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Modular synthesis of polyfunctionalized axial-chiral 2-arylpyridines *via* cobalt-catalyzed asymmetric [2 + 2 + 2] cycloaddition of diynes and nitriles

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ABSTRACT

Polyfunctionalized axial-chiral 2-arylpyridines are important class of chiral entities in organic synthesis and drug discovery. However, practical synthesis of such structures remains underdeveloped. Herein, we report a novel synthetic method *via* simple cobalt-catalyzed [2 + 2 + 2] cycloaddition reactions of easily accessible diynes with nitriles. A wide range of polyfunctionalized axial-chiral 2-arylpyridine derivatives were obtained with high enantioselectivities and atom economy by using 2-methyl tetrahydrofuran (2-MeTHF) as a green solvent. Notably, the axial-chiral 2-arylpyridine *N*-oxide compounds and chiral bispyridine derivatives have been accessible in high efficiency using this simple protocol. The mechanism and origin of regioselectivity of this reaction was revealed using DFT calculation profiles.

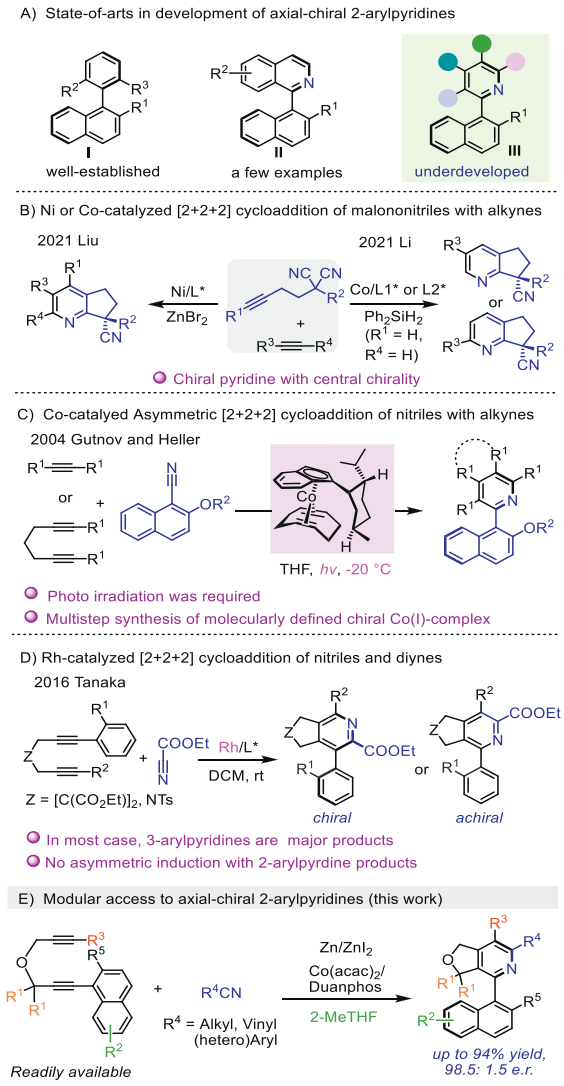
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Atropisomerism represents one of the most important chirality elements in nature [1–5]. Atropisomers with different configurations could vary drastically in their biological activities and functions [6–12]. In particular, axial-chiral 2-arylpyridine derivatives are important structural motifs that are not only widely available in bioactive molecules [13], but also frequently used as ligands or catalysts such as Quinazolinap, Quinap, and Quinox (Scheme 1A) [14–17]. Contrary to the development of protocols accessing to axially chiral biaryls [18–20] as depicted as **I**, much less attention has been paid to the methods producing densely functionalized axial-chiral 2-arylpyridines **III**. Hence, the development of the efficient methods for accessing to diverse polyfunctionalized axial-chiral 2-arylpyridine derivatives could potentially extend the chemical and biological space of this privileged scaffolds, as well as provide new opportunities in asymmetric catalysis.

