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Enantiospecific Synthesis of 1,3-Disubstituted Allenes from Propargylic Carbonates through a Borylation-1,2-Elimination Process

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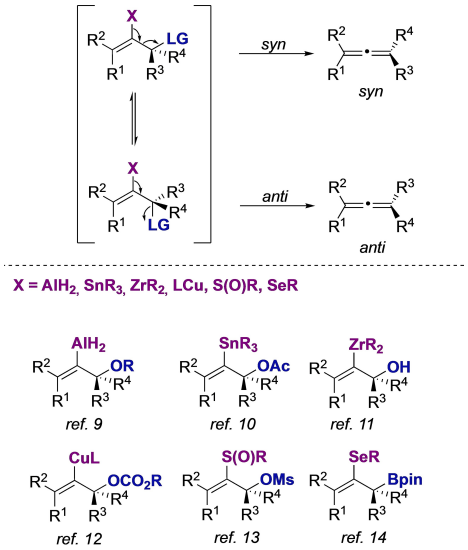
Abstract: An array of enantioenriched vinyl boronates bearing an allylic carbonate moiety have been prepared through selective hydroboration of propargyl carbonates. Treatment of these vinyl boronates with TBAF in THF affords enantioenriched 1,3-disubstituted allenes through a novel 1,2-*anti* elimination in good yields and high enantiospecificities.

Keywords: allenes; boronates; 1,2-*anti*-elimination; TBAF; propargylic carbonates

The unique reactivity of allenes, quite different to other dienes, along with their relatively high rotational barriers that result in significant configurational stability, makes allenes valuable chiral precursors to build more complex molecules.[1] This functional group is present in more than 150 natural products, all of them isolated in enantiopure or enantioenriched form.[2] Additionally, a great number of pharmacologically active compounds also contain allene functionalities, like the marketed drugs Enprostil,[3] gastric HCl inhibitor, Fenprostalene[4] and Prostalene,[5] used for veterinary medicine. Due to the growing interest in allenes new asymmetric protocols for their synthesis have been developed.[6]

Although the most studied methods to prepare allenes are based on nucleophilic substitutions,[7] one of the best approaches to obtain both enantiomers of the same allene from an enantiopure substrate is the 1,2-elimination of properly functionalized alkenes, that can occur through a *syn*- or *anti*-periplanar pathway (Scheme 1).[8] In an early example, Olsson and Claesson,[9] reported the insertion of lithium aluminum hydride in propargylic derivatives followed by *anti*- or *syn*-1,2-elimination, depending on reaction temperature, to afford enantioenriched allenes.

Similarly, other vinyl metals have been involved as reactive groups in stereospecific 1,2-eliminations such as vinyl stannanes using fluoride anions from TBAF,[10] vinyl zirconium derivatives available from reaction of Schwartz's reagent and propargylic alcohols[11] and vinyl copper intermediates generated by addition of copper hydrides to propargylic carbonates.[12] Alkenyl sulfoxides have also been reported as intermediates in 1,2-eliminations to obtain allenes, either by addition of Gilman cuprates to allylic sulfinyl mesylates or by sulfoxide-metal exchange promoted by an excess of a Grignard or an alkyllithium reagent.[13] Recently, Aggarwal and col.[14] reported the synthesis of di-, tri- or tetrasubstituted allenes from allylic β-selenoboronates. The absolute configuration of the resulting allenes can be controlled by the choice of the reaction



Scheme 1. Stereochemical course of *syn*- and *anti*-periplanar 1,2-eliminations.

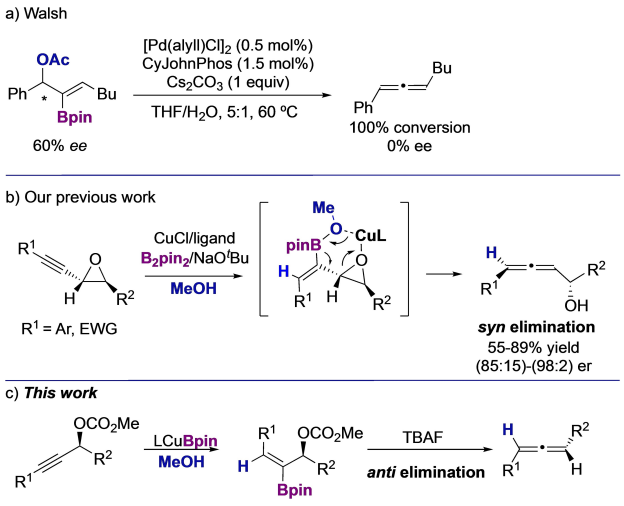
conditions resulting in selective 1,2-*syn* or 1,2-*anti* elimination.

Alkenyl boronates have received less attention to prepare allenes through 1,2-eliminations.[15,16] In 2014, Walsh and col.[16] synthesized α-hydroxy (*E*)-vinyl boronates to prepare disubstituted allenes using a palladium catalyst (Scheme 2, a). Although the products were obtained in good yields, the authors showed with one example that the allene racemized under the reaction conditions.

