

Bronsted Acid/Organic Photoredox Cooperative Catalysis: Easy Access to Tri- and Tetrasubstituted Alkenylphosphorus Compounds from Alcohols and P–H Species

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Supporting Information

ABSTRACT: A Bronsted acid/organic photoredox cooperative catalytic system toward P–C bond formation from alcohols and P–H species is developed. With the assistance of visible light and TBHP, the reactions proceeded smoothly in an environmentally benign manner to give various alkenylphosphorus compounds in high efficiency.

A lkenylphosphorus compounds, due to their manifold applications in organic synthesis,¹ distribution in material science,² and biological activity,³ have received increasing attention in the past few years. Therefore, tremendous effort has been directed toward the development of efficient methods to access this skeleton. In this context, transition-metalcatalyzed P–C bond formation reactions of P–H species with alkynes⁴ (Scheme 1a) or specifically functionalized alkenes⁵ (Scheme 1b) have been established as the dominant strategies. For example, the pioneering research of Han has realized the transition-metal-catalyzed direct phosphorylation of

Scheme 1. Strategies for the Preparation of Alkenylphosphorus Compounds

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a) Transition-metal-catalyzed addition of P-H species to alkynes

$$R \longrightarrow H^{-} R^{-} R^{1} \xrightarrow{(Rh, Pd etc.)} R^{-} P^{(O)R^{1}R^{2}}$$

b) Transition-metal-catalyzed cross-coupling strategies

$$Ar \xrightarrow{FG} + H \xrightarrow{R^2} R^1 \xrightarrow{(Pd, Cu, Ag \text{ etc.})}_{(FG = Br, COOH, NO_2 \text{ etc.})} Ar \xrightarrow{P(O)R^1R^2}$$

c) Transition-metal-catalyzed oxidative cross-coupling

 $Ar \xrightarrow{R} + H \xrightarrow{P_{-}R^{1}} \frac{cat. (Ag, Cu, Cu/Fe)}{oxidants, additives} \xrightarrow{R} P(O)R^{1}R^{2}$

d) Cooperative catalysis for P-C bond constriction (this work)



Metal-free
 Environmentally benign
 Novel catalytic system



alkynes^{4a} (Scheme 1a). The need to use additives,^{4a} excess base,^{5c} and well-defined ligands⁴ in addition to problems of stereo- and regioselectivity control encourages further research in this area. Considering the easy accessibility of alkenes, transition-metal-catalyzed reactions of alkenes and P–H species have also been reported.^{6,7} However, addition products rather than oxidative cross-coupling was predominant, irrespective of the transition-metal promoters⁶ or radical initiators.⁷ Until now, only terminal styrenes were identified as suitable partners with H-phosphonates/diarylphosphine oxides in the transitionmetal-catalyzed oxidative cross-coupling⁸ (Scheme 1c). Although much progress has been made in this area, problems associated with narrow substrate scope, the need to use expensive transition metals, a stoichiometric amount of additives, and poor regio- and stereocontrol have encouraged researchers to search for new and more efficient methods.

We envisaged that the reaction of the P–H species using the cooperative catalysis⁹ of Bronsted acid and photocatalysts under environmentally friendly conditions with readily available feedstock such as alcohols¹⁰ will provide an attractive and practical approach to access alkenylphosphorus compounds. Although photocatalysis has been selected as promoting a myriad of synthetic transformations with a range of applications in a green manner,¹¹ to the best of our knowledge, no suitable organic photoredox catalysis has been developed toward alkenylphosphorus compounds from alcohols. Furthermore, organic photoredox catalysis¹² might be compatible with Bronsted acids due to its acidic nature. In this paper, we report an efficient and environmentally friendly method for the construction of P–C bonds via a Bronsted acid/organic

Received: April 19, 2018

photoredox cooperative catalysis reaction between alcohols and P–H species.¹³ This metal-free system was carried out under green solvent (2-propanol) to yield *t*-BuOH and water as the only byproducts will provide an effective method for the synthesis of alkenylphosphorus compounds (Scheme 1d).