Transition metal-catalyzed [2 + 2 + 2] cycloaddition of alkynes and nitriles has emerged as an atom economic process and allows the formation of multisubstituted pyridines using readily available starting materials in a single step [21–24]. The great progress has recently been made in the construction of chiral pyridines with central chirality by the groups of Liu [25] and Li [26] independently (Scheme 1B), however, the methods for highly enantioselective construction of axial-chiral 2-arylpyridines yet remains underdeveloped. In this context, the groups of Gutnov, Heller, and Hapke developed methods for the synthesis of enantioenriched atropisomers of 2-arylpyridines (Scheme 1C) [27–30]. However, multi-step synthesis of the molecularly defined chiral indenyl-based cobalt(I) complex involving resolution was required, and the external photo irradiation was necessary for the reactions to take place. In the related area of the research, Tanaka *et al.* have demonstrated that Rh(I)/H_8-Binap-catalyzed asymmetric [2 + 2 + 2] cycloaddition has enabled access to axial-chiral pyridines (Scheme 1D) [31], however, in most cases, the axially chiral 3-arylpyridines were obtained in their system. Subsequently, Song *et al.* disclosed an asymmetric [2 + 2 + 2] cycloaddition reaction of B,N-diynes and nitriles for the synthesis of C-B axial chirality using rhodium catalysis system, the reactions favorably delivered axial-chiral 3-arylpyridines compounds [32]. Very recently, Liu *et al.* developed a nickel-catalyzed asymmetric [2 + 2 + 2] cycloaddition reactions for the efficient construction of axially chiral 4-arylpyridine products [33]. Therefore, a modular and practical catalytic system that utilizes

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Scheme 1. Asymmetric [2+2+2] cycloaddition of alkynes and nitriles.

commercially available catalysts, and works for the asymmetric [2+2+2] cycloaddition for accessing densely functionalized axialchiral 2-arylpyridines **III** (Scheme 1A) is still highly desirable, which remains a challenge in organic synthesis. Herein, we report our discovery and development of cobalt-catalyzed highly enantioselective [2+2+2] cycloaddition reactions of readily available diynes and nitriles by using commercially available cobalt salt and chiral bisphosphine as the catalyst system (Scheme 1E) [34–62]. A broad range of axial-chiral 2-pyridine derivatives with diverse substitutions were achieved in good yields and excellent enantioselectivities. DFT calculation profiles have identified the mechanism of this cobalt-catalyzed asymmetric [2+2+2] cycloaddition reaction for further clarifying the origin of regioselectivity.

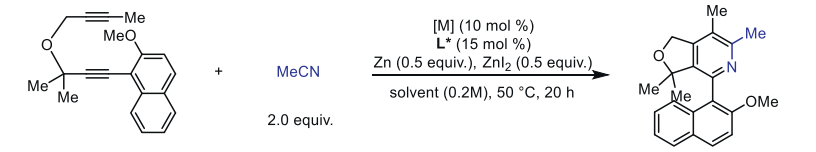
The reaction conditions were optimized using readily available diyne **1a** and commercially available acetonitrile **2a** (Table 1). Initially, a variety of metal salts which have been demonstrated to be effective in previous [2+2+2] cycloaddition reactions were chosen to tested for validation of our hypothesis, however, no desired product was observed (Table 1, entries 1–4). Interestingly, the desired product was achieved by using $Co(OAc)_2$ as the catalyst, albeit with 50% yield (Table 1, entry 5). Subsequently, a variety of commercially available chiral nitrogen-based ligands (Table 1, entries 6 and 7) and chiral phosphine ligands (Table 1, entries 8–13) were tested for this cycloaddition reaction. To our delight, it was found that duanphos **L5** offered the best reaction efficiency and selectivity, yielding the desired product **3a** with 85% yield, 95:5 *e.r.* (Table 1, entry 10). Since different anions in the cobalt salts may lead to different electronic properties, we evaluated various cobalt salts. However, the yields of **3a** were dramatically decreased in the presence of CoF_2, $CoCl_2$, $CoBr_2$, or CoI_2 as the cobalt source (Table 1, entries 14–17) compared to $Co(acac)_2$ (Table 1, entry 18). Additionally, $Co(OTf)_2$ also showed a slight decrease both in yield and stereoselectivity (75% yield, 87:13 *e.r.*) (Table 1, entry 19). Different solvents, including acetonitrile, toluene, dioxane, chlorobenzene, cyclopentyl methyl ether, and 2-methyl tetrahydrofuran, were tested to optimize the stereoselectivity (Table 1, entries 20–25). The results revealed that excellent efficiency and stereoselectivity (87% yield, 97:3 *e.r.*) could be achieved by using 2-methyl tetrahydrofuran (Table 1, entry 25). It is worth noting that the reaction failed to engage without the addition of either Zn or ZnI_2. In addition, we have identified that temperature has a great impact on enantioselectivity of desired product, 50 °C was found to be the best reaction temperature, the enantioselectivity of desired product decreased dramatically with higher temperature.

