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Cu-catalyzed regioselective diborylation of 1,3-enynes for the efficient synthesis of 1,4-diborylated allenes

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ABSTRACT

Borylation of 1,3-enynes with bis(boronate) compounds often ends up with the formation of hydroborylated products, leaving the diborylation of 1,3-enynes for the formation of 1,4-diborylated allenes to be challenging. Herein, a copper-catalyzed chemo-, regio-, and stereo-selective diborylation of 1,3-enynes for the efficient construction of 1,4-diborylated allenes under base-free conditions was reported. A wide range of 1,3-enynes bearing various functional groups can participant in the reaction and afforded the corresponding 1,4-diborylated allenes in good to excellent yields, which was enabled by the protocol of Bpin to BF_3K conversion. the borylcopper species was supposed to selectively attack the C–C triple bond of the 1,3-enynes.

Organoboron compounds have been recognized as highly versatile reagents in organic synthesis [1–5]. They are not only widely applied in Suzuki–Miyaura couplings [6,7] and Petasis reaction [8] for the construction of C–C bond as essential carbon nucleophiles, but can be easily oxidized by alkaline peroxides to afford sp^3-hybridized alkanols. Moreover, it is found that the boron atom also plays a vital role in design of the approved drugs and drug candidates [9,10], such as tavaborole [11], crisaborole [12], ixazomib [13], vaborbactam [14], and bortezomib [15]. Because of their reliable stability, low toxicity, moderate reactivities, and easy-to-handle properties, bis(boronate) compounds, such as bis(pinacolato)diboron (B_2pin_2), are expected to be very useful synthetic organoboron reagents for concise synthesis of complex borylated molecules [16–24]. These bis(boronate) compounds have been particularly used in the synthesis of various aryl and alkenylboron compounds due to their highly importance and much effort has been devoted to the formation of C-B bonds with borylmetal (M-***B***) species [25–27] serving as the key intermediates [28–32].

On the other hand, 1,3-enynes [33–35] are common and useful building blocks in synthetic chemistry, which enable straightforward transformations for rapid assembling of molecular complexity [36–39]. Direct borylation of 1,3-enynes by using the bis(boronate) reagent B_2pin_2, will afford several kinds of organoboron compounds (Scheme 1) [40]. For examples, Sasaki *et al.* developed a copper-catalyzed hydroborylation of 1,3-enynes with B_2pin_2. The 1,3-dienylboronates and 3-alkynylboronates were regio- and stereo-selectively afforded as major products and the different regioisomeric preference was supposed to be determined by both of the substrates and the catalytic complex [41]. Alfaro *et al.* found that the borylcopper species generated from ligand exchange with B_2pin_2 preferred to add on C–C triple bond of the 1,3-enynes even with a terminal C–C double bond [42], which is distinct from Cu-catalyzed hydroboration of 1,3-enynes through the Cu–H species [43,44]. Interestingly, Kuang *et al.* reported a regio- and stereo-divergent copper-catalyzed mono-hydroboration and di-hydroboration of 1,3-enynes for the synthesis of 1,4-hydroborylated allenes, 1,3-diborylated alkenes, and 1,4-diborylated alkenes [45]. Although various methods for borylation of 1,3-enynes have been nicely developed [46–49], approaches for direct diborylation of 1,3-enynes, forming 1,4-diborylated allenes, have not yet been reported.

