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Regio- and Chemoselective Mono- and Bisnitration of 8-Amino quinoline Amides with $Fe(NO_3)_3·9H_2O$ as Promoter and Nitro Source

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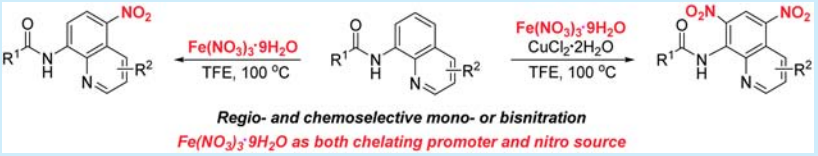
Supporting Information



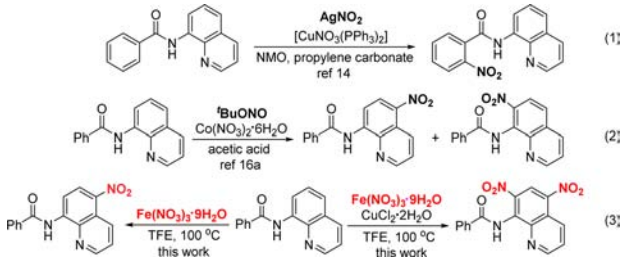
ABSTRACT: An efficient and regioselective remote C(5)−H nitration of 8-aminoquinoline amides by using the economical and nontoxic $Fe(NO_3)_3·9H_2O$ as promoter and nitro source has been developed. Furthermore, when $CuCl_2·2H_2O$ was used as a catalyst, 8-aminoquinoline amides dominantly underwent bisnitration to give 5,7-dinitro-8-aminoquinoline amides. Notably, this is the first example in which $Fe(NO_3)_3·9H_2O$ plays a dual role as both chelating promoter and nitration reagent, and $CuCl_2·2H_2O$ acts as an efficient catalyst for the bisnitration of quinolines.

Aromatic nitro compounds are key precursors or subunits of a variety of dyes, plastics, explosives, pharmaceuticals, and natural products.[1,2] Classically, they were prepared through electrophilic nitration by using a combination of HNO_3/KNO_3 and H_2SO_4 as the nitration reagent.[1,3] Unfortunately, this method often suffers from poor regioselectivity, limited functional group tolerance, and overnitration owing to the harsh reaction conditions employed therein. As an alternative, *ipso*-nitration of aryl halides, triflates, or arylboronic acids by using metal nitrites as nitro sources has been well established.[4] While this method is efficient and reliable, the necessity of pre-introduction and subsequent loss of the stoichiometric amount of halide, triflate, or boronic acid function groups might compromise its sustainability. In recent years, transition-metal-catalyzed and chelation-directed *ortho*- or *meta*-nitration of aryl C(sp^2)−H bonds by using various mild nitro sources is rapidly prevailing.[5,6] This strategy has advantages such as excellent regioselectivity, mild reaction conditions, and good functional group tolerance.

Quinoline and its derivatives have attracted tremendous interest since they are the essential scaffolds of numerous natural products, pharmaceuticals, and functional materials.[7] Among them, nitro-substituted quinolines have been identified as effective cathepsin B inhibitors, anticancer agents, endothelial cell proliferation inhibitors, fluorescent probes, and versatile intermediates for organic synthesis.[8−10] To date, some reliable methods for the preparation of nitro-substituted quinolines are available.[8,11] Nevertheless, new methods with high regioselectivity and atom-economy by using cheap and nontoxic nitro sources are still highly desirable.

Since Daugulis' pioneering work,[12] 8-aminoquinoline has been frequently used as a bidentate ancillary directing group for the functionalization of *ortho* C−H bonds.[13] In this regard, Gooßen reported a copper-catalyzed, chelation-assisted *ortho* C(sp^2)−H nitration of aromatic carboxylic acids directed by the 8-aminoquinoline auxiliary (Scheme 1, 1).[14] In contrast, C−H

