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Palladium/Xu-Phos-catalyzed enantioselective arylalkoxylation reaction of γ-hydroxyalkenes at room temperature

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ABSTRACT

Metal-catalyzed alkene arylalkoxylation is a powerful complexity-building strategy for the synthesis of oxygen heterocycles from simple γ-unsaturated alcohols, but only a few examples of catalytic enantioselective methods exist. Herein, an efficient palladium-catalyzed enantioselective arylalkoxylation of γ-hydroxyalkenes with aryl halides is reported. The salient features of this transformation include a remarkable broad substrate scope, mild reaction conditions, and good functional group tolerance, delivering a series of chiral tetrahydrofurans containing a tertiary or quaternary stereocenter in good yields with up to 95% *ee*. The **Xu10** ligand with a suitable side-arm was responsible for the high reactivity and good enantioselectivity of this transformation.

Alkenes regarding as fundamental and readily available compounds, are useful building blocks in synthetic organic chemistry [1–13]. The difunctionalization of unactivated alkenes have been utilized as a powerful method to construct two distinct substituents on the two carbon atoms of the alkene moiety in one step, which has attracted increasing attention in academia and industry due to their low cost and high efficiency [14–16]. In the past several decades, inter/intramolecular difunctionalization of alkenes tethered a heteroatom-based nucleophile is particularly attractive, which offers various oxygen and nitrogen heterocycles (*e.g.*, tetrahydrofuran, tetrahydropyran, lactone, pyrrolidine, and oxazine) [17–28]. Among them, hydroxy alkenes as important synthons have been disclosed to achieve hydroalkoxylation, haloalkoxylation, oxytrifluoromethylation, alkoxyalkenylation, and arylalkoxylation. In contrast to enantioselective hydroetherification and haloetherification reactions of alkenes [29–49], few examples have focused on the enantioselective carboalkoxylation reactions of alkenes, which can install C–O and C–C bonds at the same time. For instance, Chemler [50,51] and co-workers disclosed the enantioselective copper-catalyzed intramolecular carboetherification reactions of 4-pentenols with the use of box ligands. However, these processes demanded large amounts of oxidants and harsh conditions.

Powerful synthetic access has enabled arylation strategies incorporation into numerous efforts. As early as 2004, Wolfe's group uncovered the palladium-catalyzed arylalkoxylation of readily available γ-hydroxyalkenes with aryl halides to access the cascade cyclization/coupling products [52]. Until 2016, an enantioselective variant was achieved by using a new TADDOL/2-arylcyclohexanol-derived chiral phosphite ligand **L1*** (Scheme 1a) [53]. However, the reaction was carried out at high temperature (90 °C) and in only six examples *ee* could be obtained with ≥90%. After that, Tang [54] and co-workers successfully developed a new AntPhos-derived ligand **L2*** for furnishing benzooxazines and chromans containing a quaternary stereocenter *via* Pd-catalyzed alkene aryloxyarylation reaction (Scheme 1b).

Owing to the great potential, the development of efficient processes to optical active 2,2′-multisubstituted tetrahydrofurans under mild conditions with a wild substrate scope is highly desired. Recently, we have reported a Pd/Xu-Phos-catalyzed enantioselective alkoxy-alkenylation of γ-hydroxyalkenes with alkenyl halides, delivering various chiral tetrahydrofurans in good yields with up