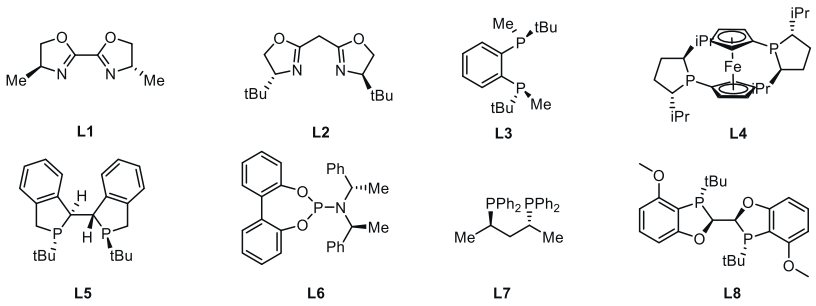
With the optimal reaction conditions in hand, the generality of this procedure for the stereoselective synthesis of axial-chiral 2-arylpyridines was investigated. Both alkyl nitriles and aryl nitriles were successfully worked in this reaction, forming the desired products in good yields and enantioselectivities under standard conditions with the same procedure (Scheme 2). In addition to simple acetonitrile, the nitriles with different functional groups such as ester, boronic ester, sulfone, cyano, and indole moieties could all produce chiral 2-arylpyridines **3c-3g** in good yields and enantioselectivities. Furthermore, arylnitriles bearing electron-donating and electron-withdrawing groups on the *para*- or *meta*-positions of the phenyl ring also participated in this reaction smoothly, producing species **3h-3m** in 48%–82% yields with 95:5–97:3 *e.r.* More importantly, nitriles with heterocycles such as furan or thiophene successfully achieved the corresponding cycloaddition products **3n**, **3o** with 74%, 71% yields in 97:3 *e.r.* In addition, 3-cyano flavone was also involved in this reaction with good enantioselectivity, albeit with a lower yield. The absolute configuration of **3g** was unambiguously assigned by single-crystal X-ray diffraction analysis.

The substrate scope of diynes **1** is shown in Scheme 3. The diynes containing (hetero)cyclic or open-chain structure (R^1) delivered the desired products **4a-4h** in high yields and enantioselectivities. However, when R^1 was replaced with benzene rings, the yield of product **4i** remained well, however the enantioselectivity decreased dramatically. Chiral 2-arylpyridine products **4j**, **4k** bearing alkene, and 3-acetal group on 6-position of naphthalene ring were achieved in 70%, 80% yield and 97:3, 95:5 *e.r.*, respectively. The diynes attached with an aryl group on the 6-position of naphthalene could proceed reactions efficiently, yielding the products **4l-4p** in good efficiency. The diyne with a pyridinyl group also proceeded this [2+2+2] cycloaddition reaction smoothly, affording the desired product **4q** with good enantioselectivity, albeit with a lower yield. Furthermore, the diyne installing a methylquinoline moiety could produce product **4r** with good yields and moderate enantioselectivity. Additionally, substrates containing longer alkyl chains at R^3 produced good yields and stereoselectivities with the axial chiral 2-arylpyridine products **4s**, **4t**. When R^3 is a phenyl group on the diyne, both the reactivity and enantioselectivity were largely affected, only affording the product **4u** in 35% yield, 61:39 *e.r.* Variation of substituents on 2-position of naphthalene ring led to the desired products **4v**-**4x** with poor enantioselectivity.

To further showcase the practicability of the protocol, a large-scale reaction was demonstrated by using 2.0 mmol dienyne **1a**

Table 1
Optimization of reaction conditions.[a]



