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One-Pot Synthesis of 4-Quinolone via Iron-Catalyzed Oxidative Coupling of Alcohol and Methyl Arene

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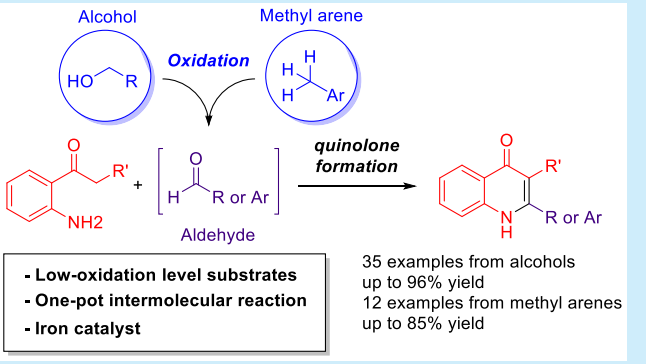
ABSTRACT: Herein, we describe the iron(III)-catalyzed oxidative coupling of alcohol/methyl arene with 2-amino phenyl ketone to synthesize 4-quinolone. Alcohols and methyl arenes are oxidized to the aldehyde in the presence of an iron catalyst and di-*tert*-butyl peroxide, followed by a tandem process, condensation with amine/Mannich-type cyclization/oxidation, to complete the 4-quinolone ring. This method tolerates various kinds of functional groups and provides a direct approach to the synthesis of 4-quinolones from less functionalized substrates.



Nitrogen-containing heterocycles are widely found in many biologically active compounds and natural products. Among them, 4-quinolone is regarded as a privileged scaffold that imparts a variety of biological activities.[1] Many antibiotics contain 4-quinolone moieties due to their significant antibacterial activity.[2] In particular, 2-aryl-4-quinolone, an aza analogue of flavone, was known to show an inhibitory effect on tubulin polymerization and play a crucial role in antitumor activity.[3] In addition, recent studies revealed that certain 2-aryl-4-quinolone derivatives possess potent antiviral,[4] antimalarial,[5] and antiplatelet[6] activity as well as effects on cathepsin inhibition[7] and xanthine oxidase.[8] Additionally, these derivatives have been used as important building blocks for various biologically active quinolone and quinoline compounds because of the facile functionalization of their reactive centers at positions 1, 3, and 4.[9] The attractive biological profile and synthetic importance have encouraged researchers to develop various synthetic methods for this scaffold.[10] The most typical method is cyclocondensation, such as Conrad−Limpach and Niementowski cyclization, which proceeds via condensation of amine and carbonyl groups followed by cyclization.[11] However, most of these classical methods suffer from harsh conditions and a limited substrate scope. Another method is base-promoted cyclization of *N*-(*o*-ketoaryl)amides, known as the Camps cyclization.[12] This reaction has been widely used in the synthesis of quinolones but usually proceeds through two steps to employ an *N*-amide group. Some improved methods were developed using *o*-haloaryl acetylenic ketones/amines,[13] *o*-aminoaryl acetylenic ketones,[14] or *N*-benzyl-2-aminoacetophenone,[15] providing a milder route toward 2-aryl-4-quinolone. However, these methods still require multiple steps or specific substrates, which have to be prepared separately (Scheme 1).

In 2017, Huang's group reported an oxidative intermolecular synthetic route for 2-aryl-4-quinolone from 2-amino-acetophenones and benzaldehydes.[16] However, only acetophenone derivatives are applied as a starting material, and aldehyde oxidation level starting materials still limit the generality of the reaction. As part of our continuing studies in the synthesis of heterocycle scaffolds using simple and less functionalized substrates, herein, we report an intermolecular synthesis of 2-substituted 4-quinolones using 2-amino phenyl ketones via iron-catalyzed oxidative annulation. Recently, various types of base metal-catalyzed oxidative annulation using peroxide have been applied to the construction of heterocycles.[17] Among the base metals, iron is the most abundant transition metal with a low price and low toxicity. Moreover, alcohol and hydrocarbon oxidation level materials are more easily available than aldehydes and are usually supplied as feedstocks. Many kinds of aldehydes are prepared by the oxidation of the corresponding alcohol. Therefore, direct use of these substrates instead of labile aldehyde is more attractive for the generality of the reaction scope. This procedure features a simple one-pot operation using readily available starting materials and catalysts. To the best of our knowledge, this is the first