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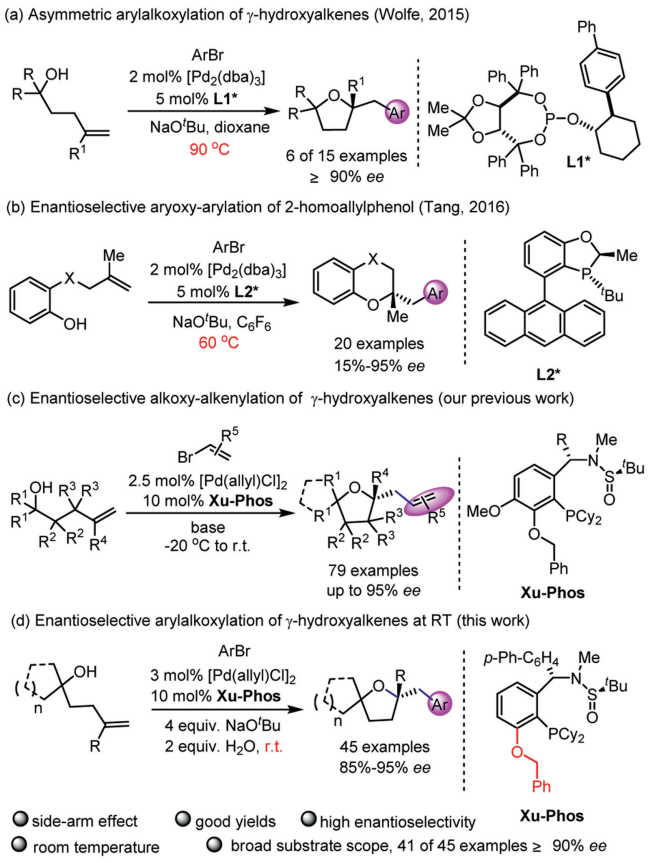
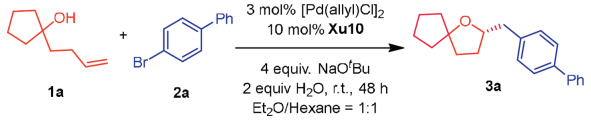


Scheme 1. Pd-catalyzed enantioselective alkene carboetherification reactions for synthesis of chiral *O*-heterocycles.

to 95% *ee* (Scheme 1c) [55]. Inspired by the work of Wolfe, Tang and our group [56–66], a growing interest in the cascade alkoxylation/coupling reaction with aryl bromide has emerged. However, there are some challenges, such as harsh conditions, relatively poor reactivity, low enantioselectivity, narrow substrate scope and competitive side reaction, *i.e.*, double bond migration of the γ-hydroxyalkene encountered in palladium-catalyzed asymmetric alkene alkoxy-alkenylation of γ-hydroxyalkenes with alkenyl halides. We speculated that the suitable ligand and optimized reaction condition could serve as the key to solve these challenges. Herein we report our efforts on palladium-catalyzed asymmetric arylalkoxylation of γ-hydroxyalkenes with aryl halides to construct enantioenriched 2,2′-multisubstituted tetrahydrofuran derivatives (Scheme 1d).

In our initial study, γ-hydroxyalkene **1a** and aryl bromide **2a** were selected as the model substrates. A series of privileged chiral ligands **L1-L12** were investigated such as (*R*)-TolBINAP (**L1**), (*R*)-2-dicyclohexylphosphino-2′-methoxybiphenyl (**L2**), (*R*)-MeO-BIPHEP (**L3**), (*R*)-BIDIME (**L4**), spirochiral ligands (**L5**-**L7**), (*R*,*R*)-^{i}Pr-FOXAP (**L8**), (*S*,*S*)-DIOP (**L9**), (*S*,*S*)-Chiraphos (**L11**) and other chiral phosphine ligands (see more details in Supporting information). All these ligands failed to obtain the product **3a** with good enantioselectivity except ligand **L12** with 57% *ee* but in low yield (Fig. 1). Next, we turned attention to our developed sulfinamide-phosphine (Sadphos) ligands, such as Ming-Phos, Xiang-Phos, PC-Phos and Xu-Phos. Unfortunately, all of these ligands with free N–H bond were ineffective. To our delight, the *N*-Me-Xu-Phos (**Xu3**) could deliver desired product **3a** in 78% yield albeit with 33% *ee* (Fig. 2), indicating that the variation of *N*-R group in Xu-Phos might tune the reactivity and enantioselectivity. With this hypothesis, we altered the *N*-R group to allyl (**Xu4**) and benzyl (**Xu5**) but both led

Table 1
Optimization of reaction conditions.[a]



