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Pd-Catalyzed Tandem Cyclization via C−H Arylation and Acylation for the Construction of Polycyclic Scaffolds

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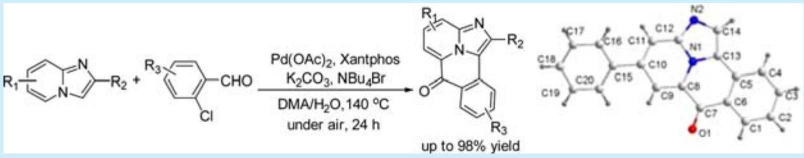
S *Supporting Information*

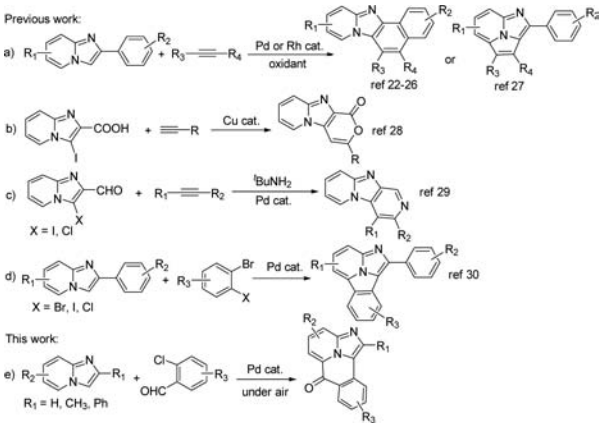


ABSTRACT: The first Pd-catalyzed tandem cyclization of imidazo[1,2-*a*]pyridines with 2-chlorobenzaldehydes through C−H arylation and acylation is presented for the efficient synthesis of novel 6*H*-benzo[*b*]imidazo[5,1,2-*de*]quinolizin-6-ones. The direct acylation reaction proceeded smoothly without the aid of directing groups and in the presence of air as a clean and free terminal oxidant.

Imidazo[1,2-*a*]pyridine is an important motif found in many natural products and pharmaceuticals.[1] These derivatives exhibit a wide range of biological activities such as antiulcer,[2] antiinflammatory,[3] antiprotozoal,[4] anticancer,[5] antibacterial,[6] antiviral,[7] and antifungal activities.[8] Moreover, drug formulations containing the imidazo[1,2-*a*]pyridine moiety include alpidem,[9] zolpidem,[9] olprinone,[10] zolimidine,[11] GSK812397,[12] necopidem, and saripidem[13] currently available on the market. In addition, imidazo[1,2-*a*]pyridines are also applied in N-heterocyclic carbene chemistry[14] and materials chemistry.[15] In view of their importance, a variety of synthetic approaches including condensation,[16] oxidative coupling,[17] C−H arylation,[18] and decarboxylative coupling[19,20] have been developed for the construction of imidazo[1,2-*a*]pyridine derivatives with a variety of substituents at the 2- and 3-positions of this moiety in the past decade.[1]

Recently, transition-metal-catalyzed dehydrogenative annulation of simple substrates has emerged as one of the most versatile and powerful synthetic strategies to construct polycyclic aromatic and heteroaromatic compounds.[21] In this regard, fused N-heterocycles naphtho[1′,2′:4,5]imidazo[1,2-*a*]pyridine and imidazo[5,1,2-*cd*]indolizine were synthesized via Pd/Rh-catalyzed annulation of imidazo[1,2-*a*]pyridines with alkynes by the groups of Li,[22] Cheng,[23] Song,[24] Fan,[25] Lee,[26] and Hajra[27] respectively in 2015 (Scheme 1a). In 2011, Abarbri[28] reported the synthesis of pyrano[3′,4′:4,5]imidazo[1,2-*a*]pyridin-1-ones via Cu(I)-mediated cross-coupling and heterocyclization reaction of 3-iodoimidazo[1,2-*a*]pyridine-2-carboxylic acid with terminal alkynes (Scheme 1b). In 2005, Chavignon[29] described a palladium-catalyzed annulation of 3-haloimidazo[1,2-*a*]pyridine-2-carbaldehyde with ^{t}BuNH$_2$ and alkynes, providing dipyrido[1,2-*a*;3′,4′-*d*]imidazoles (Scheme 1c). In 2015, Peng[30] developed the construction of benzo[*a*]imidazo[5,1,2-*cd*]indolizine derivatives from 2-arylimidazo[1,2-*a*]pyridines and *o*-dihaloarenes (Scheme 1d). Despite the significance, the present methods for the synthesis of polycyclic scaffolds containing the imidazo[1,2-*a*]pyridine moiety were restricted to the use of presynthesized imidazo[1,2-*a*]pyridine derivatives and diarylalkynes or *o*-dihaloarenes as starting materials. Therefore, the development of structurally diverse, polycyclic imidazo[1,2-*a*]pyridines from easily available starting materials are still highly desired.

