



Cite this: *RSC Adv.*, 2023, 13, 21231

Microwave-accelerated cross-dehydrogenative-coupling (CDC) of *N*-(quinolin-8-yl)amides with acetone/acetonitrile under metal-free conditions†

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A highly selective remote C(sp³)-H acetonation of *N*-(quinolin-8-yl)amide scaffolds at the C5-position under microwave irradiation has been developed. In the absence of a transition-metal-catalyst, benzoyl peroxide (BPO)-promoted cross-dehydrogenation coupling (CDC) of *N*-(quinolin-8-yl)amides with acetone/acetonitrile occurred smoothly to generate the corresponding 5-acetonated/acetonitriled *N*-(quinolin-8-yl)amides in good yields. The transformation is operationally simple, rapid, easily scaled-up to the gram scale, and shows a broad substrate scope.

Received 31st May 2023

Accepted 7th July 2023

DOI: 10.1039/d3ra03651k

rsc.li/rsc-advances

Quinolines play an important role as nitrogen-containing heterocyclic compounds in natural products, pharmaceuticals and pesticides (Fig. 1).¹ As a particular member of the quinoline compounds, *N*-(quinolin-8-yl)benzamide has an irreplaceable role in the C-H bond activation reaction system as a classical bidentate directing group or as a ligand auxiliary.² In recent years, brilliant achievements have been made in C-H bond functionalization reactions accomplished at the C-2,³ C-3,⁴ C-4,⁵ C-5 and C-8 (ref. 6) positions of quinoline and its derivatives. It is worth mentioning that the C-5 position functionalization reactions of *N*-(quinolin-8-yl)benzamide have a certain medicinal value with high activity and involve more types of reactions, such as amidation,⁷ sulfonylation,⁸ cyanoalkoxylation,⁹ allylation,¹⁰ alkylation,¹¹ halogenation¹² and so on.¹³

In the past decades, with the boom in the area of inert C-H bond activation, cross-dehydrogenation coupling (CDC)

reactions involving inert C-H bond activation have been developed,¹⁴ which was pioneered by Li in 2014.¹⁵ Currently, their group has not only constructed intermolecular C-C bond reactions by CDC of inert C(sp³)-H bonds,¹⁶ but also achieved CDC reactions of C(sp³)-H heteroarylation between alkanes and heteroarenes by visible light irradiation.¹⁷ Subsequently, CDC-type reactions have been widely used and rapidly developed (Scheme 1). Among the CDC reactions, there are even rarer dehydro-coupling reactions in which acetonitrile/acetone was used as a reaction substrate, for example, oxidant-promoted CDC reactions reported by Kianmehr,¹⁸ and Tan *et al.*¹⁹

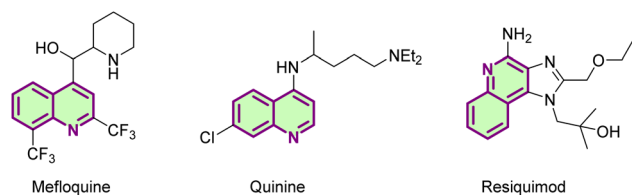
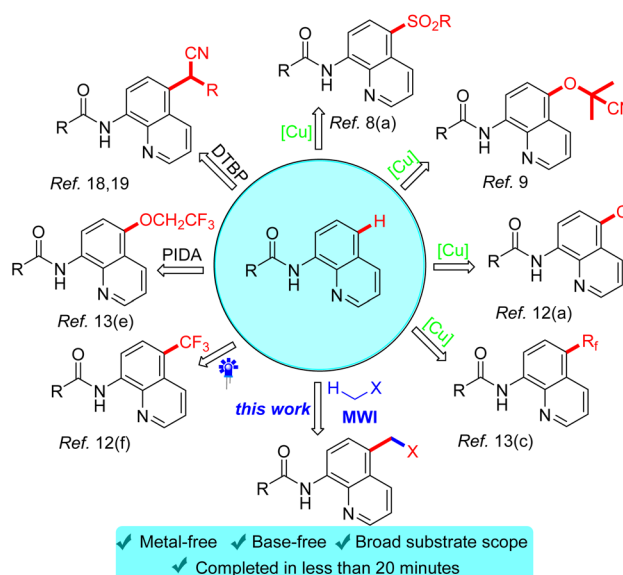


Fig. 1 Representative drug candidates and natural products with quinoline skeleton.

