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Pd-catalyzed enantioselective and regioselective asymmetric hydrophosphorylation and hydrophosphinylation of enynes

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ABSTRACT

The chemo-, regio-, and enantio-controlled synthesis of P-chiral phosphines in a general and efficient manner remains a significant synthetic challenge. In this study, a Pd-catalyzed hydrofunctionalization is developed for the highly selective synthesis of P-stereogenic alkenylphosphinates and alkenylphosphine oxides *via* conjugate addition of enynes. Notably, this methodology is suitable for both phosphine oxide and phosphinate nucleophiles, providing a versatile approach for the construction of diverse P-chiral organophosphosphorus compound.

Phosphorous compounds have numerous applications in medicinal chemistry and photoluminescent materials, as well as in asymmetric transformations as ligands or organocatalysts [1–4]. Many chiral ligands with P-chirality have been extensively used in asymmetric catalysis [5–9]. Notable examples include (*Rc*,*Sp*)-Duanphos, (*S*,*S*,*S*,*S*)-BIBOP, (*R*,*R*)-QuinoxP* and (*R*,*R*)-BenzP* (Scheme 1A). Indeed, P-stereogenic organophosphorus compounds often outperform their chiral backbone substituted counterparts in the terms of coordination efficiency and enantio-control ability. As a result, numerous elegant methods have emerged for the construction of P-stereogenic compounds in recent years. These methods involve asymmetric addition of phosphorous nucleophiles to unsaturated bond [10–17], asymmetric C-P coupling [18–29], asymmetric C–H activation [30–52], asymmetric allylation [53–57], and other novel methods [58–74].

Alkenylphosphine derivatives serve as valuable building blocks in organic synthesis that can be easily transformed to important chiral 1,2-bisphosphine derivatives through processes such as asymmetric Hayashi-Miyaura reaction [75], asymmetric conjugate hydrophosphination [76] or double asymmetric hydrogenation [77,78]. Despite their importance, general and efficient methods for the chiral preparation of alkenylphosphine derivatives are rather limited. In recent years, the catalytic asymmetric addition of phosphorous nucleophiles to alkynes has emerged as a pivotal process for constructing alkenylphosphine derivatives with P-chirality in an efficient manner. However, due to the challenging regioselectivity of phosphorous nucleophiles' addition to alkynes and the differing reaction properties of phosphorous nucleophiles, catalytic methodology leading to diverse P-stereogenic alkenylphosphine derivatives remains rare, thereby limiting their further application.

In 2002, Tanaka and Han developed a Pd-catalyzed addition of alkynes with (R_P)-menthyl-phenylphosphinate to give enantiomerically pure P-chiral alkenylphosphinates while retaining the configuration at phosphorus atom [79]. In 2020, our group achieved asymmetric hydrophosphorylation of alkynes using a Pd/(*R*,*R*)-QuinoxP* catalyst for the synthesis of P-stereogenic phosphinates. However, secondary phosphine oxides exhibited poor yield and enantioselectivity under the same conditions [80]. Simultaneously, Zhang utilized their Pd/Xiao-Phos to give *anti*-Markovnikov addition products through enantioselective hydrophosphinylation of alkynes with phosphine oxides (Scheme 1B, a) [81]. Ni-catalysis has also proven to be a highly efficient catalyst for facilitating P-H addition. In 2021, Zhang successfully applied a Ni-catalyst in the asymmetric hydrophosphination of alkynes using a one-pot two-step process, resulting in the formation of P-stereogenic phosphines [82]. More recently, Zhang also realized *anti*-Markovnikov hydrophosphination of unactivated alkynes through Ni^{II} catalysis with the addition of AgOTf (Scheme 1B, b) [83]. Besides, the same group reported the asymmetric addition of enynes with secondary

