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Advanced Synthesis & Catalysis

One-Pot Synthesis of Benzene and Pyridine Derivatives *via* Copper-Catalyzed Coupling Reactions

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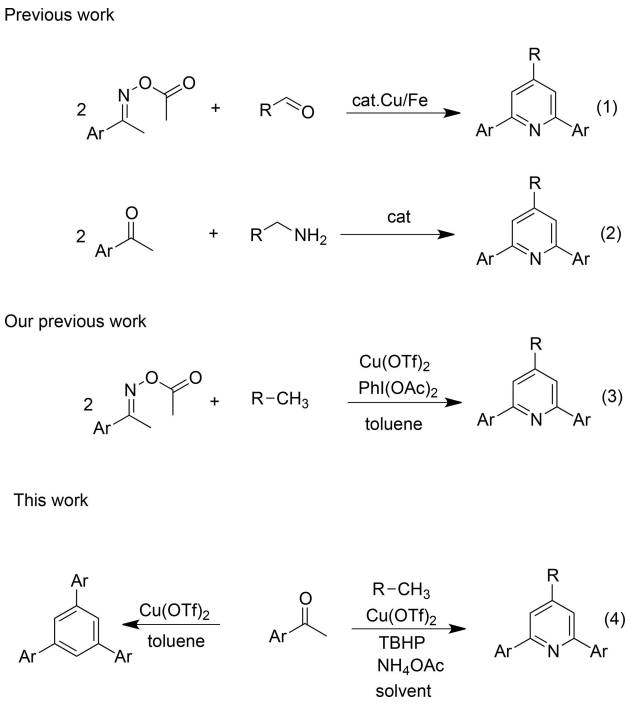
Abstract: A highly efficient one-pot synthesis of polysubstituted pyridine has been achieved through copper-catalyzed oxidative sp^3 C–H coupling of acetophenones with toluene derivatives. Besides, the polysubstituted benzene was obtained through copper-catalyzed coupling of acetophenones. Both of the reactions exhibited a good functional group tolerance to produce a 2,4,6-triphenylpyridine or 1,3,5-triarylbenzene in high yields. Compared with previous methods, these transformations allow a highly flexible and efficient preparation of substituted pyridines and benzenes.

Keywords: polysubstituted pyridine; copper-catalyzed; polysubstituted benzene; acetophenones; toluene derivatives

Pyridine is one of the most important privileged *N*-heterocyclic compounds in organic chemistry, that can be found in a vast array of natural products such as alkaloids.[1] The functionalized pyridines are widely used in industry, and have been diversified into subunits such as anti-HIV drugs, anticancer drugs, anti-oxidation materials and anti-bacterial materials in medicine, pesticides and organic materials in agriculture, etc.[2] In the past years, a variety of methods have been reported for the synthesis of pyridine heterocycles. Despite efficiency and importance,[3] these methods are still restricted to high temperature and other harsh conditions. Hence, an efficient method for easily building the pyridine motif is urgently desirable.

Pyridines with the 2,4,6-triphenylpyridine pattern play a highly important role in heterocyclic compounds.[4] Due to their excellent thermal stability, they can be used in asymmetric catalysis and coordination. In the exploration of a 2,4,6-triphenylpyridine synthesis, the most common way involves reaction of acetophenones, a nitrogen source in the presence of various catalysts.[5] Some synthesized them with 2,4,6-triphenylpyridine by oxime acetates.[6] Guan's group[6c,d] reported Cu- and Fe-catalyzed synthesis of 2,4,6-triphenylpyridine with oxime acetates in 2011 and 2017 (Scheme 1, eq 1). Jiang's group[5b] reported a Cu-catalyzed synthesis of 2,4,6-triphenylpyridine with acetophenone and benzylamine under the neat condition in 2013 (Scheme 1, eq 2). More recently, Yi's group[5e] reported a HOTf-catalyzed synthesis of 2,4,6-triarylpyridine with acetophenone and benzylamine under the neat condition in 2016 (Scheme 1, eq 2). In our group,[6a] we reported the Cu-catalyzed synthesis of 2,4,6-triphenylpyridine with oxidative sp^3 C–H coupling of oxime acetates (Scheme 1, eq 3). Encouraged by the graceful strategies mentioned above, we present an economic synthetic method of 2,4,6-triphenylpyridine through copper-catalyzed oxidative sp^3 C–H coupling of acetophenones and NH_4OAc with toluene derivatives (Scheme 1, eq 4).

