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DOI: 10.1039/C8CC01226A

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Palladium-Catalyzed Aerobic Regio- and Stereo-Selective Olefination Reactions of Phenols and Acrylates via Direct Dehydrogenative C(sp^2)–O Cross-Coupling

Received 00th January 20xx,
Accepted 00th January 20xx

DOI: 10.1039/x0xx00000x

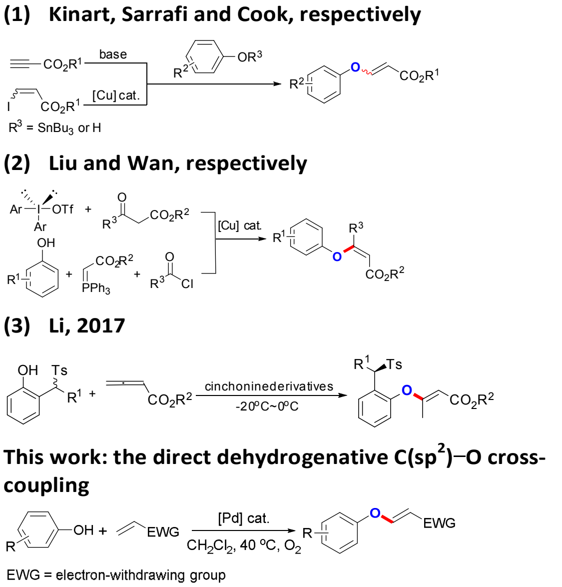
www.rsc.org/

Yun-Bin Wu,[a] Dan Xie,[a] Zhong-Lin Zang,[a] Cheng-He Zhou[a] and Gui-Xin Cai*[a,b]

An efficient olefination protocol for the oxidative dehydrogenation of phenols and acrylates has been achieved using palladium catalyst and O_2 as the sole oxidant. This reaction exhibits high regio- and stereo-selectivity (*E*-isomers) with moderate to excellent isolated yields and wide substrate scope (32 examples) including ethyl vinyl ketone and endofolliculina.

Aryl alkenyl ethers, represented by the phenoxy-acrylates, have been considered as versatile building blocks in organic and medicinal chemistry.[1] Several elegant examples for the synthesis of aryl alkenyl ethers have been reported over the last decade (Scheme 1). Kinart reported the addition reactions of organotin phenoxides and ethyl propiolate[2a] and the similar catalytic system could be used for the activation of phenols.[2b] Copper (I)-catalyzed cross coupling reaction of (*E*)- or (*Z*)-3-iodoacrylates with phenols has been pioneered by Cook.[2c,2d] Liu and Wan respectively extended the substrate scope to diaryliodonium salts[2e] and the multi-component reaction[2f] involving phenols and Wittig reagents. Li also detailed an asymmetric addition reaction of substituted phenols with allenes catalyzed by the cinchonine derivatives.[2g]

Although the chemical community has made fantastic progress in this field, current methods still possess some limitations such as stereoisomers, the lack of atom and step economy as well as the harsh reaction conditions and fairly toxic substrates. Thus it is a topic of crucial importance to explore more green and efficient approaches to constructing the scaffold of aryl alkenyl ether. C–H functionalization strategies, to be versatile and efficient, play an important role in organic chemistry.[3] Direct functionalization of inactive C–H bonds to construct new C–C or C-heteroatom (C–N, C–O, C–P) bonds has become one of the most concise and straightforward methods due to its atom- and step-economy, and this logic has underpinned the process of synthetic planning and applications.[4] Compared to the developed dehydrogenation couplings concerning phenols, the direct olefination of phenols faces the following pre-existing challenges: (1) the addition reaction of phenols and alkenes;[5] (2) the metal-catalyzed directed *ortho, meta* or *para*-selective C–H functionalization of phenols;[6] (3) the homo-coupling of phenols under an aerobic system.[7] In brief, the direct dehydrogenation of phenols and electron-deficient alkenes for the highly efficient synthesis of aryl alkenyl ethers through C–H bond activation of acrylates is quite rare, except for the intramolecular process for benzofurans[6b] and the alkoxylation of α,β-unsaturated carbonyl compounds using alcohols[4m] for the synthesis of enol ethers. Thus, we herein report a palladium-catalyzed regio- and *E*-selective direct dehydrogenative C(sp^2)–O cross-coupling reaction of phenols and acrylates using O_2 as the sole oxidant under mild conditions.



