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Synthesis of Kröhnke pyridines through iron-catalyzed oxidative condensation/double alkynylation/amination cascade strategy

Kovuru Gopalaiah*, Renu Choudhary

Department of Chemistry, University of Delhi, Delhi, 110007, India

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ABSTRACT

An efficient protocol for the synthesis of symmetrical and unsymmetrical 2,4,6-trisubstituted pyridines via oxidative cascade annulation of arylacetylenes with benzylamines has been developed. The reaction proceeds smoothly utilizing iron(II) triflate as a catalyst and molecular oxygen as an oxidant with broad substrate scope. Mechanistic studies reveal that the reaction may be experiences an oxidative condensation followed by double alkynylation and amination process.

1. Introduction

The pyridines, especially the 2,4,6-trisubstituted pyridines (Kröhnke pyridines) are important class of nitrogen containing heterocycles, shows a wide range of biological activities such as antibacterial, antifungal, vasodilator, antidepressant, antitumor, anticancer, etc. [1] These pyridines have been utilized in the synthesis of photoluminescent polymers [2], polyimides [3], chemosensors [4], ligands in catalysis [5], and also in photodynamic cancer therapy because of structural resemblance of these pyridines with symmetrical triarylthiopyrylium, telluropyrylium, and selenopyrylium photosensitizers [6]. They are useful intermediates in the preparation of drugs, agrochemicals, surfactants, and desiccants [7]. Due to their π-stacking ability, coordination properties and directional H-bonding, 2,4,6-triarylpyridines are used as substrates for the preparation of therapeutic agents and supramolecules [8]. Classic methods for the synthesis of 2,4,6-trisubstituted pyridines include (i) a multicomponent Chichibabin-type pyiridine reaction employing an aldehyde, an enolizable ketone, and ammonium acetate as a nitrogen source using various catalysts [9], (ii) condensation of keto-oximes/oxime acetates with aryl aldehydes [10] or oxiranes [11], and (iii) reaction of aldehydes with amino allenes followed by palladium-catalyzed cyclization [12]. However, harsh reaction conditions or the utilization of stoichiometric amounts of reagents or expensive catalysts are often involved in these reactions.

To address the above-mentioned drawbacks, one recent burgeoning strategy is to construct these pyridines via earth-abundant first row transition-metal catalyzed oxidative coupling/annulation reaction [13–16]. Indeed, such a strategy could complement the classic methods in terms of reactivity, reaction parameters, selectivity, functional group tolerance and substrate scope. Jiang and co-workers, for example, have first developed the copper-catalyzed oxidative coupling of ketones with aromatic methylamines for the construction of 2,4,6-trisubstituted pyridines (Scheme 1, Eq. 1) [13]. Subsequently, Chen's group reported the copper-catalyzed oxidative sp^3 C–H coupling of oxime acetates or acetophenones with toluene derivatives to afford the 2,4,6-triarylpyridines (Scheme 1, Eq. 2) [14]. Phan and co-workers disclosed an iron-organic framework (VNU-22) catalyzed oxidative cascade reaction of acetophenones with phenylacetic acids in the presence of ammonium acetate as a nitrogen source for the preparation of 2,4,6-triarylpyridines (Scheme 1, Eq. 3) [15] Recently, our research group has also demonstrated an iron-catalyzed oxidative process for the preparation of 2,4,6-trisubstituted pyridines under aerobic conditions using arylalkylketones and benzylamines as the coupling partners (Scheme 1, Eq. 4) [16]. As a continuation of our interest in the synthesis of 2,4,6-triarylpyridines [17] and in the

* Corresponding author.
E-mail address: gopal@chemistry.du.ac.in (K. Gopalaiah).