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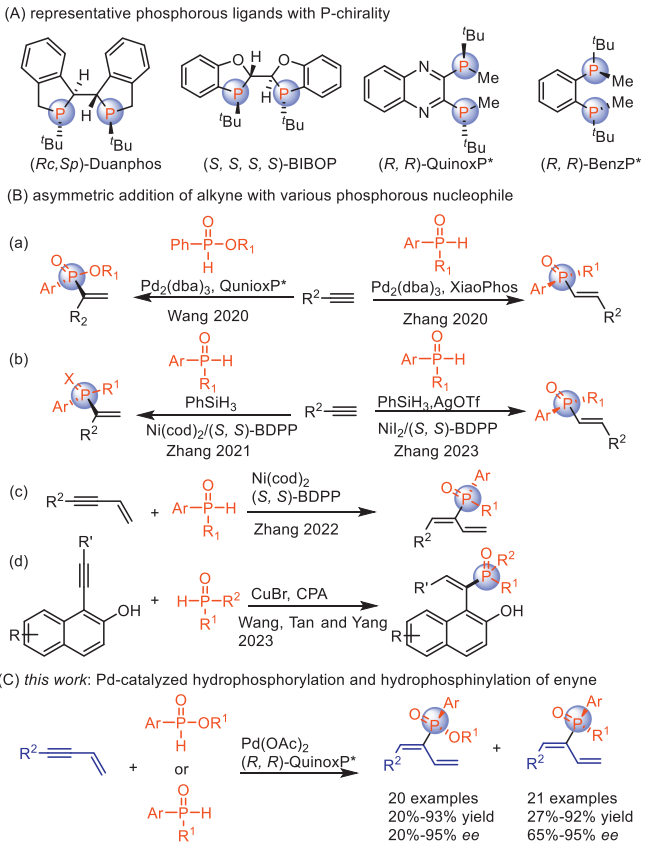
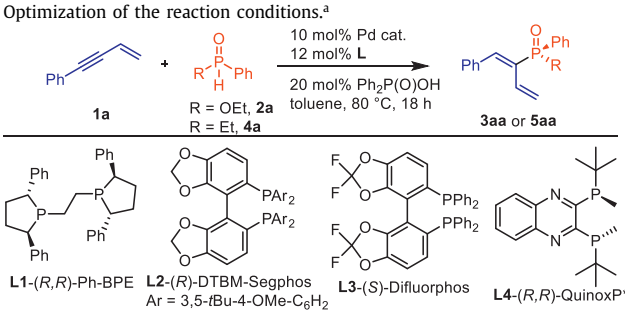


Scheme 1. Asymmetric addition of P-nucleophiles to alkynes.

phosphine oxides to give P-chiral alkenylphosphine oxides, where the alkene moiety served as a directing group by coordinating with the Ni catalyst (Scheme 1B, c) [84]. However, no examples using phosphinate as nucleophiles were provided. Recently, our group developed a Cu/CPA co-catalytic system for delivering axially chiral phosphorus-containing alkenes (Scheme 1B, d) [85]. Additionally, Duan has successfully developed a Ni-catalyzed asymmetric C(sp^2)-P coupling of secondary phosphine oxides with alkenyl bromides for generating P-stereogenic alkenylphosphine oxides with high yields and enantioselectivities [86]. The aforementioned examples are more suitable for a specific type of phosphorous nucleophile in a catalytic system, and other phosphorous nucleophiles often yield unsatisfactory results. Therefore, it is particularly desirable to develop a more general approach to realize asymmetric addition of various phosphorous nucleophiles.