Scheme 1. Dehydrogenative Annulation Strategies for Polycyclic Scaffolds Containing the Imidazo[1,2-*a*]pyridine Moiety



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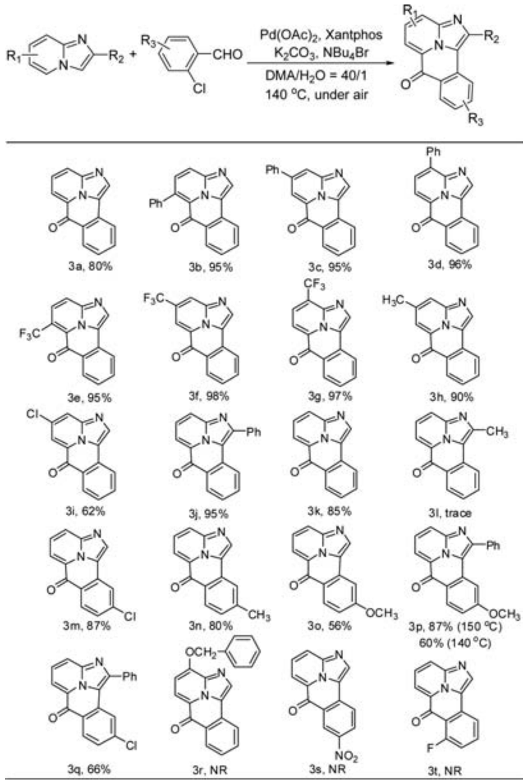
In addition, diaryl ketones are also attractive synthetic targets due to their wide applications in the fields of natural products, pharmaceuticals, agrochemicals, and functional materials.[31] The classical route to synthesize aryl ketones is Friedel−Crafts acylation of aromatic compounds.[32] However, harsh reaction conditions, limited aromatic substrates, and poor regioselectivity limit the scope of this reaction.[33] Direct acylation of aromatic sp^2 C−H bonds in proximity to various directing groups with aldehydes by transition-metal-catalyzed C−H bond activation have been successfully exploited.[34] In recent years, Li,[35] Panda,[36] and Li[37] independently reported the direct acylation of arenes with aldehydes by Rh/Ru/Cu-catalyzed C−H bond activation using TBHP or O_2 as the oxidant without the aid of directing groups. To our knowledge, Pd-catalyzed acylation of arenes with aldehydes by C−H bond activation without the aid of directing groups has not been explored.

Very recently, we have developed a convenient protocol for the synthesis of 3-aryl-imidazo[1,2-*a*]pyridines via palladium-catalyzed decarboxylative coupling under air.[20] In continuation of our interest in the construction of diverse imidazo[1,2-*a*]pyridines, herein, we attempt to develop an efficient Pd-catalyzed two-component dehydrogenative arylation and acylation strategy to construct novel polycyclic 6*H*-benzo[*b*]-imidazo[5,1,2-*de*]quinolizin-6-ones from easily available imidazo[1,2-*a*]pyridines and 2-chlorobenzaldehydes using air as the ideal oxidant (Scheme 1e). To the best of our knowledge, it is the first example of the synthesis of 6*H*-benzo[*b*]imidazo-[5,1,2-*de*]quinolizin-6-ones, and the first successful examples of Pd-catalyzed acylation of arenes with aldehydes by C−H bond activation without the aid of directing groups in the presence of air as the terminal oxidant.