Entry	Variation from the "standard" conditions	Yield (%)	*ee* (%)
1	None	89	92
2	2 equiv. NaO^{t}Bu	45	90
3	NaOH instead of H_2O	88	89
4	Ag_2CO_3 instead of H_2O	36	89
5	Without H_2O	80	88
6	$Pd(dba)_2$ instead of $[Pd(allyl)Cl]_2$	78	88
7	$Pd(MeCN)_4BF_4$ instead of $[Pd(allyl)Cl]_2$	37	87
8	$Pd(OAc)_2$ instead of $[Pd(allyl)Cl]_2$	39	86
9	$PdCl_2$ instead of $[Pd(allyl)Cl]_2$	45	85
10	KO^{t}Bu instead of NaO^{t}Bu	86	81
11	LiO^{t}Bu instead of NaO^{t}Bu	12	56
12	Cs_2CO_3 instead of NaO^{t}Bu	–	–
13	Et_3N instead of NaO^{t}Bu	–	–
14	Toluene instead of Et_2O/Hexane	62	86
15	THF instead of Et_2O/Hexane	15	70
16	Et_2O instead of Et_2O/Hexane	48	88
17	Hexane instead of Et_2O/Hexane	76	86
18	50 °C instead of RT	88	89
19	60 °C instead of RT	87	87

[a] Reaction conditions: **1a** (0.2 mmol), **2a** (0.4 mmol), NaO^{t}Bu (4 equiv.), H_2O (2 equiv.), 3 mol% $[Pd(allyl)Cl]_2$, and 10 mol% ligand in 2.0 mL Et_2O/hexane = 1:1, room temperature, under N_2 for 48 h. Isolated yield. *ee* values were determined by chiral HPLC analysis. Note: THF= tetrahydrofuran.

to lower reactivity and only a little improvement of enantioselectivity was achieved with the **Xu5** ligand. Further screening the *N*-Me-XuPhos ligand by adjusting the R^1 group, the *ee* value substantially increased and the yield remained (*e.g.*, **Xu6**: 78% yield, 59% *ee*; **Xu7**: 73% yield, 63% *ee*; **Xu8**: 78% yield, 73% *ee*). The ligand **Xu9** with an ^{i}PrO side-arm could slightly improve the enantioselectivity from 73% *ee* to 78% *ee* compared with **Xu8**. Notably, the ligand **Xu10** with a BnO side-arm could deliver much better enantioselectivity (92% *ee*) with higher reactivity (89% yield) [67]. However, when employing the bulkier naphthyl to supplant benzyl group, the ligand **Xu11** gave relatively lower enantioselectivity. On the other hand, we also attempted to introduce electron-donating MeO groups at *meta* and *para*-positions of PCy_2 group, which would enhance the electronic property of phosphine and facilitate oxidative addition of palladium to aryl bromides [68,69]. The ligand **Xu12** improved the yield and the enantioselectivity slightly compared with the ligand **Xu8**. Subsequently, further comprehensive screening of the reaction conditions *via* changing additive, palladium salt, base, solvent, temperature, but all failed to further improve the enantioselectivity (Table 1).

With the optimized reaction conditions in hand, we investigated the substrate scope of this transformation. A series of spirocyclic tetrahydrofurans were furnished in good yields with high enantioselectivities (Scheme 2). The substrate of 1-(but–3-en-1-yl)-cyclopentan-1-ol **1a** with various aryl bromides could work well to produce the desired products **3a**-**3j** in 65%–93% yields with 90%–94% *ee*. Moreover, 1-(but–3-en-1-yl)-cyclohexan-1-ol **1b** with various aryl bromides, such as electron-donating (Me, MeO) groups at different positions of aryl bromides were well tolerated to afford the corresponding chiral spirocyclic tetrahydrofurans **3k**-**3q** in 70%–89% yields with 89%–92% *ee*. Even 4-bromoveratrol furnished the corresponding product **3r** in 85% yield with 92% *ee*. Generally, substrates with halogen atom (F or Cl) at the para-position of the aromatic ring reacted well to give the corresponding products **3s**-**3t** in 76%–80% yields and 91%–92% *ee*. Modulation of the aryl skeleton of aryl bromides with a series of substituents, such as 3-