We initially investigated our reaction by employing acetophenone **1a**, NH_4OAc and toluene **2a** as model substrates. We chose $Cu(OTf)_2$ as the catalyst and O_2 as the oxidant in toluene (1.0 mL) at 100 °C for 14 h (Table 1, entry 1), and the 2,4,6-triphenylpyridine (**3aa**) was formed in a yield of 48%. We continued to optimize the reaction parameters to obtain a better yield. Compared with O_2, air, H_2O_2, $K_2S_2O_8$, PIDA and DTBP, TBHP proved to be the optimal oxidants, affording the desired product **3aa** in 89% yield (Table 1, entries 1–7). Screening of other solvents,

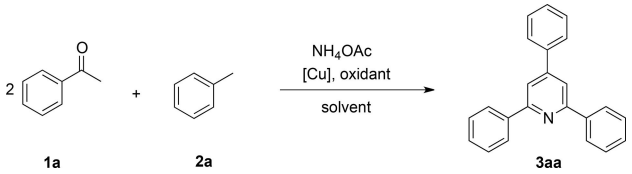


Scheme 1. Synthetic approaches to pyridines.

such as DMSO, DMF and chlorobenzene showed that toluene is the optimal solvent (Table 1, entries 8–10). With chlorobenzene as the solvent, the **3aa** yield was 66%. When the temperature increased to 120 °C, the yield of **3aa** was increased to 83% (Table 1, entries 10–11). Various inorganic nitrogen sources such as NH_4Cl and $(NH_4)_2CO_3$ were screened to confirm NH_4OAc as the best choice for the reaction system (Table 1, entries 12–13). Screening of other copper compound catalysts, such as $Cu(OAc)_2$, $CuBr_2$, $CuCl_2$, CuI and CuBr, showed that $Cu(OTf)_2$ was the optimal choice (Table 1, entries 14–19). By reducing the amount of TBHP, we discovered that the reaction with three equivalents of TBHP afforded the optimal result (Table 1, compared with entries 7 and 20). The temperature was changed to 80 °C, resulting in the product **3aa** having inferior yields. And when the temperature was increased to 120 °C, the yield of the product did not increase (Table 1, entries 21–22).

With the optimized conditions established, the generality of the reaction was explored. We initially investigated the generality of acetophenone, and the results were summarized in Table 2. This coupling reaction tolerated a wide range of substituents on the aromatic ring of acetophenone. According to the experimental results, we found that substrates with weak electron-donating groups on the aromatic ring that produced the corresponding pyridines could provide excellent yields (**3ab**–**3ad**), such as methyl regardless of the functional groups at the *para*-, *meta*-, or *ortho*-position. However, the substrates with strong

Table 1. Optimization of the Reaction Conditions[a]



1a 2a 3aa

Entry	Catalyst	Oxidant	Solvent	Yield[b] (%)
1	$Cu(OTf)_2$	O_2	Toluene	48
2	$Cu(OTf)_2$	Air	Toluene	nd
3	$Cu(OTf)_2$	$K_2S_2O_8$	Toluene	trace
4	$Cu(OTf)_2$	PIDA	Toluene	65
5	$Cu(OTf)_2$	H_2O_2	Toluene	58
6	$Cu(OTf)_2$	DTBP	Toluene	86
7	**$Cu(OTf)_2$**	**TBHP**	**Toluene**	**89**
8	$Cu(OTf)_2$	TBHP	DMSO	nd
9	$Cu(OTf)_2$	TBHP	DMF	nd
10	$Cu(OTf)_2$	TBHP	PhCl	66
11[c]	$Cu(OTf)_2$	TBHP	PhCl	83
12[d]	$Cu(OTf)_2$	TBHP	Toluene	26
13[e]	$Cu(OTf)_2$	TBHP	Toluene	48
14	$Cu(Br)_2$	TBHP	Toluene	78
15	$Cu(Cl)_2$	TBHP	Toluene	56
16	$Cu(OAc)_2$	TBHP	Toluene	66
17	CuBr	TBHP	Toluene	16
18	CuI	TBHP	Toluene	14
19	-	TBHP	Toluene	nd
20[f]	$Cu(OTf)_2$	TBHP	Toluene	83
21[g]	$Cu(OTf)_2$	TBHP	Toluene	33
22[h]	$Cu(OTf)_2$	TBHP	Toluene	87

