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A palladium-catalyzed approach to allenic aromatic ethers and first total synthesis of terricollene A†

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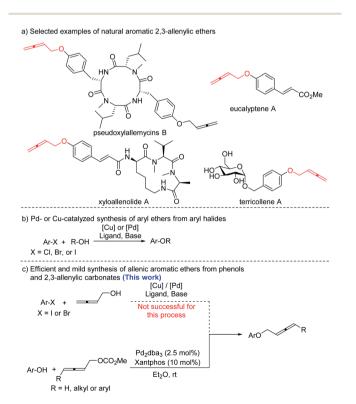
A palladium-catalyzed C-O bond formation reaction between phenols and allenylic carbonates to give 2,3-allenic aromatic ethers with decent to excellent yields under mild reaction conditions has been described. A variety of synthetically useful functional groups are tolerated and the synthetic utility of this method has been demonstrated through a series of transformations of the allene moiety. By applying this reaction as the key step, the total syntheses of naturally occurring allenic aromatic ethers, eucalyptene and terricollene A (first synthesis; 4.5 q gram scale), have been accomplished.

Introduction

Aromatic ethers are prevalent and prominent in a variety of natural products, pharmaceuticals, and agrochemicals.1 Traditional methods for the preparation of aromatic ethers include the Williamson ether synthesis,2 direct nucleophilic substitution reactions,3 Mitsunobu reaction,4 Ullman-type couplings of alkoxides with aryl halides,5 etc. On the other hand, due to their unique chemical properties as well as the substituent-loading capability derived from the distinctive structure, allenes have been demonstrated as powerful platform molecules for the efficient syntheses of other functional organic compounds.^{6,7} Interestingly, many aromatic 2,3-allenylic ethers are also found in the nature (Scheme 1a).8 Reports on the syntheses of such aromatic ethers are very limited. Thus, development of new methods for the efficient synthesis of aromatic 2,3-allenylic ethers is of high interest currently. Herein, we disclose the development of palladium-catalyzed C-O bond formation between aryl phenols and allenylic carbonates to give allenic aromatic ethers in 70-99% yields under mild reaction conditions (Scheme 1c).

Results and discussion

In contrast to the well-established metal-catalyzed coupling reactions between aryl halides and phenols or aliphatic alcohols (Scheme 1b), metal-catalyzed coupling reactions of aryl halides



Scheme 1 Naturally occurring aromatic 2,3-butadienyl ethers and the metal-catalyzed C-O bond formation for the synthesis of aromatic ethers.

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Table 1 Optimization of the reaction conditions for Pd-catalyzed reaction of p-methoxyphenol 1a with 2,3-butadienyl carbonate $2a^a$

Entry	[Pd]	Ligand	Solvent	NMR yield of 3aa (%)	Recovery of 1a (%)
1	[pdCl(= allyl)]	BINAP	Toluene	39	48
1	$[PdCl(\pi-allyl)]_2$				
2	Pd_2dba_3	BINAP	Toluene	68	26
3	$Pd(PPh_3)_4$	BINAP	Toluene	43	49
4	Pd_2dba_3	Xantphos	Toluene	80	10
5	Pd₂dba₃	DPEPhos	Toluene	28	70
6	Pd₂dba₃	BIPHEP	Toluene	10	89
7	Pd₂dba₃	SPhos	Toluene	68	13
8	Pd₂dba₃	Xantphos	MTBE	96	_
9	Pd₂dba₃	Xantphos	$\mathrm{Et_2O}$	99 (95^b)	_
10	Pd ₂ dba ₃	Xantphos	Dioxane	67	28
11	Pd_2dba_3	Xantphos	THF	49	47

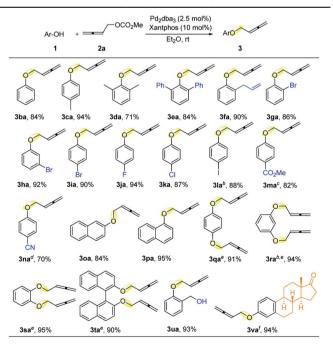
^a Yield and recovery were determined by 1 H NMR analysis using $CH_{3}NO_{2}$ as the internal standard. ^b The reaction was carried out with 1 mmol of 1a and the isolated yield is shown in parentheses.

and allenols have not been developed (Scheme 1c). In a preliminary survey, the palladium-10 and copper-catalyzed11 coupling reactions between 4-iodoanisole and allenols were studied; however, only a trace amount of the desired allenyl aryl ether product was obtained (Scheme 1c, for details, see the ESI‡). Then 4-methoxyphenol 1a was treated with 1.5 equiv. of 2,3-butadienyl carbonate 2a in the presence of 2.5 mol% $[PdCl(\pi-allyl)]_2$ and 10 mol% BINAP with toluene as the solvent at room temperature for 12 h. Interestingly, 39% yield of the expected allenol ether product 3aa was formed with 48% recovery of 4-methoxyphenol 1a. Various palladium catalysts and ligands were then screened (Table 1, entries 1-7). The combination of Pd2dba3 and Xantphos as the catalyst delivered the best result, affording 80% yield of 3aa with 10% recovery of 1a (Table 1, entry 4). Subsequent solvent screening (Table 1, entries 8-11) showed that Et₂O or MTBE was the best solvent, affording the product 3aa in 99% or 96% yield, respectively (entries 8 and 9).