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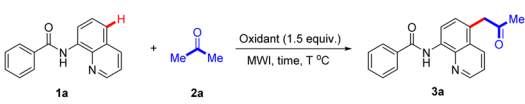
† Electronic supplementary information (ESI) available. See DOI: <https://doi.org/10.1039/d3ra03651k>



Scheme 1 The C-5 position functionalization reactions of *N*-(quinolin-8-yl)amide.



Table 1 Optimization of the reaction conditions^a

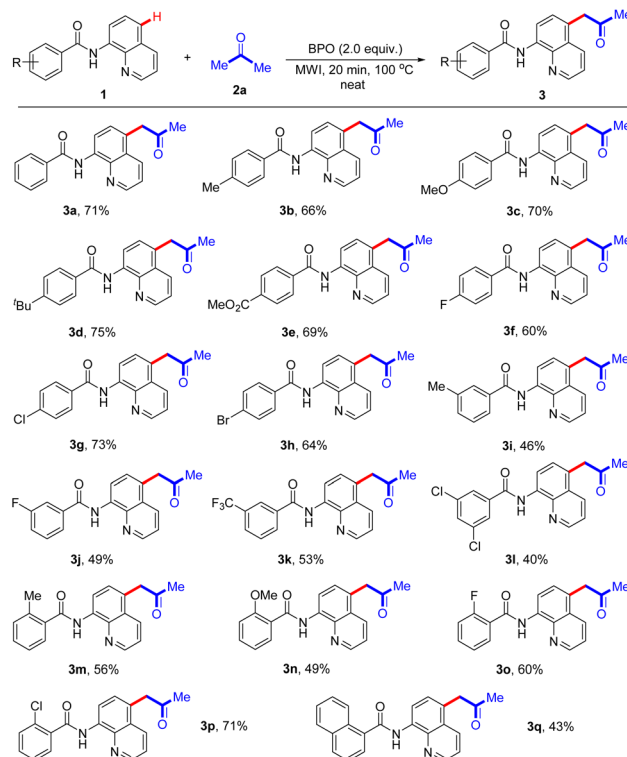
				
Entry	Oxidant	Time (min)	T (°C)	Yield ^b (%)
1	BPO	20	100	62
2	H ₂ O ₂	20	100	n.r.
3	DTBP	20	100	n.r.
4	LPO	20	100	Trace
5	TBHP	20	100	n.r.
6	BPO (1.0 equiv.)	20	100	41
7	BPO (2.0 equiv.)	20	100	71
8	BPO (2.5 equiv.)	20	100	73
9	BPO (3.0 equiv.)	20	100	72
10	BPO (2.0 equiv.)	30	100	69
11	BPO (2.0 equiv.)	15	100	60
12	BPO (2.0 equiv.)	10	100	47
13	BPO (2.0 equiv.)	20	110	70
14	BPO (2.0 equiv.)	20	120	71
15	BPO (2.0 equiv.)	20	90	40
16	BPO (2.0 equiv.)	20	80	32
17	BPO (2.0 equiv.)	12 (h)	100	18 ^c

^a Reaction conditions: *N*-(quinolin-8-yl)benzamide (**1a**, 0.10 mmol), BPO, acetone (**2a**, 3.0 mL) under microwave irradiation. ^b Isolated yield. ^c In an oil bath at 100 °C for 12 h [DTBP = di-*tert*-butyl peroxide; TBHP = *tert*-butyl hydroperoxide; LPO = dilauroyl peroxide; n.r. = no reaction].