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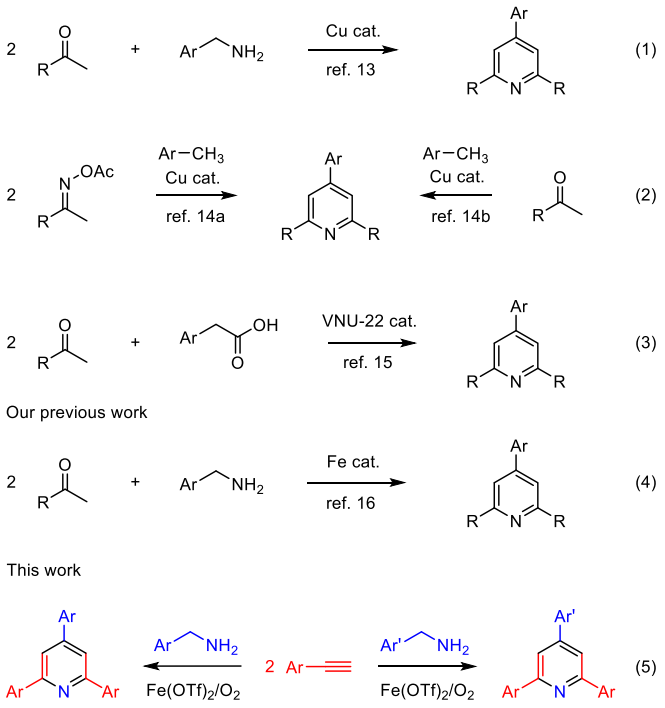


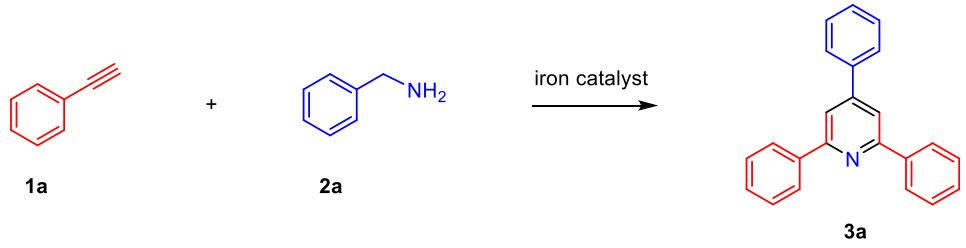
Scheme 1. Oxidative methods for the synthesis of 2,4,6-trisubstituted pyridines.

development of iron-catalyzed oxidative processes [18], we report herein a novel approach for the synthesis of 2,4,6-triarylpyridines starting from readily available arylacetylenes and benzylamines through iron-catalyzed oxidative condensation/double alkynylation and amination cascade strategy (Scheme 1, Eq. 5). Previously, Feng and co-workers studied the cascade reaction of primary amines and alkynes using $CuBr_2$ as a catalyst and TBHP as an oxidant leading to secondary propargylamines in moderate to good yields [19]. The drawback of the Cu-catalyzed oxidative reactions is the homocoupling of terminal alkynes [20], which may limit its applications.

2. Results and discussion

We began our study by examining the reaction of phenylacetylene (**1a**) and benzylamine (**2a**) at 100 °C under molecular oxygen atmosphere for 24 h. When 2 equiv of phenylacetylene reacted with 1 equiv of benzylamine in the absence of any catalyst, no desired product was obtained (Table 1, entry 1). To our delight, pyridine **3a** was obtained in the presence of $FeCl_3$ and $FeBr_3$, albeit in low yields (entries 2 and 3). The product **3a** was formed in 53% yield when $FeBr_2$ was used as a catalyst (entry 4). Interestingly, the yield of **3a** was increased to 71% when $Fe(OTf)_2$ was employed (entry 5). Low yields of **3a** were observed in the reactions of $Fe(ClO_4)_2{\cdot}xH_2O$ and $Fe(ClO_4)_3{\cdot}xH_2O$ (entries 6 and 7). Other iron salts, such as $Fe(OAc)_2$, $Fe(acac)_2$, $Fe(acac)_3$, $FeSO_4{\cdot}7H_2O$, $Fe(NO_3)_3{\cdot}9H_2O$, were not effective for the pyridine formation. We next examined the effect of reaction solvents. The reactions proceeded with low yields in toluene and 1,4-dioxane, but ineffective in polar solvents such as DMF and DMSO (entries 8–11). The effect of air or molecular oxygen as oxidant for the reaction was also examined. A lower yield of **3a** was obtained when the reaction conducted under air atmosphere, and only trace amount of pyridine product was formed in the absence of molecular oxygen (entries 12 and 13). Gratifying, the yield of the product **3a** was further improved to 84% when the reaction temperature increased to 120 °C (entry 14). Therefore, the optimal conditions for the present investigation are the following: 2.0 equiv of phenylacetylene, 1.0