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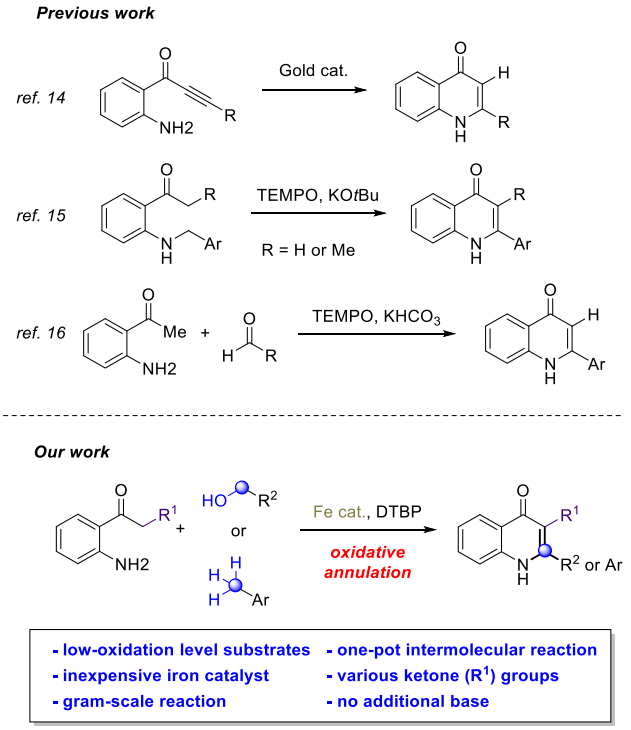
Scheme 1. Synthetic Approaches toward 2-Aryl-4-quinolones

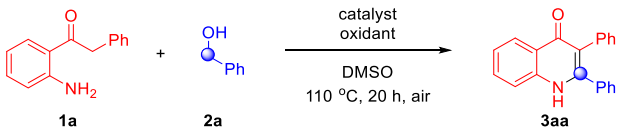


example of a one-pot synthesis of 4-quinolone starting directly from a 2-amino phenyl ketone with alcohols and methyl arene.

As shown in Table 1, we began our studies by optimizing various reaction parameters in the reaction of 1-(2-aminophenyl)-2-phenylethanone (**1a**) with benzyl alcohol (**2a**). With 20 mol % $Fe(OTf)_3$ and 3 equiv of di-*tert*-butyl peroxide (DTBP) in DMSO at 100 °C under air, the desired quinolone product **3aa** was obtained in 42% yield (entry 1). Higher conversion was achieved when the reaction temperature was increased to 110 °C (entry 2). Only trace amounts of **3aa** were observed in the DMF solvent (entry 3). Next, various kinds of oxidants and catalysts were explored in the reaction system. *tert*-Butyl hydroperoxide (TBHP) showed low efficiency, and H_2O_2 gave **3aa** in a yield similar to that of DTBP (entries 4 and 5, respectively). Among the explored catalysts, $Fe(OTs)_3 \cdot 6H_2O$ was found to be the best catalyst (entries 6–11). To find more optimized conditions, we applied O_2 or N_2 gas to the reaction system (entry 12 or 13, respectively). Interestingly, the N_2-charged reaction system gave the best yield with clean conversion, and the exposure of the reaction mixture to O_2 resulted in a lower yield. We supposed that O_2 accelerated the side reaction that originated from Baeyer–Villiger oxidation (see page S56 of the Supporting Information). In the absence of a catalyst, the reaction was very slow and was not completed (entry 14, see page S52 of the Supporting Information). This result demonstrates the important role of iron catalysts in the oxidation process.