We initiated our investigation by reacting 1,1-diphenylethanol with diphenylphosphine oxide in the presence of TsOH (20 mol %) and rhodamine B (RhBH⁺) (2 mol %) (Supporting Information (SI), Figure 1) under air with the expectation of obtaining (2,2-diphenylvinyl)phosphine oxides 3a. To our delight, the desired 3a, together with addition product 3a' was generated in 11% yield with good selectivity (SI Table 1, entry 1). Oxidants were then screened with the intention of improving the overall performance of the reaction (SI Table 1, entries 2–5). TBHP (2-hydroperoxy-2-methylpropane) gave the best result associated with yield and selectivity between 3a and 3a' (SI, entry 5). Other Bronsted acids, such as benzoic acid, trifluoroacetic acid (TFA), and HNTf₂, delivered the product in lower yield and poor selectivity (SI Table 1, entries 6-8). The organic photoredox catalysts (SI Figure 1) also have great influence on the reaction, and no positive results were obtained when Eosin Y or Eosin B was employed (SI Table 1, entries 9-11). The effect of solvent was also investigated (SI Table 1, entries 12-17). Acetonitrile, dichloromethane, DMF, toluene, as well as other alcohols disfavored this reaction, affording 3a in lower yield and poor selectivity. The cooperative catalytic system is crucial for this transformation. Either TsOH or RhBH⁺ itself could not catalyze the reaction well (SI Table 1, entries 18 and 19). Decreasing the loading of TsOH resulted in a lower yield of the desired product (SI Table 1, entry 20). In contrast, a lower amount of RhBH⁺ also can give the product in good yield, but with a decline in selectivity (SI Table 1, entry 21). A temperature of 80 °C was identified as the best temperature to obtain the product in terms of both yield and selectivity (SI Table 1, entries 22 and 23).

Under the optimized conditions, the scope of this P–C bond construction reaction was then investigated. As shown in Scheme 2, the desired products 3 can be isolated in high yield with good to excellent selectivity. The position and electronic properties of the aryl substituents on the 1,1-diarylethanols have a limited effect on the overall performance. Unsymmetrical 1,1-diarylethanols also delivered the corresponding 3h in high yields with excellent chemoselectivity (3h/3h'), although the E/Z selectivity is poor. Importantly, tetrasubstituted alkenylphosphine oxides, which were a challenge to prepare due to steric hindrance, could be conveniently obtained by using our developed method (3i-k). Thus, we can safely come to the conclusion that this was a powerful complementary method for alkenylphosphorus compounds. The reaction also can be performed well on gram scale, and 3a can be obtained in a comparable result. Unfortunately, 1-alkyl-1-arylethanols such as 2-phenylpropan-2-ol were not suitable substrates for this reaction.

We also examined the feasibility of extending this reactivation to other kinds of dialkylphosphine oxides from diphenylphosphine oxide. To the best of our knowledge, the former P–H species has not been successfully incorporated into alkenylphosphorus compounds via oxidative cross-coupling. The results are summarized in Scheme 3. Herein, we found that our method could well tolerate diverse dialkylpshosphine oxides, irrespective of their chain length (3l,o) or cyclic alkyl substitutions (3j,k), affording the corresponding products in moderate to high yields with excellent selectivity. The scope of





^{*a*}Unless otherwise specified, reactions were carried out using **1** (0.2 mmol) and **2a** (0.6 mmol) in *i*-PrOH (2.0 mL) at 80 °C for 30 min. TBHP (2.0 equiv, 5.5 mol/L in decane) was then added. After 8 h, TBHP (2.0 equiv, 5.5 mol/L in decane) was added again. Isolated yields (**3** and **3**'). The ratio of **3**/**3**' and *E*/*Z* was determined by ³¹P NMR. All of the reactions were performed under white LED (23 W).

