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COMMUNICATION

Synthesis of axially chiral 5,5'-substituted 2,2'-bipyridine ligands and their application in palladium-catalyzed asymmetric oxidative [2+2] annulation

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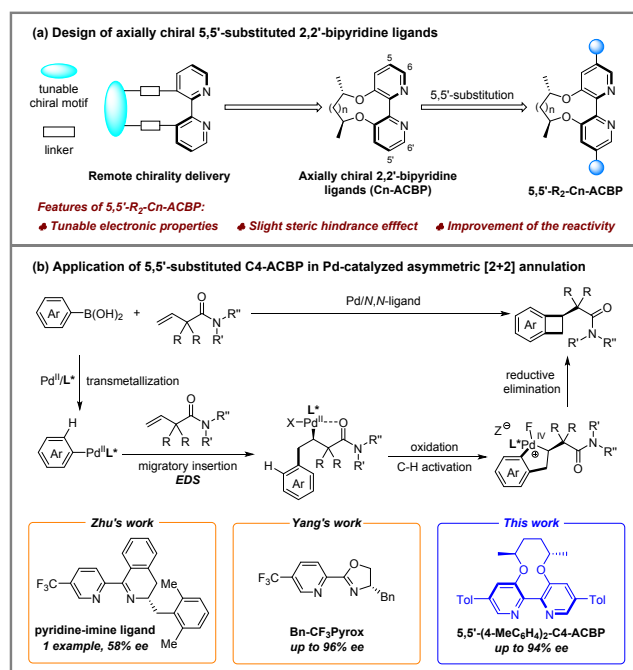
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A series of axially chiral 5,5'-substituted 2,2'-bipyridine ligands featuring tunable properties has been synthesized. They have been successfully applied to palladium-catalyzed asymmetric oxidative [2+2] annulation of arylboronic acids and alkenes, providing a diverse range of chiral benzocyclobutenes in excellent enantioselectivities with broad substrate scope.

In modern synthetic chemistry, the innovation of novel chiral ligands has been a crucial area of catalytic asymmetric catalysis.¹ Owing to their robust stability and easy functionalization, chiral *N,N*-ligands have occupied a predominant position in enantioselective synthesis.^{1a,2} Among the various types of chiral *N,N*-ligands, the chiral 2,2'-bipyridine ligands have served as one of the notable representatives. Since the first development of chiral monoalkyl-substituted 2,2'-bipyridine ligands by Botteghi's group in 1984,³ a diverse series of chiral 2,2'-bipyridine ligands have been designed and extensively applied in the last four decades.⁴⁻⁶ These privileged chiral ligands are consisted of central-chiral bipyridine ligands,⁴ planar-chiral bipyridine ligands,⁵ and axial-chiral bipyridine ligands.⁶ In 2015, our group developed axially chiral 2,2'-bipyridine ligands (abbreviated as Cn-ACBP) through the remote chirality delivery strategy (Scheme 1a).^{6a} These axially chiral ligands have been successfully utilized in versatile transition metal-catalyzed asymmetric reactions, including palladium-catalyzed enantioselective C-H functionalization of indoles^{6a} and pyrroles^{6b}, O-H insertion reaction of phenols,^{6a} intramolecular C(sp³)-H insertion of donor-donor carbenes,^{6c} arylation of *N*-tosylaryl-imines,^{6d} cyclization of aniline-tethered alkynyl cyclohexadienones,^{6e} and so on.^{6f-i} Although superior performance of our axially chiral 2,2'-bipyridine ligands has been displayed in

asymmetric catalysis, the exploration of more tunable and applicable axially chiral 2,2'-bipyridine ligands is still in demand. Conventionally, the introduction of substituents to 6,6'-positions of 2,2'-bipyridine ligands would cause remarkable steric hindrance to the transition metal center and reduce the reactivity. In order to dissolve this obstacle, we envisioned developing the axially chiral 5,5'-substituted 2,2'-bipyridine ligands (abbreviated as 5,5'-R₂-Cn-ACBP), which would fine-tune the electronic properties by regulating different substituents and exhibit slight steric hindrance effect simultaneously to ensure high reactivity and stereoselectivity (Scheme 1a).



Scheme 1. Axially chiral 5,5'-substituted 2,2'-bipyridine ligands enabled palladium-catalyzed asymmetric oxidative [2+2] annulation.

