RSC Advances

PAPER

View Article Online
View Journal | View Issue

Cite this: *RSC Adv.*, 2024, **14**, 35386

A mild and convenient protocol for the synthesis of quinoxalin-2(1*H*)-ones and benzimidazoles†

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Received 24th September 2024
Accepted 30th October 2024

DOI: 10.1039/d4ra06887d

rsc.li/rsc-advances

We present a mild and simple method for the cyclization of *N*-protected *o*-phenylenediamines with carbonyl compounds in the presence of trifluoroacetic acid. This method reliably provides various substrates of benzimidazoles and quinoxalin-2(1*H*)-ones, with all reactions conducted at room temperature, demonstrating excellent substrate adaptability and a broad substrate scope.

Introduction

Nitrogen-containing heterocyclic compounds form the foundational structures of many bioactive natural products and synthetic molecules. Benzimidazoles are extensively utilized in various pharmaceuticals due to their notable anti-inflammatory,[1] antibacterial, antiviral[2] and anticancer properties,[3] as well as their roles as antihypertensive agents, exemplified by drugs such as telmisartan[4] and bendazol.[5] Similarly, quinoxalines not only exhibit comparable therapeutic effects[6] but also display potential as antiparasitic[7] and anti-HIV agents.[8] For instance, caroverine is recognized for its antioxidant and vasodilatory activities.[9] Additionally, quinoxalines are used in various industrial materials, including organic semiconductors[10] and efficient electro-luminescent materials[11] (Fig. 1). Consequently, enhancing the synthesis methods for benzimidazoles and quinoxalines is crucial for lowering costs in pharmaceutical and industrial production. This ongoing demand makes it imperative for chemists to devise innovative synthetic pathways for the preparation of benzimidazole and quinoxaline derivatives.[12] Traditional methods for synthesizing benzimidazoles and quinoxalin-2(1*H*)-ones typically involve the reaction of aniline derivatives with carbonyl compounds under harsh conditions, often necessitating photocatalysis,[13] microwave irradiation,[14] high temperatures,[15] strong acids[16] or metal catalysts.[17] While numerous synthetic strategies have been established, we now present a straightforward and convenient approach that facilitates synthesis in an open flask at room temperature (Scheme 1).

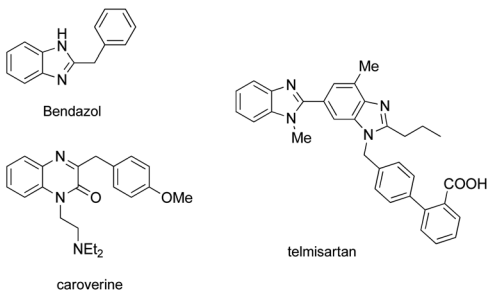
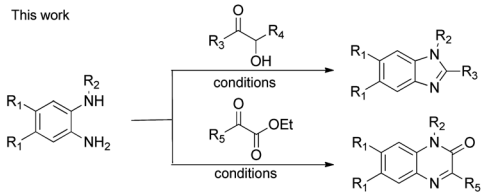


Fig. 1 Examples of bioactive quinoxalin-2(1*H*)-one and benzimidazole derivative molecules.

Results and discussion

We initiated the model cyclization by reacting *N*-benzyl-*o*-phenylenediamine (**1a**) with acetoin (**2a**) in the presence of 2 M hydrochloric acid in dichloromethane (DCM) at room temperature (Table 1, entry 1). The reaction completed within two hours, as confirmed by TLC, yielding 78% of 1-benzyl-2-methyl-1*H*-benzo[*d*]imidazole (**3a**). To optimize the reaction conditions, we explored the necessity of an oxygen atmosphere. Reactions conducted under nitrogen and oxygen atmospheres, while maintaining the same experimental conditions, showed that oxygen significantly improved the yield to 83%. In contrast, the reaction failed under nitrogen, even at 40 °C, with only minimal decomposition of *N*-benzyl-*o*-phenylenediamine (**1a**).