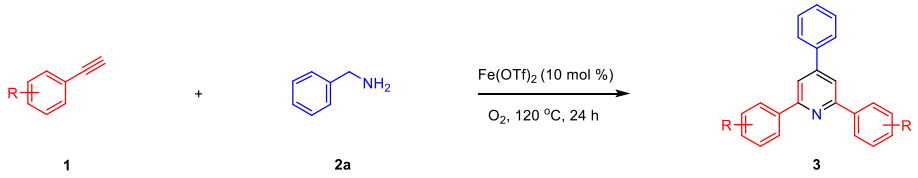
Table 1
Optimization of the oxidative cascade reaction[a].



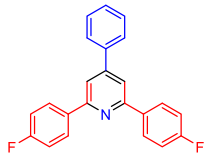
Entry	Iron catalyst	Solvent	Yield (%)[b]
1	–	–	0
2	$FeCl_3$	–	19
3	$FeBr_3$	–	30
4	$FeBr_2$	–	53
5	$Fe(OTf)_2$	–	71
6	$Fe(ClO_4)_2.xH_20$	–	45
7	$Fe(ClO_4)_3.xH_20$	–	26
8	$Fe(OTf)_2$	toluene	65
9	$Fe(OTf)_2$	1,4 dioxane	52
10	$Fe(OTf)_2$	DMF	trace
11	$Fe(OTf)_2$	DMSO	trace
12[c]	$Fe(OTf)_2$	–	48
13[d]	$Fe(OTf)_2$	–	trace
14[e]	$Fe(OTf)_2$	–	84

[a] Reaction parameters: 2.0 mmol of **1a**, 1.0 mmol of **2a**, 10 mol % of iron catalyst, 100 °C, oxygen atmosphere (O_2 balloon), 24 h.
[b] Isolated yield.
[c] The reaction was carried out under air atmosphere.
[d] The reaction was carried out in a sealed tube.
[e] The reaction was carried out at 120 °C.

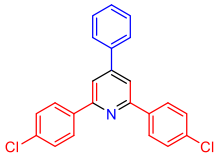
Table 2
$Fe(OTf)_2$-catalyzed oxidative cascade reaction of arylacetylenes **1** with **2a**[a,b].