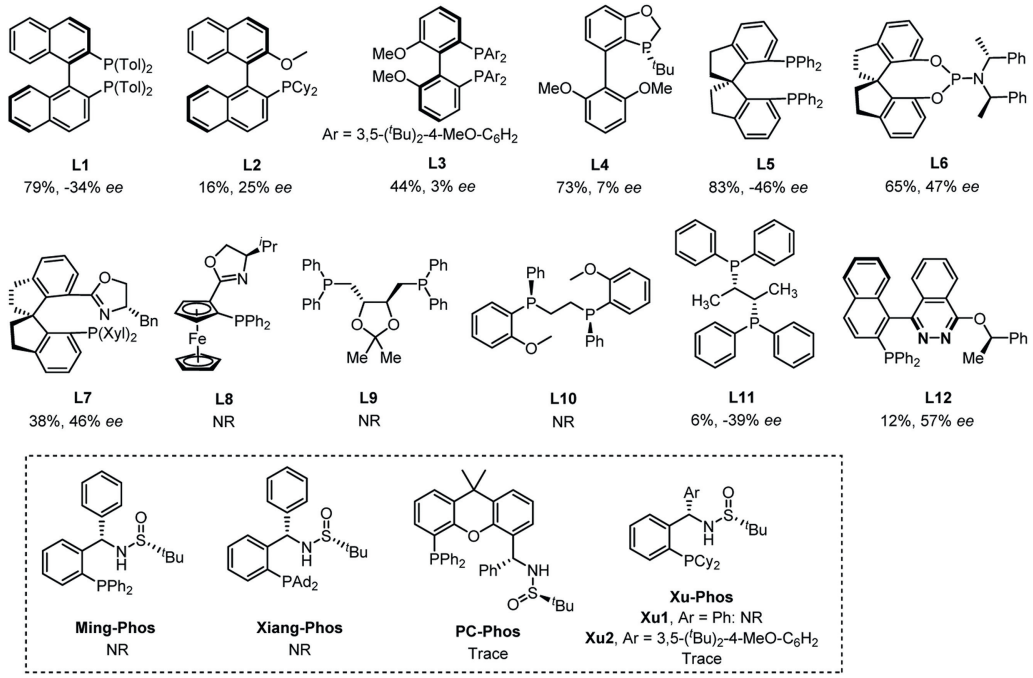


Fig. 1. Screened known ligands.