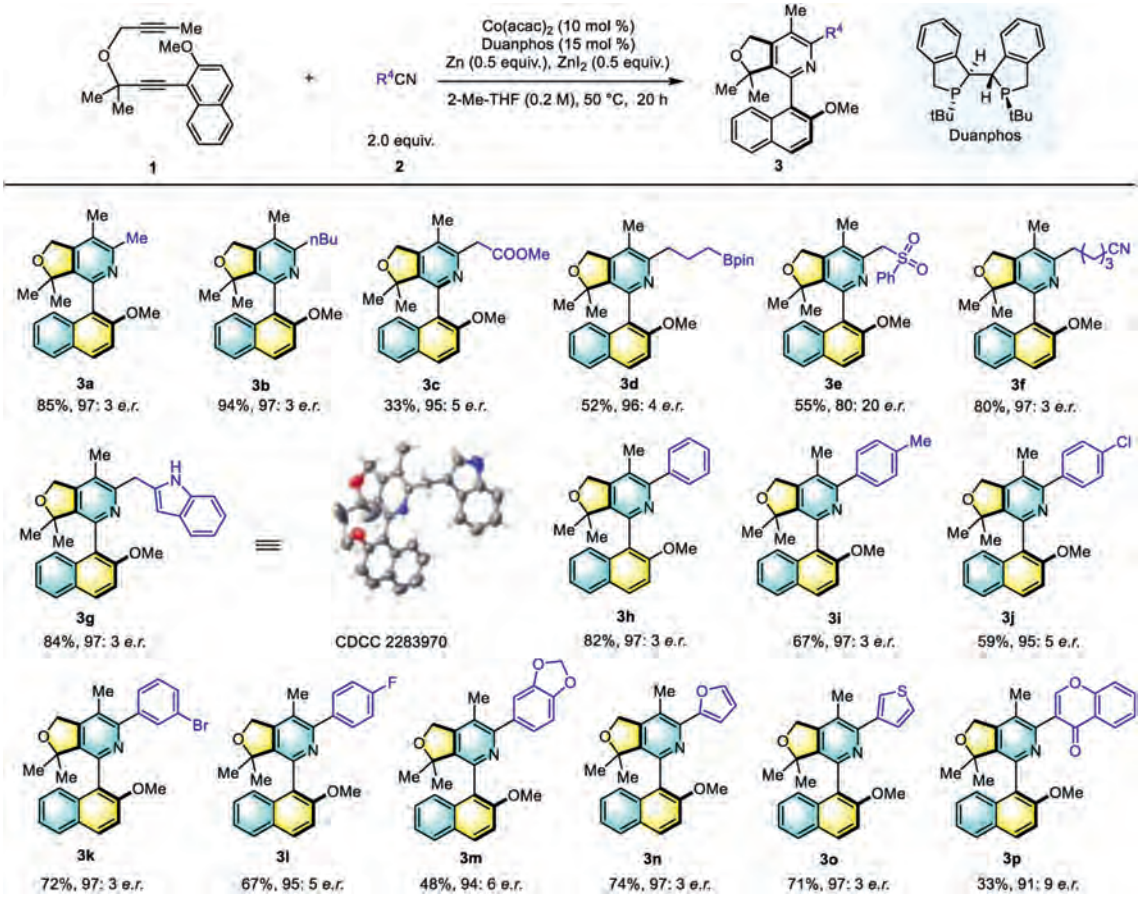


Entry	Ligand	[M]	Solvent	Yield (%)[b]	*e.r.* [c]
1[d]	DPPP	$Ni(cod)_2$	THF	<2	–
2	DPPP	$NiCl_2$	THF	<2	–
3	DPPP	$FeBr_2$	THF	<2	–
4[d]	DPPP	$[Rh(cod)_2]BF_4$	THF	<2	–
5	DPPP	$Co(OAc)_2$	THF	50	–
6	**L1**	$Co(OAc)_2$	THF	65	75:25
7	**L2**	$Co(OAc)_2$	THF	51	50:50
8	**L3**	$Co(OAc)_2$	THF	24	60:40
9	**L4**	$Co(OAc)_2$	THF	24	52:48
10	**L5**	$Co(OAc)_2$	THF	85	95:5
11	**L6**	$Co(OAc)_2$	THF	12	67:33
12	**L7**	$Co(OAc)_2$	THF	64	56:44
13	**L8**	$Co(OAc)_2$	THF	<2	–
14	**L5**	CoF_2	THF	57	95:5
15	**L5**	$CoCl_2$	THF	57	94:6
16	**L5**	$CoBr_2$	THF	48	96:4
17	**L5**	CoI_2	THF	48	95:5
18	**L5**	$Co(acac)_2$	THF	85	97:3
19	**L5**	$Co(OTf)_2$	THF	75	87:13
20	**L5**	$Co(acac)_2$	MeCN	57	90:10
21	**L5**	$Co(acac)_2$	Toluene	59	92.5:7.5
22	**L5**	$Co(acac)_2$	Dioxane	59	95:5
23	**L5**	$Co(acac)_2$	Chlorobenzene	44	94:6
24	**L5**	$Co(acac)_2$	CPME	35	92.5:7.5
25	**L5**	$Co(acac)_2$	2-Me-THF	87	97:3
26[d]	**L5**	$Co(acac)_2$	2-Me-THF	0	–
27[e]	**L5**	$Co(acac)_2$	2-Me-THF	0	–
28[f]	**L5**	$Co(acac)_2$	2-Me-THF	85	94:6
29[g]	**L5**	$Co(acac)_2$	2-Me-THF	85	57:43

