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Diastereodivergent [4+2] annulation of biphenylenes with enones *via* nickel(0)-catalyzed C–C bond activation

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ABSTRACT

Ni(0)-catalyzed regio- and diastereodivergent [4+2] annulation of biphenylenes with α,β unsaturated ketones is described. This solvent-controlled diastereodivergent reaction integrates C–C bond cleavage of biphenylene and C=C double bond insertion selectivity, offering a mild approach to all possible diastereoisomers of 9,10-dihydrophenanthrene derivatives from the same starting materials.

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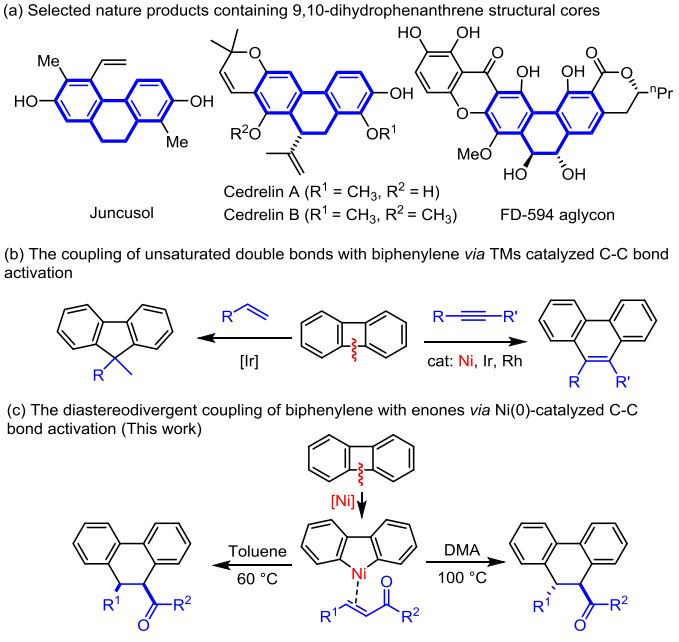
9,10-Dihydrophenanthrene derivatives are not only ubiquitous motifs in the natural products and bioactive compounds, but also important synthetic intermediates [1–6]. For example, Juncusol was isolated from the nature plant and exhibit anticancer and antimicrobial activity [7–9]. Cedrelin A was isolated from the bark of *Cedrelinga catenaeformis* Duke and has cytotoxic activity against Staphylococcus aureus and Bacillus subtilis [10–13]. The natural product FD-594 aglycon containing the *trans*-dihydrophenanthrene-diol units (Scheme 1a) [14–16]. Although 9,10-dihydrophenanthrene could be synthesized through the transformation of aryns [17–20], transition-metal catalyst annulation reactions [21–26] and others [27–31], the divergent synthesis of these structures from the same starting materials just with different reaction conditions will be much more interesting and attractive.

Transition-metal catalyzed C–C bond activation provide an efficient and straightforward approaches for the construction of complex organic structures with perfect atom- and step-economy [32–41]. Small strained rings were usually used for the cleavage of C–C bond in presence of transition-metal catalysis, and various attractive synthetic transformations have been reported [36].



Scheme 1. Transition-metal catalyzed C–C functionalization of biphenylene.