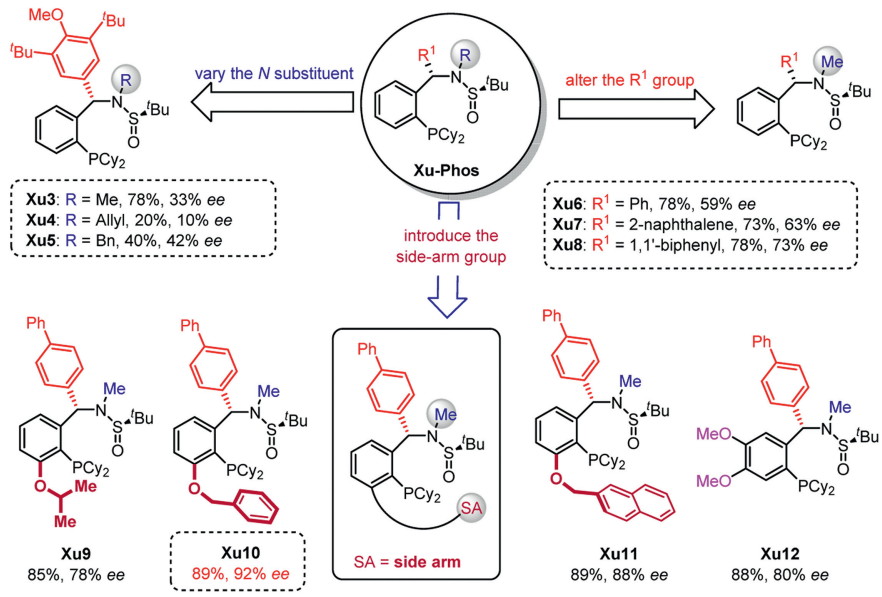
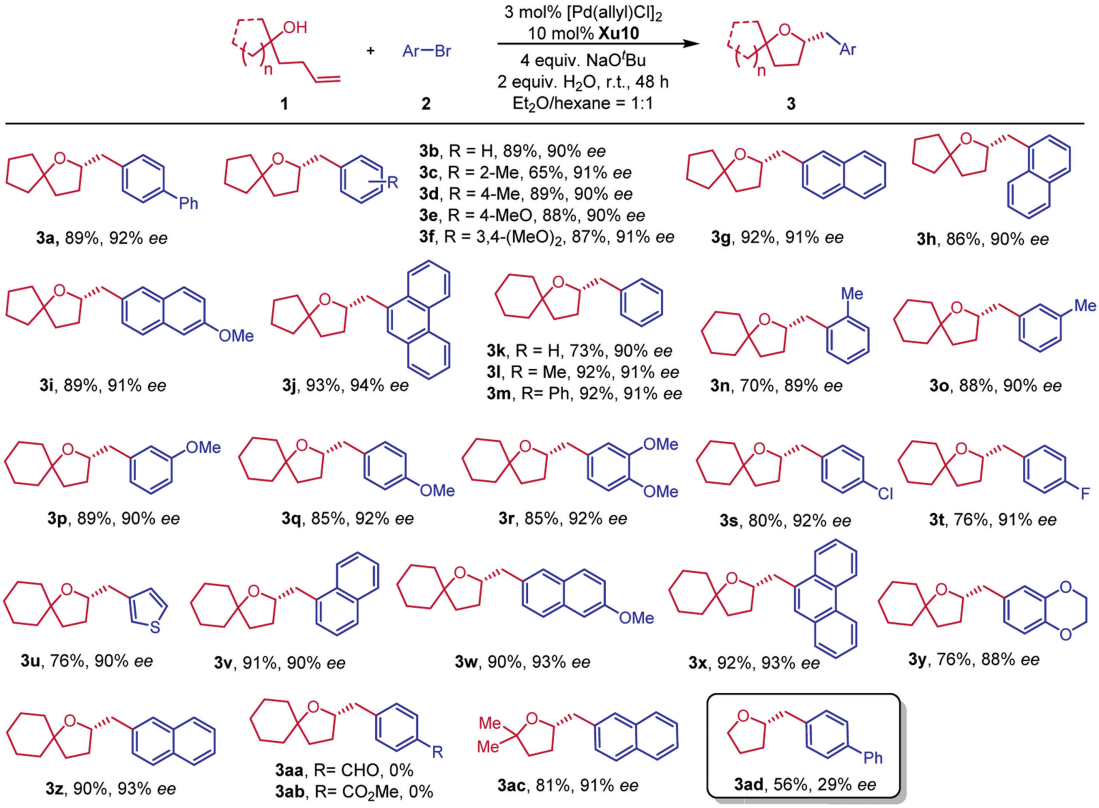


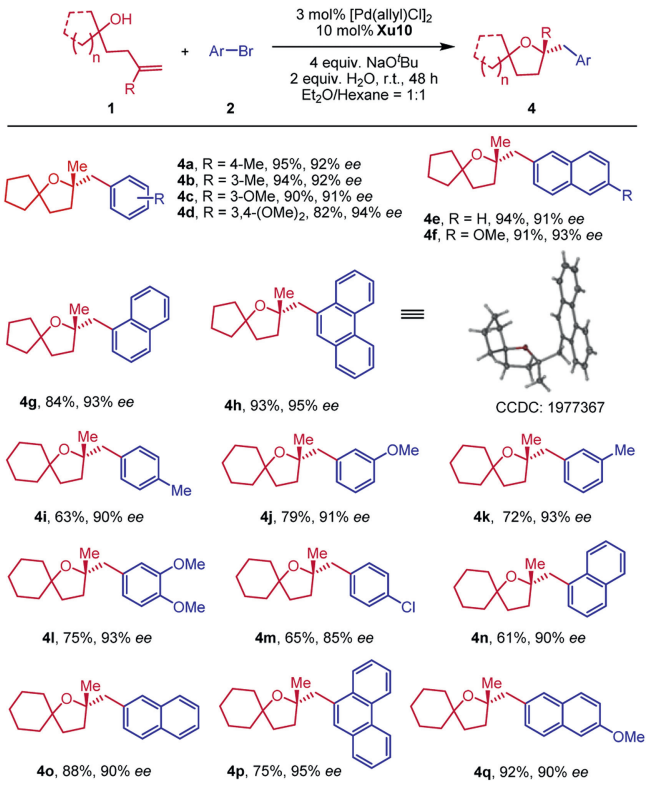
Fig. 2. Optimization of the Xu-Phos ligands.