Conjugated enynes are unique compounds that possess easily functionalized alkyne and alkene groups, allowing for modular synthesis through a 1,4-functionalization strategy to obtain chiral allenes instead of chiral 1,3-dienes [87–89]. Although Pd-catalyzed enantioselective hydrofunctionalization of conjugated enynes with N-H [90,91] or C-H [92–94] nucleophile, mediated by the *in situ* formation of catalytically active palladium(II)-hydride species, has been successful in delivering chiral allenes with good yields and enantioselectivities, the asymmetric construction of C-P bonds for the synthesis of chiral allenes or 1,3-dienes remains unexplored [95]. In recent years, our group has been dedicated to investigating the asymmetric addition of phosphorus nucleophiles to unsaturated bonds, including heterobicyclic alkenes [96], alkynes [80,85,97], allenes [98], cyclopropanes [99], alkenyl isoquinolines [100] and methylenecyclopropanes [101]. In this study, we present the catalytic enantioselective hydrophosphorylation and hydrophosphinylation of conjugated enynes using phosphinates or secondary phosphine oxides (SPO) in a Pd-catalyzed system (Scheme 1C). This methodology offers a highly efficient and versatile approach for the synthesis of P-chiral alkenylphosphinates and alkenylphosphine oxides with excellent yields and high enantioselectivities.

Table 1
Optimization of the reaction conditions.[a]

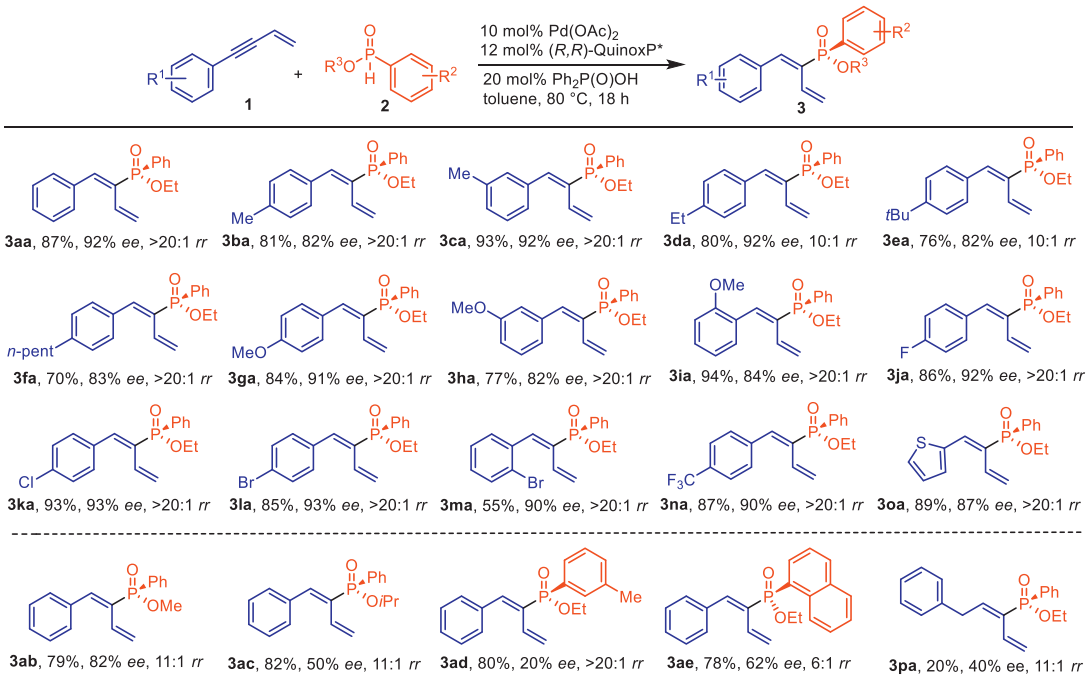


Entry	Pd	Ligand	T (°C)	Yield (%)	*ee* (%)	*rr*
1	$Pd(OAc)_2$	**L1**	80	ND	–	> 20:1
2	$Pd(OAc)_2$	**L2**	80	80	5	> 20:1
3	$Pd(OAc)_2$	**L3**	80	84	5	> 20:1
4	$Pd(OAc)_2$	**L4**	80	87	92	> 20:1
5[b]	$Pd(OAc)_2$	**L4**	80	19	92	> 20:1
6	$Pd(dba)_2$	**L4**	80	87	85	> 20:1
7	$Pd_2(dba)_3$	**L4**	80	80	90	> 20:1
8	$Pd(Piv)_2$	**L4**	80	83	67	> 20:1
9	$Pd(OAc)_2$	**L4**	70	23	92	> 20:1
10[c]	$Pd(OAc)_2$	**L4**	80	96	88	> 20:1
11[d]	$Pd(OAc)_2$	**L4**	80	90	89	> 20:1
12[e]	$Pd(OAc)_2$	**L4**	80	92	87	> 20:1

