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ARTICLE

Synthesis of symmetrical pyridines by iron-catalyzed cyclization of ketoxime acetates and aldehydes

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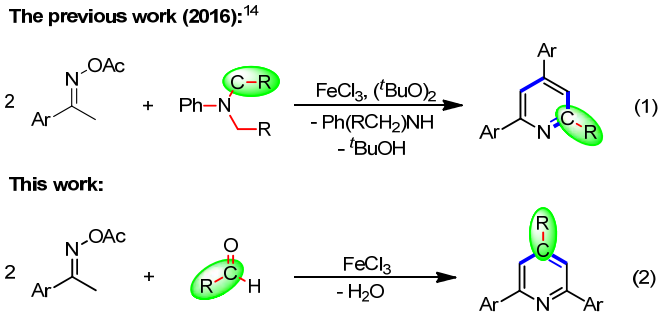
A novel and facile iron-catalyzed cyclization of ketoxime acetates and aldehydes for the green synthesis of substituted pyridines has been developed. In the presence of a $FeCl_3$ catalyst, the reaction exhibited a good functional group tolerance to produce 2,4,6-triarylsubstituted symmetrical pyridines in high yields in the absence of any additive. A gram-scale reaction sequence was performed to demonstrate the scaled-up applicability of this synthetic method.

Introduction

Ketoximes and corresponding derivatives are readily accessible and highly reactive building blocks.[1] Ketoxime acetates are frequently used for the production of aza-heterocycle due to the finding that the N-O cleavage of ketoxime acetates may easily furnish this transformation in the presence of transition-metal catalyst.[2] Pd(0)-catalyzed annulation reactions of ketoxime carboxylates for the synthesis of N-containing compounds has been developed by Narasara *et al.*[3] and other groups.[4] In the past few years, Cu(I)-catalyzed coupling reactions of ketoxime acetates have been developed by our[5] as well as other groups.[6] Recently, cyclization of ketoxime acetates with alternative coupling partners for the synthesis of substituted aza-heterocycles have emerged under Ru(II), Co(III), Rh(III)-catalyzed or transition metal-free conditions.[7-10] However, most of the reactions require noble transition metal catalysts and various environmental unfriendly additives. Therefore, the development of novel, facile and green transformations of ketoxime acetates for the synthesis of useful compounds still remains highly desirable.

Iron salts represent some of the cheapest and most environmental friendly transition metal catalysts. In the past decades, a variety of iron-catalyzed coupling or oxidative reactions have been developed and established to be particularly viable in organic synthesis.[11,12] However, Fe-catalyzed transformations of ketoxime acetates have rarely been studied.[13] Recently, we have developed a Fe-catalyzed oxidative cyclization of ketoxime acetates and tertiary anilines for the synthesis of 2,4-disubstituted pyridines (Scheme 1, eq 1).[14] In this reaction, the methylene carbon on *N,N*-dialkylanilines plays a role as the source of one-carbon synthon under oxidative conditions. Inspired by the reaction, we hypothesized that the atom-economy and efficiency of the cyclization reaction might be improved by using aldehyde as the source of one-carbon synthon, directly (Scheme 1, eq 2). If that's so, the reaction would proceed under redox-neutral conditions to produce H_2O instesd of $Ph(RCH_2)NH$ and ^tBuOH as the byproducts. However, it was found that the chemoselectivity of the reaction was different with the previous report.[14] The 2,4,6-trisubstituted symmetrical pyridines was obtained in Fe-catalyzed cyclization of ketoxime acetates and aldehydes (Scheme 1, eq 2). Additionally, we found that this Fe-catalyzed reaction of ketoxime acetates and aldehydes proceeded greenly in the absence of any additive, which is in contrast to our previous Cu-catalyzed reaction systems (*large excessive $NaHSO_3$ was required to inhibit the decomposition of ketoxime acetates*).[5] It should be noted that symmetrical pyridines is a class of valuable heterocycles which was discovered to have good biological activity in pharmaceuticals.[15] However, general, facil and green method for the synthesis of valuable symmetrical pyridines has rarely been developed,[16] albeit their are numerous protocols for the synthesis of versatile unsymmetrical substituted pyridines.[17,18] Therefore, we describe herein the development of a novel and green Fe-catalyzed cyclization of ketoxime acetates with



Scheme 1 Fe-Catalyzed green synthesis of pyridines from ketoxime acetates.