Noticing the successful developments on diborylation of alkenes and alkynes [50–55], we are curious about whether synthesis of diborylated allenes directly from diborylation of 1,3-enynes with B_2pin_2 can be achieved, which will be an efficient method for the construction of allenylborons from the viewpoint of atom and

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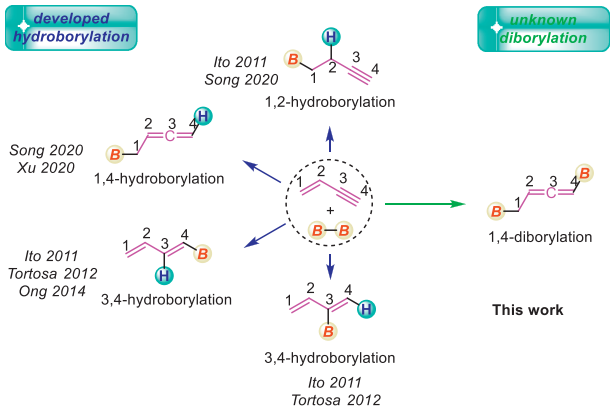
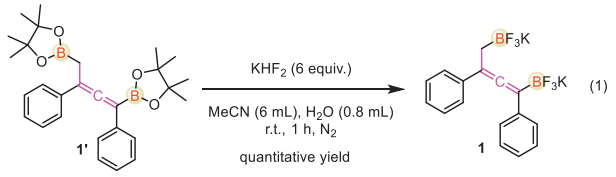


Scheme 1. Reactions of 1,3-enynes with B_2Pin_2.

step economy. However, there are several main challenges need to be addressed: (1) how to control the chemoselectivity for 1,4-diborylation of 1,3-enynes over reported types of hydroborylation; (2) how to control the regioselectivity for 1,4-diborylation over 1,2- and 3,4-diborylation [56]; and (3) how to control the stereoselectively. Herein, we reported the first copper-catalyzed diborylation of readily available 1,3-enynes with B_2pin_2 for the construction of 1,4-diborylated allenes in an efficient fashion. It was supposed that the borylcopper species regio- and stereo-selectively added on the C–C triple bond of the 1,3-enynes, rather than the less sterically-hindered terminal C–C double bond. A wide range of 1,3-enynes bearing various functional groups can participant in the reaction and afforded the corresponding 1,4-diborylated allenes in good to excellent yields.

Initially, we found that when the toluene solution of but–3-en-1–yne-1,3-diyldibenzene (**1s**) and B_2Pin_2 was stirred at 80 °C with $Cu(OAc)_2$/P(*p*-OMePh)$_3$ as the catalytic system, the desired 1,4-diborylated allene (**1′**) was produced in a yield as high as 69% (Table 1, entry 1). The result of this reaction clearly indicated that regioselective 1,4-diborylation of 1,3-enynes can be achieved. It also suggested that instead of forming the reported hydroborylation products as mentioned, this reaction might be improved as a highly efficient method for the synthesis of 1,4-diborylated allenes.

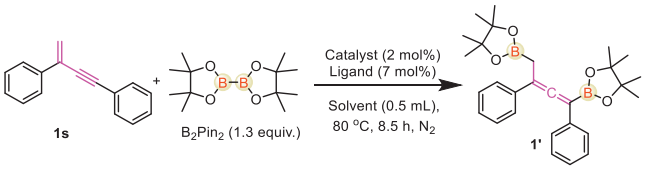
Encouraged by this result, the reaction parameters of this reaction were carefully examined, such as metal catalyst, ligand, solvent, reaction temperature, and reaction time (see Supporting information for more details). It was found that the copper salt with the carboxylate ion showed better catalytic activity (Table 1, entries 2–5 *vs.* entry 1). Ligand screening suggested that PPh_3, PCy_3, P(*p*-MePh)$_3$, and P(*p*-ClPh)$_3$ were superior to the bulkier monophosphine ligands (Table 1, entries 6–12 *vs.* entry 4), while the performances of bisphosphine ligands varied (Table 1, entries 13–16) [41]. The results in entries 17–22 indicated that polar solvents were inferior to the nonpolar ones. Further investigation of the reaction temperate and reaction time advised that the yield of product **1′** would decrease as time went by and the optimal reaction temperature was 50 °C (Table 1, entries 23–26). Finally, the reaction ran at 50 °C for 2 h offered the best yield of the desired 1,4-diborylated allene **1′** (Table 1, entry 27).