[a] Reaction conditions: **1a** (0.1 mmol), **2a** or **4a** (0.4 mmol), Pd cat. (10 mol%), ligand (12 mol%), $Ph_2P(O)OH$ (20 mol%), 0.5 mL toluene, 80 °C, 18 h.
[b] Without $Ph_2P(O)OH$.
[c] Using 1,4-dioxane as solvent.
[d] Using THF as solvent.
[e] **4a** instead of **2a**.

To begin the investigation, enyne **1a** and ethyl phenylphosphinate **2a** were chosen as the model substrates (Table 1). Based on our previous report [80], $Ph_2P(O)OH$ was included as an additive. $Pd(OAc)_2$ was selected as the catalyst for screening chiral ligands (**L1**-**L4**, entries 1-4). To our delight, (*R*,*R*)-QuinoxP* **L4** afforded chiral 1,3-dienes **3aa** in 87% yield with 92% *ee*. Omitting $Ph_2P(O)OH$ resulted in a reduced yield of 19% without loss of enantioselectivity (entry 5). Other Pd precursors, such as $Pd(dba)_2$, $Pd_2(dba)_3$ and $Pd(Piv)_2$ were tested and $Pd(OAc)_2$ was found to be the optimal choice (entry 4 *vs.* entries 6-8). Decreasing the temperature led to significantly diminished yields while enantioselectivity remained (entry 4 *vs.* entry 9). The reactions conducted in 1,4-dioxane, THF, and toluene provided product **3aa** with similar high yields, but the reaction in toluene exhibited the highest *ee* (entry 4 *vs.* entries 10-11). Surprisingly, chiral 1,3-dienes **5aa** were also obtained in 92% yield and 87% *ee* when using secondary phosphine oxide **4a** instead of phosphinate **2a** (entry 12), demonstrating the generality of this method.

With the optimal conditions, the substrate scopes were then investigated. It was found that a large range of enynes were applicable in this reaction system (Scheme 2). Aryl enynes bearing electron-donating groups at the *para*-, *meta*- or *ortho*-positions (Me, Et, *t*Bu, *n*-Pent and OMe) (**3ba**–**3ia**), as well as those substituted with electron-withdrawing groups (F, Cl, Br and CF_3) (**3ja**–**3na**), were all well-tolerated and gave satisfactory results (55%-94% yields, 82%-93% *ee*). Substrate with thiophene moieties also afforded the desired products **3oa** with good yield and good *ee* (89% yield, 87% *ee*). Encouraged by the success of enynes, we further expanded the scope of this catalytic system to include various H-phosphinates. Substrate with methyl ester were conducted



Scheme 2. Substrate scope of enyens and H-phosphinates. Reaction conditions: **1** (0.1 mmol), **2** (0.4 mmol), $Pd(OAc)_2$ (10 mol%), (*R,R*)-Quinoxp* (12 mol%), $Ph_2P(O)OH$ (20 mol%), 0.5 mL toluene, 80 °C, 18 h.