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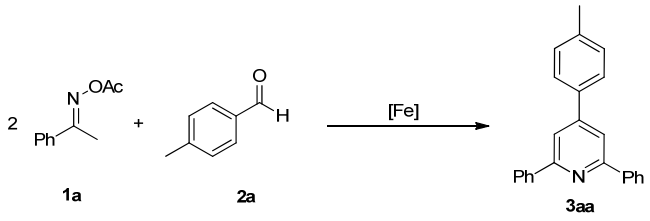
aldehydes for the synthesis of 2,4,6-trisubstituted symmetrical pyridines under very simple conditions (Scheme 1, eq 2).

Results and discussion

Our initial efforts focused on the cyclization reaction of acetophenone oxime acetate **1a** with *p*-methylbenzaldehyde **2a** in the presence of iron catalyst in 1,2-dichloroethane (DCE) at 120 °C. Only a trace amount of the pyridine product was observed (Table 1, entry 1). The same result was observed by using $Fe(OAc)_2$ as the catalyst (Table 1, entry 2). However, a symmetrical 2,6-diphenyl-4-(*p*-tolyl)pyridine **3aa** was obtained in 37% yield in the presence of $FeCl_3$ catalyst (Table 1, entry 3). Althogh the chemoselectivity of the reaction was different with our previous study,[14] this interesting result prompted us to optimize the conditions for developing a general protocol towards symmetrical pyridines. Thus, different iron(III) catalysts and green solvents were screened. It was found that the cylization of acetophenone oxime acetate **1a** and *p*-methylbenzaldehyde **2a** proceeded efficiently in the presence of $FeCl_3$ catalyst in toluene (Table 1, entry 12). Other iron catalysts, such as $FeBr_3$ and $Fe(acac)_3$ shown to be inferior (Table 1, entries 4-5). And no reaction occurred in the absence of iron catalyst (Table 1, entry 6). Diferent solvents, such as H_2O, CH_3OH, DMF, DMSO, and 1,4-dioxane, were inferior to toluene (Table 1, entries 7-11). Furthermore, the yield of **3aa** was improved to 87% when vigorous reflux of the reaction in 140 °C oil bath (Table 1, entry 13).

Table 1 Optimization of the reaction conditions[a]



1a 2a 3aa

Entry	Catalyst	Solvent	T (°C)	Yield (%)
1	$FeCl_2$	DCE	120	trace
2	$Fe(OAc)_2$	DCE	120	trace
3	$FeCl_3$	DCE	120	37
4	$FeBr_3$	DCE	120	28
5	$Fe(acac)_3$	DCE	120	0
6	---	DCE	120	0
7	$FeCl_3$	H_2O	120	0
8	$FeCl_3$	CH_3OH	120	0
9	$FeCl_3$	DMF	120	39
10	$FeCl_3$	DMSO	120	31
11	$FeCl_3$	1,4-dioxane	120	62
12	$FeCl_3$	toluene	120	76
13	**$FeCl_3$**	**toluene**	**140**	**87**

[a]Reaction conditions: **1a** (0.6 mmol), **2a** (0.2 mmol), catalyst (20 mol%), solvent (2 mL), 8 h, Ar ; isolated yield

Table 2 Substrate scope of ketoxime acetates[a]