To evaluate the substrate scope of the reaction, a wide range of alcohols **2** were reacted with **1a** under the optimized reaction conditions (Scheme 2). Benzylic alcohols with various substituents were smoothly coupled with **1a** to afford the corresponding quinolone products **3ab**–**3am** in good yields, except for the methoxy group (**3ae**). In the case of 4-methoxy benzyl alcohol, a complex product mixture was observed. We

Table 1. Optimization for the Reaction between 1-(2-Aminophenyl)-2-phenylethanone (1a) and Benzyl Alcohol (2a)[a]

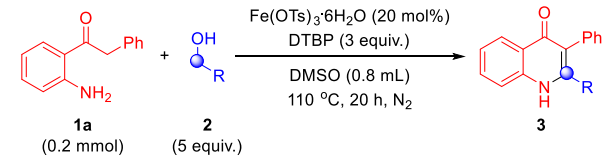


1a 2a 3aa

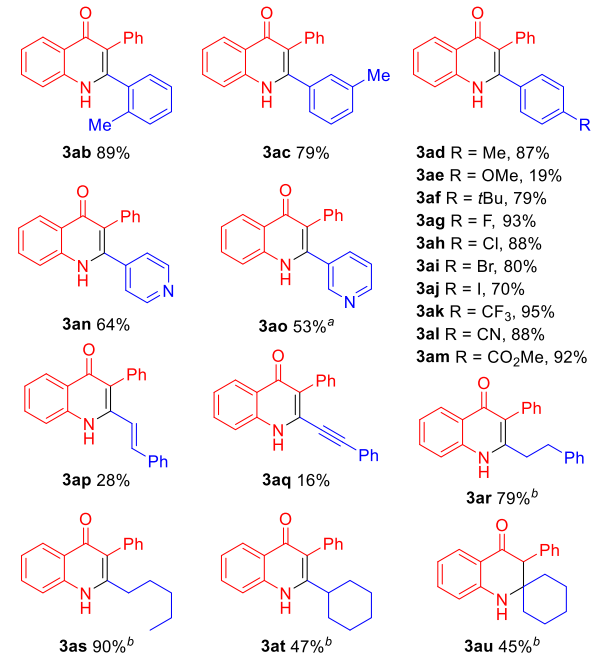
entry	catalyst	oxidant	yield[b] (%)
1[c]	$Fe(OTf)_3$	DTBP	42
2	$Fe(OTf)_3$	DTBP	74
3[d]	$Fe(OTf)_3$	DTBP	trace
4	$Fe(OTf)_3$	TBHP	50
5	$Fe(OTf)_3$	H_2O_2	73
6	$Cu(OTf)_2$	DTBP	trace
7	I_2	DTBP	5
8	$Mn(OAc)_2$	DTBP	4
9	$Fe(OAc)_2$	DTBP	13
10	$FeCl_3 \cdot 6H_2O$	DTBP	trace
11	$Fe(OTs)_3 \cdot 6H_2O$	DTBP	81
12[e]	$Fe(OTs)_3 \cdot 6H_2O$	DTBP	49
13[f]	$Fe(OTs)_3 \cdot 6H_2O$	DTBP	92
14[f]	no catalyst	DTBP	28

[a]Reaction conditions: **1a** (0.2 mmol), **2a** (1.0 mmol), catalyst (20 mol %), and oxidant (0.6 mmol) in DMSO (0.8 mL) at 110 °C for 20 h under air. [b]Isolated yield. [c]Reaction temperature of 100 °C. [d]DMF solvent. [e]O_2 balloon. [f]N_2 balloon.

Scheme 2. Scope of Alcohols 2[a]



1a (0.2 mmol) 2 (5 equiv.) 3



[a]For 40 h. [b]With 20 equiv of alcohol, 28 h.