Scheme 1. Synthetic strategy

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[a] Key Laboratory of Applied Chemistry of Chongqing Municipality, Institute of Bioorganic & Medicinal Chemistry, School of Chemistry and Chemical Engineering, Southwest University, Chongqing 400715, China.
[b] Beijing National Laboratory for Molecular Sciences, Beijing 100190, China.
E-mail of corresponding author: gxcai@swu.edu.cn.
†Electronic Supplementary Information (ESI) available: [details of any supplementary information available should be included here]. See DOI: 10.1039/x0xx00000x

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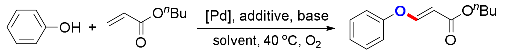
On the basis of our previous work,[8] we attempted to develop a synthetic protocol for the formation of vinyl C−O bond via direct cross-dehydrogenation coupling (CDC)[9]. Our investigation started with phenol (**1a**) and *n*-butyl acrylate (**2a**) as the model substrates to optimize the reaction parameters and the results are summarized in Table 1. When the reaction was treated using $Pd(OAc)_2$ catalyst under an O_2 atmosphere in CH_2Cl_2 at 40 °C, **3aa** was isolated in 12% yield (Table 1, entry 1). Several bases as well as $KHCO_3$ were then tested (Table 1, entries 2-7) and K_2CO_3 was chose as the optimal candidate for the following optimization. Various additives were further screened to boost the transformation. Fortunately, the product **3aa** was obtained with moderate yield in the presence of TBAC (tetrabutylammonium chloride) (Table 1, entry 8). Other tetrabutylammonium-based additives, such as TBAI (tetrabutyl-ammonium iodide) and TBAB (tetrabutylammonium bromide), were also screened and experimental results showed that TBAI was inferior in promoting this reaction, whereas TBAB showed a positive and cheerful result in 81% isolated yield (Table 1, entries 9-10). The dosage of TBAB was investigated and the results indicated that 3 equiv. of TBAB was optimum (see SI). Meanwhile, it is noteworthy that the existence of iodide ions would enormously inhibit this reaction (Table 1, entries 11-13).[10] Surprisingly, the omission of K_2CO_3 caused the significantly diminished yield, which might indicate a synergistic effect of TBAB and K_2CO_3 (Table 1, entry 14). Other additives with bromide anions were evaluated and those results showed weaker reactivity (Table 1, entries 15-17). Furthermore, commonly used oxidants in the palladium catalyst system, such as $CuBr_2$, $Cu(OAc)_2$ and $PhI(OAc)_2$ were tested (Table 1, entries 18-20). However, none of them displayed positive effects on this transformation. Compared to the results of air and Ar atmosphere (Table 1, entries 21-22), molecular oxygen (O_2) was confirmed that it had a central role in the regeneration of palladium catalyst.[11] In the absence of palladium catalyst totally restrained the reaction, which indicated its core role in the catalytic system (Table 1, entry 23). Other palladium catalysts such as $PdBr_2$, $PdCl_2$, $Pd(PPh_3)_2Cl_2$, $Pd(dppf)_2Cl_2$ and $Pd_2(dba)_3$ were also evaluated (Table 1, entries 24-28), and $Pd(OAc)_2$ was superior to them. When other solvents were used instead of CH_2Cl_2, the desired product was obtained in moderate yields and therefore CH_2Cl_2 was the optimum solvent (Table 1, entries 29-33). Finally, we treated the reaction at room temperature (25 °C), but the result was undesired (Table 1, entry 34).

With optimized conditions in hand, phenols bearing various substituents on the phenyl ring were tested (Table 2). Due to the effect of steric hindrance, 2-substituted phenols afforded the corresponding products in poor to moderate yields (**3ba**-**3fa**). No desired product **3ca** was observed in the presence of 2-methoxyphenol, which meant that palladium catalyst might be inactive under this condition because of 2-methoxyphenol as a bidentate ligand coordinating to palladium. With regard to 3- or 4-substituted phenols, electron-rich phenols generally gave better yields of the desired products than that of their electron-deficient counterparts (**3ga**-**3ta**). In addition, multi-substituted groups, owing to the steric effect, led to corresponding products in lower yields (**3ua** and **3va**). Naphthols were also compatible under standard conditions and the performance of naphthalen-2-ol was better than that of naphthalen-1-ol (**3wa** and **3xa**). To our surprise, the reaction did occur in the presence of benzylalcohol, affording **3ya** in 12% isolated yield. However, the success of this transformation couldn't be extended to pyridin-4-ol and quinolin-4-ol et al. due to the strong coordination effect of heterocyclic phenols on Pd catalyst.[6f]