diarylphosphine oxides bearing various functional groups was also checked. The electronic effect has a limited effect on the yield and chemical selectivity (3p-u). Even the hindered diarylphosphine oxides proceed well with the generation of desired product in high yield, although the selectivity was lower in some cases (3s,t). Di-1-naphthylphosphine oxides and di-2naphthylphosphine oxides can be successfully incorporated in the reaction (3v,w). 6*H*-Dibenzo[c,e][1,2]oxaphosphinin-6-ol was a suitable substrate for this reaction, which generated 3x in moderate yield with high selectivity. Moreover, this protocol could also be used in the preparation of an enantiomerically pure *P*-stereogenic alkenylphosphorus skeleton (3y-aa)species from optically pure *H*-phosphinate. Only one isomer was isolated, which was proposed with the retention of configuration at the phosphorus (see the SI for details).¹⁴

With an attempt to gain more insight into the reaction mechanism, a series of control experiments were then conducted. As shown in Scheme 4, under identical reaction conditions, 3a cannot be generated from 3a' (Scheme 4a). This result indicates that 3a' was not the intermediate for generating 3a, and the reaction pathways for 3a and 3a' were competitive. Comparable results were obtained when alcohol 1a was replaced by the corresponding ethene-1,1-diyldibenzene (Scheme 4b, middle). Thus, the alkene should be the intermediate for generating alkenylphosphine oxides. In contrast, much lower yields and selectivities were observed when the reaction was conducted without TsOH (Scheme 4b,

Scheme 3. Substrate Scope for Phosphine Oxidants^a

	Rhodamine B (2 –R ² TsOH (20 m	2 mol%) Ph O 이%)	Ph O
Ph F	hv, TBHP (4.0	equiv) Ph F	R^{1} Ph R^{1} R ¹
1a 2	<i>i</i> -PrOH, 80 °C	C, N ₂ 3 (major	r) 3' (minor)
Ph O Ph Ph Ph n-Bu n-Bu	Ph O Ph P_{H} Ph R_{H} R = cyclopentyl	Ph O Ph Ph-Cy Cy	Ph O Ph – <i>i</i> -Pr <i>j</i> -Pr
3l/3l' > 99/1 40% yield	3m/3m' > 99/1 80% yield	3n/3n' > 99/1 62% yield	3o/3o' > 99/1 66% yield
Ph O Ph Ph Ar	Ph O Ph H-Ar Ar	Ph O Ph Ph-Ar Ar	Ph O Ph P-Ar Ar
$Ar = 4-MeC_6H_4$	$Ar = 4-MeOC_6H_4$	$Ar = 4-FC_6H_4$	$Ar = 3,5$ -di MeC_6H_3
3p/3p' = 85/15 98% yield	3q/3q' = 85/15 81% yield	3r/3r' = 95/5 67% yield	3s/3s' = 85/15 86% yield
Ph O Ph O Ph Ar	Ph O Ph H-Ar Ph Ar	Ph O Ph P-Ar Ar	Ph O Ph P-Ar Ar
$Ar = 4-t-BuC_6H_4$	$Ar = 2-MeOC_6H_4$	Ar = 2-naphthyl	Ar = 1-naphthyl
3t/3t' = 87/13 75% yield	3u/3u' = 95/5 86% yield	3v/3v' = 82/18 67% yield	3w/3w' = 95/5 96% yield
Ph O Ph O Ph O Ph O Ph O Ph O Ph O Ph O			
3x/3x' > 99/1 34% yield	Ar = Mes: 20%	3z/3z' > 99/1 3 a 6 vield	aa/3aa' > 99/1 25% yield