Scheme 1. Regioselectively Diverse Nitration of *N*-(Quinolin-8-yl)benzamide with Different Nitration Reagents



transformations on the quinoline ring with the carboxamide unit as a directing group have also been explored.[15] Whiteoak reported a remote C−H nitration of 8-aminoquinoline amide giving 5-nitro-8-aminoquinoline amide or 7-nitro-8-aminoquinoline amide by using $Co(NO_3)_2·6H_2O$ as the catalyst and ^{t}BuONO as the nitro source (Scheme 1, 2).[16a] Zhang very recently reported an alternative transformation by using $Cu(NO_3)_2/NaNO_2/PhI(TFA)_2$ as the nitration system.[16b] Inspired by these elegant studies and bearing in mind that Fe(III) salt has been used as a nontoxic and inexpensive catalyst to catalyze remote C−H allylation on the quinoline ring in 8-aminoquinoline amides[15b] and that the thermal decomposition of $Fe(NO_3)_3·9H_2O$ could generate nitrogen dioxide radical ($NO_2^•$) to be used as a clean nitration reagent,[17] we proposed that $Fe(NO_3)_3·9H_2O$ might be used as both a chelating promoter

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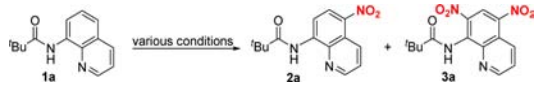
and nitro source for the regioselective C(5)−H nitration of 8-aminoquinoline amide. Gratifyingly, our study found that upon treatment with $Fe(NO_3)_3·9H_2O$, 8-aminoquinoline amide underwent the desired C(5)−H nitration to give 5-nitro-8-aminoquinoline amide in good efficiency. When $CuCl_2·2H_2O$ was used as a catalyst, interestingly, the reaction dominantly afforded the C(5)−H and C(7)−H bis-nitration product, 5,7-dinitro-8-aminoquinoline amide (Scheme 1, 3). Herein, we report our detailed studies and the corresponding results.

Our study was initiated by treating *N*-(quinolin-8-yl)-pivalamide (**1a**) with $Fe(NO_3)_3·9H_2O$ in DCE at 100 °C for 5 h to give the expected *N*-(5-nitroquinolin-8-yl)pivalamide (**2a**) in 51% yield together with *N*-(5,7-dinitroquinolin-8-yl)-pivalamide (**3a**) in 14% yield (Table 1, entry 1). To improve

Table 1. Optimization Studies[a]



entry	promoter & nitro source	catalyst	solvent	*t* (°C)	yield (%)[b] 2a	3a
1	$Fe(NO_3)_3·9H_2O$	-	DCE	100	51	14
2	$Bi(NO_3)_3·5H_2O$	-	DCE	100	28	8
3	$Co(NO_3)_2·6H_2O$[c]	-	DCE	100	42	15
4	$Cu(NO_3)_2$[c]	-	DCE	100	40	13
5	$Fe(NO_3)_3·9H_2O$	-	dioxane	100	25	8
6	$Fe(NO_3)_3·9H_2O$	-	DMF	100	trace	trace
7	$Fe(NO_3)_3·9H_2O$	-	CH_3CN	100	40	17
8	$Fe(NO_3)_3·9H_2O$	-	TFA	100	trace	trace
9	**$Fe(NO_3)_3·9H_2O$**	**-**	**TFE**	**100**	**72**	**18**
10	$Fe(NO_3)_3·9H_2O$	-	TFE	80	42	14
11	$Fe(NO_3)_3·9H_2O$	-	TFE	120	66	20
12	$Fe(NO_3)_3·9H_2O$[d]	-	TFE	100	70	24
13	$Fe(NO_3)_3·9H_2O$	$CuBr_2$	TFE	100	29	55
14	**$Fe(NO_3)_3·9H_2O$**	**$CuCl_2·2H_2O$**	**TFE**	**100**	**trace**	**86**
15	$Fe(NO_3)_3·9H_2O$	$Cu(OTf)_2$	TFE	100	36	52
16	$Fe(NO_3)_3·9H_2O$	$Cu(OAc)_2$	TFE	100	22	62
17	$Fe(NO_3)_3·9H_2O$	$PdCl_2$	TFE	100	15	73