Table 1. Optimization of the reaction conditions[a]



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

Entry	Catalyst	Additive/ Oxidant	Base	Solvent	**3aa** (%)[b]
1	$Pd(OAc)_2$	-	-	CH_2Cl_2	12
2	$Pd(OAc)_2$	-	NEt_3	CH_2Cl_2	14
3	$Pd(OAc)_2$	-	DABCO	CH_2Cl_2	32
4	$Pd(OAc)_2$	-	Cs_2CO_3	CH_2Cl_2	35
5	$Pd(OAc)_2$	-	Na_2CO_3	CH_2Cl_2	24
6	$Pd(OAc)_2$	-	K_2CO_3	CH_2Cl_2	37
7	$Pd(OAc)_2$	-	$KHCO_3$	CH_2Cl_2	28
8	$Pd(OAc)_2$	TBAC[d]	K_2CO_3	CH_2Cl_2	51
9	$Pd(OAc)_2$	TBAI[e]	K_2CO_3	CH_2Cl_2	trace
10	**$Pd(OAc)_2$**	**TBAB[f]**	**K_2CO_3**	**CH_2Cl_2**	**81**
11	$Pd(OAc)_2$	TBAB[g]	K_2CO_3	CH_2Cl_2	trace
12	$Pd(OAc)_2$	KI	K_2CO_3	CH_2Cl_2	19
13	$Pd(OAc)_2$	TBAB[h]	K_2CO_3	CH_2Cl_2	20
14	$Pd(OAc)_2$	TBAB	-	CH_2Cl_2	20
15	$Pd(OAc)_2$	LiBr	K_2CO_3	CH_2Cl_2	trace
16	$Pd(OAc)_2$	NaBr	K_2CO_3	CH_2Cl_2	7
17	$Pd(OAc)_2$	KBr	K_2CO_3	CH_2Cl_2	22
18[i]	$Pd(OAc)_2$	$CuBr_2$	K_2CO_3	CH_2Cl_2	trace
19[i]	$Pd(OAc)_2$	$Cu(OAc)_2$	K_2CO_3	CH_2Cl_2	trace
20[i]	$Pd(OAc)_2$	$PhI(OAc)_2$	K_2CO_3	CH_2Cl_2	NP[c]
21[i]	$Pd(OAc)_2$	TBAB	K_2CO_3	CH_2Cl_2	71
22[j]	$Pd(OAc)_2$	TBAB	K_2CO_3	CH_2Cl_2	29
23	-	TBAB	K_2CO_3	CH_2Cl_2	NP[c]
24	$PdBr_2$	TBAB	K_2CO_3	CH_2Cl_2	32
25	$PdCl_2$	TBAB	K_2CO_3	CH_2Cl_2	34
26	$Pd(PPh_3)_2Cl_2$	TBAB	K_2CO_3	CH_2Cl_2	8
27	$Pd(dppf)_2Cl_2$	TBAB	K_2CO_3	CH_2Cl_2	13
28	$Pd_2(dba)_3$	TBAB	K_2CO_3	CH_2Cl_2	35
29	$Pd(OAc)_2$	TBAB	K_2CO_3	DCE	41
30	$Pd(OAc)_2$	TBAB	K_2CO_3	$CHCl_3$	78
31	$Pd(OAc)_2$	TBAB	K_2CO_3	THF	36
32	$Pd(OAc)_2$	TBAB	K_2CO_3	CH_3CN	42
33	$Pd(OAc)_2$	TBAB	K_2CO_3	dioxane	53
34[k]	$Pd(OAc)_2$	TBAB	K_2CO_3	CH_2Cl_2	50

[a] Reaction conditions: phenol **1a** (0.4 mmol), *n*-butyl acrylate **2a** (0.2 mmol), catalyst (0.02 mmol), additive or oxidant (3 equiv.), base (0.06 mmol), solvent (1 mL) in a schlenk tube under O_2 balloon at 40 °C for 36 h. [b] Isolated yields. [c] NP = no desired product. [d] TBAC = tetrabutylammonium chloride. [e] TBAI = tetrabutylammonium iodide. [f] TBAB = tetrabutylammonium bromide. [g] Extra TBAI (3 equiv.) was added. [h] Extra KI (3 equiv.) was added. [i] Under air. [j] Under Ar balloon. [k] RT.