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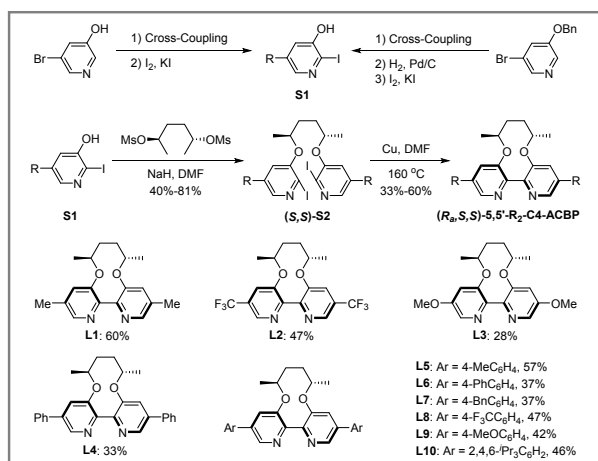
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Supplementary Information available: [details of any supplementary information available should be included here]. See DOI: 10.1039/x0xx00000x



Benzocyclobutenes have emerged as vital structural motifs in natural products and bioactive molecules.⁷ Due to the significant importance of benzocyclobutenes, considerable attention has been received to them and various synthetic methods have been developed.⁸⁻⁹ Recently, palladium-catalyzed oxidative [2+2] annulation has been a useful and straightforward protocol to construct benzocyclobutenes.¹⁰ In 2022, Zhu and coworkers disclosed an elegant synthesis of the functionalized benzocyclobutenes *via* palladium-catalyzed oxidative [2+2] annulation of arylboronic acids with alkenes using an achiral 2,2'-bipyridine as ligand.¹¹ They also reported a single catalytic asymmetric version of this reaction, delivering the target chiral benzocyclobutene product with 58% ee in the presence of a chiral pyridine-imine ligand. Last year, Yang's group realized a Pd/Pyrox-catalyzed asymmetric oxidative [2+2] annulation to afford a wide range of chiral benzocyclobutenes (Scheme 1b).¹² Based on the exceptional catalytic efficacy of chiral *N,N*-ligands in this reaction and the advantages of axially chiral 5,5'-substituted 2,2'-bipyridine ligands, we envisaged applying the 5,5'-R₂-Cn-ACBP ligands in palladium-catalyzed asymmetric annulation to ensure both reactivity and enantioselectivity. Herein, we reported the synthesis of axially chiral 5,5'-substituted 2,2'-bipyridine ligands and their application in palladium-catalyzed asymmetric oxidative annulation of arylboronic acids with alkenes for synthesis of benzocyclobutenes with excellent enantioselectivities (Scheme 1b).



Scheme 2. Synthesis of axially chiral 5,5'-substituted 2,2'-bipyridine ligands.

The axially chiral 5,5'-substituted 2,2'-bipyridine ligands were synthesized through the approach depicted in Scheme 2. A wide range of 5,5'-R₂-Cn-ACBP ligands were conveniently synthesized through two steps from the easily available chiral (2*R*,5*R*)-hexane-2,5-diyl dimethanesulfonate and the variable 5-substituted 2-iodo-3-hydroxypyridines **S1** which can be obtained from 3-hydroxy-5-bromopyridine or 3-(benzyloxy)-5-bromopyridine *via* cross-coupling and iodination. Initiated by S_N2 reaction and followed by Ullmann coupling, various 5,5'-R₂-Cn-ACBP ligands **L1-L10** bearing diverse alkyl and aryl substituents at 5,5'-positions were obtained in moderate 28-60% yields.

With the axially chiral 2,2'-bipyridine ligands in hand, we began our investigation on palladium-catalyzed asymmetric oxidative [2+2] annulations. Initially, 2,2-dimethyl-1-morpholinobut-3-en-1-one **1a** and phenylboronic acid **2a** were chosen as the model substrates to perform the reaction at 40 °C using (*R_a*,*S*,*S*)-3,3'-(1,4-dimethylbutanedioxy)-2,2'-bipyridine (abbreviated as **C4-ACBP**) as ligand and benzotrifluoride as solvent. Satisfyingly, the target product **3aa** was obtained in 38% yield with 82% ee (Table 1, entry 1). Further evaluation of solvents revealed that tetrahydrofuran (THF) afforded comparable results to benzotrifluoride (entry 2). 1,2-dichloroethane (DCE) proved optimal, delivering the product **3aa** in 40% yield with 95% ee (entry 3).