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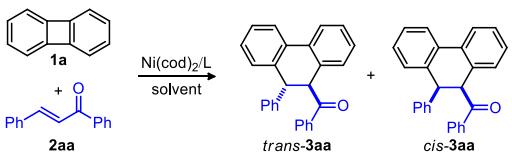
Biphenylenes are an attractive four-member carbon ring for the C–C bond cleavage and usually used as C4 synthon for the annulation reaction with many types of unsaturated units [42–53]. For instance, the alkynes were frequently used in the [4 + 2] annulation reaction with biphenylene to give phenanthrenes in presence of nickel [42–47], iridium [48] or rhodium catalysis [49]. Although, other unsaturated components such as alkenes [50], CO [51] and isocyanides [52,53] were also successfully applied in the ring-opening of biphenylene, the intermolecular coupling of unsaturated double bonds to give the 9,10-dihydrophenanthrene derivatives is still undeveloped (Scheme 1b). In 2016, the Shibata group developed the iridium-catalyzed C–C activation of biphenylenes and coupling with alkenes to give [4 + 1] cycloaddition products [54]. As part of our continuing interest in the intermolecular coupling system through C–C activation using low-cost earth-abundant nickel catalysis [55–57]. Herein, we report the nickel-catalyzed diastereodivergent formal [4 + 2] annulation between biphenylenes and enones, where the C=C bond participated regioselectively to afford *trans* or *cis* diastereoisomer of 9,10-dihydrophenanthrenes just with different reaction conditions (Scheme 1c). Despite the tremendous progress developed in the transition-metal catalyst C–C bond activation, such solvent-controlled diastereodivergent coupling systems for the diverse synthesis of small organic molecular is still rarely reported. This diastereodivergent system would increase the competitiveness and sustainability of transition-metal catalyzed C–C activation.

General procedure A: The mixture of **1a** (0.10 mmol), **2** (0.10 mmol), $Ni(cod)_2$ (1.4 mg, 0.005 mmol), PPh_3 (2.6 mg, 0.01 mmol), and DMA (1.0 mL) were charged into a reaction tube. The reaction mixture was stirred at 100 °C heated by metal sand bath for 18 h. After the reaction completed (18 h), the reaction mixture was extracted with ethyl acetate (2.0 mL) and saturated NaCl aqueous solution (3 × 4.0 mL). The combined organic layers were dried over Na_2SO_4 and filtrated, concentrated, and purified by silica gel chromatography using PE/EA (10:1) to afford *trans*-**3**.

General procedure B: The mixture of **1a** (0.10 mmol), **2** (0.10 mmol), $Ni(cod)_2$ (1.4 mg, 0.005 mmol), PPh_3 (2.6 mg, 0.01 mmol), and toluene (1.0 mL) were charged into a reaction tube. The reaction mixture was stirred at 60 °C heated by metal sand bath for 18 h. After the reaction completed (18 h), the reaction mixture was filtered through a pad of celite, eluted with ethyl acetate, concentrated, and purified by silica gel chromatography using PE/EA (10:1) to afford *cis*-**3**.

We selected biphenylene **1a** and chalcone **2aa** as model substrates to explore the reaction parameters (Table 1). The issues of both *chemo*- and diastereoselectivity arised when coupling with unsaturated alkenes comparing to the alkynes. It was found that the reaction delivered to the double bond insertion selectivity *trans*-product **3aa** in 90% yield and excellent diastereoselective ratio (*trans/cis* = > 20:1) with $Ni(cod)_2/PPh_3$ catalysis using the DMA as solvent (entry 1). When decreasing the temperature to 60 °C in the DMA solvent, the *trans/cis* value decreased to 9:1 (entry 2). Further study showed that the solvent effect was dramatic (entries 3–9). Low diastereoselective ratio was obtained when the DMF was used as solvent (entry 3). The starting materials were recovered with DCE as solvent (entry 4). Very interestingly, the DME, MeO^{t}Bu, THF or dioxane gave another isomer of the [4 + 2] annulation product *cis*-product **3aa** in excellent yield, even with low diastereoselective ratio (entries 5–8). To our delight, excellent diastereoselectivity could be obtained for *cis*-**3aa** when the toluene was used as solvent (entry 9, 72% yield, *trans/cis* = 1:9), higher yield and dr value were obtained when the reaction temperature decreased to 60 °C (entry 10, 88% yield, *trans:cis* = 1: > 10), further decrease the temperature to 30 °C in toluene led to low efficiency (entry 11). Up to now, the solvent-controlled diastereodivergent [4 + 2] cycloaddition system was established, in which the

