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Copper-catalyzed remote double functionalization of allenynes†

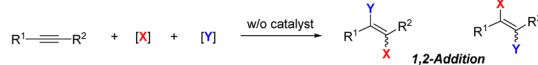
Yulong Song, Chunling Fu, Jian Zheng * and Shengming Ma *

Addition reactions of molecules with conjugated or non-conjugated multiple unsaturated C–C bonds are very attractive yet challenging due to the versatile issues of chemo-, regio-, and stereo-selectivities. Especially for the readily available conjugated allene compounds, the reactivities have not been explored. The first example of copper-catalyzed 2,5-hydrofunctionalization and 2,5-difunctionalization of allenynes, which provides a facile access to versatile conjugated vinylic allenenes with a C–B or C–Si bond, has been developed. This mild protocol has a broad substrate scope tolerating many synthetically useful functional groups. Due to the highly functionalized nature of the products, they have been demonstrated as platform molecules for the efficient syntheses of monocyclic products including poly-substituted benzenes, bicyclic compounds, and highly functionalized allene molecules.

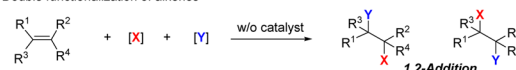
Unsaturated hydrocarbons are a class of very important compounds due to their ubiquity in organic synthesis, natural products, materials, and pharmaceuticals.^{1–4} Of particular interest, studies on versatile reactivities of C–C multiple bonds have been drawing more and more attention from organic, medicinal, and materials chemists.^{5–7} The reactions of the C–C double bond and C–C triple bond, including hydrofunctionalization and difunctionalization reactions, have been extensively developed with attractive regio- and stereo-selectivity (Schemes 1A and B).^{8–42} Recently, the addition reactions of allenenes, which deliver stereodefined olefins with decent regio-selectivities, have also been established (Scheme 1C).^{43–60} Such reactions of molecules with multiple unsaturated C–C bonds are very attractive yet challenging due to the versatile issues of chemo-, regio-, and stereo-selectivities (Scheme 1D).^{61–63} Although conjugated enynes and dienes have been studied,^{64–66} the reaction of allenynes merging an alkyne unit and an allene unit in a conjugated manner has not been studied.^{67–69} It would be challenging to control the related selectivities forming different 1,2-, 2,3-, 2,5-, 4,5-addition products with different unsaturated C–C bonds (Scheme 1E). Herein, we wish to disclose our recent observation on copper-catalyzed highly regioselective 2,5-boryl- and silyl-cupration of readily available conjugated allene-yne for efficient synthesis of conjugated vinylic allenenes with a versatile C–B or C–Si bond (Scheme 1F).

We initiated the study with the reaction of allenyne **1a**^{70–89} and B₂(pin)₂ **2a** in the presence of MeOH with NaO^tBu as the

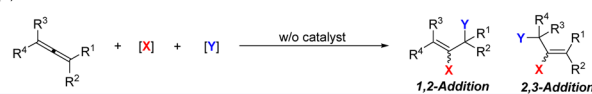
(A) Double functionalization of alkynes



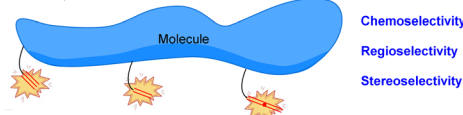
(B) Double functionalization of alkenes



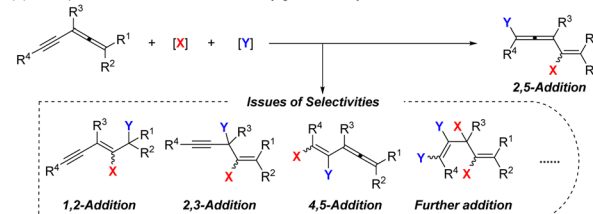
(C) Double functionalization of allenenes



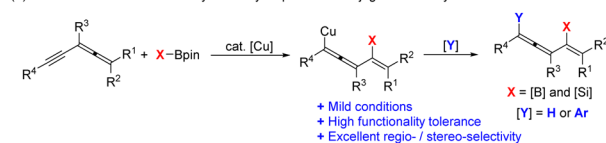
(D) Molecules with multiple different unsaturated carbon-carbon bonds



(E) Concept for double functionalization of conjugated allene-yne and issues of selectivities



(F) This work: Chemo-defined boryl- and silyl-cupration of conjugated allene-yne



Scheme 1 Double functionalization of alkynes, alkenes, allenenes, and conjugated allene-yne.