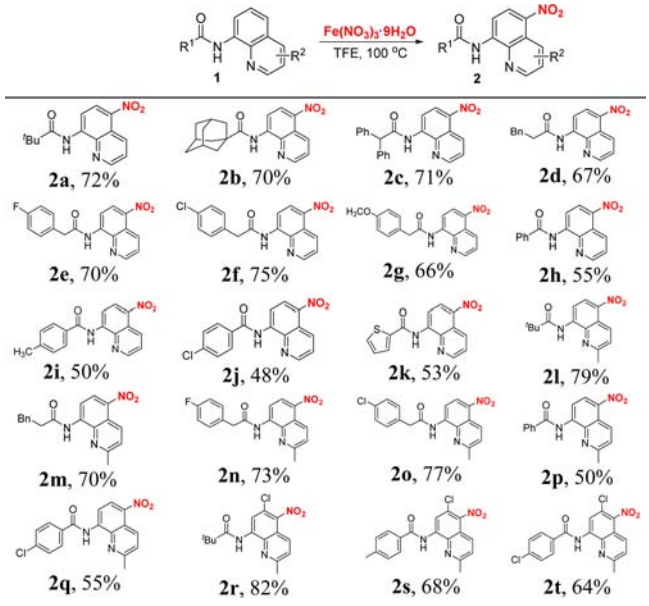
[a]Reaction conditions: **1a** (0.5 mmol), promoter, and nitro source (0.5 mmol), catalyst (0.1 mmol), solvent (2.5 mL), 100 °C, 5 h, sealed tube. [b]Isolated yield. [c]0.75 mmol. [d]1 mmol.

the efficiency, $Bi(NO_3)_3·5H_2O$, $Co(NO_3)_2·6H_2O$ and $Cu(NO_3)_2$ were also tested, but they were found to be less effective than $Fe(NO_3)_3·9H_2O$ (entries 2−4). To study the effect of solvent, dioxane, DMF, CH_3CN, trifluoroacetic acid (TFA), and trifluoroethanol (TFE) were used (entries 5−9). Among them, TFE was found to be the most efficient, affording **2a** in 72% yield (entry 9). Moreover, temperatures lower or higher than 100 °C resulted in reduced yields of **2a** (entries 10−11).

After establishing an efficient synthesis of **2a**, we continued our study to find reaction conditions favoring the formation of **3a** instead of **2a**. For this purpose, 2 equiv of $Fe(NO_3)_3·9H_2O$ was used, but the selectivity of **3a** over **2a** did not improve obviously (Table 1, entry 12 vs 9). Inspired by a recent study in which copper salt was found to be an efficient catalyst for the *ortho*-nitration of *N*,1-diaryl-5-aminotetrazoles with $Fe(NO_3)_3·9H_2O$ as a nitration reagent,[18] we then tried $CuBr_2$, $CuCl_2·2H_2O$, $Cu(OTf)_2$, and $Cu(OAc)_2$ as possible cocatalysts to assist the C(7)−H nitration in improving the yield of **3a**. To our pleasure, addition of copper salts increased the yield of **3a** significantly (entries 13−16). In particular, with $CuCl_2·2H_2O$ as a catalyst, **3a** was obtained in 86% yield while **2a** was formed only in a trace amount (entry 14). As a comparison, when $PdCl_2$ was used as a catalyst, **3a** could be obtained in a yield of 73% (entry 17).

With the optimized reaction conditions in hand, the scope of substrates for the formation of **2** was explored. The results listed in Scheme 2 showed that replacement of the *tert*-butyl unit in **1a**

Scheme 2. Substrate Scope for the Preparation of 2[a,b]



[a]Reaction conditions: **1** (0.5 mmol), $Fe(NO_3)_3·9H_2O$ (0.5 mmol), TFE (2.5 mL), 100 °C, 5 h, sealed tube. [b]Isolated yield.

with adamantyl, diphenyl methyl, phenethyl, and benzyl groups afforded **2b−g** in 66−75% yields. In addition to alkyl amides, substrates with an aryl amide scaffold also reacted with $Fe(NO_3)_3·9H_2O$ to generate **2h−k** in relatively lower yields, and the electronic nature of the aryl ring did not render obvious effect. It is worth noting that nitration on the aryl ring of the aryl amide unit was not observed in all cases. In addition to good tolerance of different kinds of ancillary acyl group, this reaction was also amenable to substrates bearing a 2-methyl group on the quinoline ring and afforded **2l−q** in 50−79% yields. Finally, substrates bearing a 6-chloro group on the quinoline scaffold generated products **2r−t** without showing an obvious steric effect. The above results demonstrated that this is an efficient and versatile method for the preparation of diversely substituted 5-nitro-8-aminoquinoline amides.