Table 1
Optimization of reaction conditions.[a]

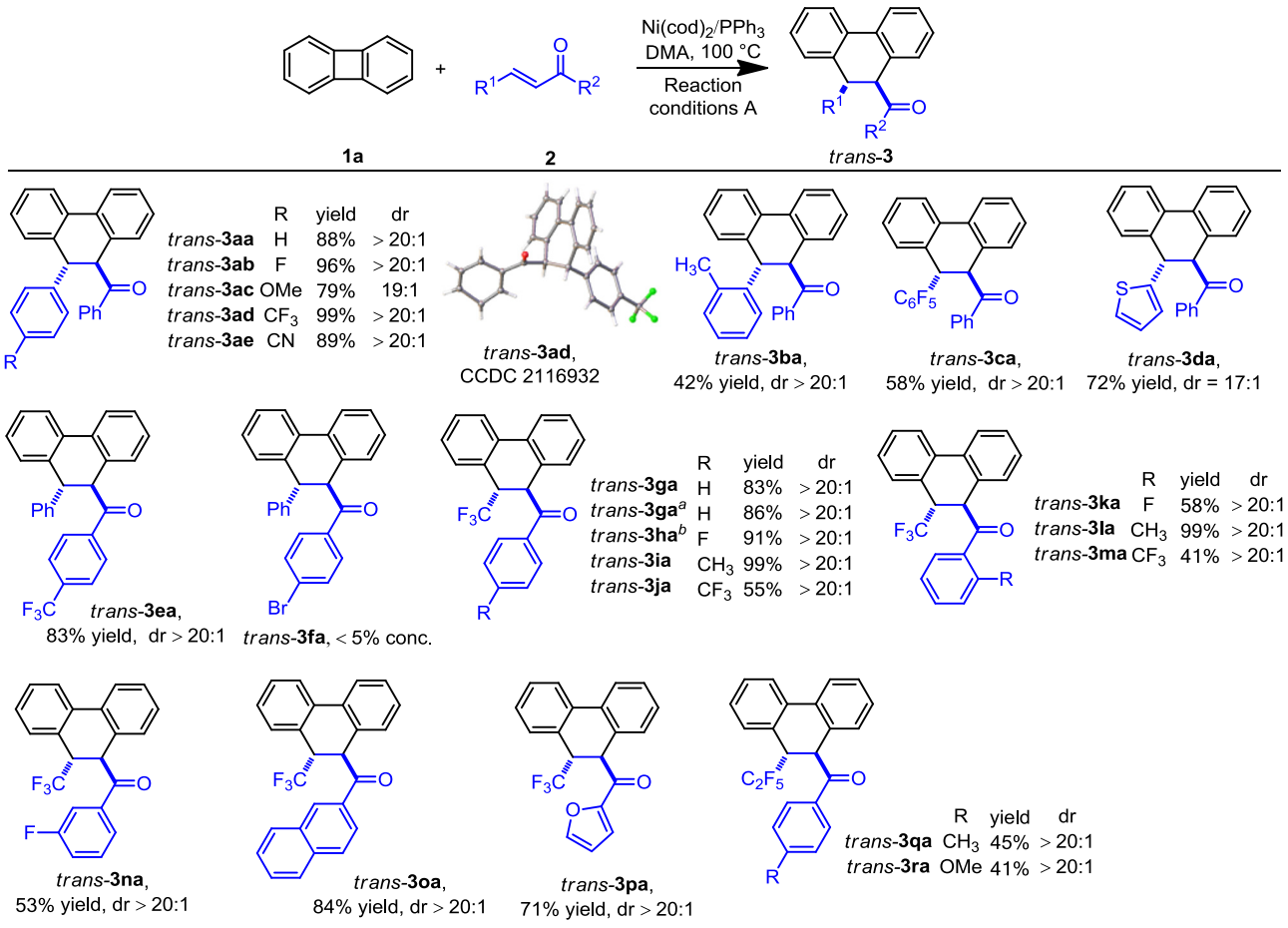


Entry	Solvent	L	T (°C)	Yield (%)	dr (*trans/cis*)
1	DMA	PPh_3	100	90	>20:1
2	DMA	PPh_3	60	99	9:1
3	DMF	PPh_3	100	90	8:1
4	DCE	PPh_3	100	trace	–
5	DME	PPh_3	100	94	1:5
6	MeO^{t}Bu	PPh_3	100	93	1:3
7	THF	PPh_3	100	91	1:3
8	Dioxane	PPh_3	100	99	1:6
9	Toluene	PPh_3	100	72	1:9
10	Toluene	PPh_3	60	88	1:>10
11	Toluene	PPh_3	30	trace	–
12	Toluene	Bpy	100	trace	–
13	Toluene	*rac*-BINAP	100	trace	–
14	Toluene	–	60	trace	–
15	DMA	–	100	trace	–
16[b]	DMA	PPh_3	60	NR	–

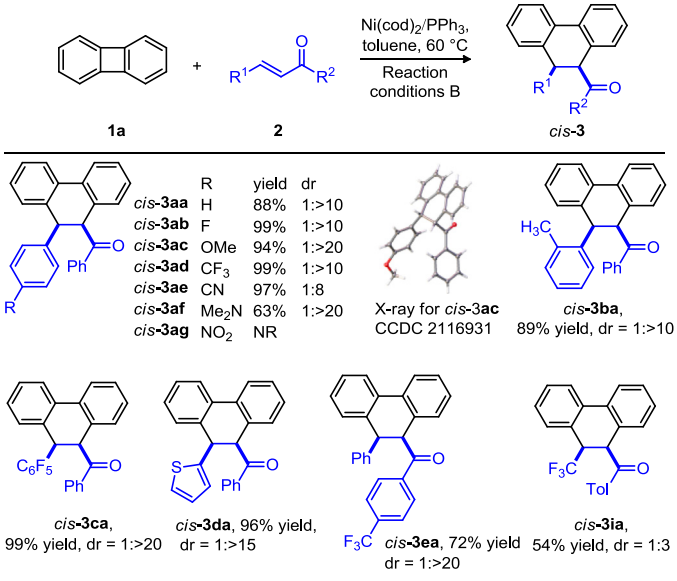
[a] Reaction conditions: **1a** (0.1 mmol), **2aa** (0.1 mmol), $Ni(cod)_2$ (5 mol%), L (10 mol%) in solvent (1.0 mL) under argon, 18 h.
[b] w/o $Ni(cod)_2$.

trans-**3aa** was obtained in polar solvent DMA (entry 1, 90% yield, *trans:cis* = > 20:1) and *cis*-**3aa** was obtained in toluene (entry 10, 88% yield, *trans:cis* = 1: > 10). Noteworthy, these reaction conditions only afforded the C=C insertion products. Other ligands such as Bpy or *rac*-BINAP did not promote the annulation (entries 12 and 13), and no corresponding products observed without the addition of PPh_3 (entries 14 and 15). Our control experiments also showed that the $Ni(cod)_2$ was essential for the transformation (entry 16).