At the outset of our studies, we explored the effect of different reaction parameters on the dehydrogenative annulation of imidazo[1,2-*a*]pyridine **1a** with 2-chlorobenzaldehyde **2a**, which included palladium catalysts, ligands, bases, solvents, additives, and temperature (Table S1; see Supporting Information (SI)). Palladium-catalyzed C−H arylation of imidazo[1,2-*a*]pyridines with aryl chlorides has generally been performed under basic and nonoxidative conditions,[38] but transition-metal-catalyzed acylation of arenes with aldehydes proceeded using an external oxidant.[35−37] Therefore, a suitable external oxidant is required to mediate dehydrogenative annulation between arylation and acylation in our designed route. Accordingly, the model reaction was performed in the presence of 5 mol % of $Pd(OAc)_2$, 10 mol % of S-Phos and 3 equiv of K_2CO_3 in 4 mL of DMF/H_2O (40/1) under air as the oxidant at 150 °C for 24 h, and the desired product was obtained in 60% LC yield (entry 1). Some traditional surfactants were selected, and NBu_4Br was found to be more effective than the other additives in this reaction to afford 77% LC yield (entries 2−5). Other bases such as Na_2CO_3, K_3PO_4, $NaHCO_3$, and Cs_2CO_3 were also investigated, but they could only give lower yields (entries 6−9). Then, the replacement of $Pd(OAc)_2$ with $Pd(CF_3CO_2)_2$, $PdCl_2$, $Pd(acac)_2$, $Pd_2(dba)_3$, or $(C_3H_5)_2Pd_2Cl_2$ leads to a drop in yield (entries 10−14). A variety of ligands such as $BuAd_2P$, PCy_3, $PBu_3{\cdot}HBF_4$, Xant-Phos, and X-Phos were also screened (Figure S1; see SI), and obviously Xantphos was identified as the best ligand in this reaction according 83% LC yield (entries 15−19). Furthermore, the temperature also affected the rate of the reaction. It was indicated that 140 °C was the best condition for this annulation (entries 20, 21). Finally, the effects of different solvents were tested, and no improvement of the yield was observed (entries 22−27). Obviously, DMA/H_2O (40/1) as the solvent is the best choice, indicating that the addition of the suitable amount of H_2O could effectively promote the reaction.

With the optimized catalytic system in hand, we next explored the scope of imidazo[1,2-*a*]pyridines (Scheme 2). As expected, a series of functional groups at C2-, C6-, C7-, or C8-substituted imidazo[1,2-*a*]pyridines, such as CF_3, Ph, CH_3, and Cl, were compatible under this transformation, and the corresponding products **3a**−**3j** were isolated in moderate to good yields. Furthermore, the structures of products **3c** and **3d** were unambiguously determined by X-ray crystallographic analysis (see SI). When 6-chloroimidazo[1,2-*a*]pyridine was employed, the product **3k** were obtained in 85% yield instead of the expected 5-chloro-6*H*-benzo[*b*]imidazo[5,1,2-*de*]quinolizin-6-one, which actually proceeded through the hydrodechlorination. However, when 2-methylimidazo[1,2-*a*]pyridine was introduced into the reaction system, only a trace amount of the product **3l** was detected by LC-MS analysis. Subsequently, the scope of 2-chlorobenzaldehydes was also explored, and the results are also summarized in Scheme 2. 2-Chlorobenzaldehydes such as 2,4-dichlorobenzaldehyde, 2-chloro-4-methylbenzaldehyde, and 2-chloro-4-methoxybenzaldehyde were also employed in the annulation reaction to give the products **3m**−**3o** in 87%, 80%, and 56% yields, respectively. To further extend the scope of this reaction, the dehydrogenative annulation of 2-phenylimidazo-[1,2-*a*]pyridine with 2-chloro-4-methoxybenzaldehyde or 2,4-dichlorobenzaldehyde could afford the corresponding products