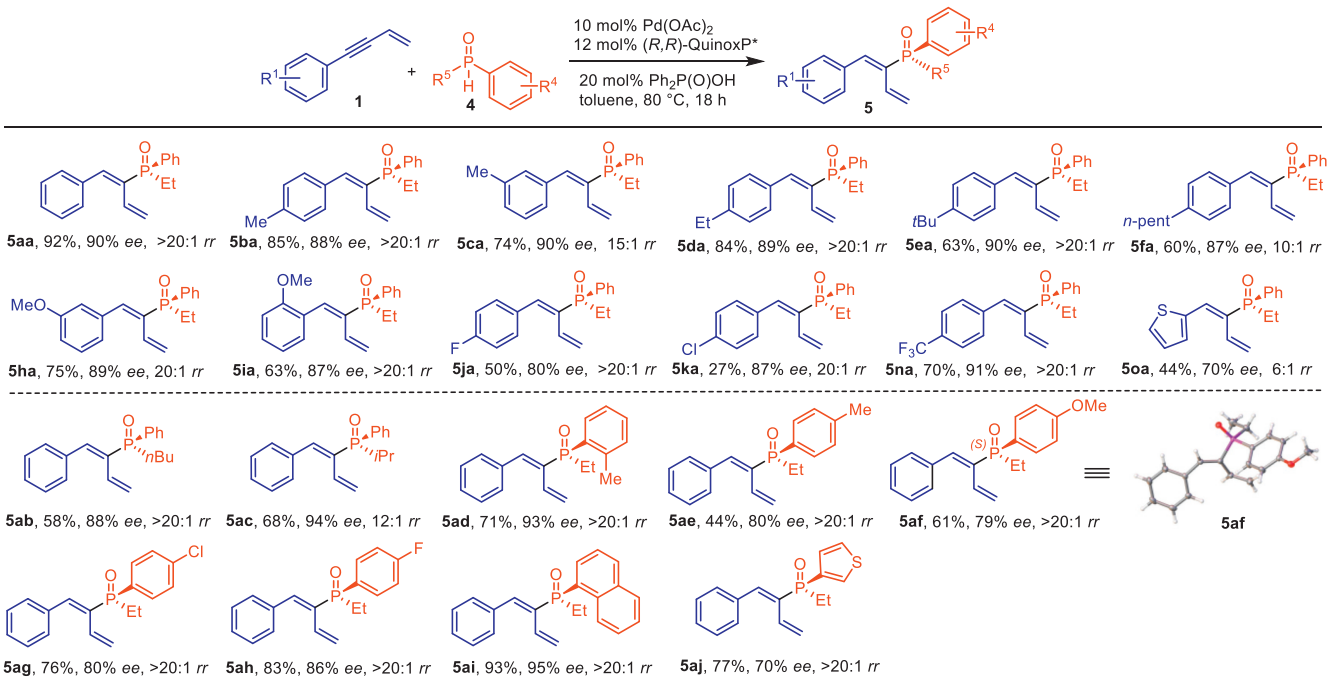
smoothly to yield the desired product **3ab** (79% yield, 82% *ee*). Isopropyl ester resulted in good yield but decreased enantioselectivity (**3ac**, 82% yield, 50% *ee*). Phenylphosphinate with a methyl substituent on the arene ring also worked and gave the product **3ad** in 80% yield but only with 20% *ee*. This transformation was also compatible with ethyl naphthalen-1-ylphosphinate, providing the desired product **3ae** in moderate yield and enantioselectivity (78% yield, 62% *ee*). Alky enyne, enzyl enyne **1p,** showed relatively poor reactivity, affording **3pa** in 20% yield with 40% *ee*.