Next, the substrate generality for the synthesis of **3** was also studied. The results listed in Scheme 3 showed that this method was amenable to **1** with either aliphatic or aromatic amide units as the ancillary group. Various functionalities such as electron-donating methoxy and methyl groups or electron-withdrawing halides were well tolerated. Remarkably, substrate with a thiopheneamide unit was also suitable for this transformation, furnishing **3h** in 65% yield.

To shed some light on the mechanism, control experiments were carried out (Scheme 4). First, 3 equiv of TEMPO was used as a radical scavenger in the reaction of **1a**. As a result, the formation of **2a** was completely suppressed, showing that the formation of **2a** might involve a radical process. Second, treatment of **1a** with 3 equiv of ^{t}BuONO, a frequently used

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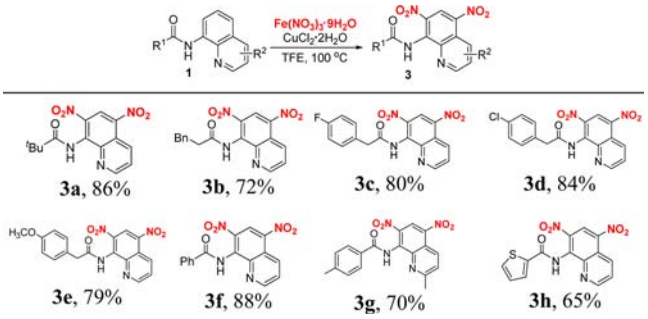
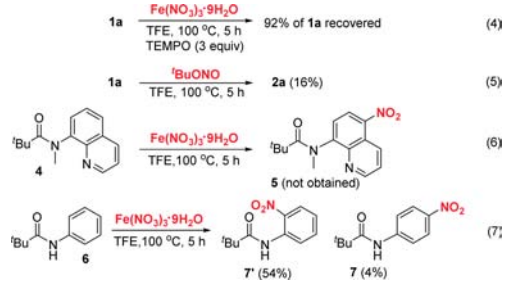
Scheme 3. Substrate Scope for the Preparation of 3[a,b]



[a]Conditions: **1** (0.5 mmol), $Fe(NO_3)_3·9H_2O$ (0.5 mmol), $CuCl_2·2H_2O$ (0.1 mmol), TFE (2.5 mL), 100 °C, 5 h, sealed tube. [b]Isolated yield.

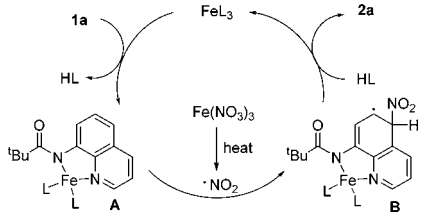
Scheme 4. Control Experiments (I)



NO_2 radical source, in the absence of any catalyst afforded **2a** in a yield of 16%, showing that the catalysis of Fe(III) is crucial for the high-yielding synthesis of **2a**. Third, when *N*-methyl-*N*-(quinolin-8-yl)pivalamide (**4**) was used, the nitration product **5** was not found, indicating that the free NH unit is indispensible. Fourth, treatment of *N*-phenylpivalamide (**6**) under standard reaction conditions afforded *N*-(4-nitrophenyl) pivalamide (**7**) in 4% yield but gave *N*-(2-nitrophenyl)pival amide (**7′**) in 54% yield, showing that the pyridine unit of the quinoline ring plays a key role for the regioselective nitration.

On the basis of the above facts and previous reports,[15d,f] a plausible pathway for the formation of **2a** is proposed in Scheme 5. First, coordination of **1a** with Fe(III) affords a chelated

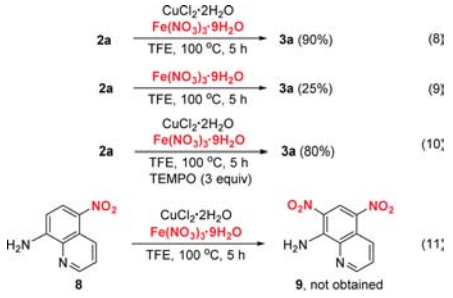
Scheme 5. Proposed Mechanism for the Formation of 2a



intermediate (**A**). Meanwhile, $NO_2^{\bullet}$ is generated from $Fe(NO_3)_3$ under thermal conditions[17] and then reacts with **A** at the C-5 position of the quinoline unit as C-5 has the largest p_z orbital occupancies as reported previously by Yang[15d] to give **B**. Aromatization and decomposition of **B** provide **2a**.