[a] Reaction conditions: **1a** (0.2 mmol), catalyst (20 mol%), oxidant (3.0 equiv.), solvent (1.0 mL), under air, 100 °C, 14 h.
[b] Yields of isolated products.
[c] 120 °C.
[d] NH_4Cl.
[e] $(NH_4)_2CO_3$.
[f] fTBHP (2.0 equiv.).
[g] 80 °C.
[h] 120 °C.
TBHP = terbutylhydroperoxide (5.0–6.0 M in decane), PIDA = $PhI(OAc)_2$, DTBP = di-t-butyl peroxide, DMSO = demethyl sulfoxide, DMF = N,N-dimethyl formamide.

electron-donating groups (methoxy group) on the aromatic did not afford a good yield (**3ae**–**3ag**). In addition, the experimental results showed that electron-withdrawing groups on the aromatic ring obtained corresponding pyridines in good to excellent yields (**3ah**–**3al**), and some of them seemed to increase the reaction yield slightly such as trifluoromethyl at the *para*-position (**3aj**). Disappointingly, the reaction with *p-nitro*-substituted acetophenone did not obtain the desired product **3ap**, indicating that the acetyl group of the aromatic ring could not cause a reaction because of the electron conjugation and induction of

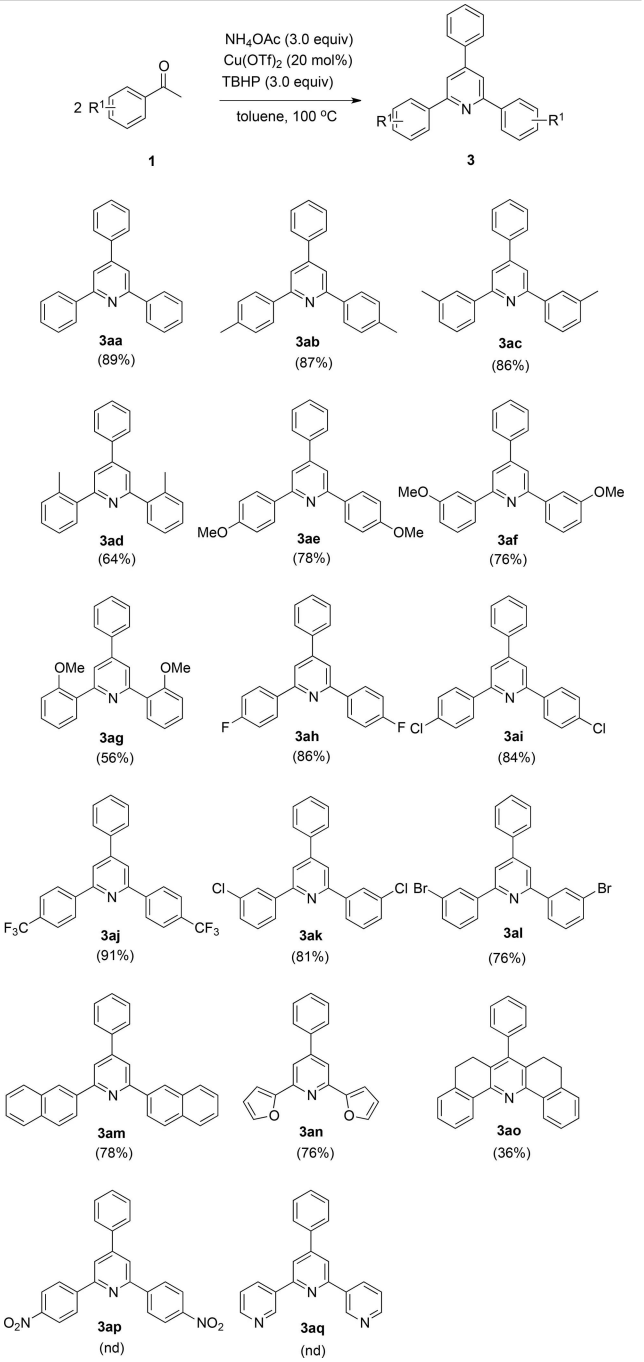
the nitro group. In the intermolecular effect, both *para* and *meta*-position had good yields. The yield of the *ortho*-position (**3ag**) was poor due to a large *ortho*-blockage. In addition, 2-acetylnaphthalene, 2-acetylfuran and α-tetralone also afforded the corresponding pyridines (**3am**–**3ao**) in moderate yields respectively. Besides, the 3-acetylpyridine was also investigated, but the desired product **3aq** was not found.

