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Copper-catalyzed efficient access to 2,4,6triphenyl pyridines *via* oxidative decarboxylative coupling of aryl acetic acids with oxime acetates[†]

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An efficient and concise approach for the synthesis of 2,4,6triphenyl pyridines has been developed through copper-catalysed oxidative decarboxylative coupling of $C(sp^3)$ aryl acetic acids with oxime acetates in DMF at 150 °C under an oxygen atmosphere. Various functional groups were well tolerated and provided the corresponding 2,4,6-triphenyl pyridines in good to excellent yields.

Introduction

Nitrogen-heterocyclic compounds are one of the most important classes of compounds and they are found extensively in natural products, pharmaceutical drugs and several functional materials. Among the various heterocycles, pyridines have been recognized as privileged scaffolds because of their widespread biological and synthetic applications.^{1*a*-*c*} Pyridines exhibit significant biological properties such as antioxidant, anticancer, anticoagulant, anti-inflammatory, anti-HIV, anti-malarial, anticonvulsant, vasodilator and antiepileptic activities.^{1*d*-*g*} In addition, some of the compounds that have pyridine core units are being used as therapeutic drugs (Fig. 1).²

On the other hand, some of the substituted pyridines are used as tuneable dye lasers, organic light-emitting diodes, and fluorescent chromophores and as ligands in coordination chemistry and aqua chemistry.³ Due to the interesting applications of pyridine derivatives, a large number of methods have been developed for their synthesis.⁴ Conventionally, these compounds are synthesized by a three-component cyclocondensation of acetophenones, benzaldehydes, and ammonium acetate in the presence of various catalysts such as AcOH,^{5a} ZrOCl₂,^{5b} TCT,^{5c} TiO₂–SO₃H,^{5d} Fe₃O₄@TiO₂@O₂PO₂(CH₂)₂NHSO₃H^{5e} etc.^{5f-h,6} However, most of the methods have some drawbacks, such as the usage of expensive transition metal catalysts, oxidants, unfriendly additives, poor yields, and harsh conditions. Recently, oxime acetates were proved to be versatile building blocks for the construction of various heterocyclic compounds, in particular triphenylpyridines.⁷ Thus, plenty of methods have been reported for the synthesis of tri-substituted pyridines from oxime acetates by the coupling of various precursors such as aldehydes, toluenes, benzyl amines, and *p*-toluene-sulfonylhydrazones using various transition metals as catalysts (Scheme 1a).^{8–12}

On the other hand, transition metal catalysed decarboxylative coupling of carboxylic acids,¹³ in particular, $C(sp^3)$ aryl acetic acids, with nucleophiles has recently emerged as a powerful tool for the synthesis of various heterocyclic compounds because of the readily available starting materials, nontoxic byproduct (CO₂), and easy handling. Moreover, aryl acetic acids are more stable than aldehydes.⁸ Despite the prevalence of the synthetic utility of the phenyl acetic acids in the synthesis of various heterocyclic compounds, methods that directly form

Fig. 1 Some of the bioactive compounds containing pyridine core units.



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Previous works



2,4,6-triphenyl pyridines have not been explored very well in the literature. However, Phan¹⁴ and co-workers have recently synthesized 2,4,6-triphenyl pyridines from phenyl oxime acetates by using strontium-doped lanthanum cobaltite as a recyclable pervoskite heterogeneous catalyst, and DTBP as an oxidant (Scheme 1b). However, this protocol has some drawbacks, such as the usage of less abundant, toxic and highly expensive catalysts and oxidants. Therefore, the development of an efficient and ecofriendly protocol for the synthesis of tri-substituted pyridine derivatives from oxime acetates and phenyl acetic acids is highly desirable. In continuation of our research work on the development of new synthetic methodologies for heterocyclic compounds, herein, we describe a simple and efficient method for 2,4,6-tri-substituted pyridines from oxime acetates and $C(sp^3)$ aryl acetic acids in the presence of copper chloride as a catalyst, and NaHSO3 as a base in DMF at 150 °C for 2 h under an oxygen atmosphere (Scheme 1c).

Results and discussion

Initially, we started our investigation by taking oxime acetate (1a) and phenyl acetic acid (2a) as model substrates to optimize the reaction conditions. Thus, in our first attempt, the reaction was performed with CuI (10 mol%), and NaHSO₃ (0.5 equiv.) in DMF (2 mL) at 150 °C for 2 h under an oxygen atmosphere. To our delight, the desired 2,4,6-triphenyl pyridine (3aa) was formed in 40% yield (Table 1, entry 1). Other copper catalysts including CuBr, CuCl, CuCl₂ and Cu(OAC)₂ were screened and CuCl was found to be the most effective catalyst for this