The scope of various substituents on the acrylate partner was then explored (Table 3). Satisfyingly, a range of functional groups were compatible, including *n*-butyl, methyl, ethyl, *tert*-butyl, benzyl, *n*-hexyl, cyclohexyl, trifluoroethyl, 2-methoxy-ethyl, (tetrahydrofuran-2-yl)methyl and phenyl groups, to afford the corresponding products (**3aa**-**3ak**) in modest to excellent yields (up to 84% isolated yield). While methyl at α-

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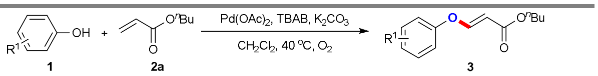
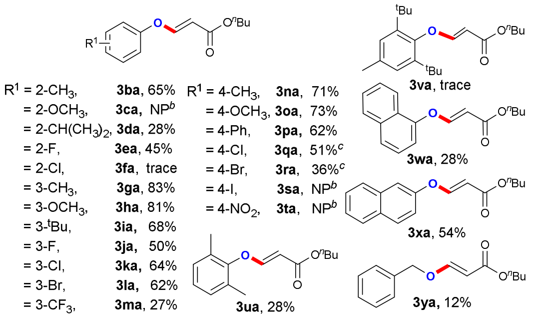
DOI: 10.1039/C8CC01226A

or β-position of ethyl acrylate respectively gave a low conversion under the standard conditions, which meant that the steric hindrance had obvious inhibitory effect on this transformation (**3al** and **3am**). Fortunately, the treatment with pent-1-en-3-one also proceeded well to obtain the desired product (**3an**) in 83% isolated yield, whereas electron-rich alkenes such as styrene showed a poor performance and gave the trace amount of product (**3ao**).

Table 2. Reactions of various phenols and *n*-butyl acrylate[a]

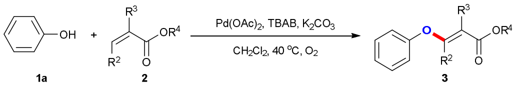


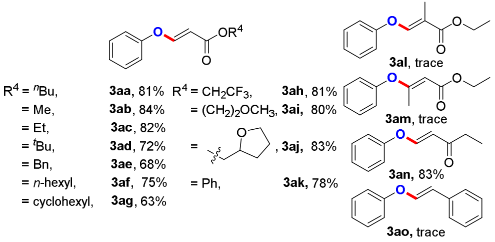


[a] Reaction conditions: phenols **1** (0.4 mmol), *n*-butyl acrylate **2a** (0.2 mmol), $Pd(OAc)_2$ (0.02 mmol), TBAB (3 equiv.), K_2CO_3 (0.06 mmol), CH_2Cl_2 (1 mL) in a schlenk tube under O_2 balloon at 40 °C reflux for 36 h with isolated yields. [b] NP = no desired product. [c] TBAB (5 equiv.).

We further had insights into the mechanism by control experiments. In many cases, the reactions of phenols involve free radicals,[12] while the results of our experiments using TEMPO (2,2,6,6-tetramethylpiperidine-1-oxyl) or ascorbic acid could exclude the free radicals[13] in this transformation (Scheme 2). We also found that **3ab** could not be obtained coming from methyl 3-phenoxypropanoate, which indicated this reaction not involved the process of the addition reaction of acrylate followed by α,β-dehydrogenation elimination of methyl 3-phenoxypropanoate. A plausible reaction mechanism was depicted in Scheme 3. First, the reaction was initiated by the coordination of $Pd(OAc)_2$ to *n*-butyl acrylate 2a to give a palladium-olefin complex **I**,[14] which underwent nucleophilic attack by phenol **1a** to generate an intermediate **II**.[15] Subsequent deprotonation and *syn* β-hydride elimination from the intermediate **II** assisted by base resulted in butyl (*E*)-3-phenoxyacrylate **3aa** exclusively and released a $HPd^{II}X$ complex. Then under the effect of base, the Pd^{II}-hydride complex experienced reductive elimination to afford a Pd^0 species, which could be oxidized by O_2 as a sole oxidant to regenerate the Pd^{II} catalyst.[11] The excellent regio- and stereo-selectivity observed in this transformation might be attributed to the favorable formation of the intermediate **II**. In addition, the role of excess TBAB (3 equiv.) in the reaction system was to avoid the deactivation of the Pd^{II} catalyst arising from the strong coordination by phenol, and therefore promoting the catalytic cycle (see SI).[16]