With the optimized conditions in hand, the scope of phenols was evaluated (Table 2). To our delight, parent phenol **1b** and electron-rich *p*-cresol **1c** gave high yields of **3ba** and **3ca**; even the reaction of highly sterically hindered 2,6-dimethyl or 2,6-diphenyl substituted phenols proceeded smoothly to afford **3da** or **3ea** in 71% or 84% yield, respectively. The *ortho*-allyl substituent also survived, allowing further modification based on the reactivity of the C–C double bond. Bromo-substituents at the *ortho*- (**3ga**), *meta*- (**3ha**), and *para*-positions (**3ia**) were all well tolerated without much difference, indicating that the steric effect is negligible and the relatively reactive C–Br bonds were tolerated. In addition, other synthetically useful halogen substituents such as F, Cl, and even I were all compatible, delivering the corresponding products **3ja**, **3ka**, and **3la** in 94%, 87%, and 88% yields, respectively. It is worth noting that the

highly reactive C–I bond is generally less compatible in palladium-catalyzed reactions. ¹² We reasoned that the relatively strong coordination effect of the allene unit brought the palladium catalyst to the carbonate, so that the oxidative addition of

Table 2 The substrate scope: phenols or naphthols^a



^a Reaction conditions: **1** (1.0 mmol), **2a** (1.5 mmol), Pd_2dba_3 (2.5 mol%), and Xantphos (10 mol%) in Et_2O (5 mL) at rt; isolated yields are shown. ^b In MTBE at 35 °C. ^c With 5 mol% $Pd(PPh_3)_4$ in dioxane at 35 °C. ^d With 5 mol% $Pd(PPh_3)_4$ in MTBE at 40 °C. ^e With 2.5 mmol of **2a**. ^f In MTBE at 60 °C in a sealed tube.

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the palladium catalyst occurred exclusively to the carbonate unit, leaving the C-Br/I bond intact.¹³ Furthermore, phenols with electron-withdrawing and synthetically versatile substituents such as CO2Me and CN could also deliver the corresponding products 3ma and 3na in good to excellent yields upon changing the catalyst to Pd(PPh₃)₄. Moreover, 1- or 2naphthols could also afford the corresponding products 30a or 3pa in 84% and 95% yield, respectively. With 2.5 equiv. of allene 2a, the coupling reactions of hydroquinone, resorcinol, catechol, and 1,1'-binaphthol gave the double-coupling products 3qa-3ta in excellent yields. Interestingly, only the phenol hydroxy group was exclusively coupled leaving the benzylic hydroxyl group untouched to afford 3ua. Such a coupling reaction of the hydroxyl group in estrone was also realized by changing the solvent to MTBE for a reaction at 60 °C to afford 3va in an excellent yield.

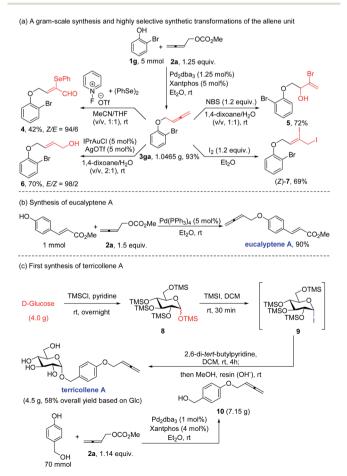
We then evaluated the scope of 2,3-allenylic carbonates (Table 3): carbonates with 4-alkyl and isopropyl substituents delivered the coupling products 3bb, 3pc, and 3pd in decent to excellent yields. Benzyl ether, which is a common and practical protecting group for the hydroxyl group, was also viable in this transformation affording 3oe in 96% yield. 4-Phenyl-2,3butadienyl carbonate 1f reacted with phenol to afford 3bf in an almost quantitative yield. Moreover, electron-donating groups such as p-methyl and p-methoxy and electron withdrawing yet potentially useful p-Cl, p-Br, 13 p-I, 13 p-CF₃, p-NO₂, and p-CN were all accommodated yielding the corresponding products 3pg-3pn in decent yields. Last but not the least, 4-

Table 3 The substrate scope: 2,3-allenylic carbonates^a

heteroaryl substituents such as a furyl and a thienyl group in allenylic carbonates 2 were also successfully tolerated affording corresponding products 3bo and 3pp in excellent yields.