Encouraged by the success with H-phosphinates, the investigations were extended to include various secondary phosphine oxides (SPO) for the synthesis of alkenylphosphine oxides (Scheme 3). Employing the same condition, HP(O)EtPh **4a** exhibited excellent reactivity with various aryl enynes substituted with electron-donating groups (Me, Et, *t*Bu, *n*-Pent and OMe) (**5ba**–**5ia**) or electron-withdrawing groups (F, Cl and CF_3) (**5ja**–**5na**) at the *para*-, *meta*- or *ortho*-positions, and gave the desired products with good yields with high *ee* values (27%-92% yields, 80%-91% *ee*). Notably, a thiophene-enyne also furnished the desired product in 44% yield and 71% *ee* (**5oa**). Second phosphine oxides with *n*-Bu or *i*-Pr were successfully transformed to alkenylphosphine oxides (**5ab**-**5ac**) without any significant steric hindrance effects. Furthermore, second phosphine oxides bearing various substituents on phenyl ring, such as Me, OMe, F, and Cl (**5ad**-**5ai**, 44%-83% yield, 79%-95% *ee*), were well tolerated with the reaction conditions. Interestingly, the introduction of bulky groups did not impact the reactivity. The use of secondary phosphine oxides with *ortho*-Me or 1-Nap substituent resulted in good yields and enantioselectivity (**5ad** and **5ai**). Ethyl(thiophen-2-yl)phosphine oxide was also compatible, affording the desired product **5aj** in 77% yield and 70% *ee*. The absolute configuration of **5af** was confirmed as *S*-configuration by X-ray crystallography (CCDC: 2322969).

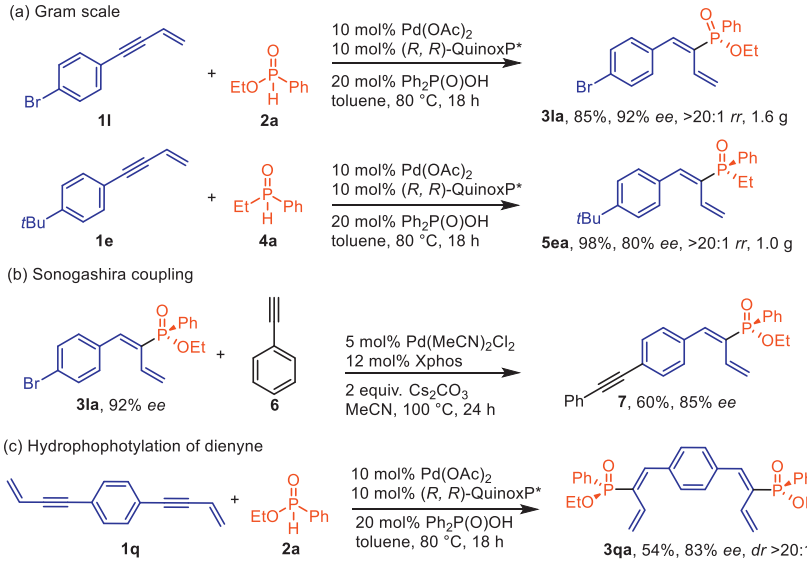
To estimate the synthetic potential of this catalytic system, gram–scale reactions were performed, giving **3la** and **5ea** in good yields without loss of enantioselectivity (Scheme 4a). Further transformations of the hydrophosphorylation products were also discovered. Product **3la** underwent Sonogashira coupling to deliver compound **7** smoothly (Scheme 4b). In addition, the dihydrophosphorylation of dienynes **1q** led to the formation of C_2-symmetric products **3qa** in 54% yield and 83% *ee* (Scheme 4c).

