

Research Article

Catalytic Application of Iron for Towards Synthesis of Quinoxaline

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Received: September 14, 2025

Accepted: October 13, 2025

Published: October 20, 2025

Abstract

The study investigates the synthesis of substituted quinoline derivatives known for their biological activities and structural versatility which serve as pivotal building blocks in organic synthesis. A series of substituted quinoline derivatives were synthesized by one-pot cyclocondensation between substituted phenyldiamine with phenylacetyl bromide by Fe(acac)₃ as catalyst at 60 °C for 50–80 min. afforded quinoxalines (3a–3m) with good to excellent yields (88–94 %) with high purity. The used techniques provide the practical benefits of facile isolation, high yields and cascading one pot condensation.

Keywords: Cyclocondensation; Phenyldiamine; Phenylacetyl bromide; Fe(acac)₃; Quinoxalines

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 quinoxaliny moiety as a key pharmacophoric 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 Figure 1, showcasing the relevance of this heterocyclic core in modern pharmaceutical chemistry. Quinoxalines are nitrogen-containing heterocycles found in various natural and synthetic compounds.

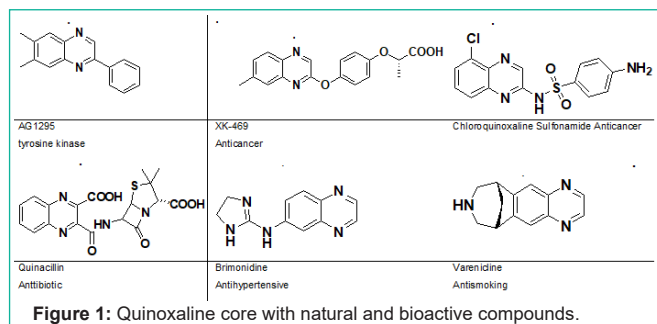


Figure 1: Quinoxaline core with natural and bioactive compounds.

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 metal [8]. 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*-phenylenediamine with 1,2-dicarbonyl compounds. Among the aforementioned protocols, condensation of *o*-phenylenediamine with phenylacetyl bromides in the solid phase is highly preferred [9–15], (NH₄)₆Mo₇O₂₄·4H₂O [16], PVPPOTf [17] and Ga(ClO₄)₃ [18]. As well as by using transition metal catalysts [19,20] and also using various substrate to synthesis of quinoxaline includes, Synthesis of spiro-indeno[1,2-*b*]quinoxalines via a g-Fe₂O₃@Oxo-triazolidin-sulfone nanocatalyst, CuBr/O₂ catalytic system, oxidative cyclization between deoxybenzoins and 1,2-phenylenediamines in the presence of a catalytic amount of a Cu(II)-complex of a zwitterionic calix, Ni(II)/1,10-phenanthroline-catalyzed dehydrogenative coupling reaction for the synthesis of quinoxalines, quinoxalines by using

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

Sr. No	Solvent	Catalyst	Yield %
1	H ₂ O	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	DMF	Fe(acac) ₃	64
7	Dioxane	Fe(acac) ₃	58
8	EtOH	No catalyst	20

NR: no reaction. ^aReaction conditions: Phenacyl bromide (0.001 mol), *o*-phenylenediamine (0.001 mol), 20mol % Fe(acac)₃ in 10 ml EtOH, at 60 °C for 50 min. ^bIsolated yields.

Figure 2: Plausible Mechanism for synthesis of Quinoxaline.

Sr.no	Amine	Phenacyl bromide	Product	Yield
1	<i>o</i> -phenylene diamine	4-Chloro Phenacylbromide		88
2	<i>o</i> -phenylene diamine	4-Fluoro phenacylbromide		90
3	<i>o</i> -phenylene diamine	4-Chloro Phenacylbromide		94
4	<i>o</i> -phenylene diamine	3,4 dichloro Phenacylbromide		93
5	<i>o</i> -phenylene diamine	4-bromo Phenacylbromide		92
6	<i>o</i> -phenylene diamine	3-nitro Phenacylbromide		89
7	<i>o</i> -phenylene diamine	4-Nitro Phenacylbromide		88
8	<i>o</i> -phenylene diamine	4-Cyano Phenacylbromide		92

heterogeneous solid zinc oxide nanoparticle loaded mesoporous silica (ZnOKIT-6).

Experimental Section

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. ¹H NMR spectra and ¹³C NMR spectra were recorded Bruker Avance 300-400 (FT-NMR) and Bruker DRX-300 instruments, respectively, using CDCl₃ 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 [21-30].

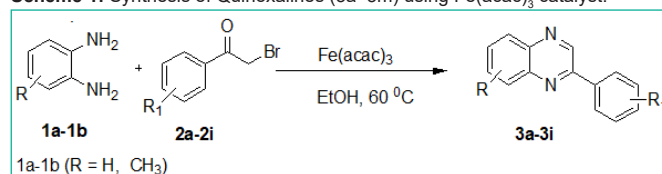
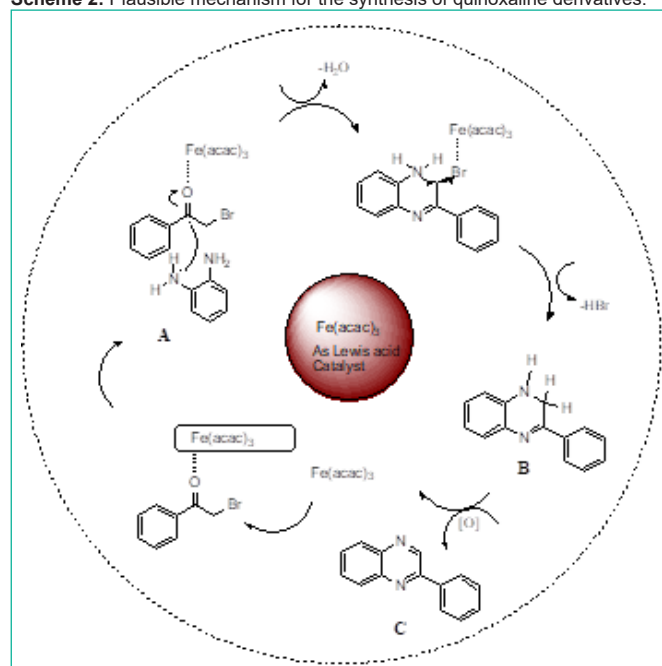
General Experimental Procedure for Synthesis of Benzothiazole