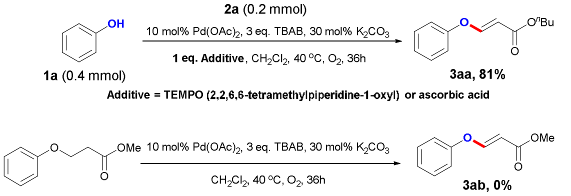
Table 3. Reactions of phenol and various acrylates[a]



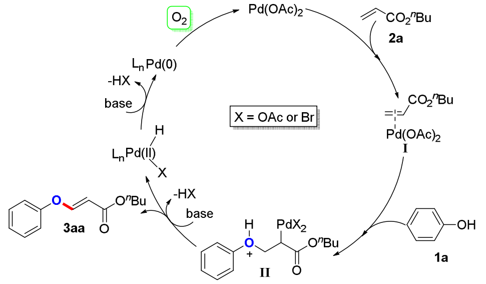


[a] Reaction conditions: phenol **1a** (0.4 mmol), acrylates **2** (0.2 mmol), $Pd(OAc)_2$ (0.02 mmol), TBAB (3 equiv.), K_2CO_3 (0.06 mmol), CH_2Cl_2 (1 mL) in a schlenk tube under O_2 balloon at 40 °C reflux for 36 h with isolated yields.

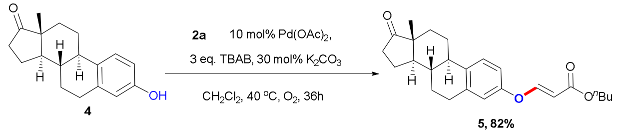
Furthermore, to demonstrate the synthetic application of our approach, we investigated the transformation of natural product (8*S*,9*R*,13*R*,14*R*)-3-hydroxy-13-methyl-6,7,8,9,11,12,13,14,15,16-decahydro-17*H*-cyclopenta[*a*]phenanthren-17-one **4** with *n*-butyl acrylate **2a** to prepare a novel functionalized molecule and gave the corresponding target product **5** in 82% isolated yield.



Scheme 2. Control experimental results



Scheme 3. Plausible mechanism



Scheme 4. The application of our approach

Conclusions

DOI: 10.1039/C8CC01226A

In conclusion, we have developed an efficient and mild Pd-catalyzed direct dehydrogenation C(sp^2)–O cross-coupling reaction of phenols and acrylates under oxygen atmosphere, providing a highly regio- and stereo-selective approach to synthesize (*E*)-alkenyl aryl ether compounds. A wide variety of products could be synthesized in moderate to excellent yields. And the optimized catalytic system can be used for functionalization of ethyl vinyl ketone and endofolliculina to afford the desired products with high stereo-selectivity and excellent isolated yields.

We are grateful to the National Natural Science Foundation of China (Grants 21372186 and 21672173), Beijing National Laboratory of Molecular Sciences (BNLMS) (20140130), the Doctoral Fund of Southwest University (SWU111075), and the Research Funds for the Central Universities (XDJK 2017B015) for support of this research.

Conflicts of interest

There are no conflicts to declare.

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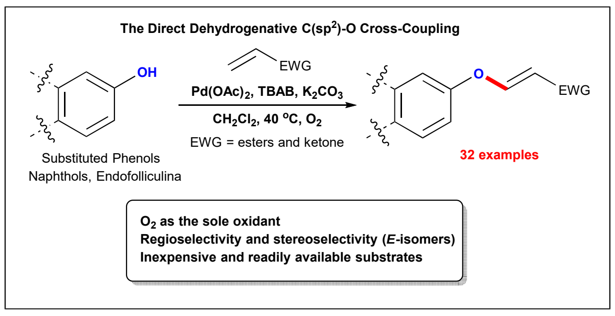

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DOI: 10.1039/C8CC01226A