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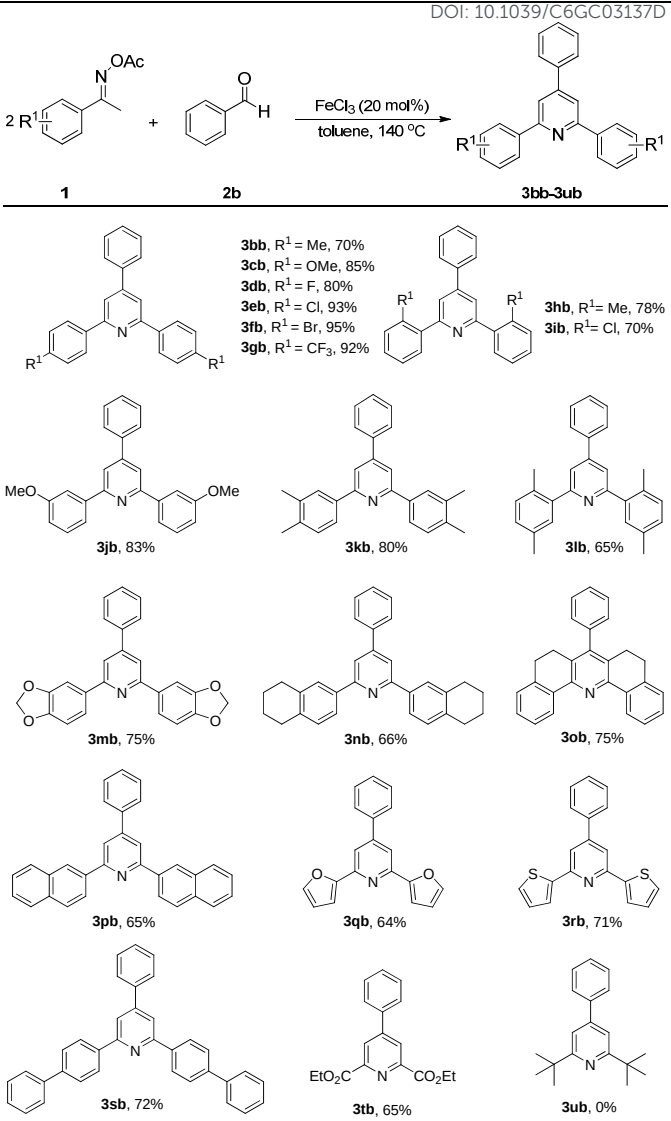
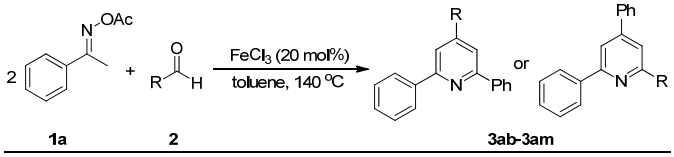


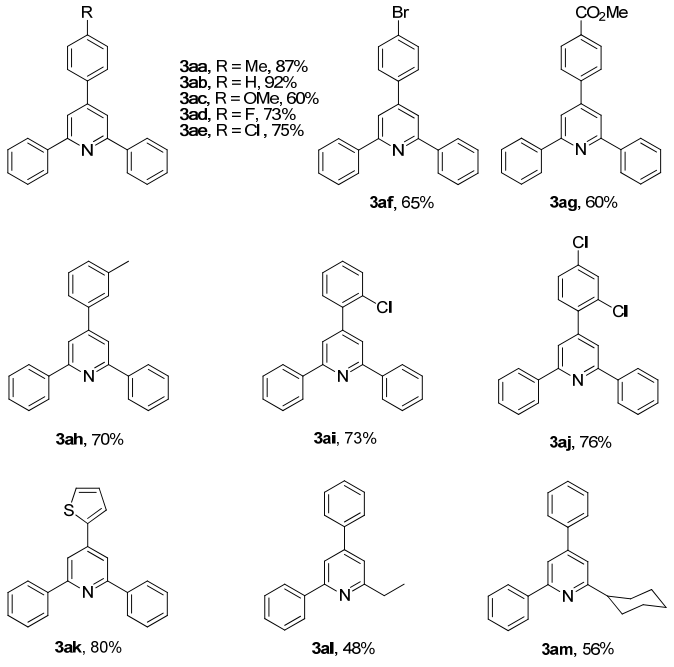
[a]Reaction conditions: **1** (0.6 mmol), **2b** (0.2 mmol), $FeCl_3$ (20 mol%), toluene (2 mL), 6-8 h, Ar ; isolated yield.

With optimized reaction conditions in hands, the scope of ketoxime acetates was investigated (Table 2). The reaction showed good functional group tolerance and proved to be a general method for the synthesis of 2,4,6-trisubstituted symmetrical pyridines. Ketoxime acetates with methyl, methoxyl, fluoro, and sensitive fuctional groups, e.g. chloro and bromo on aryl rings, all provided excellent yields of the corresponding pyridines **3bb-3fb**. In this manner, the resulting Cl or Br substituted products **3eb–3fb** could be further expanded to a wider variety of functionalized pyridines by undergoing subsequent cross-coupling reactions. The strongly electron-withdrawing trifluoromethyl group on the aromatic ring did not exhibit a negative effect on this transformation and the desired product **3gb** was produced with a yield of up to 92%. In addition, *ortho*-methyl and chloro substituted substrates reacted smoothly and resulted in the target symmetrical pyridines **3hb-3ib** in 70%-78% yields. The *meta*-methoxyl substituted ketoxime acetate **1j** resulted in **3jb** in a