Although the optimal reaction conditions were well established, the purification of **1′** was challenging. This is because the 1,4-diborylated allene **1′** was found to be more fragile than other organoboron compounds with Bpin moiety. It would decompose simultaneously during the separation on silica gel and alumina as well. This problem was finally solved when the Bpin group was transformed into its fluoroborate form (Eq. 1). Upon treatment with KHF_2 in aqueous acetonitrile [57,58], the 1,4-diborylated allene **1′** was converted into the corresponding fluoroborate **1** in a quantitatively yield. Furthermore, this protocol can be directly used in a one-pot process after the completion of 1,4-diborylation of 1,3-enynes without isolation of the allene products with two Bpin moieties and the fluoroborate precipitated as a pure product.

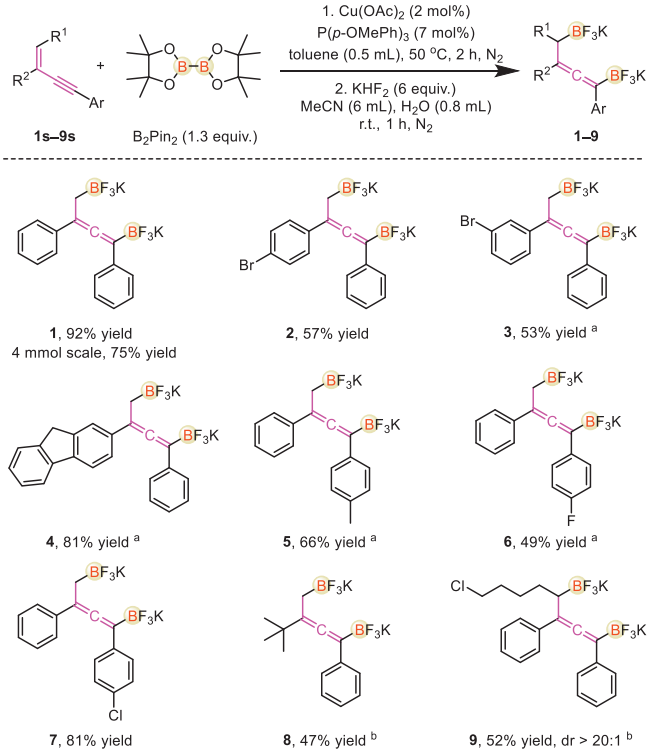
With the optimal reaction conditions and the protocol for BPin to BF_3K conversion in hand, we investigated the scope of 1,3-enynes (Scheme 2). First, 1,3-enynes with an aromatic moiety attached on the terminal side of alkynyl group was studied. While the diarylated 1,3-enynes afforded the desired 1,4-diborylated allenes **1–7** in good to excellent yields, substrate **8s** with an alkyl substitution delivered the corresponding 1,4-diborylated allene **8** in a fair yield. Several functional groups, such as Br, F, Cl, and methyl group were tolerated, and 1,3-enyne **4s** decorated with an activated methylene group performed well to produce allene **4** in 81% yield without affecting the methylene group. Notably, when a trisubstituted 1,3-enyne **9s** was applied to this reaction, the corresponding 1,4-diborylated allene **9** can be obtained in 52% yield and with a diastereomeric ratio higher than 20:1, which might be determined by the *Z*-selective substrate and/or the steric hindrance.

Table 1
Optimization of reaction conditions.[a]