We next explored the scope of this divergent coupling system (Scheme 2). The generality of *trans*-products synthesis was investigated firstly. The reaction tolerated with the electron-donating or -withdrawing groups at the *para* position of the benzene ring in the enones with excellent regio- and diastereoselectivity (*trans*-**3aa** to *trans*-**3ae**, 79%–99% yield, dr = 19:1 to > 20:1). The configuration of *trans*-**3ad** was determined by X-ray crystallography (CCDC: 2116932). The reaction also worked smoothly for *ortho*-substituted methyl group (*trans*-**3ba**, 42% yield, dr > 20:1) and for pentafluorobenzyl-substituted enones (*trans*-**3ca**, 58% yield, dr > 20:1). The thiophenyl-substituted enone also worked well to give the product *trans*-**3da** in 72% yield and 17:1 dr. The reaction for the electron-withdrawing group CF_3 at *para*-position of the benzene ring gave the corresponding product *trans*-**3ea** in 83% yield and > 20:1 dr. The bromide-substituted enone did not give desired product (*trans*-**3fa**, < 5% conc.). To our delight, when extension of the chalcones to the β-CF_3 substituted enones, the reaction also worked smoothly to give the desired products. Various electron-donating and -withdrawing groups at the different position of the benzene ring tolerated (*trans*-**3ga** to *trans*-**3na**, 41%–99% yield and dr > 20:1), When the *cis*-enone was used, similar results with *trans*-enone obtained probably owing to the isomerization of *cis*-**2ga** to *trans*-**2ga** in the catalyst systems (*trans*-**3ga**, 86% yield, dr > 20:1). The naphthyl- or furyl-substituted enones all coupled with biphenylene gave the [4 + 2] annulation product with excellent diastereoselectivity (*trans*-**3oa**, 84% yield, dr > 20:1 and *trans*-**3pa**, 71% yield and dr > 20:1). The β-C_2F_5 substituted enones gave the corresponding products with a slight low yield (*trans*-**3qa**, 45%



Scheme 2. Scope of substrates for the synthesis of *trans*-products. Reaction conditions A: Reaction conditions: **1a** (0.1 mmol), **2** (0.1 mmol), $Ni(cod)_2$ (5 mol%), PPh_3 (10 mol%), in DMA (1.0 mL), 18 h, 100 °C, argon. [a]from the *cis*-enone substrates. [b]$Ni(cod)_2$ (5 mol%), PPh_3 (5 mol%) in DMA (1.0 mL), dr = the ratio of *trans*/*cis*.



Scheme 3. Scope of substrates for the synthesis of *cis*-products. Reaction conditions B: **1a** (0.1 mmol), **2** (0.1 mmol), $Ni(cod)_2$ (5 mol%), PPh_3 (10 mol%), in toluene (1.0 mL), 18 h, 60 °C, argon. dr = the ratio of *trans*/*cis*.

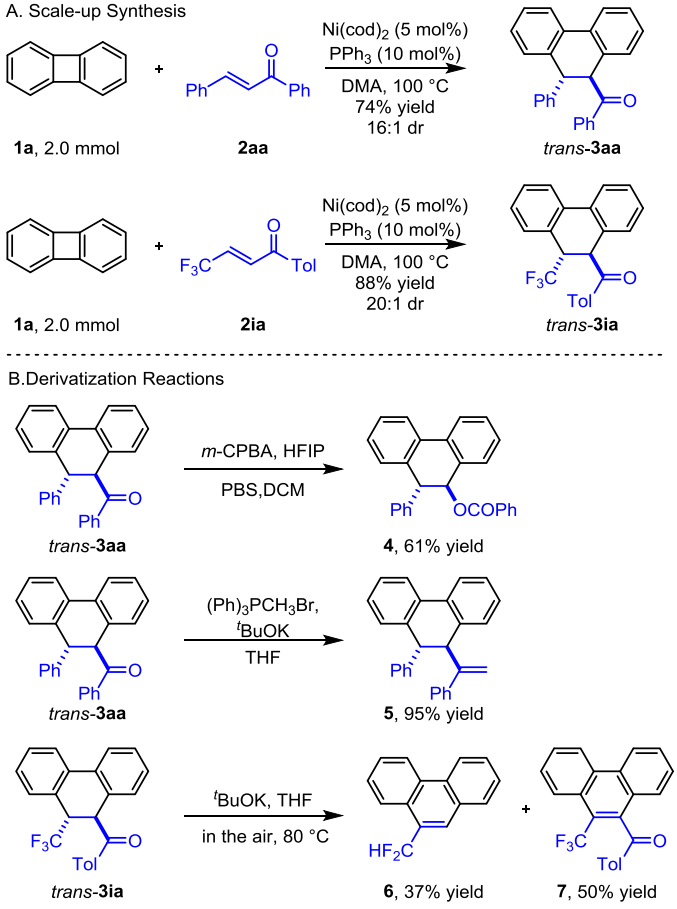
yield, dr > 20:1 and *trans*-**3ra**, 41% yield, dr > 20:1). However, the ethyl cinnamate was found to be unreactive.