Scheme 2. Dehydrogenative Annulation of Imidazo[1,2-*a*]pyridines with 2-Chlorobenzaldehydes[a]



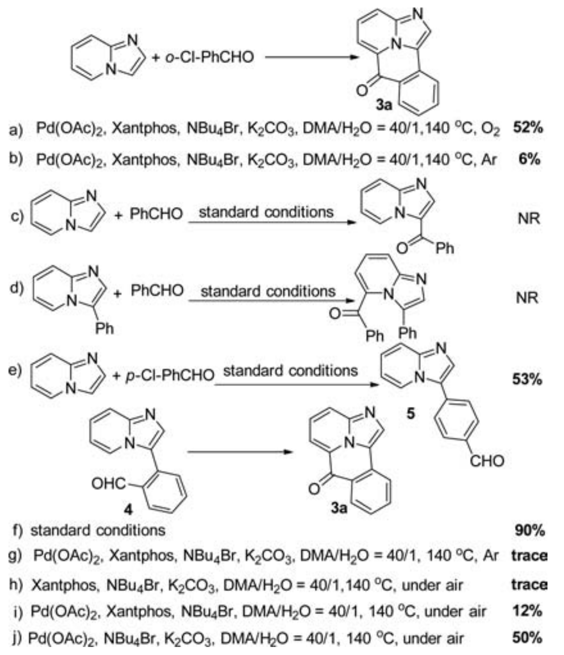
[a] Reaction conditions: 0.3 mmol of imidazo[1,2-*a*]pyridines, 0.9 mmol of 2-chlorobenzaldehydes, 0.9 mmol of K_2CO_3, 5 mol % of $Pd(OAc)_2$, 10 mol % of Xantphos, 0.9 mmol of NBu_4Br, 4 mL of DMA, and 0.1 mL of H_2O at 140 °C under air for 24 h. Isolated yields.

3p and **3q** in 60% and 66% yields, respectively. When the annulation of 2-phenylimidazo[1,2-*a*]pyridine with 2-chloro-4-methoxybenzaldehyde was performed at a higher temperature of 150 °C, the yield increased to 87% yield (product **3p**). Unfortunately, the corresponding products **3r**−**3t** were not detected when imidazo[1,2-*a*]pyridine bearing strong electron-donating group or 2-chlorobenzaldehydes with strong electron-withdrawing groups were used in the reactions, such as 8-(benzyloxy)imidazo[1,2-*a*]pyridine, 2-chloro-5-nitrobenzaldehyde, and 2-chloro-6-fluorobenzaldehyde. Finally, when another similar substrate such as imidazo[1,2-*a*]pyrimidine was introduced into the reaction system, the corresponding product was not detected by LC-MS analysis.

In addition, some preliminary investigations on fluorescence properties of the target products **3a**, **3g**, **3h**, and **3i** were carried out in $CHCl_3$. The fluorescence emission spectra were measured under excitation at 460 nm (Figures S2−S5; see SI). They emitted fluorescence at about 500 nm.

To gain insight into the mechanism, some controlling experiments were carried out and are presented in Scheme 3.

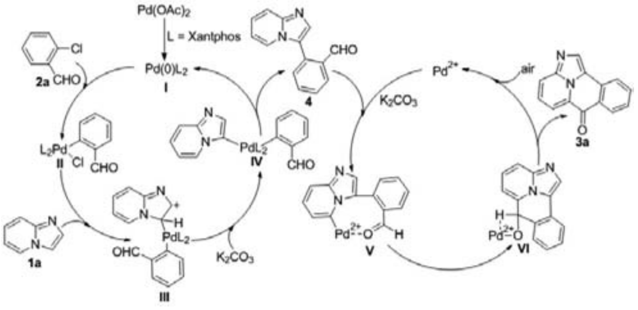
Scheme 3. Comparative Experiments under Different Reaction Conditions