To gain insights into the potential reaction mechanism, deuterium-labelling experiments were conducted using D_2-**1a** or *D*-P(O)EtPh (*D*-**4a**) as starting materials. Since *D*-P(O)EtPh readily converted to H-P(O)EtPh at room temperature in the presence of $Ph_2P(O)OH$, deuterium labelling experiment were conducted without the addition of $Ph_2P(O)OH$. Firstly, enyne **1a** and *D*-P(O)EtPh were subjected to the optimized reaction conditions without $Ph_2P(O)OH$ at 90 °C, due to low reactivity of *D*-P(O)EtPh in standard condition. 80% deuterium incorporation was observed at the 4-position. Surprisingly, 30% deuterium incorporation was also observed at the terminal alkene positions, along with 20% incorporation of deuterium at the 2-position (Scheme 5A, a). These results suggested that the hydrophosphinylation reaction of enyne existed other reversible pathway. These results were different with previous report on hydrofunctionalization of conjugated enynes to give diene [84] or allenes [90–94], suggesting the reaction is not a direct hydrophosphinylation of the alkyne, but involves other reversible pathways. Then, when enyne D_2-**1a** and *D*-P(O)EtPh were used as the starting material, deuterium incorporation was observed at the 4-positions to a 70% extent, deuterium incorporation at the terminal alkene positions to 70% (reduced from 87%), and 30% incorporation of deuterium at the 2-position (*D*-**5aa-2**). These results indicate that the hydropalladation of alkene in the enyne is a reversible process (Scheme 5A, b). Finally, when enyne D_2-**1a** reacted with H-P(O)EtPh, the resulting product *D*-**5aa-3** exhibited 10% incorporation of deuterium at the 2-position and 75% incorporation of deuterium at the terminal alkene positions. This observation confirms that the deuterium incorporation at the 2-position originates from the terminal alkene of D_2-**1a**, further supporting the reversibility of the hydropalladation of the alkene in the enyne (Scheme 5A, c).

Based on previous reports about hydrofunctionalization of conjugated enynes [90–94], Pd-catalyzed intermolecular functionalization of conjugated enynes typically leads to the formation of al-



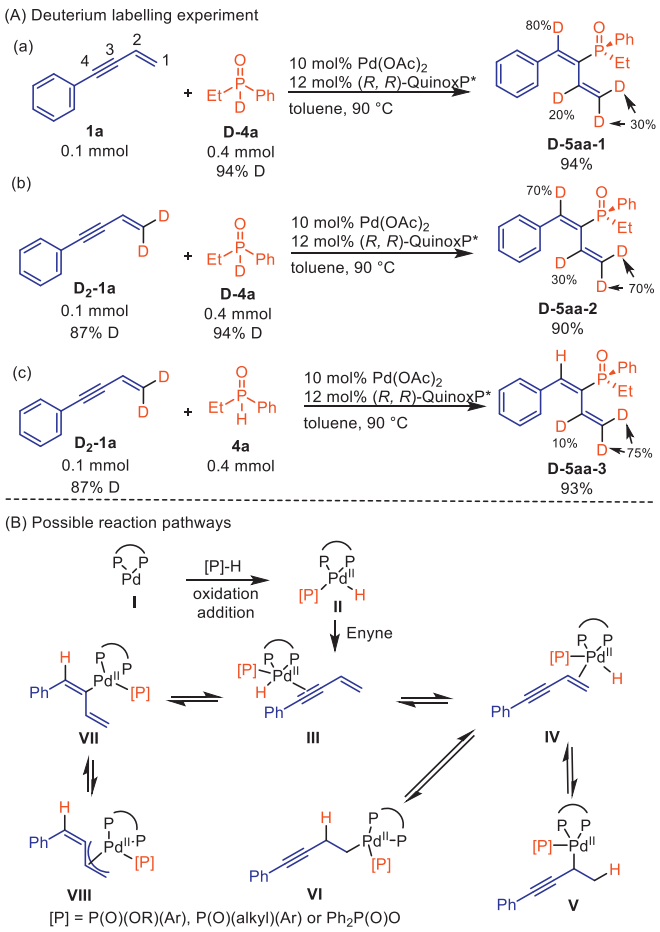
Scheme 3. Substrate scope of enyens and SPO. Reaction conditions: **1** (0.1 mmol), **4** (0.4 mmol), $Pd(OAc)_2$ (10 mol%), (*R,R*)-Quinoxp* (12 mol%), $Ph_2P(O)OH$ (20 mol%), 0.5 mL toluene, 80 °C, 18 h.