Table 1. Optimization of reaction conditions

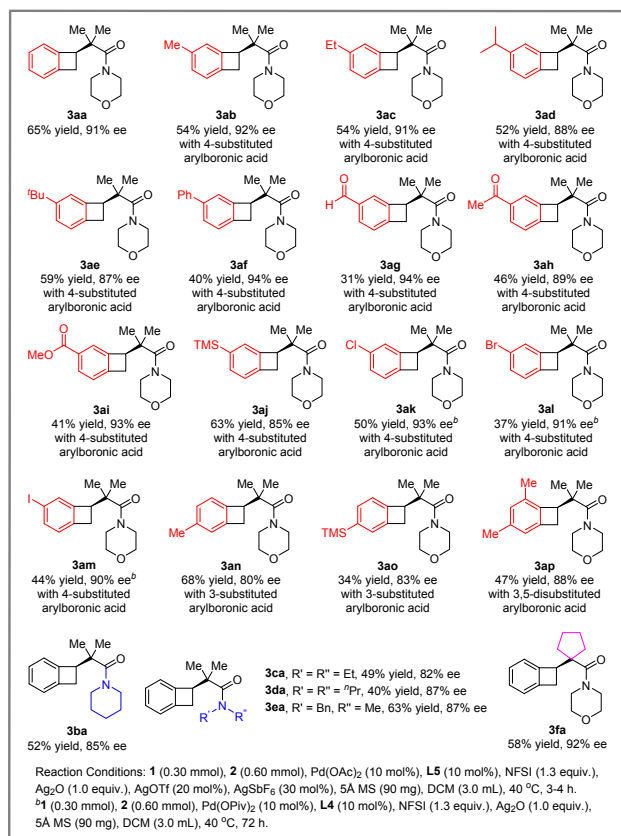
entry ^a	solvent	L [*]	yield (%) ^b	ee (%) ^c
1	PhCF ₃	C4-ACBP	38	82
2	THF	C4-ACBP	39	84
3	DCE	C4-ACBP	40	95
4	DCE	C2-ACBP	45	53
5	DCE	C3-ACBP	37	91
6	DCE	L1	39	93
7	DCE	L2	33	83
8	DCE	L3	37	92
9	DCE	L4	46	90
10	DCE	L5	52	91
11	DCE	L6	47	80
12	DCE	L7	40	89
13	DCE	L8	48	92
14	DCE	L9	41	91
15	DCE	L10	40	93
16 ^d	DCE	L5	56	89
17 ^e	DCE	L5	49	93
18 ^f	DCE	L5	65 ^g	91

^a Reaction conditions: **1a** (0.10 mmol), phenylboronic acid **2a** (0.20 mmol), Pd(OAc)₂ (10 mol%), L^{*} (12 mol%), NFSI (1.3 equiv.), Ag₂O (1.0 equiv.), 5A MS (30 mg), solvent (1.0 mL), 40 °C, 22 hours. ^b Yield was measured by analysis of ¹H NMR spectra, using diphenylacetonitrile as the internal standard. ^c Determined by chiral HPLC. ^d AgOTf (20 mol%) was added. ^e AgSbF₆ (30 mol%) was added. ^f Reaction was conducted on a 0.30 mmol scale, **L5** (10 mol%), AgOTf (20 mol%) and AgSbF₆ (30 mol%) were added, 3 h. ^g Isolated yield.

Afterwards, (*R_a*,*S*,*S*)-3,3'-(1,2-dimethylethylenedioxy)-2,2'-bipyridine (abbreviated as **C2-ACBP**) and (*R_a*,*S*,*S*)-3,3'-(1,3-dimethylpropanedioxy)-2,2'-bipyridine (abbreviated as **C3-ACBP**) ligands were evaluated. Compared with **C4-ACBP** ligand, **C2-ACBP** ligand diminished enantioselectivity, although slightly increased the yield (entry 4). **C3-ACBP** ligand resulted in both diminished yield and enantioselectivity (entry 5). In order to further improve the yield and maintain the enantioselectivity, a number of 5,5'-R₂-C4-ACBP ligands bearing diverse substituents were screened. Ligands bearing alkyl groups **L1-L3** exhibited



inferior results compared to **C4-ACBP** (entries 6-8). When phenyl substituted ligand **L4** was utilized, the yield could be enhanced to 46% and the ee value was 90% (entry 9). Notably, **L5** bearing *p*-methylphenyl groups was the most efficient ligand, giving the desired product in 52% yield and 91% ee (entry 10). **L6-L9** containing aromatic substituents with different electronic properties behaved worse than **L5** (entries 11-14). **L10** with bulky groups reduced the yield of the reaction (entry 15). Since silver salts additives play a crucial role in palladium-catalyzed C-H activation,¹³ the investigation of silver salt additives was conducted. It was found that the addition of AgOTf could improve the yield of the reaction and slightly decrease the enantioselectivity (entry 16). The addition of AgSbF₆ could improve the enantioselectivity to 93%, while reduce the yield to 49% (entry 17). To achieve the better yield and enantioselectivity at the same time, the combination of AgOTf and AgSbF₆ was adopted (see the Supporting Information for details). When both of AgOTf and AgSbF₆ were added and the loading of ligand **L5** was reduced to 10 mol%, the enantiomeric excess of **3aa** was maintained at 91% with a significant improvement of yield to 65% (entry 18).