Laboratory of Molecular Recognition and Synthesis, Department of Chemistry, Zhejiang University, Hangzhou 310027, Zhejiang, People's Republic of China. E-mail: zhengj88@zju.edu.cn; masm@sioac.ac.cn

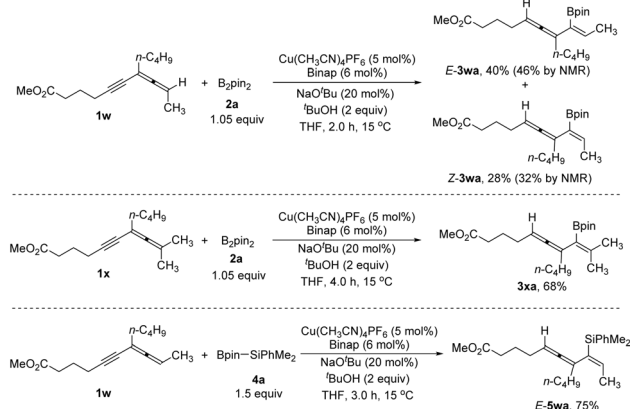
† Electronic supplementary information (ESI) available. As well as CCDC 2085439 and 2119437 available. For ESI and crystallographic data in CIF or other electronic format see DOI: <https://doi.org/10.1039/d4sc00034j>



79%. As for different substitutions at the R² position, **1u** (R² = ClC₃H₆-) and **1y** (R² = PhC₂H₄-) were tested to afford the corresponding silane products **5ua** in 61% yield and **5ya** in 71% yield, respectively.

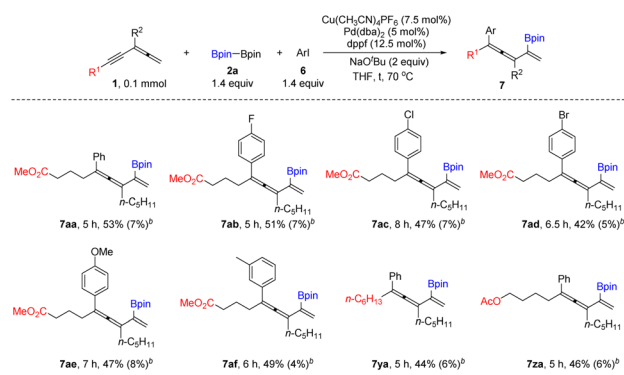
In addition, trisubstituted allenyne (**1w**) and tetrasubstituted allenyne (**1x**) are also suitable for affording the corresponding *Z*- and *E*-isomers of 1,3,4-trienyl boronates **3wa** in 68% yield and **3xa** in 68% yield. Furthermore, the reaction of **1w** also works with Me₂PhSiBpin **4a** affording 1,3,4-trien-2-yl silanes *E*-**5wa** in 75% yield (Scheme 2). When we tried iodobenzene as the electrophile under standard conditions, the target arylborylation product **7aa** was not produced. Interestingly, **7aa** was formed when an extra catalytic amount of Pd(dba)₂ was introduced (Table 3). Further screening of the temperature and ligand led to the formation of **7aa** in 53% yield. Aryl iodides with different electron-donating or -withdrawing groups afforded **7ab**, **7ac**, **7ad**, **7ae**, and **7af** in 42–51% yields. Allenynes with different alkyl substituents (R² = *n*-C₆H₁₃-, AcOC₄H₈-) also afforded the desired products **7ya** and **7za** (Table 3). 2,5-Hydrofunctionalization byproducts **3** were observed in all the cases, indicating that the Cu-catalyzed borylation reaction occurred before the Pd cross-coupling reaction with ArI.