The yield of product **3a** decreased significantly when the reaction was performed under an O_2 atmosphere, and only a trace amount of product **3a** was observed under an argon atmosphere, thus suggesting that air as the oxidant is essential for this Pd-catalyzed two-component reaction (Scheme 3a and 3b). Then, the reactions of imidazo[1,2-*a*]pyridine or 3-phenylimidazo[1,2-*a*]pyridine with benzaldehyde were carried out under the standard conditions, but the acylation reaction did not take place (Scheme 3c and 3d). Imidazo[1,2-*a*]pyridine and 4-chlorobenzaldehyde afforded the corresponding arylation product **5** in 53% yield under the standard conditions (Scheme 3e). When the treatment of the presynthesized 2-(imidazo[1,2-*a*]pyridin-3-yl)benzaldehyde **4** was performed under the standard conditions, the product **3a** was obtained in 90% isolated yield (Scheme 3f). This result showed that compound **4** may be an intermediate in this transformation. In the following experiments, the presynthesized 2-(imidazo[1,2-*a*]pyridin-3-yl)-benzaldehyde **4** was reacted under an argon atmosphere or in the absence of $Pd(OAc)_2$, and only a trace amount of product **3a** was observed (Scheme 3g and 3h). In contrast, the annulation product **3a** was obtained in 12% LC yield without K_2CO_3, and in an LC yield of 50% without Xantphos (Scheme 3i and 3j). The results indirectly indicated that the catalytic amount of $Pd(OAc)_2$, air as the oxidant, and K_2CO_3 as the base may play a crucial role in the dehydrogenative acylation. These observations suggested that imidazo[1,2-*a*]pyridines and 2-chlorobenzaldehydes transformed to 6*H*-benzo[*b*]imidazo-[5,1,2-*de*]quinolizin-6-ones probably through C−H arylation and the sequential intramolecular C−H oxidation/acylation pathway.

On the basis of the above-mentioned experimental results and previous literature reports,[38,39] a possible mechanism of Pd-catalyzed tandem cyclization by using imidazo[1,2-*a*]pyridine **1a** and 2-chlorobenzaldehyde **2a** as an example is outlined in Scheme 4. The first step is the release of Pd(0) species from $Pd(OAc)_2$ to form the catalytically active $Pd(0)L_2$ **I** with the assistance of the phosphine ligand. Then, the oxidative addition of **2a** to $Pd(0)L_2$ takes place to form the intermediate **II**. Subsequently, it involves an electrophilic attack by the intermediate **II** on **1a** to generate the intermediate **III**. The deprotonation of intermediate **III** with K_2CO_3 takes place to give intermediate **IV**, which then undergoes reductive elimination to afford the arylation product **4** and regenerates the catalytically active catalyst $Pd(0)L_2$ to fulfill the catalytic cycle. Thereafter, the reaction may proceed through the palladation of the arylation product **4** with the aid of K_2CO_3 to form the intermediate **V**, and subsequent insertion of the C═O bond of the aldehyde into the C−Pd bond of the intermediate **V** produces intermediate **VI**. Finally, the *β*-H elimination of the intermediate **VI** leads to the product **3a** and a Pd(0) species, which is oxidized by air to regenerate the Pd(II) catalyst.

Scheme 4. Proposed Mechanism of Palladium-Catalyzed Dehydrogenative Annulation of Imidazo[1,2-*a*]Pyridine with 2-Chlorobenzaldehyde



In summary, we first demonstrated the Pd-catalyzed tandem dehydrogenative annulation of imidazo[1,2-*a*]pyridines with 2-chlorobenzaldehydes in aqueous medium under air for the efficient synthesis of novel polycyclic 6*H*-benzo[*b*]imidazo-[5,1,2-*de*]quinolizin-6-ones. $Pd(OAc)_2$ could be used as a tandem catalyst to trigger C−H arylation and to promote the subsequent intramolecular C−H acylation/cyclization sequences. It is worth noting that the direct acylation reaction proceeded smoothly without the aid of directing groups using cheap 2-chlorobenzaldehydes as the starting materials and air as the terminal oxidant. Ongoing work seeks to gain further insights into the mechanism of this reaction and to expand the reaction scope of other fused heterocycles.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.6b02571.

> Experimental details, characterizations, NMR spectra, and X-ray crystallographic analysis of the products (PDF)

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Notes

The authors declare no competing financial interest.

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