Since microwave contain many advantages such as short reaction time and environmental friendliness which have been used in organic reactions, it has already attracted the attention of chemists and achieved remarkable results in the field of C–H bond activation.²⁰ In the past few years, microwave-facilitated organic synthesis has developed into one of the most powerful tools in organic chemistry. Herein we wish to report a microwave-accelerated cross-dehydrogenative-coupling (CDC) of *N*-(quinolin-8-yl)amides with acetone/acetonitrile in the presence of benzoyl peroxide (BPO) as oxidant under metal-free conditions, delivering good yields of C-5-acetonated *N*-(quinolin-8-yl)amides as products.

We initiated our study with microwave irradiation of *N*-(quinolin-8-yl)benzamide (**1a**) and acetone (**2a**) in the presence of benzoyl peroxide (BPO). After 20 min at 100 °C, the desired acetonation product **3a** was obtained in 62% yield (Table 1, entry 1). The reaction did not proceed with other oxidants such as H₂O₂, DTBP, TBHP and LPO (entries 2–5). Screening of oxidant loading indicated that 2.0 equiv. is the most effective choice for the reaction, afforded the product **3a** in 71% yield (entries 6–9). Investigation of the reaction time and temperature gave no obvious improvement in the product yield of **3a** (entries 10–16). Interestingly, when the model reaction was carried out in an oil bath at 100 °C for 12 h, only 18% yield of **3a** was obtained (entry 17). For detailed optimization of reaction conditions, please see the (Tables S1–S4 in ESI).[†]

Under optimal reaction conditions, we next investigated the substrate scope of acetonation reaction (Scheme 2). A variety of

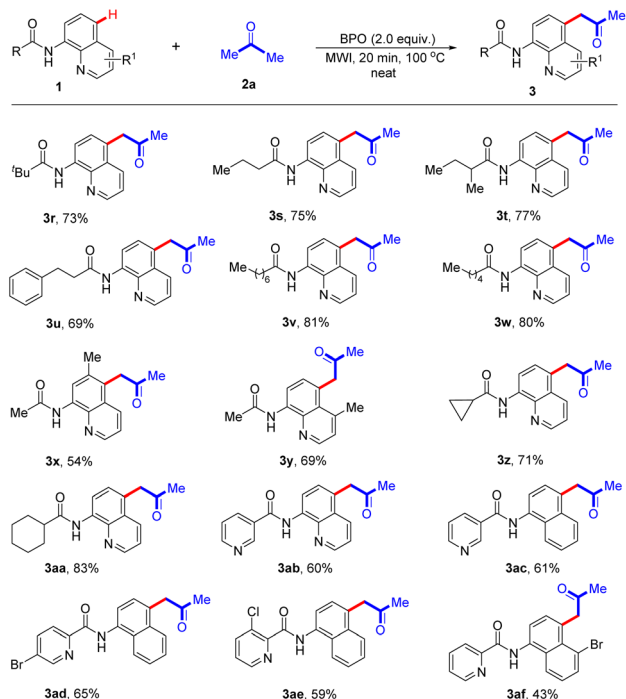


Scheme 2 The substrate scope of *N*-(quinolin-8-yl)arylamides [reaction conditions: **1** (0.10 mmol), BPO (2.0 equiv.) and acetone (**2a**, 3.0 mL) under microwave irradiation at 100 °C for 20 min; isolated yield of the product].