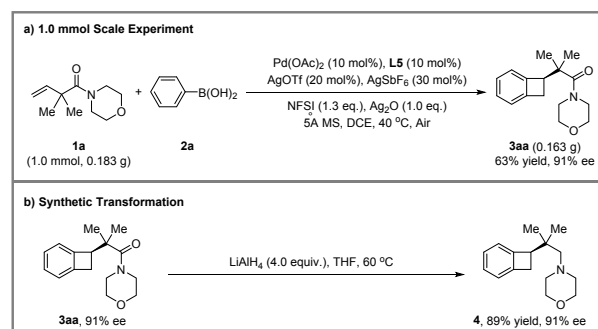


Scheme 3. Substrate scope: alkenes **1** and arylboronic acids **2**.

Upon establishing the reliable reaction conditions, we explored the substrate scope of this enantioselective [2+2] annulation by varying alkenes **1** and arylboronic acids **2** (Scheme 3). First, we investigated the arylboronic acid components. 4-Alkyl substituted arylboronic acids furnished the benzocyclobutenes **3ab-3ae** in moderate yields with 87%-92% ee value. 4-

Phenyl substituted arylboronic acid **2f** was accommodated. Arylboronic acids with electron-withdrawing substituents were appropriate reaction partners. (4-Formylphenyl)boronic acid **2g** and (4-acetylphenyl)boronic acid **2h** and smoothly proceeded the annulation with 94% and 89% ee value. (4-(Methoxycarbonyl)phenyl)boronic acid **2i** afforded desirable benzocyclobutene with excellent 93% of enantioselectivity. (4-(Trimethylsilyl)phenyl)boronic acid **2j** was also tolerated, giving good enantioselectivity and moderate yield. When arylboronic acids bearing halogen at the 4-position were used, the corresponding benzocyclobutenes (**3ak-3am**) were obtained in moderate yields and excellent enantioselectivities using Pd(OPiv)₂ and **L4** as the catalyst. 3-Substituted (**2n, 2o**) and 3,5-disubstituted (**2p**) aryl-boronic acids were suitable for asymmetric reaction, giving the desired products **3an-3ap** with good enantioselectivities. Moreover, a range of alkenes **1** were examined with phenylboric acid. Amide moiety in alkene **1** derived from piperidine (**1b**) and linear amides (**1c-1e**) were tolerated. Finally, the alkene with cyclic *gem*-disubstituted group (**1f**) could also afford the corresponding benzocyclobutene in moderate yields with 92% ee value.

To demonstrate the scalability of the tactic, a 1.0 mmol scale experiment was performed to provide the desirable chiral product **3aa** in 63% yield with 91% ee, which is consistent with the result of 0.30 mmol scale (Scheme 4a). Then the product **3aa** underwent reduction with LiAlH₄ to give chiral amine in 89% yield and 91% ee (Scheme 4b).



Scheme 4. Experiment at a 1.0 mmol scale and synthetic transformation.

Conclusions

In conclusion, we have successfully synthesized a class of axially chiral 5,5'-substituted 2,2'-bipyridine ligands, featuring tunable properties. These ligands would finely regulate the electronic characters by introducing different substituents to 5,5'-positions and slightly affect the steric hindrance. These ligands were applied in palladium-catalyzed oxidative [2+2] annulation of arylboronic acids and alkenes to give chiral benzocyclobutenes in excellent enantioselectivities. These results suggested the hopeful potential of axially chiral ligands as a useful type of chiral *N,N*-ligands. Further explorations on the application of these ligands in asymmetric catalysis are ongoing in our laboratory.



Author contributions

Kai Xue: Methodology, investigation and writing – review & editing. Jian Chen: Validation. Yu-Qing Bai: Validation. Bo Wu: conceptualization, validation, supervision, and writing – review & editing. Yong-Gui Zhou: conceptualization, validation, supervision, and writing – review & editing.

Conflicts of interest

There are no conflicts to declare.

Data availability

The data supporting this article have been included as part of the ESI.

Acknowledgements

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Data availability

The data supporting this article have been included as part of the ESI.