[a] **1a** (0.1 mmol, 1.0 equiv.), **2a** (0.2 mmol, 2.0 equiv.), [M] (0.010 mmol, 10 mol%), ligand (0.015 mmol, 15 mol%), Zn (0.05 mmol, 0.5 equiv.), ZnI_2 (0.05 mmol, 0.5 equiv.) in solvent (0.5 mL, 0.2 mol/L) at 50 °C for 20 h under N_2 atmosphere. DPPP: 1,3-Bis(diphenylphosphino)propane.
[b] Yield was determined by GC and *n*-dodecane was used as the internal standard.
[c] Determined by chiral HPLC.
[d] Reaction without Zn.
[e] Reaction without ZnI_2.
[f] Reaction temperature is 60 °C.
[g] Reaction temperature is 100 °C.

and 4.0 mmol acetonitrile **2a** to give 587 mg of the corresponding axial-chiral 2-arylpyridines **3a** (Scheme 4A, left). To demonstrate the utility of this strategy, several transformations of **3a** were conducted (Scheme 4B). Firstly, the pyridine moiety of **3a** could be readily oxidized using *m*-CPBA, generating N-oxide containing product **5** in high yield and good stereoselectivity (78 % yield, 95:5 *e.r.*), which was subsequently demethylated using $HPPh_2$ and *t*BuOK to produce the naphthol product **6** in a moderate yield with excellent stereoselectivity (53 % yield, 96:4 *e.r.*). It should be noted that using **3a** as the starting material to do demethylation reaction, a complete racemization issue occured, yielding racemic naphthol product. Considering the importance of chiral bispyridines in organic synthesis [63], we also adopted this simple protocol to synthesize the bisaxial-chiral 2-arylpyridines skeleton **7** using 1,5-dicyanopentane as the nitrile source (Scheme 4C).

We performed DFT calculations to reveal the mechanism of this Co-catalyzed asymmetric [2 + 2 + 2] cycloaddition to clarify the origin of regioselectivity. Importantly, all the transition states and intermediates calculated in the catalytic cycle are triplet states. The unfavorable singlet free-energy profiles are depicted in Scheme S1 (Supporting information). As shown in Scheme 5, The diyne coordinated triplet cationic Co(I) species ^{3}**INT1** was used as the active

