

## Copper-Catalyzed Borylative Cyclization of Substituted *N*-(2-Vinylaryl)benzaldimines

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**Supporting Information** 



**ABSTRACT:** A *t*-BuOCu-initiated reaction sequence of styrene borometalation and intramolecular imine addition has been achieved using a  $Cu(OTf)_2/dppf$  combination as catalyst. The product of this reaction cascade is a useful 2,3-disubstituted indoline bearing a versatile boryl moiety and is formed with sole *cis*-selectivity. To account for the observation of the exclusive formation of *cis*-stereoisomers, a transition state featuring copper—imine coordination is suggested. The application to the synthesis of antioxidant tetrahydroindenoindoles is described.

ecently, 1,2-carboboration of  $\pi$ -bonds has received Rattention, as these are transformations that could convert readily available alkenes, alkynes, or allenes into valuable, diversely functionalized organic boronates through concurrent construction of C–B and C–C bonds.<sup>1,2</sup> The C–B motif formed in such reactions can serve as a facile point for further chemical manipulation.<sup>3</sup> Carboboration of unsaturated bonds<sup>4</sup> initiated by borylcopper<sup>5</sup> has emerged as a practical means for this purpose because these reactions can proceed in a chemo-, regio-, and stereocontrolled manner by the use of properly ligated metal catalysts derived from readily available copper reagents.<sup>6,7</sup> As outlined briefly in Scheme 1, the catalytic cycle generally begins with the formation in situ of a copper alkoxide, typically copper tert-butoxide I, which undergoes transmetalation with boron compounds, such as 1 giving rise to the nucleophilic borylcopper species II. Subsequent chemoselective migratory insertion into





the double or triple bond in 3 forms the key organocopper intermediate III which couples with the carbon electrophile 4 to give IV and the carboboration product 5 with concomitant regeneration of complex I which continues the catalytic cycle. Carbon electrophiles that can trap the organocopper intermediate III include alkyl halides or sulfonates,<sup>8</sup> aryl halides,<sup>9</sup> alkyl carbonates/phosphates,<sup>10</sup>  $\alpha$ , $\beta$ -enonates,<sup>11</sup> and carbonyl groups.<sup>12</sup> Herein, we report an intramolecular carboboration reaction of aryl alkenes with an imino group to give Nheterocycles containing a C–B group.

We became interested in carboboration of alkenes with imino carbon electrophiles as a result of the utility of the boronated alkylamines. To our knowledge, there are only a few related published examples, and no intramolecular precedent has been reported.<sup>13</sup> On the other hand, Buchwald et al. in 2015 reported a CuH-catalyzed asymmetric intramolecular hydrocarbonation of alkenes with aldmines.<sup>14</sup> The model substrate *N*-(vinylphenyl)aldimine **6a** reacting with  $B_2(pin)_2$  was taken as a proof of concept and was used to optimize the reaction conditions. On the basis of earlier work, cuprous triflate Cu(OTf)<sub>2</sub> was quickly identified as a superior catalyst and t-BuOLi/t-BuOH as an optimal choice of base/proton source combination. Phosphorus ligands were found to be necessary for the reaction to proceed smoothly, giving 3-(borylmethyl)-2-phenylindoline 7a. The stereochemistry of 7a was established by NMR spectroscopy, which revealed a considerable <sup>1</sup>H-<sup>1</sup>H NOESY correlation, as shown between the methylene protons and benzyl protons. Experiments aimed at screening the reaction conditions are summarized in Table 1. In the absence of a phosphoruscontaining ligand, the imine **6a** fails to react with  $B_2(pin)_2$  (1.1)



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# Table 1. Condition Optimization for Intramolecular Carboboration of *o*-Iminostyrene<sup>a</sup>

Bpin Cu(OTf) <sub>2</sub> (10 mol %), L (10 mol %) - t-BuOL i (x equiv)/t-BuOH (1 1 equiv)				
solvent, rt, overnight				
entry	ligand	solvent	x (equiv)	yield <sup>b</sup>
1	none	THF	1.1	NR
2	dppm	THF	1.1	trace
3	dppe	THF	1.1	trace
4	dppp	THF	1.1	9%
5	dppb	THF	1.1	34%
6	Xantphos	THF	1.1	33%
7	dppf	THF	1.1	51%
8	Xphos	THF	1.1	trace
9	Johnphos	THF	1.1	trace
10	PCy <sub>3</sub>	THF	1.1	trace
11	SPhos	THF	1.1	53%
12	Davephos	THF	1.1	50%
13	dppf	THF	2.0	70%
14	dppf	THF	2.5	83%
15	dppf	THF	3.0	58%
16	dppf	CH <sub>3</sub> CN	2.5	60%
17	dppf	MTBE	2.5	55%
18	dppf	PhMe	2.5	34%
	Ph <sub>2</sub> P $(n = 0, dppm)$ n = 0, dppm n = 1, dppe n = 2, dppp n = 3, dppb	PPh <sub>2</sub> Pl XantPhos	Cy <sub>2</sub> P <sup>i</sup> Pr ph <sub>2</sub> <sup>i</sup> Pr XPhos	- <sup>i</sup> Pr
$\begin{array}{c c} & & & & \\ P('Bu)_2 & & & \\ & & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ $				