In order to acquire a variety of polyfunctional pyridines, we next investigated the scope of various methylarenes (Table 3). Toluene was not suitable as a solvent for this reaction. It has been found that chlorobenzene is a relatively better solvent for this purpose (Table 1, entry 11). The optimal temperature for the reaction was determined to be 120 °C and this method was further expanded by the utilization of a wide range of substituted toluenes. The experimental results showed that both the electron-donating groups and the electron-withdrawing groups on the aromatic ring proceeded successfully to come up with the desired products in good yields (**3ba**–**3bk**). Disappointingly, the reaction with 2-nitrotoluene did not obtain the desired product **3bl** indicating that the 2-nitrotoluene cannot cause the reaction because of the electron conjugation and induction of the nitro group. In the intermolecular effect, the corresponding pyridines were obtained in good yields, regardless of the functional groups at the *para*-, *meta*-, *or ortho*-position.

To widen the diversity of substrates for polysubstituted pyridines, we tried to use propiophenones and methyl heterocycles as substrates for this transformation (Scheme 2). Propiophenones and 2-methylfuran afforded the corresponding pyridines (**4aa** and **5aa**) in moderate yields respectively. Disappointingly, the reaction with 2- methylpyridine and 2-methylquinoline did not obtain the desired product (**5ab** and **5ac**).

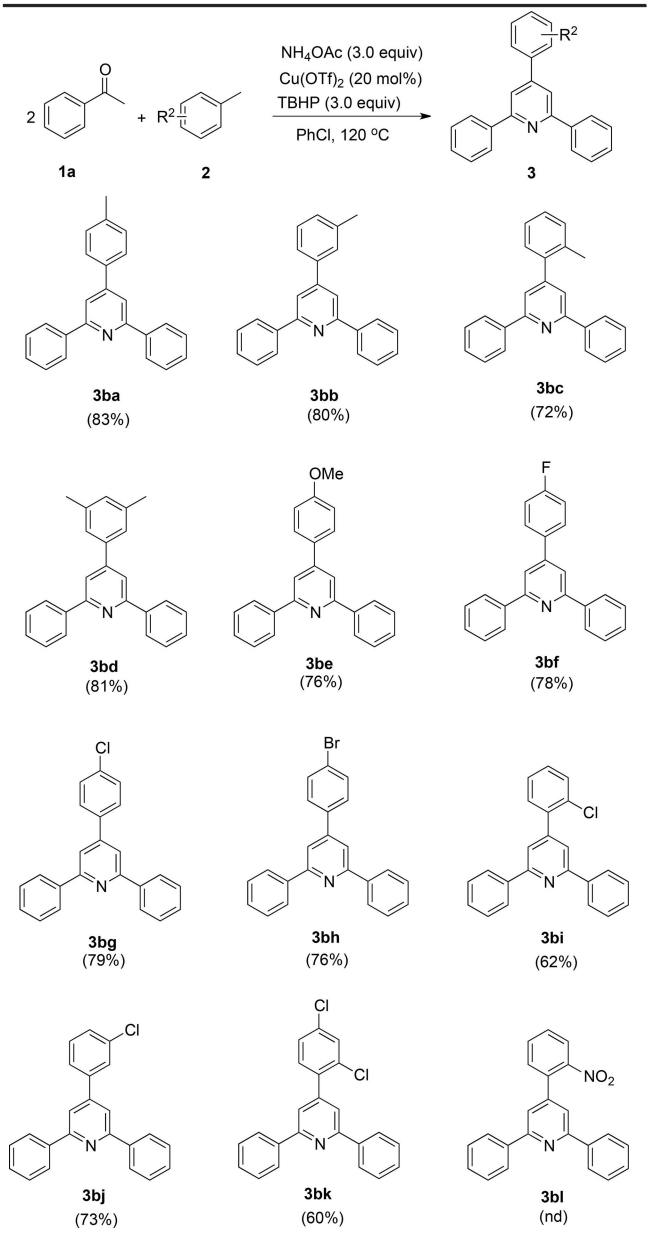
Considering the wide substrate selectivity by the $Cu(OTf)_2$ catalysis, a series of experiments were carried out to explore the mechanisms of reaction. As shown in Scheme 3, we conducted a controlled trial. First, we found no reaction occurring when the catalyst was absent. To our surprise, the trisubstituted benzene (**6a**) scaffold was obtained in a 83% yield, when only adding in acetophenone **1a** and the catalyst with the toluene as solvent (Scheme 3a). At the same time, other corresponding acetophenones successfully obtained the desired products in good yields (**6b**–**e**). The results showed that the epimerization of the enol form of the protonated ketone might be a key intermediate in the catalytic cycle. In addition, it opened up a new method for an efficient way to synthesize benzene. The reaction was not carried out when acetophenone **1a**, the catalyst and ammonium acetate were added to the reaction system with toluene as the solvent. And when the acetophenone **1a**, the catalyst, the ammonium acetate and the benzaldehyde (Scheme 3b) or the benzyl alcohol (Scheme 3c) were added with toluene as the solvent, the 2,4,6-triphenylpyridine was obtained in a good yield. This indicated that the reaction may be toluene