Table 1 Optimization of the conditions^a

| | N ^{OAc} | соон | | | | Ph |
|-------|------------------|---------|---|--------------------|----------------|--------------------------------|
| + | | | Catalyst Base, Oxidant T °C, Solvent, 2 h | | ĺ | |
| | |] | | | | |
| | | | | | | 0 |
| 1a 2a | | | , , | | | заа |
| Entry | Catalyst (mol%) | Oxidant | Base | Solvent | $T(^{\circ}C)$ | $\operatorname{Yield}^{b}(\%)$ |
| 1 | CuI (10) | O_2 | NaHSO ₃ | DMF | 150 | 40 |
| 2 | CuBr (10) | O_2 | NaHSO ₃ | DMF | 150 | 58 |
| 3 | CuCl (10) | O_2 | NaHSO ₃ | DMF | 150 | 65 |
| 4 | $CuCl_2$ (10) | O_2 | NaHSO ₃ | DMF | 150 | 25 |
| 5 | $Cu(OAc)_2$ (10) | O_2 | NaHSO ₃ | DMF | 150 | 32 |
| 6 | CuCl (10) | O_2 | Cs_2CO_3 | DMF | 150 | 30 |
| 7 | CuCl (10) | O_2 | Na_2CO_3 | DMF | 150 | 35 |
| 8 | CuCl (10) | O_2 | K_2CO_3 | DMF | 150 | 32 |
| 9 | CuCl (10) | O_2 | Et ₃ N | DMF | 150 | 40 |
| 10 | CuCl (10) | DTBP | $NaHSO_3$ | DMF | 150 | 50^c |
| 11 | CuCl (10) | TBHP | NaHSO ₃ | DMF | 150 | NR^{d} |
| 12 | CuCl (10) | PIDA | NaHSO ₃ | DMF | 150 | NR^d |
| 13 | CuCl (10) | Air | NaHSO ₃ | DMF | 150 | 15^e |
| 14 | CuCl (10) | N_2 | NaHSO ₃ | DMF | 150 | Trace ^e |
| 15 | CuCl (10) | Ar | NaHSO ₃ | DMF | 150 | Trace ^e |
| 16 | CuCl (10) | O_2 | NaHSO ₃ | CH ₃ CN | 150 | NR |
| 17 | CuCl (10) | O_2 | NaHSO ₃ | DCE | 150 | NR |
| 18 | CuCl (10) | O_2 | NaHSO ₃ | DMSO | 150 | 35 |
| 19 | CuCl (10) | O_2 | NaHSO ₃ | Dioxane | 150 | NR |
| 20 | CuCl (10) | O_2 | NaHSO ₃ | Toluene | 150 | NR |
| 21 | CuCl (10) | O_2 | NaHSO ₃ | DMF | 120 | 40 |
| 22 | CuCl (10) | O_2 | NaHSO ₃ | DMF | 160 | 52 |
| 23 | CuCl (20) | O_2 | NaHSO ₃ | DMF | 150 | 90 |
| 24 | CuCl (30) | O_2 | NaHSO ₃ | DMF | 150 | 86 |
| 25 | CuCl (20) | O_2 | _ | DMF | 150 | NR |
| 26 | | O_2 | $NaHSO_3$ | DMF | 150 | NR |

^{*a*} Reaction conditions: **1a** (0.2 mmol), **2a** (0.2 mmol), catalyst (20 mol%), base (0.5 equiv.), oxidant (1.0 equiv.), solvent (2.0 mL), 150 °C, under an oxygen atmosphere, 2 h. ^{*b*} Isolated yield. NR = no reaction. ^{*c*} For 6 h. ^{*d*} For 8 h. ^{*e*} 12 h.

reaction (Table 1, entries 2-5). Subsequently, several bases such as Cs₂CO₃, Na₂CO₃, K₂CO₃ and Et₃N were explored, which showed that NaHSO3 was a suitable base and other bases were inferior (Table 1, entries 6-9). Screening of a series of oxidants revealed that the oxidants such as DTBP, TBHP and PIDA disfavored the transformation, and molecular oxygen was the best choice (Table 1, entries 10-12). Moreover, only 15% of the desired product was formed after 12 h (Table 1, entry 13), when we carried out the reaction under an air atmosphere. This might be due to the formation of acetophenone as a byproduct via the hydrolysis of oxime acetate. We also performed the reaction under nitrogen and argon atmospheres without adding an oxidizing agent, but, unsatisfactory results were found in both cases (Table 1, entries 14 and 15). Therefore, an oxidizing agent is very important in this reaction to get the product in high yields, and the oxidizing agent could accelerate the reaction by the oxidation of phenyl acetic acid to benzaldehyde in the presence of a copper catalyst. Replacing DMF with other solvents, such as DMSO, CH3CN, DCE, dioxane or toluene, no significant improvement in the yield of the desired product was observed (Table 1, entries 16-20). Then, we tested the temperature; the yield of 3a dropped to 40% when the reaction temperature was reduced to 120 °C (Table 1, entry 21). Next, we focused on the loading of the catalyst; fortunately, the yield of **3a** was remarkably increased to 90%, when the catalyst loading was increased to 20 mol%. However, no significant improvement in the yield of **3a** was observed at 30 mol% of the catalyst (Table 1, entries 23 and 24).

No product was observed in the absence of either catalyst or a base, which suggests that a metal/base combination is required for the reaction to proceed (Table 1, entries 25 and 26).