yield of 83%. Ketoxime acetates **1k-1n** were also found to be suitable for this transformation, affording **3kb**-**3nb** with yields between 65-80%. The *α*-tetralone oxime acetate **1o** and 2-acetylnaphthalene oxime acetate **1p** underwent the desired reaction to provide the corresponding pyridines **3ob** and **3pb** in good yields. Moreover, hetero-aromatic substrates such as furan and thiophene ketoxime acetates **1q** and **1r** were also obtained with good results, providing 64% and 71% yields of the desired products, respectively. Expectedly, the symmetrical pyridines **3sb-3tb** were also obtained in 72% and 65% yields when *para*-phenyl substituted ketoxime acetate **1s** or ethyl 2-(acetoxyimino)propanoate **1t** was used as the substrate. However, no reaction occurred when the aliphatic ketoxime acetate **1u** was used as the substrate.

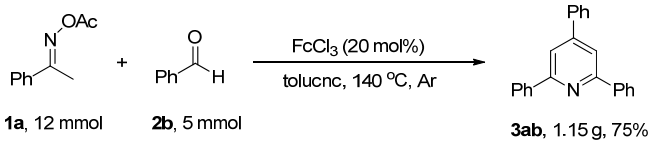
Table 3 Substrate scope of aldehydes[a]





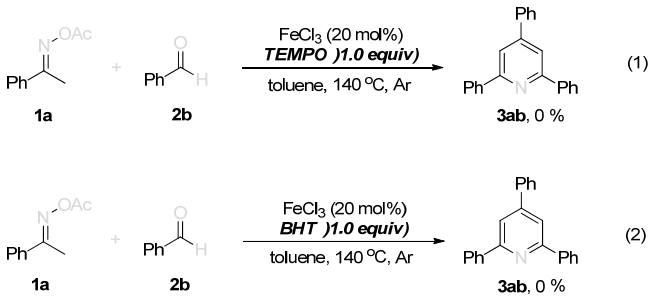
[a]Reaction conditions: **1a** (0.6 mmol), **2** (0.2 mmol), $FeCl_3$ (20 mol%), toluene (2 mL), 6-8 h, Ar ; isolated yield.

Next, we have studied the scope of the aldehydes (Table 3). Different *para*-substituted aromatic aldehydes, containing electron-donating and electron-withdrawing groups, could be converted to the corresponding pyridines in 60-92% yields (**3aa**-**3af**). Noteworthy in this context is the finding that methyl 4-formylbenzoate **2g** was appropriate for this process, providing the corresponding pyridine **3ag** in 60% yield. Furthermore, *meta*-methyl, *ortho*-chloro and 2,4-dichloro substituted aryl aldehydes all exhibited a good reactivity and provided satisfying yields of the desired products (**3ah**-**3aj**). In addition, the use of 2-thiophene aldehyde afforded the symmetrical pyridine **3ak** in 80% yield. Interestingly, unsymmetrical 2,4,6-trisubstituted pyridines **3al**-**3am** were obtained in 48% and 56% yields when the aliphatic aldehydes such as propylaldhyde **2l** and cyclohexanecarbaldehyde **2m** were used as the substrates.



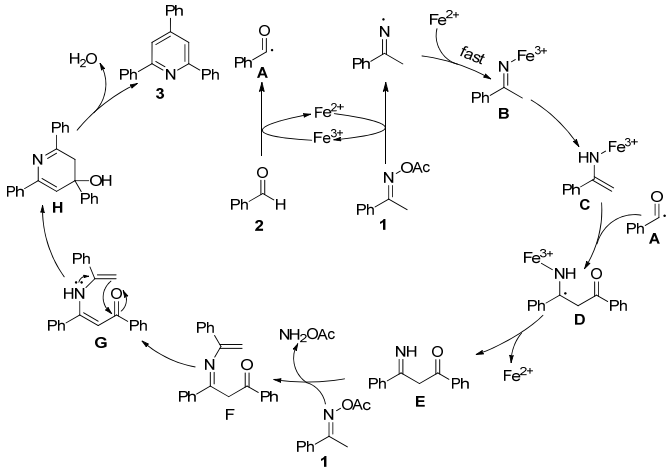
Scheme 2 A gram-scale preparation of 2,4,6-triphenylpyridine.

