c

S. No.	Solvent	Catalyst (10 mol%)	Yield %
1	H_2O	$Fe(acac)_3$	40
2	MeOH	$Fe(acac)_3$	87
3	**EtOH**	**$Fe(acac)_3$**	**95**
4	DCM	$Fe(acac)_3$	75
5	AcCN	$Fe(acac)_3$	78
6	$CHCl_3$	$Fe(acac)_3$	64
7	Dioxane	$Fe(acac)_3$	58
8	EtOH	No catalyst	20

[a]Reaction conditions: Phenacyl bromide (0.001 mol), o-pheneylenediamine(0.001 mol), 10 mol % $Fe(acac)_3$in 10 ml EtOH, at 60 °C for 50 min.
[b]Isolated yields

The influence of two key experimental parameters, catalystand solvent, was systematically studied. Additionally, the reaction scope was explored by varying phenacyl bromide and and *o*-pheneylenediaminesubstrates, as illustrated in Scheme 2 and summarized in table 3. The synthesis of quinoxalines was carried out by reacting substituted phenacyl bromides/benzil (2a–2i) (0.001 mol) and *o*-pheneylenediamine(1a–1b) (0.001 mol) in the presence of $Fe(acac)_3$ as a catalyst. Further, the reaction mass was stirred at 60 °C for 40–80 min. afforded quinoxalines (3a–3k) with excellent yields and high purity.

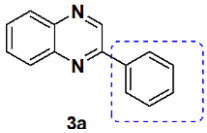
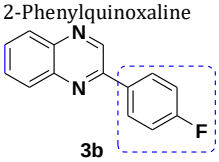
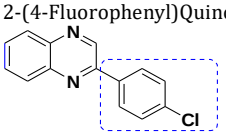
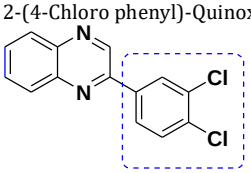


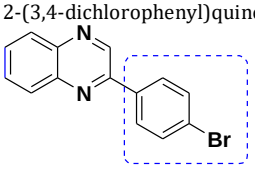
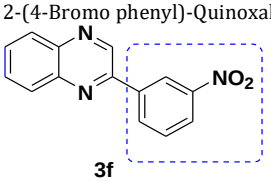
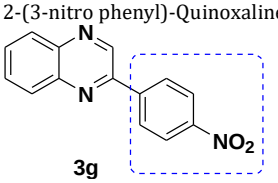
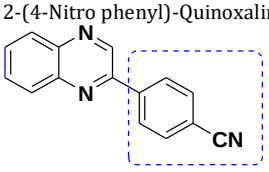
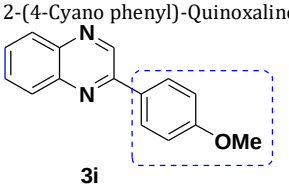
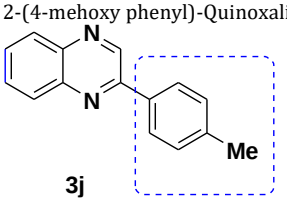
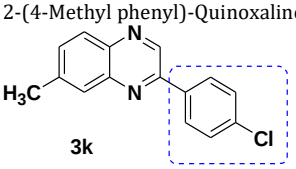
1a-1b **2a-2i** **3a-3k**

1a-1b (R = H, CH_3)

Scheme 2: Synthesis of quinoxalines (3a–3k) using $Fe(acac)_3$ catalyst

Table 2: Screening of reaction condition with respect to substrate

S. No.	Amine	Phenacyl bromide	Product	Yield[b]
1	*o*-pheneylene diamine	4-Chloro Phenacylbromide	 **3a** 2-Phenylquinoxaline	88
2	*o*-pheneylene diamine	4-Fluro phenacylbromide	 **3b** 2-(4-Fluorophenyl)Quinoxaline	90
3	*o*-pheneylene diamine	4-Chloro Phenacylbromide	 **3c** 2-(4-Chloro phenyl)-Quinoxaline	94
4	*o*-pheneylene diamine	3,4 dichloroPhenacylbromide	 **3d**	93