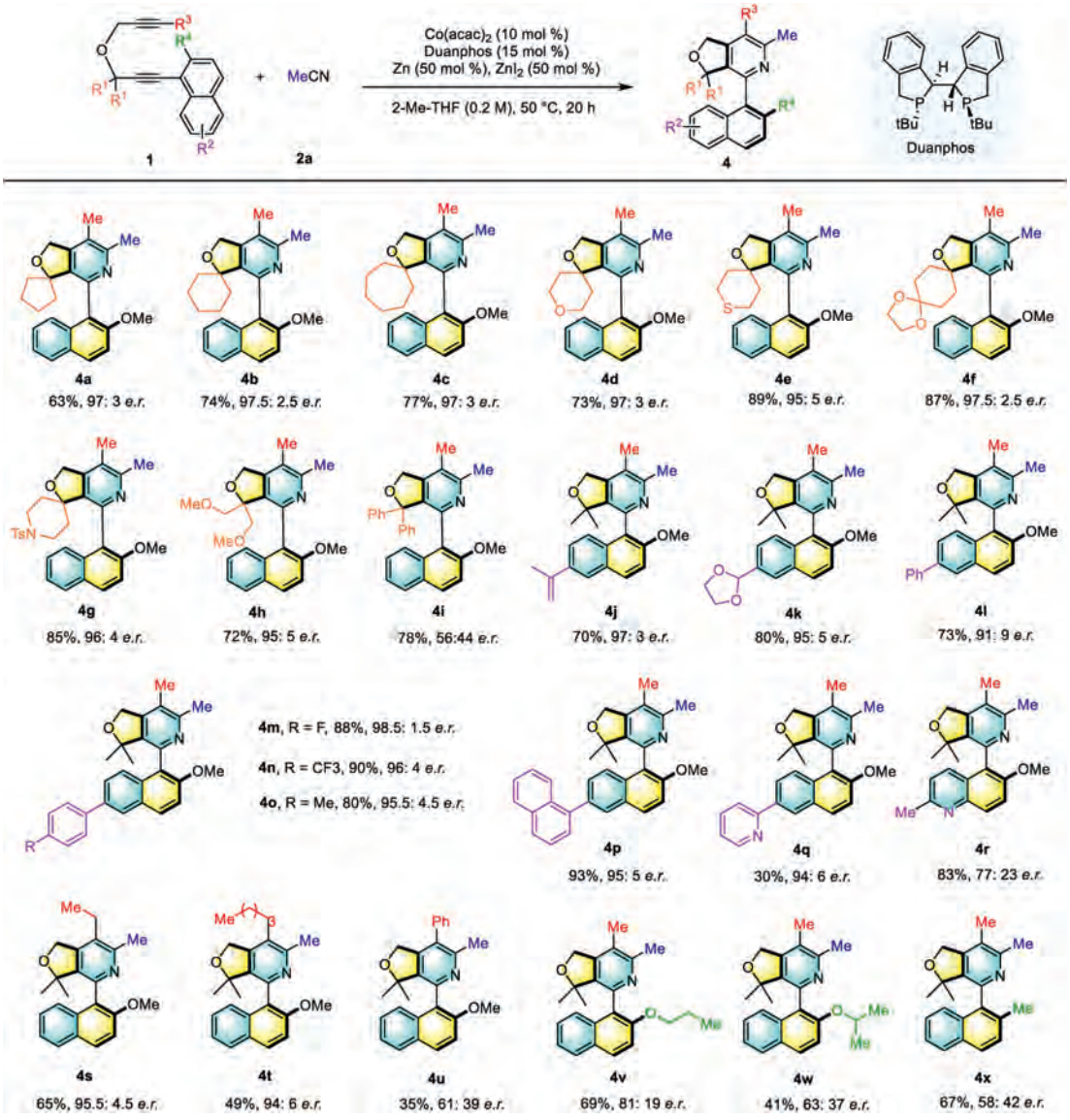


Scheme 2. Substrate scope of Co-catalyzed [2 + 2 + 2] cycloaddition reaction. Reaction conditions: **1** (0.2 mmol, 1.0 equiv.), **2** (0.4 mmol, 2.0 equiv.), $Co(acac)_2$ (0.020 mmol, 10 mol%), duanphos (0.030 mmol, 15 mol%), Zn (0.1 mmol, 0.5 equiv.), ZnI_2 (0.1 mmol, 0.5 equiv.) in 2-Me-THF (1.0 mL, 0.2 mol/L) at 50 °C for 20 h under N_2 atmosphere. Isolated yields; *er* values were determined by HPLC using chiral columns. The absolute configuration of **3g** was confirmed by X-ray.

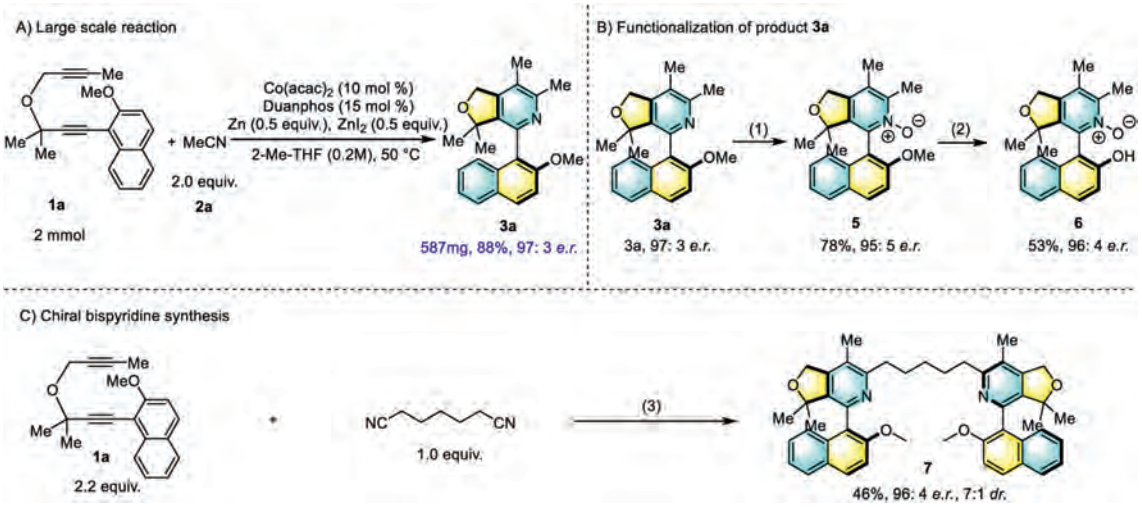
catalytic complex for the catalytic cycle. The oxidative cyclization occurred *via* transition state **^{3}TS1** with a free energy barrier of 13.2 kcal/mol to give the cyclometallic triplet intermediate **^{3}INT2**. The length of the forming C–C bond in **^{3}TS1** is 1.97 Å. The formation of **^{3}INT2** is exergonic by 2.2 kcal/mol, where the oxidative state of cobalt center is increased to +3. The following cyano migratory insertion would occur *via* transition state **^{3}TS2** or **^{3}TS2′**, respectively, with a free energy barrier of 24.4 or 37.5 kcal/mol. The calculated results indicated that cyano insertion into methyl alkenyl cobalt site is favorable, compared to the naphthylalkenyl part, which dominates the origin of regioselectivity. The steric repulsion of nitrile with naphthyl contributed to the high free energy barrier of **^{3}TS2′**. After this step, a seven-membered cobaltacycle **^{3}INT3** was formed. The axial-chiral 2-arylpyridines product **3a** was generated by C–N reductive elimination *via* transition state **^{3}TS3** with energetic span of 3.6 kcal/mol. The length of the forming C–N bond is 2.32 Å. The atroposelective product **3a** was predicted as the major product in our DFT calculation, which is coincident with experimental observations. Furthermore, the [4 + 2] cycloaddition pathway has also been considered. As shown in Scheme 5, the [4 + 2] cycloaddition transition state **^{1}TS4** entailed a free energy barrier of 56.4 kcal/mol, which is 32.0 kcal/mol higher than that of **^{3}TS2**. The calculation results showed that the [4 + 2] cycloaddition pathway was unfavorable.