To demonstrate the synthetic applicability of this reaction, a gram scale reaction was performed under standard conditions. The desired 2,4,6-triphenylpyridine **3ab** was obtained in 75% yield (Scheme 2).



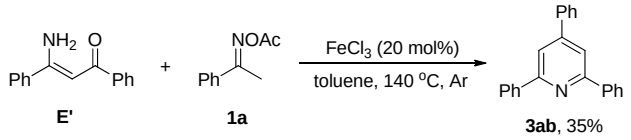
Scheme 3 Control experiments.

To gain insights into the reaction mechanism, control experiments were performed as highlighted in Scheme 3. When the radical scavenger TEMPO (2,2,6,6-tetramethylpiperdine-1-oxyl) or BHT (butylated hydroxytoluene) was added to the reaction mixture, no reaction occurred (Scheme 3, eq 1 and 2). These results indicate that a radical pathway might be involved.



Scheme 4 Plausible mechanism for Fe-catalyzed cyclization of ketoxime acetates with aldehydes for green synthesis of pyridines.

Based on the above results and previous reports,[14,19] a plausible mechanism for the Fe-catalyzed cyclization could be proposed as shown in Scheme 4. Initially, benzoyl radical **A** is formed from benzaldehyde **2** by a single-electron transfer (SET) process with Fe^{3+}.[19b] Subsequently, the reductive cleavage of the N-O bond of ketoxime acetate **1** by a Fe^{2+} species via a two-step SET process forms the Fe^{3+} imine anion complex **B**.[14] Tautomerization of **B** affords **C**, which combines with a benzoyl radical **A** to produce radical **D**.[19] Through a SET process, intermediate **D** leads to the production of intermediate **E** upon releasing Fe^{2+}. Then, the condensation of intermediate **E** with a second molecular of ketoxime acetate **1** forms intermediate **F**.[5,14] Next, tautomerization of **F** produces complex **G** and intramolecular cyclization of intermediate **G**, assisted by $FeCl_3$ forms intermediate **H**. The latter species **H** then undergoes a dehydration step promoted by $FeCl_3$ to provide pyridine **3.**



Scheme 5 Investigation of the reaction mechanism.

To further gain insights into the reaction mechanism, 3-amino-1,3-diphenylprop-2-en-1-one **E'** was synthesized to undergo cycloaddition reaction with acetophenone oxime acetate **1a** under the standard conditions. The desired symmetrical pyridine **3ab** were obtained in morderate yields (Scheme 5). These results further confirm the proposed reaction mechanism.

Conclusions

In summary, we have developed a facile, efficient and green iron-catalyzed cyclization of ketoxime acetates and aldehydes for the synthesis of diverse 2,4,6-triaryl substituted pyridines. In comparation with our previous pyridine synthesis, the current reaction proceeded smoothly under redox-neutral conditions to produce H_2O instesd of $Ph(RCH_2)NH$ and ^tBuOH as the byproducts. The reaction tolerates a wide range of functional groups and provides a simple protocol for the rapid synthesis of valuable 2,4,6-trisubstituted symmetrical pyridines. In addition, grams-scale reaction was performed to demonstratethe scaled-up applicability of this synthetic method. Further scope and mechanistic studies of the reaction are underway in our laboratory.

Experimental

Procedure for the Synthesis of Pyridines

In a 10 mL round bottom flask, the ketoxime acetates **1** (0.6 mmol), aldehydes **2** (0.2 mmol) and $FeCl_3$ (20 mol%, 6.5 mg) was stirred in toluene (2.0 mL) under Ar at 140 °C. When the reaction was completed (detected by TLC), the mixture was cooled to room temperature, extracted with EtOAc (2 × 10 mL) and washed with brine (10 mL). The combined organic layers were dried over anhydrous Na_2SO_4 and then evaporated in vacuo. The residue was purified by column chromatography on silicagel to afford the corresponding pyridines **3** with hexane/ethyl acetate as the eluent.

Acknowledgements

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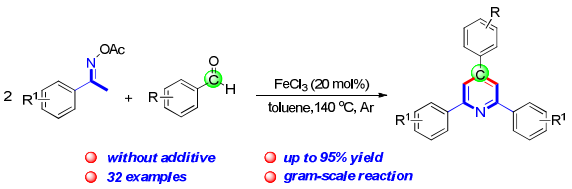

A facile iron-catalyzed cyclization of aldehydes with ketoxime acetates for synthesis of multisubstituted symmetrical pyridines has been developed.