In two a neck round bottom flask was charged with a mixture of Phenacyl bromide (2a-m) (0.001mol) and Fe(acac)₃ catalyst (40 mg) were dissolved in EtOH (5 mL) at room temperature for 10 min. Then *O*-phenyldiamine (1a-b) (0.001mol) was added slowly to the reaction mixture at 60°C. The progress of the reaction was monitored by thin-layer chromatography (TLC). The reaction mixture was diluted with ethyl acetate (20 mL) and diluted HCl (5 mL). The combined organic layers were dried using anhydrous MgSO₄, filtered, and the solvent was removed by evaporation. The crude product was purified by crystallization using ethanol to afford the pure 2-phenyl quinoxalines (3a-3m). All desired product characterised by IR, ¹H-NMR, ¹³CNMR and melting points of the desired products were found to be in good agreement with those reported in the literature [31-41].

Phenylquinoxaline (3a)^a

Isolated as a Yellow Solid. Yield 90 %; Mp. 88 °C (Lit¹⁴ 75-76 °C). IR (KBr, cm⁻¹): $\nu_{\max}$ 3447, 3059, 2921, 2852, 1631, 1544, 1487, 1314, 1224, 1028, 956, 766 cm⁻¹. ¹H 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

Scheme 1: Synthesis of Quinoxalines (3a-3m) using Fe(acac)₃ catalyst.

**Scheme 2:** Plausible mechanism for the synthesis of quinoxaline derivatives.

(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 $\text{C}_{14}\text{H}_{10}\text{N}_2$ (M+H)⁺: 207.0923; found 207.0926.

(4-Fluorophenyl) benzothiazole (3b)⁴

Isolated as a Yellow Solid, Yield 90%; Mp. 113-119 °C (Lit¹⁴ 112-118 °C). IR (KBr, cm^{-1}): ν_{max} 3421, 2927, 1633, 1583, 1534, 1475, 1418, 1101, 1073, 955, 830, 759 cm^{-1} . ^1H 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 $\text{C}_{14}\text{H}_9\text{FN}_2$ (M+H)⁺: 225.0823; found 225.0852.

(4-Chloro phenyl)-Quinoxaline (3c)¹

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} . ^1H NMR (400 MHz, CDCl_3) 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. (CH₃)₀. MS (ESI): m/z calcd for $\text{C}_{14}\text{H}_9\text{ClN}_2$ [M+H]⁺: 241.3113, found 241.0512.

(4-Bromo phenyl)-Quinoxaline (3c)

Isolated as a pale yellow solid, Yield 92%; Mp. 128-134 °C IR (KBr, cm^{-1}): ν_{max} 3443, 2925, 1634, 1583, 1536, 1481, 1421, 1307, 1121, 1070, 954, 827, 710 cm^{-1} . ^1H NMR (400MHz, CDCl_3) 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 $\text{C}_{14}\text{H}_9\text{BrN}_2$ [M+H]⁺: 287.21, found 287.07.

Results and Discussion

The synthesis of quinoxalines was carried out by reacting substituted phenacyl bromides/benzil (2a–2m) (0.001 mol) and *o*-phenylenediamine (1a–1b) (0.001 mol) in the presence of $\text{Fe}(\text{acac})_3$ as a catalyst. Further, the reaction mass was stirred at 60 °C for 50–80 min. afforded quinoxalines (3a–m) with excellent yields and high purity.

This reaction is outlined in Scheme 1. The structures of all the synthesized quinoxalines (3a–m) are shown in Figure 2. The model reaction was carried out in presence of *o*-phenylenediamine (1a) and phenacyl bromide (2a) as shown in Scheme 1.

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). no reaction was observed when water was used as the solvent.

The plausible mechanism for the quinoxalines synthesis was depicted in Scheme 3, which involves the protonation of the carbonyl group of phenacyl bromide over $\text{Fe}(\text{acac})_3$ catalyst (A). Later on, it reacts with *o*-phenylenediamine that involves dehydration and dehalogenation simultaneously resulting in the formation of cyclic product B, which is readily oxidized in air to form desired product C.

Conclusion

In conclusion, we have developed a mild, efficient, and environmentally benign synthetic protocol for the synthesis of quinoxalines (3a–m) from substituted phenacyl bromides and *o*-phenylenediamines using $\text{Fe}(\text{acac})_3$ catalyst. The key feature of the current protocol involves simple reaction conditions, no side reaction with the formation of the desired product in high yield. The present method is an alternative to the conventional processes for the synthesis of quinoxalines. The catalyst could be recovered several times without loss of catalytic activity, which makes the process cost-effective. 72-95%.

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