thienyl, 1,4-benzodioxane-6-yl, phenanthren-9-yl, 2-naphthyl, and 1-naphthy, delivering the desired **3u**-**3z** in 76%–92% yields with 88%–93% *ee*. It is a pity that no desired products **3aa** and **3ab** could be obtained by using aryl bromides bearing strong electron-withdrawing groups, such as aldehyde and ester groups. We proposed that the reduction elimination process is difficult to occur in the reaction of aryl electrophiles bearing electronically deficient groups. Fortunately, acyclic γ-hydroxyalkene **1c** reacted smoothly with 2-bromonaphthalene to furnish the multisubstituted tetrahydrofuran **3ac** in 81% yield with 91% *ee*. Notably, pent–4-en-1-ol was also tolerated, giving **3ad** in 56% yield with 29% *ee*. This result indicated that *gem*–dialkyl groups at 2,2-positions in the hydroxyalkenes influenced strongly the stereoselectivity, which could be explained by the steric effect of *gem*–dialkyl groups in favor of the formation of five-membered heterocycles.

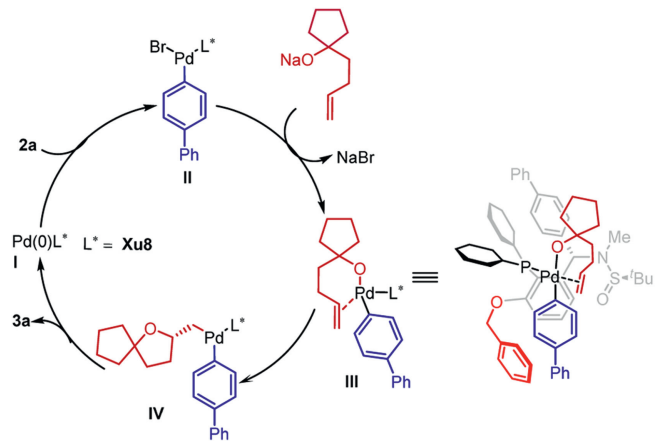
To further explore the scope of this reaction, a range of chiral spirocyclic tetrahydrofurans bearing a quaternary stereocenter were formed in excellent yields with high enantioselectivities (Scheme 3). For example, the substrate of 1-(3-methylbut-3-en-1-yl)-cyclopentan-1-ol **1d** with various aryl bromides could work well to produce the desired products **4a**-**4h** in 82%–95% yields with 90%–95% *ee*. Moreover, the reactions of 1-(3-methylbut-3-en-1-yl)cyclohexan-1-ol **1e** with various aryl bromides bearing electron-donating groups (Me, OMe) at the *meta*- and *para*-position went well to furnish the corresponding products **4i**-**4l** in 63%–79% yields with 90%–93% *ee*. The electron-withdrawing group such as halide (Cl) was compatible to afford the corresponding products **4m** in 65% yield with 85% *ee*. 1-Naphthyl bromide, 2-naphthyl bromide and 9-bromophenathrene could also be employed to deliver the desired chiral spirocyclic tetrahydrofu-



Scheme 2. Scope with respect to the aryl halides. Reaction conditions: **1a** (0.2 mmol), **2a** (0.4 mmol), NaO^{t}Bu (4 equiv.), H_2O (2 equiv.), 3 mol% [Pd(allyl)Cl]$_2$, and 10 mol% ligand in 2.0 mL Et_2O/hexane = 1:1, room temperature, under N_2 for 48 h. The yield is isolated yield. *ee* was determined by chiral HPLC analysis.