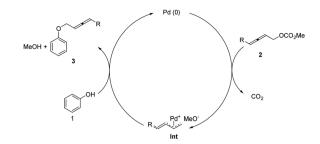
A gram-scale reaction between 1g and 2a delivered 1.0465 g of 3ga in 93% yield with a reduced loading of both the catalyst and ligand (Scheme 2a). As a class of synthetically useful chemicals, the versatile allenyl unit could be transferred into different structure units: selenohydroxylation and oxidation of 3ga with 1-fluoropyridinium/1,2-diphenyldiselane could deliver 2-selenoenal 4 with a Z/E selectivity of 94/6;14 bromohydroxylation of this allenyl group in aqueous dioxane afforded terminal 2-bromoallylic alcohol 5 as the sole product in 72% yield;15 the allenyl group may also be selectively transformed into the corresponding primary allylic alcohol 6 with an E/Z selectivity of 98/2 by a gold-catalyzed hydration reaction;16 iodination of the allenyl group exclusively gave diiodide product (Z)-7 in 69% yield. 17 It should be noted that these products are difficult to synthesize through traditional transition-metal catalyzed coupling reactions. 10,18

Finally this method has been applied to the efficient syntheses of two naturally occurring allenes: eucalyptene A has been successfully synthesized under the catalysis of 5 mol% Pd(PPh₃)₄ in 90% yield from methyl 4-hydroxycinnamate and 2,3-butadienyl methyl carbonate (Scheme 2b); 70 mmol scale



Scheme 2 Synthetic applications

^a Reaction conditions: 1 (1.0 mmol), 2 (1.5 mmol), Pd₂dba₃ (2.5 mol%), and Xantphos (10 mol%) in Et₂O (5 mL) at rt; isolated yield.



Scheme 3 Proposed mechanism

reaction of *p*-hydroxymethylphenol with 1.14 equiv. of 2,3-butadienyl methyl carbonate 2a afforded the corresponding *p*-hydroxymethylphenyl 2,3-butadienyl ether 10. p-Glucose (4.0 g) was etherified with chlorotrimethylsilane in pyridine to give penta-*O*-trimethylsilyl-p-glucopyranose 8, which was then reacted with iodotrimethylsilane to generate the glucosyl iodide 9 within 30 min at room temperature. The reaction of allene 10 (7.15 g) with 9 in the presence of 2,6-di-*tert*-butylpyridine in dry dichloromethane at room temperature for 4 h followed by treatment with methanol and resin(OH⁻) to remove the silyl protecting groups afforded 4.5 g of terricollene A in 58% overall yield as the sole anomer, which is the first total synthesis of this natural product (Scheme 2c).¹⁹

A plausible mechanism is proposed (Scheme 3): the oxidative addition of Pd(0) with 2,3-allenylic carbonate would yield, after releasing CO₂, an α -methylene- π -allylic Pd intermediate Int, ^{20,13c,d} which would subsequently undergo a nucleophilic allenylation reaction with phenol to deliver the allenic aromatic ether 3 with regeneration of the catalytically active Pd(0) to finish the catalytic cycle.

Conclusions

In conclusion, we have described a general method of palladium-catalyzed C–O bond formation reaction between phenols and allenylic carbonates. This first example of such a C–O bond formation reaction exhibits a broad scope of both phenols and carbonates under very mild conditions, providing an attractive complement to traditional C–O bond formation reactions. The attractive reactivities of the allene unit in the products have been demonstrated. Moreover, the present catalytic system offers a convenient route for the efficient synthesis of naturally occurring aromatic 2,3-allenylic ethers, eucalyptene A and terricollene A, which exhibited cytotoxic activity against KB/KBv200 cells and HeLa/MCF-7 cells, respectively. Further studies are currently under way in our laboratories.

Data availability

The electronic supplementary information include experimental detail, NMR data, MS data, IR data, elemental analysis data, and all the spectra.

Author contributions

C. H. and S. M. conceived the idea. C. H. conducted the most of experiments. B. Y. supervised the total synthesis of terricollene A. C. H. and F. S. co-synthesized the terricollene A. Y. C., C. L., J. L., Q. L., A. Q., H. W., G. W., P. W., J. X., H. X., Y. Y., Y. Z., W. Z., and Y. Z. co-synthesized part of substrates and they contribute equally to this paper. C. H. and S. M. co-wrote the paper. All the authors discussed the results and commented on the manuscript.

Conflicts of interest

There are no conflicts to declare.

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