To gain insight into the mechanism, a deuterium-labelling experiment was conducted. When the reaction was performed with a stoichiometric amount of ⁴BuOD, 70% of [D]-**3aa** with 75% deuterium incorporation at the allenic position was observed (Scheme 3A). When the R¹ group in **1z'** is a highly sterically hindered TMS group, the reaction failed to afford the 1,3,4-trienes. Instead, 1,4-enyne [D]-**3za'** with 66% deuterium incorporation was formed, indicating that the steric hindrance of TMS plays a critical role in determining the regioselectivity (Scheme 3B). On the basis of these experimental data, we proposed a possible mechanism (Scheme 3C). Initially, the copper-boryl complex **I** is generated *in situ* via the reaction of B₂(pin)₂ with the Cu precursor. Then, the C=C bond in the allene next to the C-C triple bond highly regioselectively inserted into the copper-boryl bond in **I** with the boron connected to the middle carbon atom to afford the propargylic copper species **II**, which would rapidly isomerize to allenyl copper species **III** and subsequently get trapped with ⁴BuOD to



Scheme 2 Scope of trisubstituted and tetrasubstituted allenynes

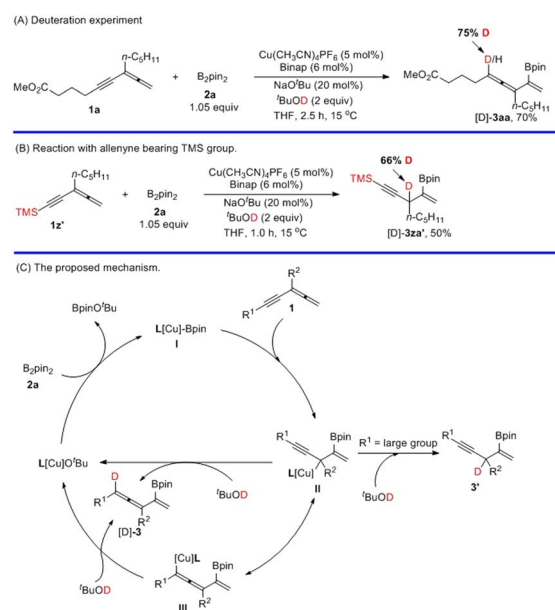
Table 3 Scope of 2,5-arylboration^a



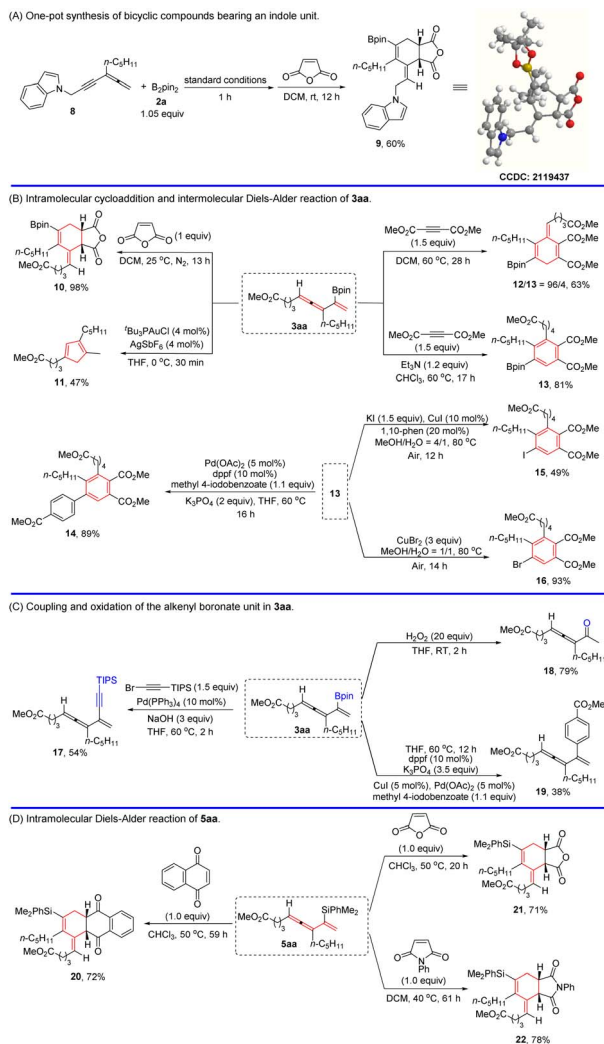
^a Reaction conditions: **1** (0.1 mmol), **2a** (1.4 equiv.), **6** (1.4 equiv.), Cu(CH₃CN)₄PF₆ (7.5 mol%), Pd(dba)₂ (5 mol%), dppf (12.5 mol%), and NaO^tBu (2 equiv.) in THF (1 mL) at 70 °C on a 0.1 mmol scale. ^b ¹H NMR yield of 2,5-hydroboration by-products **3aa**, **3ya**, and **3za** in parentheses.