N-(quinolin-8-yl)arylamides with *para*-substituents, such as Me, MeO, *t*Bu, CO₂Me, F, Cl and Br groups, in differed electronic properties are well tolerated (**3a–3h**), affording the desired C-5-acetonated *N*-(quinolin-8-yl)arylamides in good yields. Some *meta*-substituted groups including Me, F and CF₃ on the benzene ring in *N*-(quinolin-8-yl)arylamides were examined, and exhibited good reactivity in the reaction, providing the corresponding products (**3i–3k**) in 46–53% yields. When disubstituted *N*-(quinolin-8-yl)arylamide **1l** was employed in this transformation, the corresponding product **3l** was afforded in 40% yield. In addition, the reactions with an electron-donating group (Me or OMe) or an electron-withdrawing group (F or Cl) at the C2-position of the aryl rings generated the products **3m–3p** with 49–71% yields. Notably, *N*-(quinolin-8-yl)-1-naphthamide reacted with acetone, affording the desired product **3q** in 43% yield.

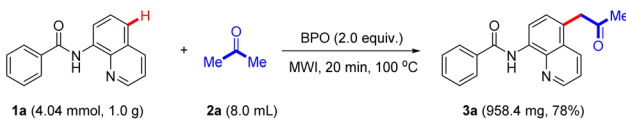
It is gratifying that the aliphatic-derived *N*-(quinolin-8-yl) amides reacted acetone under optimal conditions, indicating that it is not affected by the length of the aliphatic carbon chain, and the corresponding products **3r–3w** were isolated in 69–81% yields (Scheme 3). It is also worth mentioning that the presence of a methyl substituent at the C-6 or C-3 position of the quinoline ring could also formed the desired products (**3x** and **3y**) in 54% and 69%, respectively. Additionally, cyclopropanamide and cyclohexanamide also participated in the reaction and the acetonation products **3z** and **3aa** could be obtained in good yields (71% and 83%, respectively). It should be noted that *N*-



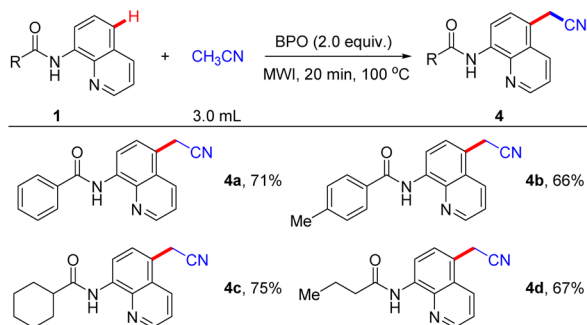


Scheme 3 The substrates scope of *N*-(quinolin-8-yl)aliphatic amides and *N*-(quinolin-8-yl)heteroarylamides [reaction conditions: **1** (0.10 mmol), BPO (2.0 equiv.) and acetone (**2a**, 3.0 mL) under microwave irradiation at 100 °C for 20 min; isolated yield of the product].

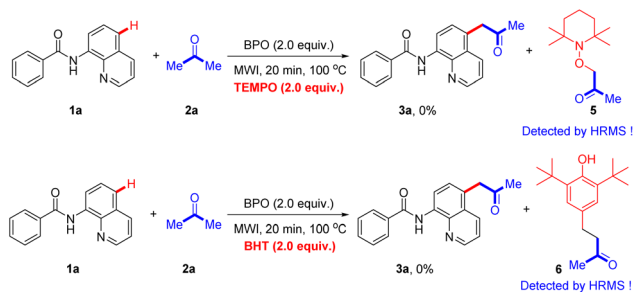
(quinolin-8-yl)nicotinamide **1ab** underwent the reaction smoothly with **2a** to give the corresponding product **3ab** in 60% yield. Encouragingly, when the quinoline moiety was replaced with naphthalene one, the obtained compounds were also compatible with this reaction, giving the anticipated products



Scheme 4 Gram-scale reaction.



Scheme 5 The substrates scope of *N*-(quinolin-8-yl)arylamides and *N*-(quinolin-8-yl)aliphatic amides [reaction conditions: **1** (0.10 mmol), BPO (2.0 equiv.) and acetonitrile (3.0 mL) under microwave irradiation at 100 °C for 20 min; isolated yield of the product].