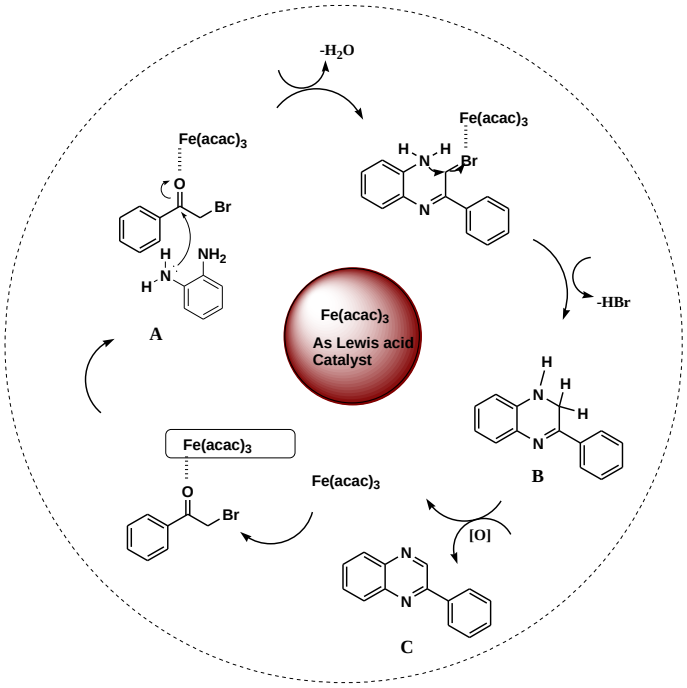
5	*o*-pheneylene diamine	4-bromo Phenacylbromide	2-(3,4-dichlorophenyl)quinoxaline  **3e**	92
6	*o*-pheneylene diamine	3-nitro Phenacylbromide	2-(4-Bromo phenyl)-Quinoxaline  **3f**	89
7	*o*-pheneylene diamine	4-Nitro Phenacylbromide	2-(3-nitro phenyl)-Quinoxaline  **3g**	88
8	*o*-pheneylene diamine	4-Cyano Phenacylbromide	2-(4-Nitro phenyl)-Quinoxaline  **3h**	92
9	*o*-pheneylene diamine	4-Methoxy Phenacylbromide	2-(4-Cyano phenyl)-Quinoxaline  **3i**	89
10	*o*-pheneylene diamine	4-methyl Phenacylbromide	2-(4-mehoxy phenyl)-Quinoxaline  **3j**	90
11	4-methyl-o-pheneylenediamine	4-Chloro Phenacylbromide	2-(4-Methyl phenyl)-Quinoxaline  **3k** 2-(4-Chloro-phenyl)-7-methyl-quinoxaline	91

[a]Reaction conditions: Phenacyl bromide (0.001 mol), o-phenylenediamine (0.001 mol), 10 mol % $Fe(acac)_3$ in 10 ml EtOH, at 60 °C for 50 min.
[b]Isolated yields (3a-3k)

It was observed that both electron-donating and electron-withdrawing substituents on phenacyl bromide substrates facilitated smooth reactions, yielding the desired products (3a-3k) in high yields (88–94%). However, phenacylbromide derivatives containing electron-withdrawing groups such as nitro, fluoro, chloro, bromo, cyano, and exhibited a faster reaction rate compared to those with electron-donating groups such as methyl and methoxy. This suggests that electron-withdrawing substituents enhance the reactivity of phenyl diamine in this transformation. All synthesized products were thoroughly characterized using (IR) spectroscopy, (^{1}H NMR), (^{13}C NMR), and mass spectrometry, for confirming their structures. All data can agreement with reported data Lit [1, 4, 8, 21]

Plausible mechanism for synthesis of quinoxaline by $Fe(acac)_3$ catalyst

The plausible mechanism for the quinoxalines synthesis was Proposes in scheme 3, which involves the protonation of the carbonyl group of phenacyl bromide over $Fe(acac)_3$ catalyst (A). Later on, it reacts with *o*-pheneylenediaminethat involves dehydration and dehalogenation simultaneously, resulting in the formation of cyclic product B, which is readily oxidized in air to form desired product C [20].



Scheme 3: Plausible mechanism for the synthesis of quinoxaline derivatives

CONCLUSION

In conclusion, we have developed a mild, efficient, and environmentally benign synthetic protocol for the synthesis of quinoxalines (3a–3k) from substituted phenacyl bromides and *o*-pheneylenediamines using $Fe(acac)_3$ as a catalyst. A key feature of this protocol is its simple reaction conditions and the absence of side reactions, leading to the formation of the desired products in high yields. This method serves as an effective alternative to conventional processes for the synthesis of quinoxalines with good to excellent yields (88-94%).

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Nil

AUTHORS CONTRIBUTIONS

The authors are equally contributed to the design, optimization, referencing analysis, interpretation, drafting, and final approval of the manuscript.

Design and Experimental work Suraj Ade, Analysis of Data Sunil Choudhare and Santosh Padghan and Manojkumar Chopadedrafting and Allreaming things as corresponding Author

CONFLICT OF INTERESTS

Declared none

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