produce the final product [D]-**3**, regenerating LCuO^tBu to complete this catalytic cycle. Meanwhile, protonation of another isomeric form **II** at the γ position would also afford product [D]-**3**. When the R group is the sterically bulky TMS group, **II** would directly undergo protonolysis to form 1,4-enyne [D]-**3za'**. In the first step, the allene was inserted into the copper-boryl bond in intermediate **I** since the allene unit is more reactive than the alkyne unit. The regioselectivity for the protonolysis depended on the steric hindrance of the R¹ group in copper species **II** or **III**.

In order to showcase the synthetic utility of the products, further transformations of the 1,3,4-trien-2-yl boronates/silanes were demonstrated (Scheme 4): The one-pot sequential 2,5-



Scheme 3 Deuteration experiment and the proposed mechanism.



Scheme 4 Synthetic applications.

borylcurpuration of allenyne **8** and a Diels–Alder reaction with maleic anhydride afforded stereodefined bicyclic lactonyl borate **9** (Scheme 4A).⁹⁰ Similarly, allenene **3aa** could also be readily converted to corresponding bicyclic lactone product **10**, 1,4-cyclohexadiene with a stereodefined exo-cyclic C=C double bond **12**, and penta-substituted benzene **13** via a Diels–Alder reaction with dimethyl but-2-ynedioate under different conditions. Such differently polysubstituted benzenes are difficult to prepare yet very useful.⁹¹ In order to show the reactivity of the C–B bond, the Diels–Alder product **13** was employed for further transformation. Firstly, **13** was successfully coupled with methyl 4-iodobenzoate to afford **14** in 89% yield.⁹² Secondly, the iodination of **13** with KI afforded the iodination product **15** in 49% yield catalyzed by copper iodide under air.⁹³ Thirdly, bromination of the C–B bond in **13** has been realized to afford **16** in an excellent yield by its treatment with copper bromide.⁹³ The intramolecular cycloisomerization of **3aa** also proceeded smoothly to give the corresponding cyclopentadienyl boronate **11** (Scheme 4B).⁹⁴ Moreover, **3aa** could be readily converted into 2-alkynyl-1,3,4-triene **17** via alkynylation with alkynyl bromide⁷⁹

and allenyl ketone **18** via oxidation with H₂O₂.⁹⁵ In addition, the C–B bond in **3aa** was successfully coupled with methyl 4-iodobenzoate to afford **19** in 38% yield even in the presence of a highly reactive allene unit (Scheme 4C). Meanwhile, other dienophiles, such as 1,4-naphthoquinone, maleic anhydride, and *N*-phenylmaleimide, could all undergo the Diels–Alder reaction with silane **5aa** to produce the corresponding products **20**, **21**, and **22** in decent yields (Scheme 4D).⁹⁰

Conclusions

In summary, we have developed the first example of copper-catalyzed 2,5-boryl- or silyl-cupration of allenynes, providing an efficient protocol for substituted 1,3,4-trien-2-yl boronates or silanes with excellent regioselectivity. This method has advantages of a readily available catalyst and starting materials, high catalytic activity, mild conditions, a broad substrate scope tolerating many synthetically useful functional groups, and synthetically useful functionalities. The synthetic utility of this protocol has been demonstrated via the versatile transformations of the resulting products to furnish valuable functionalized molecules. Further studies are being actively pursued in our laboratory.

Data availability

The data supporting this article have been uploaded as the ESI.†

Author contributions

S. M. and J. Z. conceived and designed the experiments. Y. S., J. Z. performed the experiments. Y. S., C. F., J. Z., and S. M. wrote the manuscript. S. M. and J. Z. directed the research.

Conflicts of interest

There are no conflicts to declare.

Acknowledgements

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