Table 2. Synthesis of pyridines from different acetophenone[a,b]

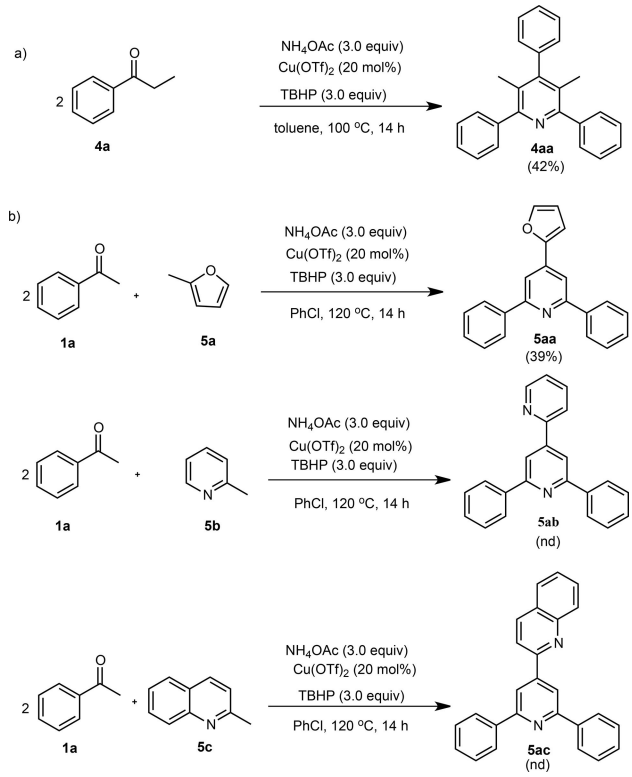


[a] Reaction conditions: **1** (0.2 mmol), $Cu(OTf)_2$ (20 mol%), NH_4OAc (0.6 mmol) and TBHP (0.6 mmol) in Toluene (1.0 mL) at 100 °C for 14 h.
[b] Isolated yields.

Table 3. Synthesis of pyridines from diverse arylmethane[a,b]



[a] Reaction conditions: **1a** (0.2 mmol), **2** (0.6 mmol), $Cu(OTf)_2$ (20 mol%), NH_4OAc (0.6 mmol) and TBHP (0.6 mmol) in PhCl (1.0 mL) at 120 °C for 14 h.
[b] Isolated yields.