^{*a*}Unless otherwise specified, reactions were carried out using 1a (0.2 mmol) and 2 (0.6 mmol) in *i*-PrOH (2.0 mL) at 80 °C for 30 min, TBHP (2.0 equiv, 5.5 mol/L in decane) was then added. 8h later, TBHP (2.0 equiv, 5.5 mol/L in decane) was added again. Isolated yields (3 and 3'). The ratio of 3/3' was determined by ³¹P NMR. All of the reactions were performed under white LED (23 W).





top). These results indicate that TsOH not only promotes the initial dehydration process, but also influences the whole catalytic cycles. Oxides **3a** were obtained in a lower yield and poor selectivity when the reaction was performed at room temperature with TsOH (Scheme 4b, bottom). This result reveals that a higher temperature was required for the overall transformation, beyond dehydration. Under the optimized

conditions, no reaction occurred when 2.0 equiv of TEMPO was added (Scheme 4c). This outcome, to some extent, means the reaction proceeded via a radical process.

The (2,2-diarylvinyl)phosphines have been identified as key ligand skeletons in palladium-catalyzed aromatic amination reactions.¹⁵ However, transformation and tedious purification processes were generally required for their preparation. Theoretically, the (2,2-diarylvinyl)phosphines oxides prepared in this investigation can be used as key feedstocks for the aforementioned (2,2-diarylvinyl)phosphines. We selected alkenylphosphine oxides **3a** as the model substrate. As is shown in Scheme 5, the desired (2,2-diphenylvinyl)phosphines **4** can be conveniently obtained in excellent yield.





To explore the mechanism of the visible-light-mediated P-C bond construction, we conducted the Stern–Volmer fluorescence quenching experiments. A 581 nm fluorescence launched by rhodamine B was observed when the sample was excited at 571 nm. The fluorescence intensity dramatically decreased when diphenylphosphine oxide **2a** was added (see the SI for details). This investigation indicated the single-electron transfer between rhodamine B and **2a**. Based upon the aforementioned investigations, we proposed a plausible reaction pathway, as shown in Scheme 6. The reaction started with the

Scheme 6. Proposed Mechanism



single electron oxidation of diphenylphosphine oxide by photoexcited rhodamine B (PC^*) to give radical cation **A** and reduced rhodamine B ($PC^{\bullet-}$). The deprotonation occurred with the generation of phosphinoyl radical **B**, which was followed by the anti-Markovnikov addition to the in situ generated alkenes to deliver radical **D**. The $PC^{\bullet-}$ was quenched by TBHP to yield rhodamine B (PC), *t*-BuO[•], and OH⁻. OH⁻ might be captured immediately by TsOH with the generation of TsO⁻. In the presence of proton, *t*-BuO[•] reacted with radical **D** by SET to give the cation intermediate **E**. With the assistance of TsO⁻, the deprotonation process then occurred to give the final product **3**.

In summary, we have developed an efficient and practical method for the construction of the P-C bond under Bronsted acid/organic photoredox cooperative catalysis. A variety of

alkenylphosphine oxides could be obtained in moderate to high yields with good to excellent selectivities. It should be noted that the challenging tetrasubstituted alkenylphosphorus compounds could also be successfully accessed by using our developed method. This protocol proceeded in an environmentally benign manner, generating water and *tert*-butyl alcohol as the only byproducts. The Bronsted acid/organic photoredox cooperative catalysts systems will shed light on related cooperative catalyst systems designed toward oxidative cross-coupling reactions involving alcohols, even though the alcohols are currently limited to α , α -diaryl alcohols.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.or-glett.8b01173.

Experimental procedures, screening reaction conditions, and NMR spectra of products (PDF)

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Notes

The authors declare no competing financial interest.

ACKNOWLEDGMENTS

We greatly acknowledge financial support by the National Natural Science Foundation of China (21702108), the State Key Program of the National Natural Science Foundation of China (21432009), the Natural Science Foundation of Jiangsu Province, China (BK20160977), Nanjing Tech University, and the SCIAM Fellowship by the Jiangsu National Synergetic Innovation Center for Advanced Material.

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