After having the optimized reaction conditions in our hands. the scope and limitations of the reaction were investigated. As shown in Scheme 2, various oxime acetates (1) smoothly reacted with phenylacetic acid (2a), and provided the corresponding products (3aa-3ai) in excellent yields under optimal reaction conditions. Unsubstituted oxime acetate reacted with phenylacetic acid (2a) and gave the corresponding product in 90% yield (3aa). Oxime acetates having electron-donating groups such as 4-methyl and 4-methoxy groups on the aromatic ring smoothly reacted with phenylacetic acid 2a and afforded the respective pyridines in 92%, and 94% yields, respectively (3ab and 3ac). Oxime acetates having electron-withdrawing groups such as 4-fluoro, 4-chloro and 2,4-dichloro could also be converted into the desired products in good yields (3ad-3af). However, 4-nitro substituted oxime acetate could not serve as a viable substrate, as it could not provide the intended product. It might have occurred because of the strong electronic effect of the nitro group. It is worth mentioning that aliphatic ketoxime acetate such as isobutyl oxime acetate also reacted smoothly and gave the desired product in good yields (3ah). Moreover, hetero-aromatic ketoxime acetate, *i.e.* furan ketoxime acetate, could efficiently get transferred to the desired product (3ai).



Encouraged by the above results, we employed a series of phenyl acetic acids for further extension of the substrate scope (Scheme 3). To our delight, a wide variety of commercially available phenyl acetic acids having electron-donating or electron-withdrawing groups at different positions were accommodated and gave the corresponding pyridines in moderate to high yields. Phenyl acetic acids **2b** and **2c** bearing halogen groups on the C-4 or C-2 positions could react with various oxime acetates and gave the products in good yields. Notably, phenyl acetic acid **2d** having a strong electron-withdrawing group like nitro on the C-4 position could proceed to provide the corresponding pyridine in 65% yield. Moreover, phenylacetic acid **2e** with an electron-donating group was also proven to be a suitable substrate for this transformation, affording the corresponding product in 67–88% yields.

To probe the reaction mechanism, several control experiments have been performed (Scheme 4). Since the reaction involves oxygen, radical trapping experiments were conducted by treating TEMPO or BHT with phenyl acetic acid and oxime acetate under the optimal conditions. The results showed that the reaction was completely suppressed in the presence of either TEMPO or BHT (Scheme 4, eqn (1)). Based on previous reports9 and our control experiments (Scheme 4, eqn (2)), it could be concluded that aldehyde is the key intermediate in this reaction and it was



Scheme 2 Substrate scope of various oxime acetates. Reaction conditions: 1 (0.2 mmol), 2a (0.2 mmol), CuCl (20 mol%), and NaHSO₃ (0.5 equiv.) in DMF (2.0 mL) at 150 °C, under an oxygen atmosphere, 2–4 h. Isolated yields.

Scheme 3 Substrate scope of various oxime acetates and phenyl acetic acids. Reaction conditions: 1 (0.2 mmol), 2 (0.2 mmol), CuCl (20 mol%), NaHSO₃ (0.5 equiv.) in DMF (2.0 mL) at 150 °C, under an oxygen atmosphere, 2–4 h. Isolated yields.





formed from phenyl acetic acid *via* oxidative decarboxylation. Moreover, as shown in (Scheme 4, eqn (3)), when benzaldehyde was subjected to the reaction with oxime acetate under the optimized conditions, a 94% yield of the desirable product **3aa** was obtained.

Based on the above results and previous literature,^{15–17} a plausible mechanism of the reaction is illustrated in Scheme 5. First, the reductive cleavage of the N–O bond of **1a** is initiated by copper salts to generate an imino-copper(π) complex **A** that undergoes tautomerization to afford copper enamide intermediate **B**. On the other hand, benzaldehyde (**C**) is formed from phenylacetic acid *via* oxidation in the presence of CuCl and NaHSO₃ under an oxygen atmosphere by loss of CO₂ as a byproduct.¹⁷ Subsequently, nucleophillic addition of **B** to aldehyde **C** gives the imine intermediate **D**. Subsequently, imine intermediate **D** reacts with another molecule of oxime acetate **1a** and generates intermediate **E** by the loss of ammonium acetate. Then, beta elimination of intermediate **E** gives intermediate **F**. Finally, **F** undergoes electro cyclization followed by oxidation to form 2,4,6-tri-substituted pyridines.

Conclusion

In conclusion a simple and efficient protocol has been disclosed for the synthesis of 2,4,6-triphenyl pyridines *via* the

decorboxylative coupling of $C(sp^3)$ arylacetic acids with oxime acetates in the presence of CuCl as a catalyst in DMF at 150 °C under an oxygen atmosphere. In this reaction various oxime acetates and phenylacetic acids are well tolerated, and they provided the 2,4,6-triphenyl pyridines in moderate to good yields. This method not only used easily available starting substrates but also avoided the usage of expensive ligands and oxidants.

Conflicts of interest

There are no conflicts to declare.

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