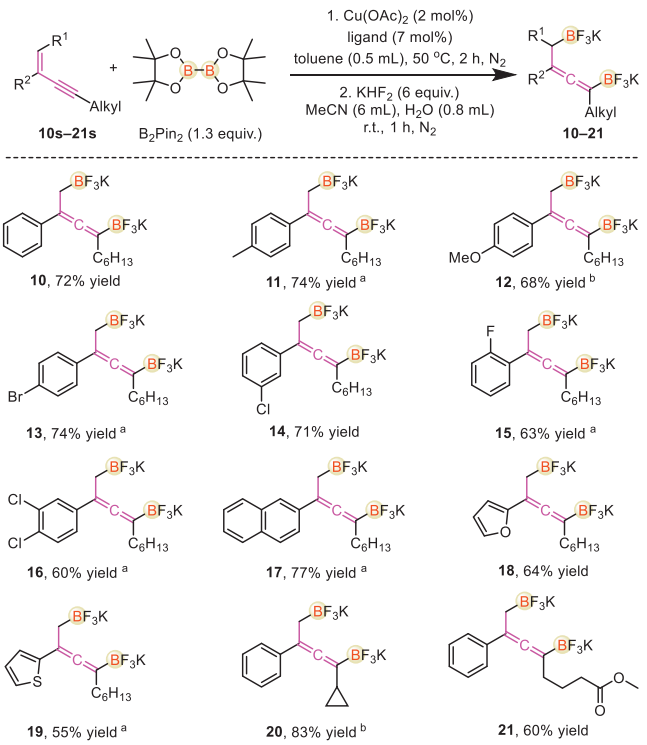


Entry	Catalyst	Ligand	Solvent	Temp (°C)	Yield (%)[b]
1	$Cu(OAc)_2$	P(*p*-OMePh)$_3$	Toluene	80	69
2	CuI	P(*p*-OMePh)$_3$	Toluene	80	trace
3	$Cu(OTf)_2$	P(*p*-OMePh)$_3$	Toluene	80	trace
4	CuTc	P(*p*-OMePh)$_3$	Toluene	80	36
5	CuOAc	P(*p*-OMePh)$_3$	Toluene	80	50
6	$Cu(OAc)_2$	PPh_3	Toluene	80	60
7	$Cu(OAc)_2$	PCy_3	Toluene	80	49
8	$Cu(OAc)_2$	P(*p*-MePh)$_3$	Toluene	80	66
9	$Cu(OAc)_2$	P(*p*-ClPh)$_3$	Toluene	80	52
10	$Cu(OAc)_2$	*t*BuXPhos	Toluene	80	trace
11	$Cu(OAc)_2$	X-Phos	Toluene	80	trace
12	$Cu(OAc)_2$	CyJohnPhos	Toluene	80	15
13 [c]	$Cu(OAc)_2$	BINAP	Toluene	80	trace
14 [c]	$Cu(OAc)_2$	DPPP	Toluene	80	25
15 [c]	$Cu(OAc)_2$	DPPB	Toluene	80	58
16 [c]	$Cu(OAc)_2$	DPPF	Toluene	80	33
17	$Cu(OAc)_2$	P(*p*-OMePh)$_3$	CH_3CN	80	trace
18	$Cu(OAc)_2$	P(*p*-OMePh)$_3$	DMF	80	25
19	$Cu(OAc)_2$	P(*p*-OMePh)$_3$	DME	80	59
20	$Cu(OAc)_2$	P(*p*-OMePh)$_3$	DCE	80	56
21	$Cu(OAc)_2$	P(*p*-OMePh)$_3$	THF	80	49
22	$Cu(OAc)_2$	P(*p*-OMePh)$_3$	1,4-Dioxane	80	48
23	$Cu(OAc)_2$	P(*p*-OMePh)$_3$	Toluene	70	53
24	$Cu(OAc)_2$	P(*p*-OMePh)$_3$	Toluene	60	58
25	$Cu(OAc)_2$	P(*p*-OMePh)$_3$	Toluene	50	66
26	$Cu(OAc)_2$	P(*p*-OMePh)$_3$	Toluene	40	61
27 [d]	$Cu(OAc)_2$	P(*p*-OMePh)$_3$	Toluene	50	91

[a] Reaction was conducted with 1,3-enyne **1s** (0.5 mmol, 1 equiv.), B_2Pin_2 (0.65 mmol, 1.3 equiv.), catalyst (2 mol%), and ligand (7 mol%) in solvent (0.5 mL) at given temperature under the atmosphere of nitrogen.
[b] ^1H NMR yield with dibromomethane as the internal standard.
[c] Ligand (3.5 mol%).
[d] The reaction was run at 50 °C for 2 h.