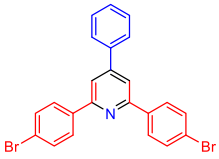
1 **2a** **3**



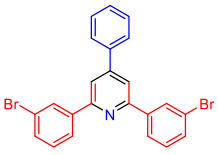
3b, 81 %



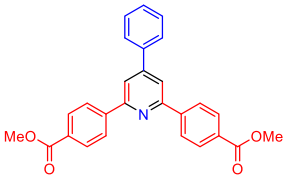
3c, 85 %



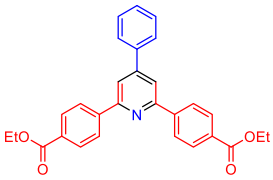
3d, 79 %



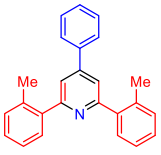
3e, 77%



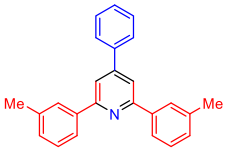
3f, 31 %



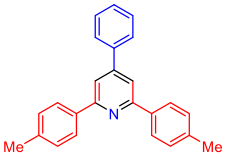
3g, 28 %



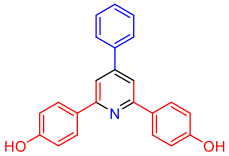
3h, 82 %



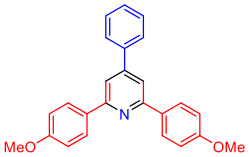
3i, 84 %



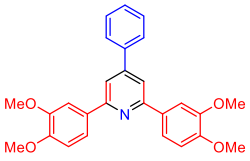
3j, 85 %



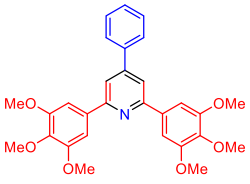
3k, 82 %



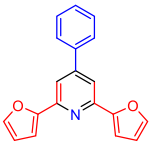
3l, 88%



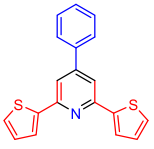
3m, 89 %



3n, 86 %

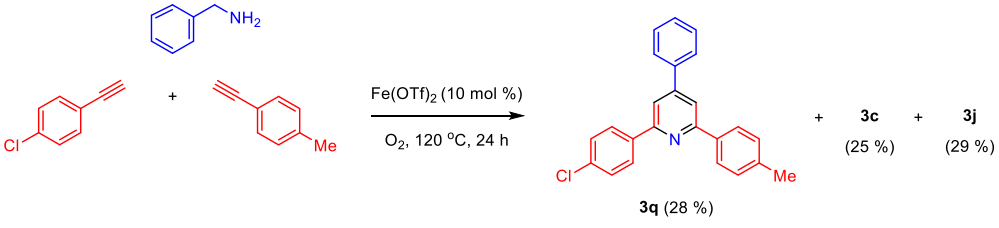


3o, 70 %



3p, 73 %

[a] Reaction parameters: 2.0 mmol of **1**, 1.0 mmol of **2a**, 10 mol % of $Fe(OTf)_2$, 120 °C, O_2 balloon. [b] Isolated yields.



3q (28 %)

Scheme 2. Reaction of two different alkynes with benzylamine.

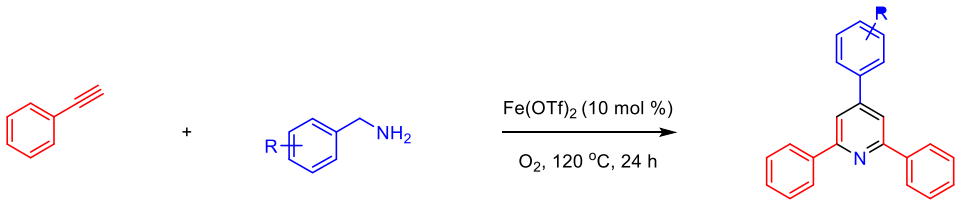
equiv of benzylamine, 10 mol % $Fe(OTf)_2$ at 120 °C under O_2 atmosphere for 24 h.