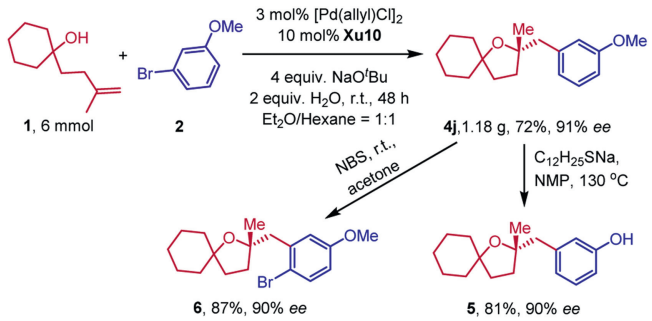
Scheme 3. Enantioselective formation of quaternary centers. Reaction conditions: **1a** (0.2 mmol), **2a** (0.4 mmol), NaO^{t}Bu (4 equiv.), H_2O (2 equiv.), 3 mol% [Pd(allyl)Cl]$_2$, and 10 mol% ligand in 2.0 mL Et_2O/hexane = 1:1, room temperature, under N_2 for 48 h. The yield is isolated yield. *ee* was determined by chiral HPLC analysis.



Scheme 4. Proposed catalytic cycle and stereochemical model.

rans bearing a quaternary stereocenter **4n**-**4q** in 61%–92% yields with 90%–95% *ee*.

Based on the studies by Wolfe and Tang as well as our previous work, the proposed catalytic cycle is shown in Scheme 4. The Pd^0 species undergoes oxidative addition with the aryl bromide (**2a**) to afford Pd^{II} complex **II**. With NaO^{t}Bu as the base, ligand exchange between the Pd^{II} complex **II** and alcohol (**1a**) would lead to the formation of Pd–O bond and give intermediate **III**. And intermediate **III** undergoes migratory insertion, giving the Pd complex **IV**. This process was proposed as the stereoselectivity-determining step. Subsequent reductive elimination occurred to form the desired product **3a** with concomitant regeneration of the Pd(0)L* catalyst. A stereochemical induction model is proposed. The BnO side-



Scheme 5. Gram-scale synthesis and transformations of products.

arm, the *N*-Me moiety, and the substituent R^1 of **Xu10** all contributed to this well-defined stereochemical process.

To display the synthetic utility of this methodology, a gram-scale reaction and derivatizations of **4j** were accomplished (Scheme 5). Under standard conditions, 1.18 g of **4j** was obtained in 72% yield with 91% *ee*. The enantiopure **4j** could undergo a demethylation reaction to produce compound **5** with retention of the enantiomeric purity. Furthermore, a bromination product **4j** was obtained in 87% yield with 90% *ee*.

In summary, we have developed an efficient palladium-catalyzed enantioselective arylalkoxylation of γ-hydroxyalkenes with aryl halides, which provided a facile access to a series of chiral tetrahydrofurans containing a tertiary or quaternary stereocenter in good yield with up to 95% *ee*. The salient features of this transformation include mild reaction conditions, readily available starting materials, a remarkable broad substrate scope, and good functional group tolerance. The chiral sulfnamidephosphine ligand **Xu10** with a suitable side-arm was responsible for the high reactivity and enantioselectivity of this transformation. This study further confirmed that the introduction of a side-arm is an efficient strategy for tuning the enantioselectivity of a certain asymmetric reaction.

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

Shuai Zhu: Data curation, Writing – original draft. **Mingjie Chen:** Data curation, Methodology. **Haichao Shen:** Data curation, Validation. **Hanming Ding:** Supervision. **Wenbo Li:** Supervision, Writing – review & editing. **Junliang Zhang:** Funding acquisition, Supervision, Writing – review & editing.

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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.2024.109879.

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