We next explored the generality of the *cis*-products formation system (Scheme 3). To our delight, introduction of halogen-, MeO-, CF_3-, CN-, Me_2N- group into the *para* position of the β-phenyl ring in the chalcone and other chalcones afforded the desired *cis*-products with excellent yield and diastereoselective ratio (*cis*-**3aa** to *cis*-**3ea**, 63%–99% yield, dr = 1:8 to 1:>20). The configuration of *cis*-**3ac** was determined by X-ray crystallography (CCDC: 2116931). Only starting materials were recovered with the NO_2 group substituted enone (*cis*-**3ag**). While, when the CF_3-substituted enone was employed, the [4 + 2] cycloaddition product *cis*-**3ia** was obtained in 54% yield with low diastereoselectivity (dr = 1:3).

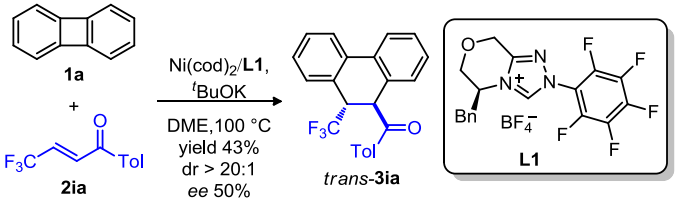
The synthetically useful of this [4 + 2] annulation system was briefly investigated (Scheme 4). The coupling of **1a** and **2aa** was readily scaled up to 2.0 mmol, affording the product *trans*-**3aa** in excellent yield and diastereoselectivity. Similarly, the β-CF_3 substituted enone **2ia** could also be scale up to 2.0 mmol (Scheme 4A). The ester **4** was obtained in 61% yield through Baeyer-Villiger oxidation of *trans*-**3aa**. Wittig reaction of *trans*-**3aa** gave the olefination product **5** in 95% yield. These two transformations with the retention of the *trans* configuration. Interestingly, when the CF_3-substituted dihydrophenanthrene *trans*-**3ia** was treated with ^{t}BuOK, CF_2H-substituted phenanthrene **6** (37% yield) and CF_3-substituted phenanthrene **7** (50% yield) were obtained in one pot under the air atmosphere (Scheme 4B).

We initially realized the enantioselective variant of this C–C activation system of biphenylene using β-CF_3 substituted enone **2ia** as coupling partner (Scheme 5). With the chiral NHC ligand **L1**, the desired [4 + 2] annulation product *trans*-**3ia** was obtained in 43% yield and 50% *ee*.

To determine the kinetic and thermodynamic control property of the reaction, we studied the isomerization of the two



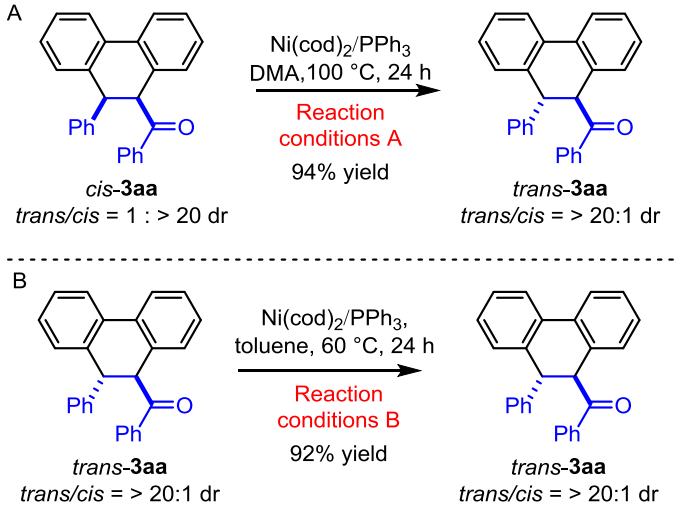
Scheme 4. Scale-up synthesis and derivatization.