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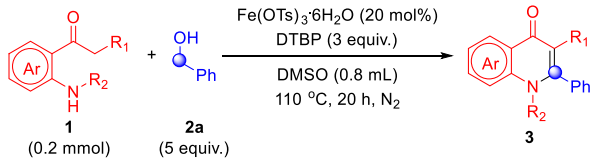
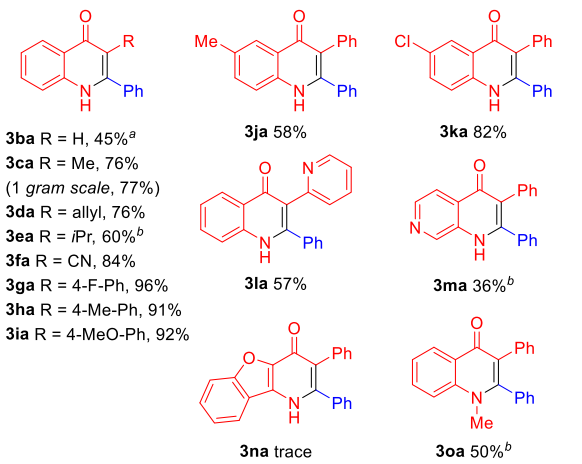
supposed that a side reaction originating from Baeyer−Villiger oxidation could occur competitively in some cases. Other substituents, such as *tert*-butyl, halogen, nitrile, and ester, were well tolerated under the reaction conditions. The pyridine group was employed at position 2 of the 4-quinolone products in moderate yield (**3an** and **3ao**). For further expansion of the alcohol scope, allylic, propargylic, and aliphatic alcohols were also explored. Cinnamyl and 3-phenylpropargyl alcohol could participate in the reaction; however, products **3ap** and **3aq** were obtained in low yields. Aliphatic alcohols also afforded the desired products **3ar**−**3at**, even though an excess amount of alcohol was required. For the secondary alcohol, the expected 2,3-dihydro-4-quinolone product **3au** was formed in moderate yield.

Next, we employed a series of 2-amino aryl ketones **1** for further extension of the substrate scope (Scheme 3). From most simple methyl ketone **1b**, the desired product **3ba** was obtained in moderate yield. In addition to methyl ketones, variously substituted ketone substrates could be successfully applied for 4-quinolone formation (**3ca**−**3ia**). Generally, a substituent that can activate the α-position of the ketone showed a high yield, such as nitrile and aryl groups (**3fa**−**3ia**). For the isopropyl group, the reaction was slow due to steric effects; thus, a longer reaction time was required for a satisfactory yield (**3ea**). The 5-methyl substituent on the phenyl group of substrate **1** showed a negative result compared with the chloride substituent (**3ja** and **3ka**). The introduction of pyridine instead of a phenyl group resulted in a lower yield of the desired products (**3la** and **3ma**), and a small amount of unoxidized dihydroquinolone remained. In the case of 2-amino benzofuran-3-ketone **1n**, the tricyclic product **3na** was obtained in a trace amount. N-Substituted **1o** could also participate in oxidative cyclization and afford the *N*-methyl-4-quinolone product **3oa**. The broad range of substrates demonstrates the synthetic potential of this method for the synthesis of 4-quinolones. Furthermore, the practical utility of the developed method was demonstrated on a 1 g scale (6.7 mmol scale) reaction for **3ca** (77%).

Scheme 3. Scope of 2-Amino Aryl Ketones 1[a]

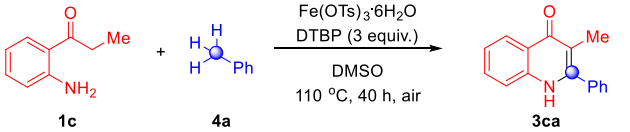




[a]With 10 equiv of alcohol. [b]For 40 h.

After successful synthesis of 4-quinolones using alcohol, our interest moved to methyl arene as a coupling partner. On the basis of our previous synthesis of quinazolinone,[17a] we envisioned that methyl arene can also be oxidized to the corresponding aldehyde under these conditions and participate in oxidative annulation. As shown in Table 2, a preliminary study was performed with the cyclization between 2-aminopropiophenone (**1c**) and toluene (**4a**). In contrast to the reaction with alcohol, methyl arene was less reactive; thus, competitive Baeyer−Villiger oxidation is often observed in substrate **1a**. The phenyl group of **1a** was replaced with a methyl group to suppress migration during Baeyer−Villiger oxidation. Considering the lower reactivity of methyl arene, excess **4a** was used as a cosolvent. Fortunately, **4a** was also readily applied in the annulation and gave desired product **3ca** in 52% yield (entry 1). On the basis of our experience and previous reports,[17a] we expected that O_2 gas would help the oxidation of methyl arene to the aldehyde. As expected, when the reaction mixture was exposed to air, an improved yield was obtained with clean conversion (entry 2). After the **4a**/solvent ratio had been controlled, the optimized condition was selected as entry 4 (72%).