From the above results and previous transition metal- catalyzed [2 + 2 + 2] cycloaddition reports [64–67], the proposed mechanisms for this cobalt-catalyzed [2 + 2 + 2] cycloaddition of the diynes **1** with nitriles **2** are depicted in Scheme 6. Initially, the cationic Co(I) species is generated *in-situ* from the reduction of Co(II) with zinc powder and ZnI_2 before it was coordinated with the diyne **1a**, followed by cyclization to form the cationic Co(III) intermediate **A** [68–73]. Subsequently, acetonitrile **2a** coordinated with **A** to give the cationic Co(III) complex **B**. Complex **B** undergoes migratory insertion to generate the Co(III) intermediate **C**, followed by reductive elimination to give the final product **3a** while the reactive cationic Co(I) species was released for the next catalytic cycle. Due to the steric hindered interaction between the aryl group in **A** and the methyl group in acetonitrile **2a**, cyano insertion into naphthyl alkenyl part is unfavourable.

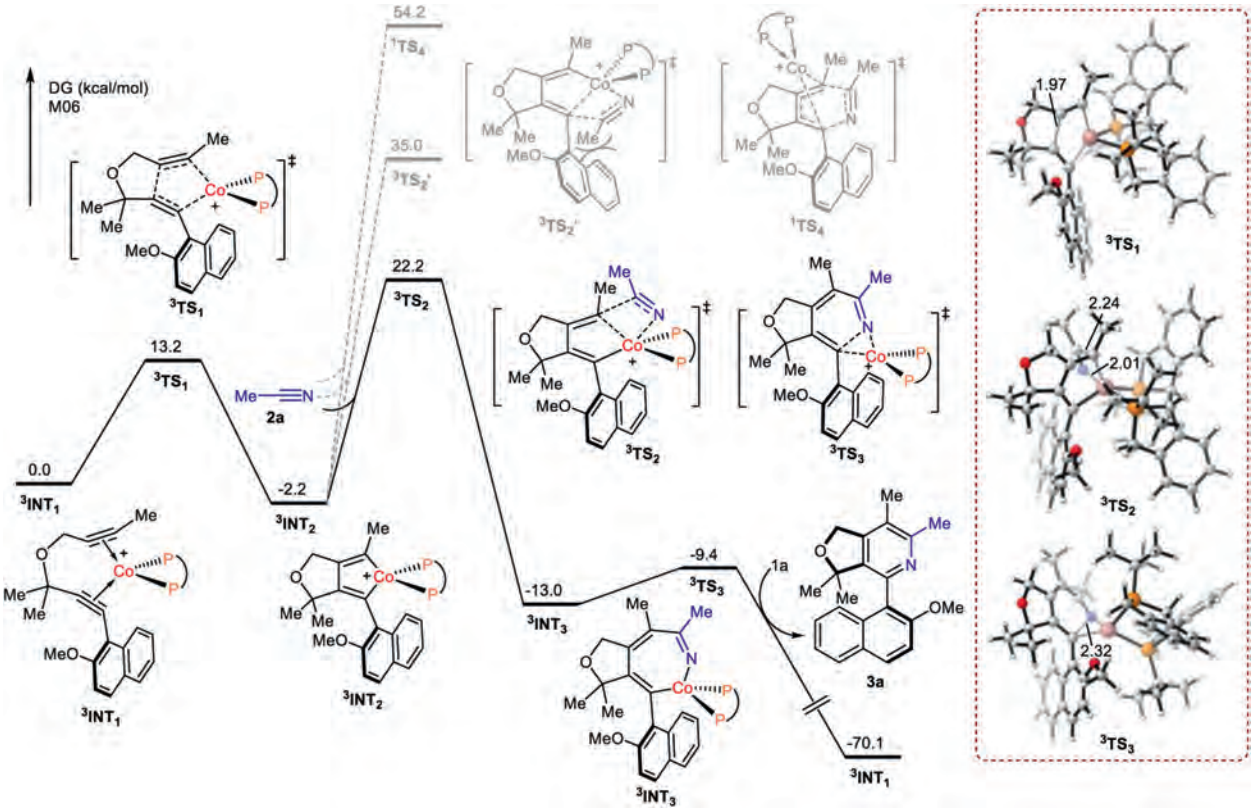
In summary, we have developed an efficient approach to synthesis of polyfunctionalized axial-chiral 2-arylpyridines through asymmetric [2 + 2 + 2] cycloaddition of diynes with nitriles using commercially available cobalt catalyst system. A wide range of axial-chiral 2-arylpyridines products with highly diverse substitution patterns have been achieved in high efficiency by using 2-methyl tetrahydrofuran as a green solvent. This reaction exhibits high modularity, excellent stereoselectivity and regioselectivity, good functional tolerance. The gram-scale synthesis also demonstrated its practicability. Furthermore, the efficient accessing axial-chiral 2-pyridine *N*-oxide and chiral bispyridine skeletons were also accomplished. The DFT calculation profiles have elucidated the mechanism of this cobalt-catalyzed asymmetric cycloaddition reaction and clarified the origin of regioselectivity. Further applications of this simple cobalt catalysis system to other [2 + 2 + 2] cycloaddition for synthesis of novel nitrogen containing chiral molecules are currently underway in our laboratories.