Scheme 1 Synthesis of quinoxalin-2(1*H*)-ones and benzimidazoles.

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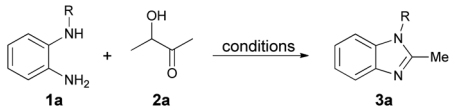
† Electronic supplementary information (ESI) available: Experimental procedures, characterization data, and copies of NMR spectra for all compounds. See DOI: https://doi.org/10.1039/d4ra06887d

Table 1 Optimization of the reaction conditions[a]



Entry	R	Acid	Solvent	T/°C	Yield (%)
1	Bn	HCl	DCM	r.t.	78
2	Bn	HCl	DCM	r.t.	83[b]
3	Bn	HCl	DCM	r.t.	0[c]
4	BOC	HCl	DCM	r.t.	0
5	Ts	HCl	DCM	r.t.	0
6	Ac	HCl	DCM	r.t.	0
7	Bn	H_2SO_4	DCM	r.t.	55
8	Bn	CF_3COOH	DCM	r.t.	81
9	Bn	CF_3SO_3H	DCM	r.t.	65
10	Bn	CF_3COOH	THF	r.t.	83
11	Bn	CF_3COOH	EtOH	r.t.	82
12	Bn	CF_3COOH	DMF	r.t.	79
13	Bn	CF_3COOH	MeCN	r.t.	91
14	Bn	CF_3COOH	MeCN	−15	41
15	Bn	CF_3COOH	MeCN	0	93
16	Bn	CF_3COOH	MeCN	40	57
17	Bn	CF_3COOH (2.0 eq.)	MeCN	r.t.	72
18	Bn	CF_3COOH (1.5 eq.)	MeCN	r.t.	83
19	Bn	CF_3COOH (0.5 eq.)	MeCN	r.t.	47
20	Bn	CF_3COOH (0.0 eq.)	MeCN	r.t.	0

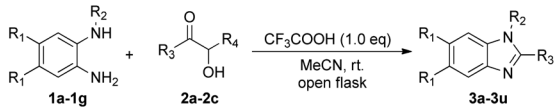
[a] Reaction conditions: **1a** (0.6 mmol), **2a** (0.6 mmol), acid (1.0 eq.), solvent (2.0 mL) in open flask, 2 h. [b] O_2 balloon (1.0 atm). [c] N_2 balloon (1.0 atm).

This confirmed that the reaction requires oxygen (Table 1, entries 2 and 3). With this critical information, we continued to further optimize the reaction conditions. Unfortunately, some *N*-protected *o*-phenylene-diamines did not yield products, likely because electron-withdrawing substituents hindered the cyclization reaction (Table 1, entries 4–6). We treated **1a** with **2a** in the presence of several acids in dichloromethane at room temperature (Table 1, entries 7–9). Although each acid was examined and the target product **3a** was obtained, trifluoroacetic acid demonstrated superior yields compared to other acids. Moreover, a much higher yield was achieved from the reaction conducted in acetonitrile tested respectively under oxygen atmospheres (Table 1, entries 10–13). Regarding temperature, we noted that lower temperatures generally enhanced the yield, although the effect was not substantial (Table 1, entries 14–16). Conversely, at elevated temperatures, the reaction became complex, leading to an increase in by-products and a consequent decrease in the desired product yield. Finally, we assessed the influence of varying amounts of CF_3COOH on the reaction. Our findings revealed that the optimal quantity of CF_3COOH was 1.0 equivalent (Table 1, entries 17–20). Notably, increasing the amount of acid resulted in shortened reaction times but lower yields, while reducing the acid amount inhibited the reaction's progression. Thus, we established concise and efficient conditions for this cyclization: *N*-protected *o*-phenylenediamines (**1a**, 1.0 eq.) and acyloin (**2a**, 1.0 eq.) in the presence of CF_3COOH (1.0 eq.) in acetonitrile, conducted in an open flask at room temperature (Table 1, entry 13).