<sup>*a*</sup>Reactions were conducted on a 0.25 mmol scale in 2 mL of solvent at rt. <sup>*b*</sup>Isolated yields are reported. NR: no reaction.

equiv) at rt for 12 h (Table 1, entry 1), but a trace amount of 7a is formed after introduction of 10 mol % of a bidentate phosphorus ligand such as dppm, dppe, or dppp (Table 1, entries 2–4). The yield increases dramatically to ~33% when dppb or XantPhos is used as the ligand (Table 1, entries 5 and 6) and is further increased to 51% when dppf is used (Table 1, entry 7). While the monodentate ligands XPhos, JohnPhos, and PCy<sub>3</sub> are ineffective in this transformation (Table 1, entries 8–10), SPhos and DavePhos provide much improved outcomes (Table 1, entries 11 and 12). A detectable side reaction involves alkene hydroboration, which leads to the product from the protonation of the related copper intermediate III.

It is reasonable to predict that an increase in the *t*-BuOLi/*t*-BuOH ratio should slow the C-protonation side process and thus favor the imine addition event, which produces the desired intermediate IV. Indeed, doubling the amount of the base results in a 20% enhancement of the yield and 2.5 times the amount of *t*-BuOLi gives the highest yield of 83% (Table 1, entries 13 and 14). However, a drastic decrease in the yield, to 58%, was

observed with further increase up to a factor of 3 in the base loading (Table 1, entry 15). The reaction also proceeds in MeCN, MTBE, and toluene but is less efficient (Table 1, entries 16-18).

The highly efficient formation of 7a is remarkable given the unfavorable 5-*endo-trig* cyclization step that forms the indoline ring. With the optimal conditions in hand, the substrate scope was examined with 2-vinylanilinylaldimines 6b-q derived from the appropriate aromatic aldehydes (Scheme 2). Generally, the

Scheme 2. Substrate Scope Imines Derived from Various Aryl Aldehydes  $^{a,b}$ 



<sup>a</sup>Reactions were conducted out on a 0.25 mmol scale in 2 mL of THF. <sup>b</sup>Isolated yields are reported.

reaction affords 2,3-disubstituted borylindolines with good to excellent yields. In all cases, the cis-diastereomers are observed exclusively. Aryl halides and aryl cyanide are well tolerated with this transition-metal-catalyzed process as is exhibited by good yields in the case of 6b-e, which produces 7b-e in 57-72% yield. Substrates with 4-CF<sub>3</sub>, 4-Me, or 4-*t*-Bu at the C4 position give comparable yields (7f-g, 7i: 62-71%), while a methyloxyl group at this position produces a higher yield (7h: 81%). Substitution at the meta position has little effect on the outcomes of this transformation (7j,k: 55% and 60%), but interestingly, ortho-substitution of this ring increases the yield to approximately 80% (7l,m). Aromatic groups other than the phenyl ring are also viable in this reaction, and 2-naphthyl-, 2-pyridyl-, 2thiophene-yl-, and 2-furanylindolines 7n-q are formed with satisfactory yields (50–64%). Unfortunately, *o*-iminopropenylbenzene 6r fails undergo this transformation, probably because the borocupration step encounters increased steric problems.