With the established reaction conditions in hand, we first evaluated the scope of alkynes bearing different aromatic and heteroaromatic rings (Table 2). As illustrated, the phenylacetylenes containing electron-withdrawing groups such as fluoro, chloro and bromo at the *para* or *meta* positions of the phenyl ring were efficiently reacted with benzylamine, affording the desired products with good yields (**3b**-**3e**). The phenylacetylenes containing ester group also gave the pyridine products **3f-3g** albeit in low yields, but nitro substrate namely 1-ethynyl-4-nitrobenzene failed to afford the desired product. Phenylacetylenes bearing electron-donating groups such as methyl, hydroxy, methoxy functionalities at the *ortho*, *meta* or *para* positions also smoothly underwent the oxidative cascade reaction to furnish the corresponding products in good yields (**3h**-**3l**). Electron-rich disubstituted and trisubstituted phenylacetylenes also furnished the desired products **3m** and **3n** in 89% and 86% yields, respectively. Additionally, the heteroaryl-substituted alkynes also served as suitable reacting partners to offer good yields, as exemplified by **3o** and **3p**. Unfortunately, 1-hexyne and 1-decyne failed to give the desired products. It is noteworthy to mention that the synthesized products **3d**, **3j**, **3o** and **3p** are useful building blocks for the preparation of photoluminescence polymers [21], liquid crystalline polymers [22], 4-arylpyridines [23], and conjugated polymer chemosensors and OLEDs [24], respectively.

To construct unsymmetrical 2,4,6-trisubstituted pyridines with different groups at 2- and 6-positions, we carried out the reaction of two different alkynes, namely, 1-chloro-4-ethynylbenzene and 1-ethynyl-4-methylbenzene, with benzylamine under the standard reaction conditions (Scheme 2). It gave a mixture of three products **3q**, **3c** and **3j**, which are separated and characterised by spectroscopic techniques.

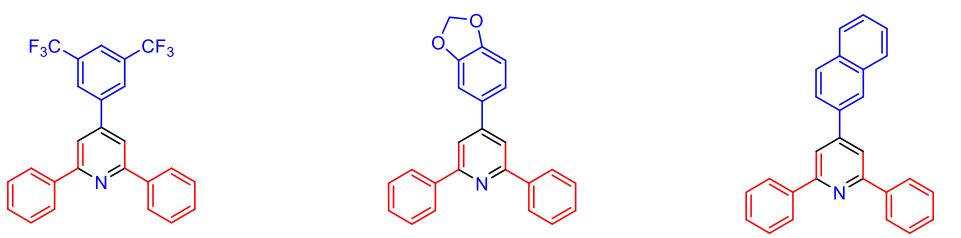
Next, we examined the reactions of **1a** with various substituted benzylamines (Table 3). The benzylamines bearing electron-donating groups (methyl, *iso*-propyl, methoxy, phenyl) and electron-withdrawing substituents (fluoro, chloro, bromo) on the aryl ring were proceeded well to give the desired products in good yields (**4a**-**4i**). The substituents at different positions (*para*, *meta* or *ortho* positions) on the arene ring of benzylamine did not much affect the reaction efficiency (**4a**-**4c**). It is noteworthy that the halogen-substituted benzylamines tolerated well, leading to halogen-substituted 2,4,6-triarylpyridines (**4g**-**4i**), which could be used further in various classical metal-catalyzed cross-coupling reactions. Besides, the method was equally effective for strong electron-withdrawing trifluoromethyl groups containing amine, namely, 3,5-bis(trifluoromethyl)benzylamine to give the corresponding product **4j** in 71% yield under the standard reaction conditions. Furthermore, piperonylamine also smoothly underwent the oxidative reaction to furnish the desired product in good

Table 3
$Fe(OTf)_2$-catalyzed oxidative cascade reaction of **1a** with benzylamines **2**[a,b].



1a + **2** → **4**

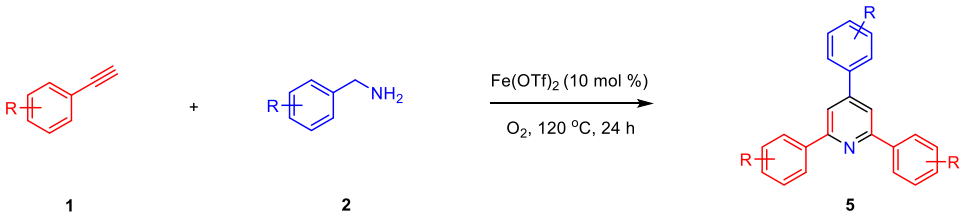
4a	R = 4-Me	86 %
4b	R = 3-Me	86 %
4c	R = 2-Me	84 %
4d	R = 4-*i*-Pr	85 %
4e	R = 4-OMe	89 %
4f	R = 4-Ph	81 %
4g	R = 4-F	82 %
4h	R = 4-Cl	83 %
4i	R = 4-Br	80 %