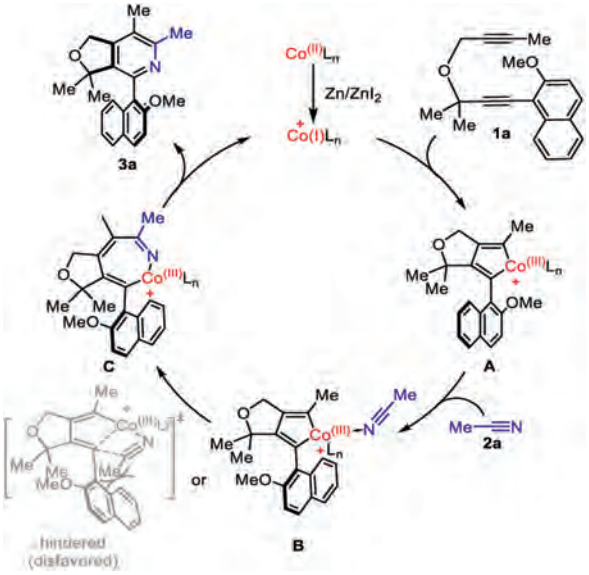
Scheme 3. Substrate scope of Co-catalyzed [2 + 2 + 2] cycloaddition reaction. Reaction conditions: **1** (0.2 mmol, 1.0 equiv.), **2** (0.4 mmol, 2.0 equiv.), $Co(acac)_2$ (0.020 mmol, 10 mol%), duanphos (0.030 mmol, 15 mol%), Zn (0.1 mmol, 0.5 equiv.), ZnI_2 (0.1 mmol, 0.5 equiv.) in 2-Me-THF (1.0 mL, 0.2 mol/L) at 50 °C for 20 h under N_2 atmosphere. Isolated yields; *er* values were determined by HPLC using chiral columns.



Scheme 4. Large scale reaction and applications of products. (1) *m*-CPBA, CH_2Cl_2, 0 °C to r.t., 1 h; (2) $HPPh_2$, *t*BuOK, DMF, 50 °C, 12 h; (3) $Co(acac)_2$, (1*S*,1′*S*,2*R*,2′*R*)-duanphos, Zn, ZnI_2, 2-Me-THF, 50 °C, 72 h.



Scheme 5. Free-energy profiles and structure information for the Co-catalyzed asymmetric [2 + 2 + 2] cycloaddition. Bond lengths are labeled with angstroms (Å).



Scheme 6. Proposed catalytic cycle.

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

Zhen-Qi Wang: Investigation, Conceptualization. **Lin-Wen Wei:** Writing – original draft, Methodology, Investigation, Formal analysis. **Zhao-Qing Wang:** Methodology, Investigation, Formal analysis. **Yan-Jie Yang:** Investigation. **Yu Zhao:** Writing – review & editing. **Song Liu:** Software, Investigation. **Yuan Huang:** Writing – review & editing, Project administration, Investigation, Funding acquisition, Conceptualization.

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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.2025.111377.

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