Table 2. Optimization for the Reaction between 2-Aminopropiophenone (1c) and Toluene (4a)[a]



entry	4a	DMSO (mL)	yield[b] (%)
1[c]	1.2 mL (60 equiv)	0.8	52
2	1.2 mL (60 equiv)	0.8	57
3	1.9 mL (90 equiv)	0.8	67
4	1.9 mL (90 equiv)	1.0	72
5	1.9 mL (90 equiv)	1.2	64

[a]Reaction conditions: **1c** (0.2 mmol), **4a**, $Fe(OTs)_3·6H_2O$ (20 mol %), and DTBP (0.6 mmol) in DMSO at 110 °C for 40 h under air. [b]Isolated yield. [c]N_2 balloon.

Then, we investigated the substrate scope for the synthesis of 4-quinolone using methyl arene (Scheme 4). Under the optimized conditions, various ketone-substituted starting materials **1** reacted with toluene **4a** and afforded the desired quinolone products in moderate to good yield (**3aa**−**3da** and **3fa**). Similar to the reaction using an alcohol substrate, the 3-pyridyl quinolone product **3la** was obtained in low yield along with unoxidized intermediate **3la′**. Initially, we had expected the pyridine group to be oxidized in the reaction, but any N-oxidation products were not detected. Thus, we proposed another possibility that Lewis basic pyridine captures Lewis acidic iron salt and disrupts the iron-mediated oxidation from the dihydroquinolone intermediate to the quinolone product. The phenylethanone substrate (**1a**) smoothly reacted with toluene and gave **3aa** in good yield. However, some inseparable mixture was obtained in the reaction with other methyl arenes, which is presumed to originate from Baeyer−Villiger-type side reactions. On the contrary, propiophenone substrate **1c** could react with various methyl arenes, and

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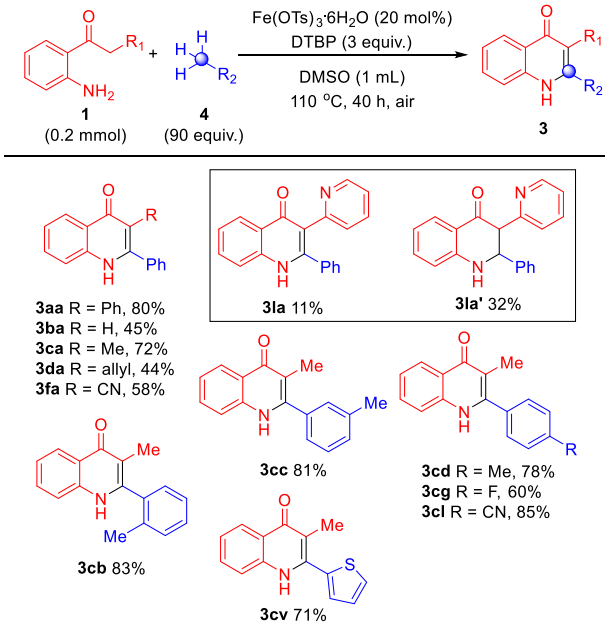
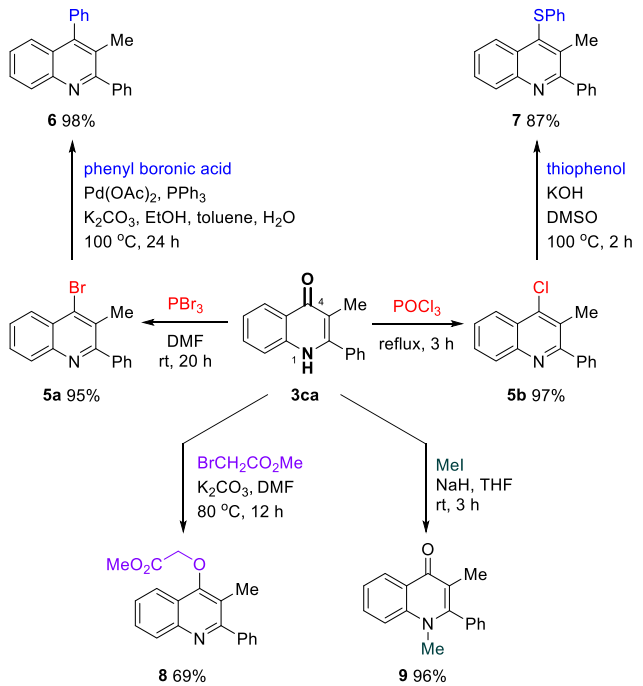
Scheme 4. Substrate Scope in the Reaction between 2-Amino Phenyl Ketones 1 and Methyl Arene 4