With the optimal reaction conditions established, we first explored the substrate scope of *N*-protected *o*-phenylenediamines, and the results were summarized in Table 2. A variety of *N*-benzyl-1,2-diaminobenzenes, featuring both electron-donating and electron-withdrawing substituents, successfully underwent cyclization with **2a** in an open flask, yielding modest to good amounts of the corresponding benzimidazoles (**3a–c**). Next, we evaluated the scope of α-hydroxy ketones under the established conditions. These substrates delivered high yields, including 1-hydroxycyclohexyl phenyl ketone (**2c**), despite bearing bulky substituent, **3d–i** still achieved moderate yields. Similarly, various *N*-methyl-1,2-diaminobenzenes with different electron-donating and electron-withdrawing substituents underwent smooth cyclization with a range of α-hydroxy ketones, resulting in moderate to good yields (**3j–r**). Furthermore, the reaction of *N*1-phenyl-1,2-diaminobenzene (**1g**) with α-hydroxy ketones (**2a–c**) successfully afforded the target products in good yields (**3s–u**).

We then investigated the reactions of various *N*-protected *o*-phenylenediamines (**1**) with α-ketoesters (**4**) under the optimized conditions, as detailed in Table 3. Although the yields were slightly lower when using an open flask, this method was proven relatively straightforward and was therefore preferred (Table 3, entries 1 and 2). The reactions of various *N*-protected *o*-phenylenediamines containing electron-donating or electron-withdrawing substituents with α-ketoesters proceeded smoothly, furnishing the corresponding quinoxaline derivatives

Table 2 Synthesis of the benzimidazoles[a]



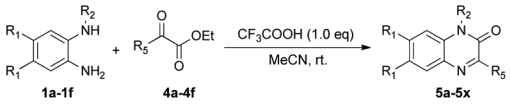
Entry	1, R1, R2	2, R3, R4	3, yield (%)
1	**1a**, H, Bn	**2a**, Me, Me	**3a**, 91
2	**1b**, Me, Bn	**2a**, Me, Me	**3b**, 81
3	**1c**, F, Bn	**2a**, Me, Me	**3c**, 83
4	**1a**, H, Bn	**2b**, Et, Et	**3d**, 89
5	**1b**, Me, Bn	**2b**, Et, Et	**3e**, 78
6	**1c**, F, Bn	**2b**, Et, Et	**3f**, 82
7	**1a**, H, Bn	**2c**, Ph, Cy	**3g**, 74
8	**1b**, Me, Bn	**2c**, Ph, Cy	**3h**, 59
9	**1c**, F, Bn	**2c**, Ph, Cy	**3i**, 63
10	**1d**, H, Me	**2a**, Me, Me	**3j**, 89
11	**1e**, Me, Me	**2a**, Me, Me	**3k**, 69
12	**1f**, F, Me	**2a**, Me, Me	**3l**, 68
13	**1d**, H, Me	**2b**, Et, Et	**3m**, 86
14	**1e**, Me, Me	**2b**, Et, Et	**3n**, 64
15	**1f**, F, Me	**2b**, Et, Et	**3o**, 62
16	**1d**, H, Me	**2c**, Ph, Cy	**3p**, 73
17	**1e**, Me, Me	**2c**, Ph, Cy	**3q**, 58
18	**1f**, F, Me	**2c**, Ph, Cy	**3r**, 60
19	**1g**, H, Ph	**2a**, Me, Me	**3s**, 88
20	**1g**, H, Ph	**2b**, Et, Et	**3t**, 85
21	**1g**, H, Ph	**2c**, Ph, Cy	**3u**, 73

[a] Reaction conditions: a mixture of **1** (0.6 mmol), **2** (0.6 mmol) and CF_3COOH (0.6 mmol) in MeCN (2.0 mmol) while stirring in open flask at room temperature.

in moderate yields (**5b–i**, **5m–u**). Notably, the reaction showed good tolerance to a range of functional groups, and ethyl 2-cyclohexyl-2-oxoacetates were successfully introduced into the desired products (**5j–l**, **5v–x**).