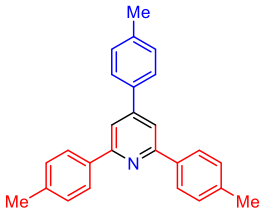
4j, 71% **4k**, 66% **4l**, 73%

[a] Reaction parameters: 2.0 mmol of **1a**, 1.0 mmol of **2**, 10 mol % of $Fe(OTf)_2$, 120 °C, O_2 balloon. [b] Isolated yields.

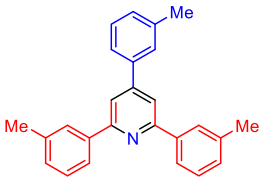
Table 4
$Fe(OTf)_2$-catalyzed oxidative cascade reaction of arylacetylenes **1** with benzylamines **2**[a,b].



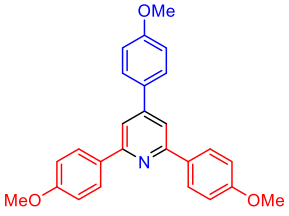
1 **2** **5**



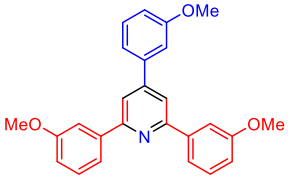
5a, 86 %



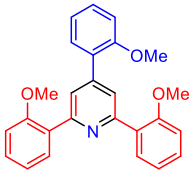
5b, 84 %



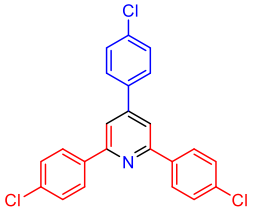
5c, 93 %



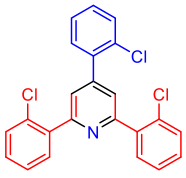
5d, 87 %



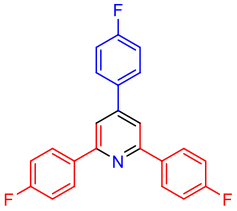
5e, 83 %