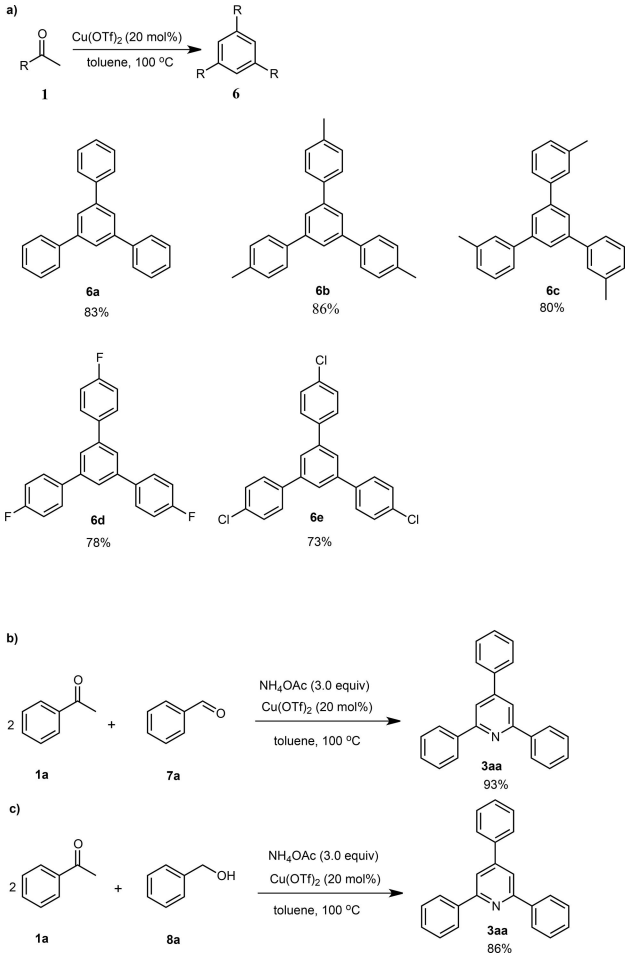


Scheme 2. Synthesis of pyridines from **4a**, **5a**, **5b** and **5c.**

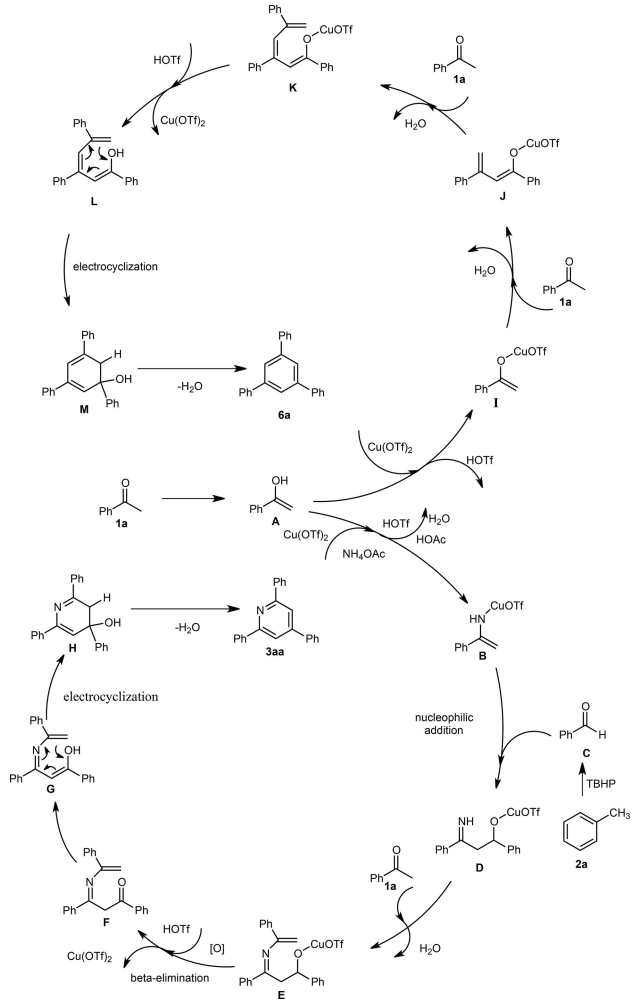
oxidation to benzaldehyde and benzaldehyde should have been another key intermediate of the reaction.