To enhance the practicality of this methodology, the optimized conditions were utilized to synthesize a series of benzimidazole and quinoxaline derivatives. We next examined the reaction of *N*-phenyl-*o*-phenylenediamine (**1a**) with phenylpyruvic acid (**6a**) under the optimized conditions (Table 4). The reaction successfully produced the target product, 1,3-dibenzyl quinoxalin-2(1*H*)-one (**7a**), with a yield of 9%, along with 1,2-dibenzyl-1*H*-benzo[*d*]imidazole (**8a**) at 76% yield, which was a precursor to the antihypertensive drug bendazol. Subsequently, the reaction of **1a** with **6a** was scaled up to 10 mmol, resulting in a yield of 76% for **8a**. *N*-Phenyl-*o*-phenylenediamines (**1a**, **1c**) bearing diverse substitutions reacted with trimethylpyruvic acid, yielding benzimidazoles (**8b**, **8c**) in 73% and 71%, respectively, along with **7b** and **7c** in 11% and 12%. In sharp contrast, the reaction of *N*1-benzyl-4,5-dimethylbenzene-1,2-diamine (**1b**) was performed under the standard conditions to isolate **7d**. Similarly, the reaction of *N*1-methyl-benzene-1,2-diamine (**1d**) with phenylpyruvic acid (**6a**) afforded 3-benzyl-1-methylquinoxalin-2(1*H*)-one (**7e**) but failed to isolate benzimidazoles. Next, we explored the reaction of *N*-methyl-*o*-phenylenediamines with various α-ketoesters; however, this

Table 3 Synthesis of quinoxalin-2(1*H*)-ones[a]

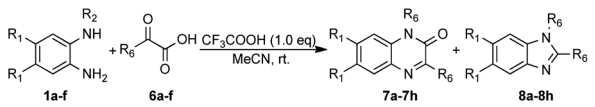


Entry	1, R1, R2	4, R5	5, yield[a] (%)
1	**1a**, H, Bn	**4a**, i-Pr	**5a**, 78
2	**1a**, H, Bn	**4a**, i-Pr	**5a**, 81[b]
3	**1b**, Me, Bn	**4a**, i-Pr	**5b**, 57
4	**1c**, F, Bn	**4a**, i-Pr	**5c**, 61
5	**1a**, H, Bn	**4b**, *n*-Pr	**5d**, 71
6	**1b**, Me, Bn	**4b**, *n*-Pr	**5e**, 55
7	**1c**, F, Bn	**4b**, *n*-Pr	**5f**, 57
8	**1a**, H, Bn	**4c**, *n*-Bu	**5g**, 55
9	**1b**, Me, Bn	**4c**, *n*-Bu	**5h**, 56
10	**1c**, F, Bn	**4c**, *n*-Bu	**5i**, 57
11	**1a**, H, Bn	**4d**, Cy	**5j**, 71
12	**1b**, Me, Bn	**4d**, Cy	**5k**, 55
13	**1c**, F, Bn	**4d**, Cy	**5l**, 51
14	**1d**, H, Me	**4a**, i-Pr	**5m**, 71
15	**1e**, Me, Me	**4a**, i-Pr	**5n**, 52
16	**1f**, F, Me	**4a**, i-Pr	**5o**, 56
17	**1d**, H, Me	**4b**, *n*-Pr	**5p**, 67
18	**1e**, Me, Me	**4b**, *n*-Pr	**5q**, 65
19	**1f**, F, Me	**4b**, *n*-Pr	**5r**, 71
20	**1d**, H, Me	**4c**, *n*-Bu	**5s**, 66
21	**1e**, Me, Me	**4c**, *n*-Bu	**5t**, 61
22	**1f**, F, Me	**4c**, *n*-Bu	**5u**, 63
23	**1d**, H, Me	**4d**, Cy	**5v**, 53
24	**1e**, Me, Me	**4d**, Cy	**5w**, 54
25	**1f**, F, Me	**4d**, Cy	**5x**, 51

[a] Reaction conditions: a mixture of **1** (0.6 mmol), **4** (0.6 mmol) and CF_3COOH (0.6 mmol) in MeCN (2.0 mmol) while stirring in open flask at room temperature. [b] N_2 balloon (1.0 atm).