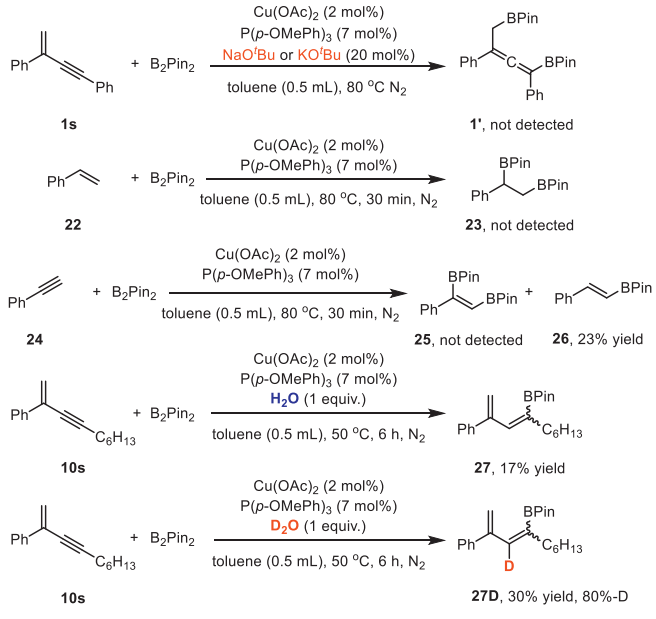
Scheme 2. Substrate scope of 1,3-enynes when R^3 is an Ar group. Reaction was conducted with 1,3-enyne (0.5 mmol), B_2Pin_2 (0.65 mmol), $Cu(OAc)_2$ (2 mol%), P(*p*-OMePh)$_3$ (7 mol%) in toluene (0.5 mL) at 50 °C for 2 h, and then with KHF_2 (3 mmol) in aqueous solution of MeCN/H_2O (6 mL:0.8 mL) at r.t. for 1 h. [a] P(p-OMePh)$_3$ was replaced by PPh_3. [b] P(*p*-OMePh)$_3$ was replaced by PCy_3.



Scheme 3. Substrate scope of 1,3-enynes when R^3 is an alkyl group. Reaction was conducted with 1,3-enyne (0.5 mmol), B_2Pin_2 (0.65 mmol), $Cu(OAc)_2$ (2 mol%), P(*p*-OMePh)$_3$ (7 mol%) in toluene (0.5 mL) at 50 °C for 2 h, and then with KHF_2 (3 mmol) in aqueous solution of MeCN/H_2O (6 mL:0.8 mL) at r.t. for 1 h. [a] P(*p*-OMePh)$_3$ was replaced by PPh_3. [b] P(*p*-OMePh)$_3$ was replaced by PCy_3.

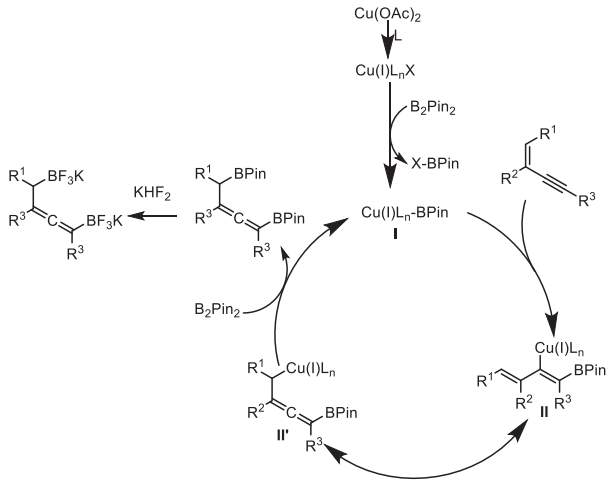
1,3-Enynes with an alkyl group attached on the terminal side of alkynyl group was also studied (Scheme 3). When the R^2 was a *para*-substituted phenyl group (**11s–13s**), a *meta*-substituted phenyl group (**14s**), an *ortho*-substituted phenyl group (**15s**), a disubstituted phenyl group (**16s**), or a 2-naphthyl group (**16s**), the reactions with these 1,3-enynes performed smoothly to afford the corresponding 1,4-diborylated allenes **11–16** in good to high yields. Heteroaryl groups such as 2-furyl and 2-thienyl can also be tolerated, and the reactions produced 1,4-diborylated allenes **18** and **19** in 64% and 55% yield, respectively. While substrate **20s** with a secondary alkyl substitution afforded the 1,4-diborylated allene **20** in 83% yield, substrate **21s** with a remote ester group-modified primary alkyl substitution generated the 1,4-diborylated allene **21** in 60% yield.