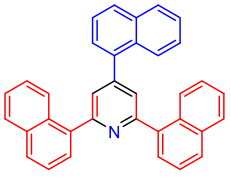
5f, 89 %



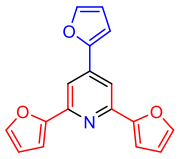
5g, 78 %



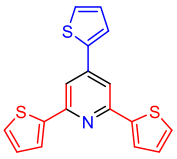
5h, 82 %



5i, 57 %



5j, 59 %

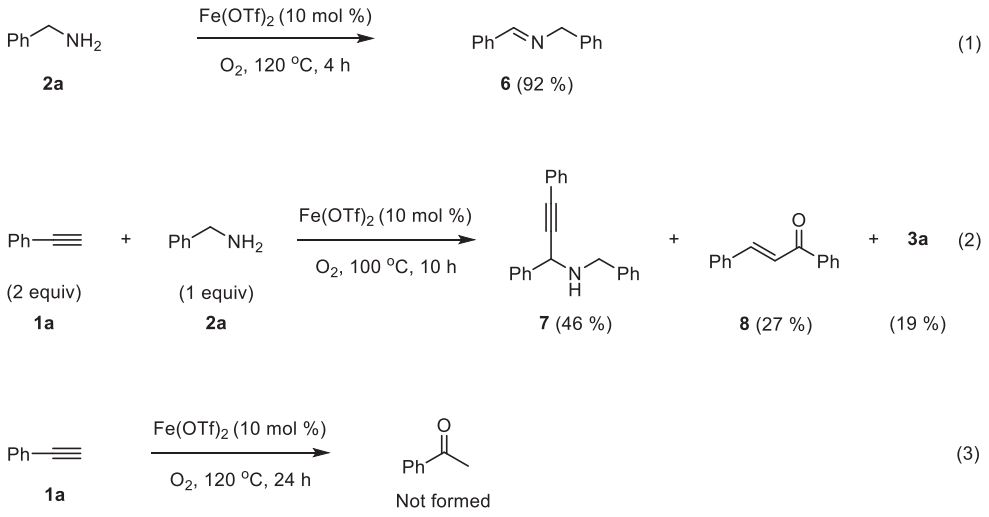


5k, 64 %

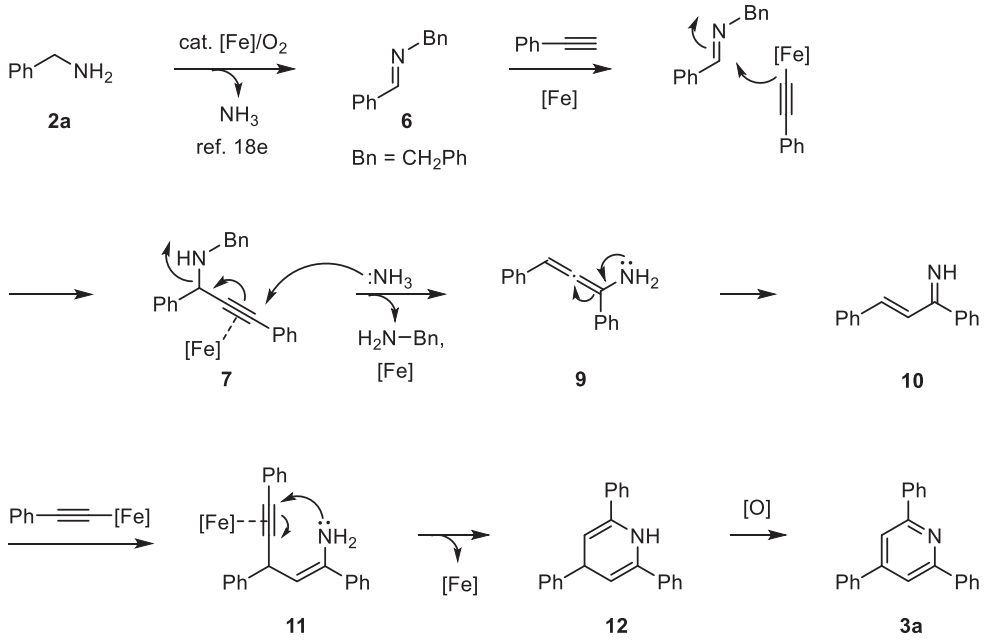
[a] Reaction parameters: 2.0 mmol of **1**, 1.0 mmol of **2**, 10 mol % of $Fe(OTf)_2$, 120 °C, O_2 balloon. [b] Isolated yields.

yield (**4k**, 66% yield). In addition to benzylamines, the naphthyl-substituted amine viz., 1-(2-naphthyl)methanamine was also compatible with the reaction conditions, affording the corresponding product **4l** in 73% yield. However, an aliphatic amine such as *n*-octylamine did not give the desired product.