Substrates with substituents in the styrene moiety were examined (Scheme 3). (*E*)-*N*-Benzylidene-2-vinylanilines 8a-c with 4-F, 4-Cl, or 4-Br at the C4 position were converted to the related boryl indolines 9a-c in yields of 58-70%. With a CH<sub>3</sub> at C4, 9d is produced in 65% yield, whereas 9e with a 4-t-Bu substituent is formed in only 40% yield from 8e. A strongly electron-donating methoxyl group at the para positon has little effect on the reaction, as demonstrated by a 58% yield obtained for 9f. A 4-phenyl group also has little influence on the reaction yield, and 9g is isolated in 61% yield. Carboxylate and nitrile groups vulnerable to nucleophiles are compatible with the borocupration–aldimine addition cascade and good yields are observed for 9h (70%) and 9i (64%). While the reaction of the

## Scheme 3. Substrate Scope-Iminostyrenes Derived from Various 2-Vinylanilines $^{a,b}$



<sup>a</sup>Reactions were conducted on a 0.25 mmol scale in 2 mL of THF. <sup>b</sup>Isolated yields are reported. <sup>c</sup>Starting materials were recovered.

4,5-dichlorinated imine **8j** gives rise to **9j** in low yield (38%), the 4-CN-5-CF<sub>3</sub>-substituted imine **8k** is an excellent substrate for the reaction and affords **9k** in 87% yield. The 6-fluorinated imine **8l** undergoes this reaction smoothly, affording the 6-F-indoline **9l** in 62% yield, while a marked decrease to 30% in the yield in the formation of **9m** occurs and is due possibly to the steric impact of the 6-methyl group on this reaction. In comparison with 6substituted imines **8l** and **8m**, 3-substitution has a more obvious steric effect on the reaction and no reaction is observed with **8o** and **8p**. It seems that electron-withdrawing groups on the styrene benefit the reaction, probably because the initial borolcuperation step is favorable for electron-deficient styrene. A satisfactory crystalline sample of **9b** was obtained, and its X-ray crystallographic analysis confirmed the validity of previous assignment of *cis*-stereochemistry of the products (Scheme 3).

We envisioned that the exclusive formation of *cis*-2,3disubstituted indolines 7 and 9 should arise from the stereoselective cyclization step, which converts the organocopper intermediate **10** into the indolyl copper **11** (Scheme 4).





Coordination of the copper atom with the  $\pi$ -orbital of the (*E*)imine group in the hypothetical transition states (*trans*-**TS** vs *cis*-**TS**) would force group R to protrude toward the imine moiety and group R' to point away from it. Accordingly, the steric repulsion between group R and the imine hydrogen would be the singular factor that could dictate the relative energetic levels of the two transition states. *Cis*-**TS** with the H atom oriented inward is more favored than its counterpart *trans*-**TS**, and this results in the observation of exclusive *cis*-selectivity. We suspect this might also account for the *cis*-selectivity reported by Buchwald et al. in the synthesis of 2,3-disubstituted indolines by hydrocarboration.  $^{\rm 14}$ 

To show the usefulness of this reaction, product 7m was submitted to a Suzuki reaction to test whether intramolecular coupling could take place (Table 2). In this case, the desired

#### Table 2. Intramolecular Suzuki Coupling of Boryl Indolines<sup>4</sup>



<sup>a</sup>Conditions for Suzuki coupling: 7 or 13a-e (1.0 equiv), Pd(OAc)<sub>2</sub> (10 mol %), Sphos (20 mol %), *t*-BuOK (3.0 equiv), toluene/H<sub>2</sub>O (10:1, 0.1 M), 80 °C, 24 h. <sup>b</sup>Isolated yields.

tetracyclic product **14m** is obtained in 92% yield when a  $Pd(OAc)_2/Sphos catalyst system is used.<sup>15</sup> Many molecules with$ *cis*-tetrahydroindenoindole scaffolds have been reported for their antioxidation activities in biological systems.<sup>16</sup> Our two-step protocol provides a facile access to this class of compounds, and additional examples are provided in Table 2. Through this two-step protocol, facile*o*-iminostyrenes**12a–e**can be converted into antioxidants**14a–e**bearing various substituents in both aromatic rings in 48–88% yields.

In summary, we reported a borometalation—imine addition cascade, which has been developed into an efficient protocol for synthesis of 2,3-disubstituted indolines. This reaction features exclusive diastereoselectivity and *cis*-2,3-disubstituted indolines carrying a boronate group can be accessed. The readily accessible copper salt used as a precatalyst causes this protocol to be extremely useful for potential applications. The products with preinstalled *o*-bromides can participate in an intramolecular Suzuki reaction to give valuable *cis*-tetrahydroindenoindoles, demonstrating the potential applications of this chemistry.

## ASSOCIATED CONTENT

#### **S** Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.8b00213.

Experimental procedures, characterization data and NMR spectra for new compounds (PDF)

#### **Accession Codes**

CCDC 1585427 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data\_request/cif, or by emailing data\_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033.

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#### Notes

The authors declare no competing financial interest.

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