It has been mentioned above that for most of the documented reactions of 1,3-enynes with B_2pin_2, the hydroboration of 1,3-enynes was dominating, leaving the 1,4-diborylation of 1,3-enynes untouched. We were quite curious about the differences in terms of chemoselectivity (hydroboration *vs.* 1,4-diborylation), regioselectivity (1,4-diborylation *vs.* 1,2- and 3,4-diborylation), and stereoselectively (as for allene **9**). In order to understand this reaction, control experiments were carried out accordingly (Scheme 4). First, for hydroboration of 1,3-enynes, the addition of a base was necessary for the smooth transformation. In this study, the addition of 20 mol% of commonly used inorganic base, such as NaO^{t}Bu or KO^{t}Bu, resulted in completely prohibition of the formation of the 1,4-diborylated allene **1′**, which might be due to the decomposition of the diborylated allene under basic conditions, spontaneously [45]. This different result might be one of the key factors for this unusual selectivity. Second, when the reaction was performed with styrene (**22**) rather than but–3-en-1–yne-1,3-diyldibenzene (**1s**) as the substrate, no diborylated product (**23**) was detected, while the reaction with phenylacetylene (**24**) afforded the 1,2-hydroborated



Scheme 4. Mechanistic studies.

product **26** in 23% yield. These results suggested that, under the base-free conditions, the borylcopper species might prefer to add on C≡C bond rather than C=C bond [41]. In addition, under the optimal conditions, the reaction with water additive produced the 3,4-hydroborylated product **27** and this was further verified by the addition of deuteroxide, forming deuterated diene **27D**. According to these above control experiments, it was supposed that the reaction might be initiated with the addition of the borylcopper species on the internal C≡C bond rather than the C=C bond.



Scheme 5. Proposed reaction mechanism.

Based on the control experiment results, a possible mechanism for this 1,4-diborylation of 1,3-enynes was proposed (Scheme 5). First, borylcopper species **I** was formed through nucleophilic δ-bond metathesis between the copper(I)L_nX complexes and B_2Pin_2, and then, Cu–***B*** species **I** was regioselectively inserted into the C≡C bond to form the copper species **II**, which would resonate to its allene form **II'** [26]. Subsequently, nucleophilic δ-bond metathesis again afforded the desired 1,4-diborylated allene product and the regenerated Cu–***B*** species **I**. However, the initial addition of the Cu–***B*** species **I** on the C=C bond cannot be excluded.

In summary, we have developed the first selective 1,4-diborylation of 1,3-enynes for the formation of 1,4-diborylated allenes by copper catalysis, which is different from the reported copper-catalyzed hydroborylation of 1,3-enynes with B_2Pin_2 as the borylating reagent. Because of the instability of these allenes with Bpin moieties on column purification, they were transformed into the corresponding stable fluoroborates *in situ*. The substrate scope was broad and the reaction conditions were mild. Results of control experiments supported that the reaction might be initiated by the addition of the Cu–***B*** species (**I**) on the C≡C bond.

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.

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.2023.108980.

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