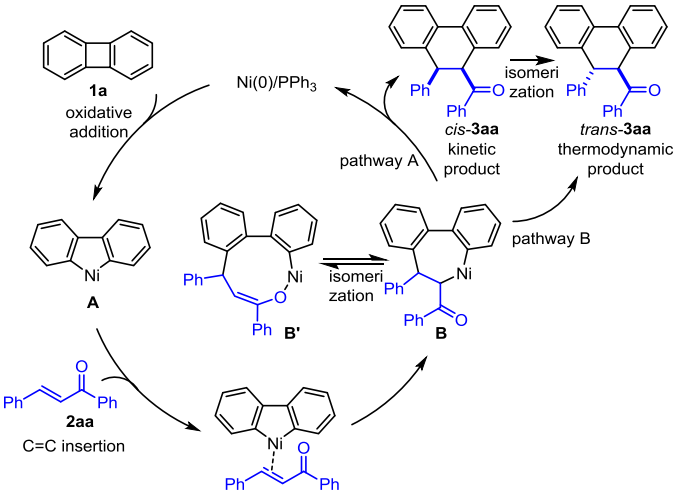
Scheme 5. Enantioselective coupling system.

diastereoisomers of products (Scheme 6). When *cis*-**3aa** was added to the *trans*-**3aa** formation conditions (Reaction conditions A), the *cis*-**3aa** isomerized to *trans*-**3aa** with >20:1 dr value (Scheme 6A). While *tran*-**3aa** could not isomerize to *cis*-**3aa** under the *cis*-products formation conditions (Reaction conditions B) (Scheme 6B). These results indicated the *trans*-**3aa** would be the thermodynamic product, and *cis*-**3aa** would be the kinetic product.

On the basis of previous reports on the transition-metal catalyzed ring-opening of biphenylenes [32–51], a plausible pathway is proposed (Scheme 7). Oxidative addition of biphenylene **1a** to Ni(0) afforded the intermediate **A**. The double bond of enone coordinated to the nickel center, which is proposed to undergo Ni-C insertion into the C=C bond of **2aa** to generate the intermediate **B**. The intermediate **B** could isomerize to intermediate **B'**, followed by reductive elimination to give the kinetic product *cis*-**3aa** in toluene solvent and regenerated the catalyst. The high steric hindrance for the *cis*-isomer might benefit the reductive elimination process from the intermediate **B** in the toluene solvent [58–60]. The *cis*-**3aa** could isomerized to thermodynamic product *trans*-**3aa** in DMA solvent (pathway A). Right now, we could not exclude



Scheme 6. Isomerization of the product.



Scheme 7. Proposed mechanism.

the pathway of direct generation of *trans*-**3aa** from the intermediate **B** in DMA solvent (pathway B).

In summary, we have realized Ni(0)-catalyzed diastereodivergent [4+2] annulation of biphenylene with enones *via* C–C bond activation. This reaction allows efficient access to the two diastereoisomers of the annulation products from the same starting materials just changing the reaction conditions. This solvent-controlled reaction proceeded with highly diastereoselectivity for both isomers (> 20:1 dr) and the C=C bond formally insertion chemoselectively in this cycloaddition. This intermolecular coupling system would be a straightforward strategy for the synthesis of 9,10-dihydrophenanthrene derivatives.

Declaration of competing interest

The authors declare no competing financial interests.

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.2022.02.079.

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