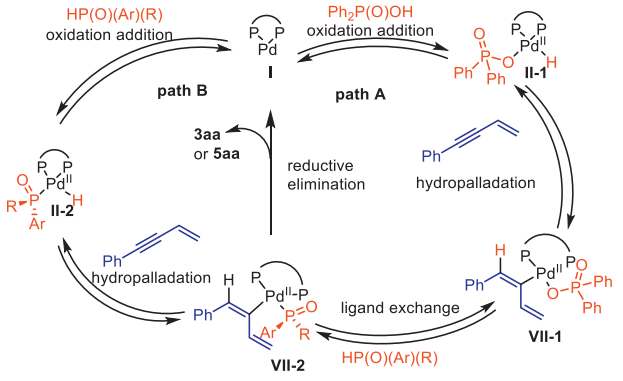
Scheme 4. Gram-scale version of the reaction and synthetic transformation.

lenes through the intermediate η^3-butadienyl–Pd (**VIII**), which is derived from η^1-butadienyl–Pd (**VII**). However, the phosphinylallene product through η^3-butadienyl–Pd (**VIII**) with phosphinates or secondary phosphine oxides were not formed in current study, showing the active species in this transformation was intermediate **VII** rather than intermediate **VIII**. Based on our previous work on palladium-catalyzed asymmetric hydrophosphorylation of alkynes [80] and deuterium labeling experiment, we hypothesized that a chiral palladium complex **I** generates from $Pd(OAc)_2$ and (*R,R*)-QuinoxP*. Then, the oxidative addition of P–H bond of phosphorous nucleophile or the O–H bond of $Ph_2P(O)OH$ to the palladium complex triggers the reaction, producing the hydropalladation intermediate **II**. Intermediate **II** coordinates with enyne to give intermediate **III** and **IV**, which undergo tautomerism. Intermediate **VII** is formed by alkyne insertion into a palladium hydride species (**III**), as confirmed by incorporation of deuterium of *D*-**5aa-1** and *D*-**5aa-2**. Additionally, the intermediates **V** and **VI** are produced ***via*** hydropalladation of alkene. These intermediate then undergo β-H-elimination to give intermediate **IV** that is coincided with the deuterium labelling experiment (Scheme 5B).

According to the deuterium labelling experiment and previous reports [80,90,102], we assumed chiral palladium complex **I** undergoes oxidative addition of $Ph_2P(O)OH$ to generate the hydropalladation **II-1**. After that, hydropalladation of enyne occurs, leading to the formation of the intermediate **VII-1**. Subsequent ligand exchange of intermediate **VII-1** with phosphinates or secondary phosphine oxides gives the internal phosphorylpalladium intermediate **VII-2** (Path A). Since the reaction also proceeds in the absence of $Ph_2P(O)OH$, an alternative pathway is also possible, in which the intermediate **II-2** is generated directly through the ox-



Scheme 5. Deuterium labelling experiment and possible reaction pathways.



Scheme 6. the proposed mechanism of hydrophosphorylation and hydrophosphinylation of conjugated enynes.

idative addition of the P–H bond of phosphorous nucleophile to palladium. This is followed by hydropalladation of enyne, resulting in the same intermediate **VII-2** (Path B). Finally, the reductive elimination of intermediate **VII-2** gives the desired product and regenerates chiral palladium complex **I** (Scheme 6).

In summary, a versatile Pd/(*R,R*)-Quinoxp* system was developed for achieving highly regio- and enantioselective construction of alkenylphosphinates and alkenylphosphine oxides with P-chirality. The mechanistic study has revealed a different pathway for the Pd-catalyzed hydrofunctionalization reaction of enyne, which refreshes the understanding of hydrofunctionalization of conjugated enynes.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

CRediT authorship contribution statement

Yanxin Jiang: Methodology. **Kwai Wun Cheng:** Methodology. **Zhiping Yang:** Writing – original draft, Project administration. **Jun (Joelle) Wang:** Writing – review & editing, Supervision.

Acknowledgments

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2024.110231.

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