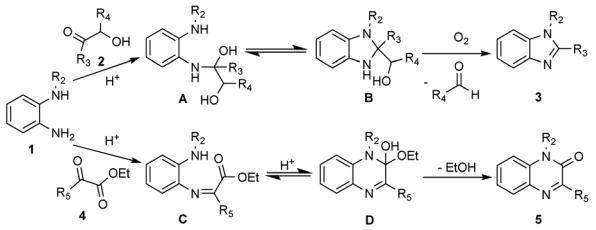
obtained only quinoxalines (**7f–i**). Benzimidazoles were not isolated even in the presence of oxygen. Clearly, substituents played a directing role in the reaction.

Table 4 Synthesis of benzimidazoles and quinoxalin-2(1*H*)-ones[a]



Entry	1, R1, R2	6, R6	7, yield (%)	8, yield (%)
1	**1a**, H, Bn	**6a**, Bn	**7a**, 9	**8a**, 76
2	**1a**, H, Bn	**6b**, *t*-Bu	**7b**, 11	**8b**, 73
3	**1c**, F, Bn	**6b**, *t*-Bu	**7c**, 12	**8c**, 71
4	**1b**, Me, Bn	**6b**, *t*-Bu	**7d**, 79	Trace
5	**1d**, H, Me	**6a**, Bn	**7e**, 69	Trace
6	**1d**, H, Me	**6b**, *t*-Bu	**7f**, 71	0
7	**1e**, Me, Me	**6b**, *t*-Bu	**7h**, 73	0
8	**1f**, F, Me	**6b**, *t*-Bu	**7i**, 59	0

[a] Reaction conditions: a mixture of **1** (0.6 mmol), **6** (0.6 mmol) and CF_3COOH (0.6 mmol) in MeCN (2.0 mmol) while stirring in open flask at room temperature.



Scheme 2 Proposed reaction mechanism.

Based on the above results and previous reports,[18–21] a probable mechanism is proposed. *N*-protected *o*-phenylenediamines **1** with α-hydroxy ketone **2** afforded intermediate **A** in the presence of acid, subsequently, intermediate **A** undergone the favored 5-*exo*-tet cyclization to generated intermediate **B**. Under oxidative conditions, intermediate **B** was converted into benzimidazole (**3**), accompanied by the generation of aldehydes or ketones (for details, see the ESI†). In addition, *N*-protected *o*-phenylene-diamines 1 with α-ketoesters **4** in the presence of acid to give intermediate **C**, which subsequently undergone 6-*exo*-trig cyclization to afford intermediate **D**. Finally, after the elimination of ethanol, quinoxalin-2(1*H*)-ones (**5**) was obtained. The reaction mechanisms for the formation of compounds **7** and **8** was similar to the aforementioned. However, we have not yet been able to provide a satisfactory explanation for the different ratios of **7** and **8**. We will continue to investigate this reaction mechanism in our laboratory (Scheme 2).

Conclusions

We have established a concise and efficient protocol for the synthesis of benzimidazoles and quinoxalin-2(1*H*)-ones in an open flask at room temperature. The method accommodated substrates with a variety of substitutions, obtaining moderate to good yields under mild reaction conditions. On going investigations in our laboratory aim to further explore the applications of this cyclization protocol for the synthesis of additional benzimidazole and quinoxaline derivatives.

Data availability

The data supporting this article have been included as part of the ESI.†

Conflicts of interest

There are no conflicts to declare.

Acknowledgements

We are grateful for financial support from the NSFC (No. 22161027); Scientific Research Foundation of Moutai Institute (No. mygccrc[2022]016, mygccrc[2022]004, mygccrc[2022]023, mygccrc[2022]027, mygccrc[2022]031); Joint Project of Zunyi Science & Technology Bureau and Moutai Institute (No. ZunShiKeHe HZ zi[2021]307).

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