To clarify the mechanism for the formation of **3a**, more control experiments were performed (Scheme 6). First, treating **2a** with $Fe(NO_3)_3·9H_2O$ and $CuCl_2·2H_2O$ gave **3a** in a yield of 90%, indicating that **2a** is most likely an intermediate for the formation of **3a** from **1a**. Second, treating **2a** with $Fe(NO_3)_3·9H_2O$ in the

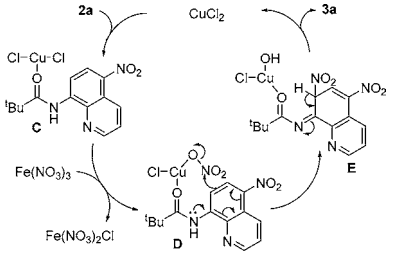
Scheme 6. Control Experiments (II)



absence of $CuCl_2·2H_2O$ afforded **3a** in 25% yield. Third, treating **2a** with $Fe(NO_3)_3·9H_2O$ and $CuCl_2·2H_2O$ in the presence of 3 equiv of TEMPO still gave **3a** in 80% yield, indicating that the nitration of **2a** may mainly proceed via an ionic pathway. Fourth, when 5-nitroquinolin-8-amine (**8**) was used, a complicated mixture, rather than the desired 5,7-dinitroquinolin-8-amine (**9**), was obtained, indicating that the presence of the acyl ancillary group is crucial for the success of the nitration. Moreover, two deuterium experiments showed that the C(5)−H and C(7)−H bond cleavage is not the key factor influencing the nitration process (see the SI for the details).

On the basis of the above facts and previous reports,[18] a plausible mechanism for the formation of **3a** from **2a** is proposed in Scheme 7. First, **2a** binds with $CuCl_2$ to give intermediate **C**,

Scheme 7. Proposed Mechanism for the Formation of 3a



which then complexes with the nitrate unit of $Fe(NO_3)_3$ to give **D**. Then, an intramolecular *ortho*-nitration of **D** via a possible electrophilic substitution pathway takes place to afford **E**. Finally, aromatization occurs with **E** to afford **3a**.

Next, we continued our study by performing the nitration on a gram scale. Thus, 5 mmol of **1n** was treated with $Fe(NO_3)_3·9H_2O$ in TFE at 100 °C for 5 h. From this reaction, **2n** was obtained in 71% yield (Scheme 8). Afterward, structural

Scheme 8. Gram-Scale Synthesis and Transformation of 2n

elaborations of **2n** were explored. It was found that the 2-(4-fluorophenyl)acetyl ancillary group in **2n** could be easily removed by treating it with NaOH in refluxing ethanol to give **10** without damaging the nitro group. On the other hand, the nitro group in **2n** could be conveniently reduced to an amine unit by subjecting it to iron in acetic acid to give **11**.

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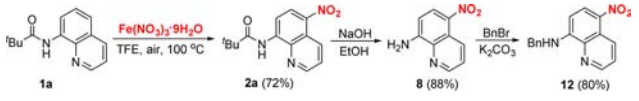
N-Benzyl-5-nitroquinolin-8-amine (**12**, Scheme 9) is one of a series of compounds developed as potential cathepsin B

Scheme 9. Alternative Synthesis of 12



inhibitors by Gobec,[8d] and therein they were prepared from 8-hydroxyquinoline via hydroxyl protection, aromatic nitration, and amination. While this synthetic route is reliable, it uses much excessive amounts of H_2SO_4 and KNO_3 for the nitration. In view of the increasing need for more economical and sustainable chemistry, we developed an alternative synthetic approach toward **12** starting from **1a** by using the nitration protocol developed herein as a key step.

In summary, we have established an efficient remote C(5)−H nitration of 8-aminoquinoline amides by using the inexpensive and nontoxic $Fe(NO_3)_3·9H_2O$ as both promoter and nitro source. Moreover, with $CuCl_2·2H_2O$ as a catalyst, the 5,7-bisnitration of 8-aminoquinoline amides was realized efficiently. In general, the reactions were accomplished under mild and neutral conditions and showed good tolerance toward a variety of functional groups. With these advantages, this relatively green and economical nitration method is expected to find applications in the synthesis of related aromatic nitro compounds.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.6b02998.

Experimental procedure, characterization data, and NMR spectra of all products (PDF)

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Notes

The authors declare no competing financial interest.

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