Subsequently, we turned our attention to synthesize pyridines possessing identical aryl functionalities at 2, 4 and 6-positions with the present synthetic protocol (Table 4). The reactions of phenylacetylenes and benzylamines bearing electron-donating groups such as methyl, methoxy at the *para*, *meta* or *ortho* positions of the phenyl rings, afforded the pyridines **5a**-**5e** in good to excellent yields. Similar results were obtained from phenylacetylenes and benzylamines substituted by electron-withdrawing groups (chloro, fluoro) in *para* or *ortho*-positions of aryl rings (**5f**-**5h**, 78–89% yields). 1-Ethynylnaphthalene and 1-naphthylmethylamine were also found to be suitable starting materials for this oxidative cascade reaction to furnish the product **5i** in 57% yield. Furthermore, the heteroaryl substituted alkynes and amines were served as good substrates for this reaction to provide good yields of **5j** and **5k**. It is also noteworthy to mention that the pyridine products **5a**



Scheme 3. Control experiments.



Scheme 4. Proposed mechanism for the synthesis of 2,4,6-trisubstituted pyridines.

and **5k** are valuable precursors for the preparation of luminescent mesoporous metal-organic frameworks [25], multibranched or star-shaped π-conjugated materials [26], oligo(2,2-bithien-5-yl)-substituted pyridine derivatives [27], and topoisomerase I inhibitors [28].

To understand the mechanism of the present oxidative annulation, we conducted some control experiments (Scheme 3). Treatment of benzylamine (**2a**) with iron(II) triflate in the presence of molecular oxygen at 120 °C for 4 h furnished the self-condensation product *N*-benzylidenebenzylamine (**6**) in 92% yield (Scheme 3, Eq. 1). Next, we carried out the reaction between phenylacetylene (**1a**) and benzylamine (**2a**) in the presence of iron(II) triflate and molecular oxygen at a lower temperature (100 °C) and interrupted the reaction after 10 h, resulting the formation of propargylamine **7** and chalcone **8** in 46% and 27% yields, respectively, along with the desired product **3a** (Scheme 3, Eq. 2). Notably, the preparation of propargylamines from alkynes and benzylamines under oxidative conditions was demonstrated by Feng and co-workers [19]. Furthermore, it is believed that the chalcone **8** could be generated by hydrolysis of the imino-chalcone **10** (Scheme 4) during the purification process. Besides, phenylacetylene was treated with iron(II) triflate in the absence of benzylamine under the standard reaction conditions, but acetophenone product was not formed (Scheme 3, Eq. 3). Thus, we contemplated a different mechanistic pathway for the present cascade reaction as compared to the previously reported oxidative annulation of arylalkylketones and benzylamines [16].

On the basis of our observations and previous studies [18e,19,29], a plausible mechanism for the formation of triarylpyridines is outlined in Scheme 4. At first, benzylamine (**2a**) would undergo the oxidative self-condensation in the presence of iron catalyst and molecular oxygen to give *N*-benzylidenebenzylamine

(**6**) with the liberation of ammonia. Addition of iron-acetylide, which is generated *in situ* from phenylacetylene and iron catalyst, to the imine **6** would produce the propargylamine **7**. Coordination of the triple bond in alkyne **7** to the iron catalyst enhances the electrophilicity of the alkyne, and subsequent nucleophilic attack of the ammonia would produce the imino-chalcone **10** through the allenylamine intermediate **9**. Next, the 1,4-addition of another iron-acetylide to the chalcone **10** leads to the formation of aminoalkyne **11**. The intermediate **11** would then undergo 6-*endo-dig* cyclization to form dihydropyridine **12**, which further experiences the oxidation under molecular oxygen to give the 2,4,6-triarylpyridine **3a**.

3. Conclusion

In summary, we have developed a one-step strategy to access 2,4,6-trisubstituted pyridines from arylacetylenes and benzylamines by Fe(II)-catalyzed aerobic oxidative cascade annulation. The method is straightforward with simple reaction set up on the benchtop. Alkynes and benzylamines with a variety of aryl and heteroaryl substituents are effective coupling partners. The developed protocol has been found to be applicable for the synthesis of pharmaceutically important compounds and building blocks for functional materials. The mechanistic investigations disclosed that this cascade annulation proceeded through imine formation by self-condensation of benzylamine, double alkynylation and amination process.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.tet.2021.132429.

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