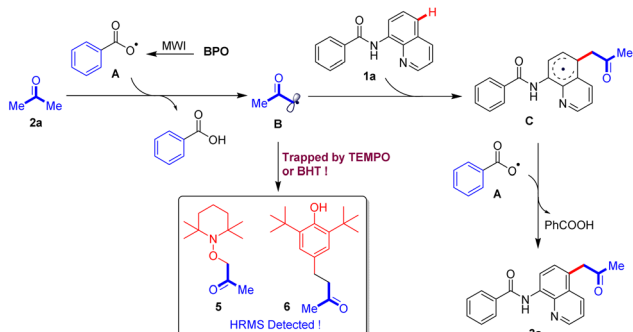
Scheme 6 Radical trapped experiments.

3ac–3ae in 59–65% yields. Moreover, the optimized conditions were also suitable for the reaction of *N*-(5-bromonaphthalen-1-yl)picolinamide with acetone (**2a**), and the expected product (**3af**) was formed in 43% yield. Furthermore, when 1.0 g of **1a** was subjected to the reaction, the targeted product **3a** was obtained in 78% yield (Scheme 4).

Remarkably, the reaction system was also capable of cyano-methylation reaction under optimal conditions, the results of which were summarized in Scheme 5. We were delighted to observe the acetonitrile was good coupling partner, enabling the production of **4a** in 71% yield. Additionally, this reaction can tolerate functional groups such as *N*-(quinolin-8-yl)arylamides with *para*-Me (**1b**) and aliphatic-derived *N*-(quinolin-8-yl)amides can afford the corresponding products in 66%, 75%, and 67% yield, respectively.

To gain insight into the reaction mechanism, the radical trapped experiments were conducted (Scheme 6). When a radical scavenger 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) or butylated hydroxytoluene (BHT) was added, no desired product was detected under the standard conditions, suggesting that a free radical was involved in this transformation. The TEMPO adduct **5** and BHT adduct **6** were detected by HRMS analysis (ESI[†]).

Based on the control experiment, a plausible mechanism is proposed as in Scheme 7. First, the benzoate radical **A** is generated as a result of microwave irradiation from BPO, and it then captures a hydrogen atom from the substrate acetone **2a** to form the carbon radical **B**, which is trapped by TEMPO or BHT to its adducts **5** or **6**. The obtained acetone radical **B** reacts with substrate *N*-(quinolin-8-yl)benzamide **1a** to afford an



Scheme 7 The proposed mechanism.



intermediate C, which further reacts with benzoate radical A to generate the final product 3a.

Conclusions

In summary, we have developed a microwave-accelerated cross-dehydrogenative-coupling (CDC) of *N*-(quinolin-8-yl)amides with acetone/acetonitrile under metal-free conditions. In the presence of benzoyl peroxide (BPO) as promoter, a cross-dehydrogenation coupling (CDC) of *N*-(quinolin-8-yl)amides with acetone underwent smoothly to generate the corresponding 5-acetonated/acetonitriled *N*-(quinolin-8-yl)amides in good yields. Compared with the traditional strategy for C-5 position coupling reactions, this reaction provides an environmentally friendly, efficient, and convenient access to C-5 position functionalization of *N*-(quinolin-8-yl)amides. Moreover, a plausible reaction mechanism is proposed based on a radical trapped experiment and HRMS studies. Further attempts to apply microwave acceleration coupling reactions are currently underway in our laboratory.

Conflicts of interest

There are no conflicts to declare.

Acknowledgements

We gratefully acknowledge the National Natural Science Foundation of China (21901081), University Natural Science Research Project of Anhui Province (KJ2021A0526, KJ2020A1199), Research Fund of Excellent Young Scientists in Higher Education Institution of Anhui Province, China (2022AH030060) and the Natural Science Foundation of Anhui Province (2008085QB90).

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