desired products **3cb**−**3cd**, **3cg**, and **3cl** were readily synthesized without side products. Thiophene was also applied to position 2 of the 4-quinolone product under the developed conditions.

To demonstrate the synthetic potential of the developed method, we tried to convert 4-quinolone products **3ca** to various quinoline compounds via functionalization of the ketone moiety at position 4 (Scheme 5). Halogenation with PBr_3 and $POCl_3$ provided the corresponding 4-haloquinolines **5a** and **5b**, respectively. Suzuki coupling introduced a phenyl group at position 4, and 4-phenyl quinoline **6** was synthesized. Furthermore, direct nucleophilic substitution with thiophenol afforded 4-sulfide quinoline **7**.[18] O-Alkylation could also be achieved directly from **3ca** using alkyl halide and K_2CO_3, and quinoline product **8** was obtained, which has an ether linkage at position 4.[19] In addition to the ketone group at position 4, the position 1 − *N* could be substituted directly with iodomethane in the presence of NaH as a base.[20] Under these conditions, N-methylated quinolone **9** was synthesized selectively in high yields.

Scheme 5. Diverse Chemical Conversions of 4-Quinolone 3ca



On the basis of the experimental results and previous studies,[17] we proposed a plausible reaction mechanism (see page S58 of the Supporting Information). Initially, the reaction begins with oxidation of alcohol **2a** to benzaldehyde. Condensation of benzaldehyde with **1** produces imine intermediate **A**, followed by Mannich-type cyclization to form dihydroquinolone **3′**. Final oxidation of intermediate **3′** affords quinolone product **3**. Iron salt can mediate each oxidation process and also act as the Lewis acid in the cyclization process. In the presence of O_2, Baeyer−Villiger oxidation of **1** is accelerated, and the corresponding alcohol **side-2** is generated after hydrolysis of ester. This alcohol **side-2** can participate in the annulation with another **1** to produce **side-3**. Methyl arene **4a** is also converted to benzaldehyde by iron-mediated oxidation; however, aldehyde formation is less favored than that of alcohol **2a**. Thus, for methyl arene, O_2 promotes aldehyde formation through benzyl hydroperoxide intermediate and has a positive effect in the reaction.

In conclusion, we have described the iron-catalyzed oxidative annulation of 2-amino phenyl ketone with a low-oxidation level substrate for the synthesis of 4-quinolone. Iron is an earth-abundant and low-toxicity metal, and all of the substrates and reagents used in the reaction were readily available. Moreover, the broad substrate scope, large scale reaction, and diverse chemical conversion of 4-quinolone products demonstrate the synthetic potential of the developed method. Importantly, this is the first report of the synthesis of 4-quinolone via intermolecular coupling of 2-amino phenyl ketone with alcohol and methyl arene. Further expansion of the oxidative annulation using low-oxidation level substrates to access other N-heterocycles is currently being investigated in our research group.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acs.orglett.0c03011.

Experimental procedures, characterization data, ^1H and ^{13}C NMR spectra, mechanistic studies, and the proposed mechanism (PDF)

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Notes

The authors declare no competing financial interest.

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