Based on previous studies and reported literature,[7–11] a plausible reaction mechanism has been proposed to explain the formation of product **3aa** and **6a** (Scheme 4). First, acetophenone **1a** would be isomerized to intermediate **A** and intermediate **B** would be formed by intermediate **A**, $Cu(OTf)_2$ and NH_4OAc, with H_2O, HOTf and HOAc being released.[7] While toluene **2a** would be easily converted to benzaldehyde **C** by the TBHP.[8] Next, nucleophilic addition of **B** to **C** would afford imine intermediate **D**.[9] The intermediate **E** would be formed by condensation reaction of intermediate **D** with another molecule of acetophenone **1a**. Then intermediate **E** through beta-elimination and oxidation would afford intermediate **F**.[10] $Cu(OTf)_2$ would be formed by getting rid of copper ions and HOTf, which completed the catalytic cycle. Next, isomerization of **F** would produce complex **G** and electrocyclization of intermediate **G**, assisted by $Cu(OTf)_2$ forms intermediate **H**. Finally, the intermediate **H** would undergo dehydration step to furnish the desired product **3aa**.

On the other hand, the interaction of Cu $(OTf)_2$ and intermediate **A** would afford an intermediate of **I**.[11] Next, the intermediate **J** would be formed by a condensation reaction of intermediate **I** with a second molecule of acetophenone **1a**. Subsequently, the condensation of intermediate **J** with a third molecule of acetophenone **1a** would form intermediate **K**, which would lead to the production of intermediate **L** upon releasing copper ions. $Cu(OTf)_2$ would be formed by getting rid of copper ions and HOTf, which completed the catalytic cycle. Then the intermediate **L** would undergo electrocyclization promoted by $Cu(OTf)_2$ to furnish intermediate **M**. Finally, the intermediate **M** through dehydration step would afford the desired product **6a**.



Scheme 3. Mechanistic studies.



Scheme 4. Possible mechanism.

In summary, we have developed a new, mild and highly efficient $Cu(OTf)_2$ catalyst for the one-pot synthesis of polysubstituted pyridines and benzene. These processes afforded some new approaches to polysubstituted pyridines and benzenes with advantages such as ready availability of the starting materials (acetophenone and toluene), tolerance to a wide range of functional groups, cheap catalysts and mild conditions. Based on the above study, further research on the details of the mechanism and the synthetic applications of this transformation are currently ongoing in our laboratories.

Experimental Section

Synthesis of 2,4,6-triphenyl-pyridine (3aa): A test tube was charged with acetophenone (0.2 mmol), $Cu(OTf)_2$ (20 mol%), NH_4OAc (0.6 mmol) and TBHP (0.6 mmol) in toluene (1.0 mL). The mixture was stirred at 100 °C for 14 h under air. When the reaction was completed (detected by TLC), the mixture was cooled to room temperature. The mixture was diluted with water and extracted with EtOAc (3 × 20 mL). The combined organic layers were dried over anhydrous Na_2SO_4 and evaporation. The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 40:1) to yield the isolated product **3aa**.

Synthesis of 4-(4-methylphenyl)-2,6-diphenylpyridine (3ba): A test tube was charged with acetophenone (0.2 mmol), para-xylene (0.6 mmol), $Cu(OTf)_2$ (20 mol%), NH_4OAc (0.6 mmol) and TBHP (0.6 mmol) in PhCl (1.0 mL). The mixture was stirred at 120 °C for 14 h under air. When the reaction was completed (detected by TLC), the mixture was cooled to room temperature. The mixture was diluted with water and extracted with EtOAc (3 × 20 mL). The combined organic layers were dried over anhydrous Na_2SO_4 and evaporation. The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 40:1) to yield the isolated product **3ba**.

Synthesis of 5′-phenyl-1,1′:3′,1″-terphenyl (6a): A test tube was charged with acetophenone (0.6 mmol) and $Cu(OTf)_2$ (20 mol%) in toluene (1.0 mL). The mixture was stirred at 100 °C for 14 h under air. When the reaction was completed (detected by TLC), the mixture was cooled to room temperature. The mixture was diluted with water and extracted with EtOAc (3 × 20 mL). The combined organic layers were dried

over anhydrous Na_2SO_4 and evaporation. The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 50:1) to yield the isolated product **6a**.

^1H NMR and ^{13}C NMR spectra were determined on 300 MHz and 75 MHz in $CDCl_3$. unknown products were further characterized by HRMS (TOF-ESI), the melting points of solid products were determined on a microscopic apparatus.

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