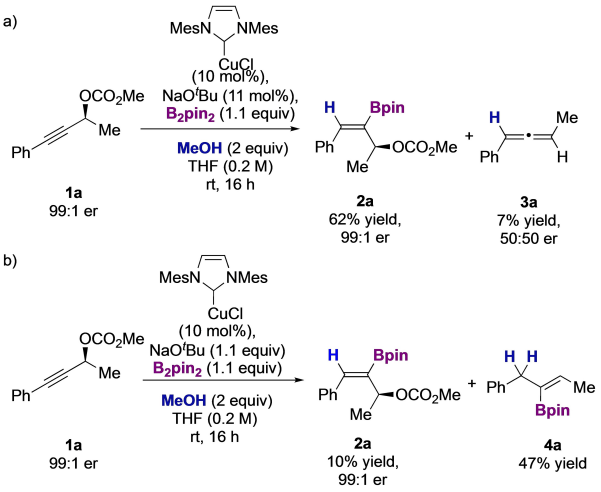
In connection with our recent synthesis of α-hydroxy allenes by copper-catalyzed borylation-*syn*-elimination of propargylic epoxides (Scheme 2, b),[17] we decided to explore if propargylic carbonates could be suitable candidates for the borylation-allenation reaction with stereocontrol. Herein we disclose our results on the borylation and subsequent 1,2-elimination of enantioenriched propargylic carbonates to prepare disusbtituted allenes with good to excellent chirality transfer (Scheme 2, c). Interestingly, we found that the stereochemical outcome of the elimination step is *anti*, opposite to that observed before for the propargylic epoxides.

Our preliminary studies began with the copper-catalyzed borylation of phenyl substituted propargyl carbonate **1a**, under the conditions used previously in our group for the synthesis of α-hydroxyallenes from propargylic epoxides.[17] It should be highlighted that the regioselectivity of the copper-catalyzed borylation of propargylic carbonates was not obvious at the outset since both carbonate and phenyl groups are known to direct the borylation with opposite regiochemistry.[18] Using a mixture of IMesCuCl (10 mol%), NaO^{t}Bu (11 mol%), B_2pin_2 (1.1 equiv.) and MeOH (2 equiv.) as a proton source, a mixture of (*Z*)-vinyl boronate **2a** and allene **3a** was obtained, indicating that the borylation had occurred with the desired regiochemistry. In contrast to the low stability of the epoxy vinyl boronates studied before,[17] vinyl boronate **2a** was isolated after purification by chromatography, in 62% yield and with the same enantiomeric ratio as the propargylic carbonate precursor. Interestingly, a small quantity of allene **3a** was isolated as a racemic product, suggesting a possible competition between *syn*- and *anti*-1,2-elimination from compound **2a** (Scheme 3, a) or racemization of **3a** under the reaction conditions.

To promote the *in situ* formation of allene **3a** we tested the hydroboration-allenation cascade using a stoichiometric amount of sodium *tert*-butoxide, hoping that the excess of base would promote the 1,2-



Scheme 2. Synthesis of allenes from vinyl boronates through 1,2-elimination.

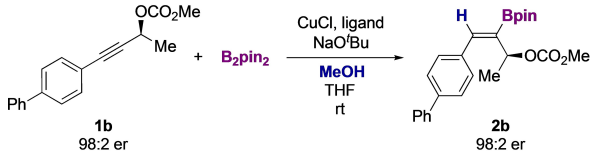


Scheme 3. Preliminary studies.

elimination pathway. Unfortunately, we found that allene **3a** was more reactive under copper-catalyzed borylation conditions than the propargyl carbonate **1a**. Indeed, a mixture of recovered starting material **1a**, vinyl boronate **2a** and vinyl boronate **4a**, derived from the undesired hydroboration of allene **3a**, was obtained (Scheme 3, b). These results prompted us to examine the optimization of a stepwise sequence with hydroboration of propargylic carbonates and subsequent stereospecific elimination of the vinyl boronates as individual steps.

Optimization of the hydroboration was carried out on carbonate **1b**, that would produce a less volatile allene than **1a**, and the results are gathered in Table 1. The conditions used before for **1a** (Scheme 3, a) gave boronate **2b** with moderate conversion and low isolated yield (Table 1, entry 1). A screening of ligands, including xantphos, triphenyl and tricyclohexyl phoshines gave improved conversions but poor isolated yields (Table 1, entries 2–4). The use of the more hindered phoshine $(o\text{-Tolyl})_3P$ produced a decrease in conversion and yield and Johnphos gave a very low conversion (Table 1, entries 5 and 6). As expected, the enantiomeric ratio of the carbonate precursor was preserved in the vinyl boronate **2b**. The variable low yields were attributed to inconsistent quantities of non-stereospecific allene formation during the purification process. The isolated yield of vinyl boronate **2b** was improved by precipitation upon addition of *n*-hexane to the reaction crude and filtration leading to pure **2b** in 92% yield (Table 1, entry 7).

Table 1. Optimization of the hydroboration conditions. Screening of ligands.[a]



1b 98:2 er — **2b** 98:2 er

Entry	Ligand	Conversion (%)[b]	Yield (%)[c]
1	Cu(IMes)Cl[d]	57	29
2	Xantphos	67	40
3	Ph_3P	100	58
4	Cy_3P	100	58
5	$(o\text{-Tolyl})_3P$	82	34
6	Johnphos	7	–
7	Cy_3P	100	92[e]

[a] Borylation conditions: **1b** (0.2 mmol), B_2pin_2 (0.22 mmol), CuCl (0.02 mmol), ligand (0.022 mmol), NaO^{t}Bu (0.04 mmol), MeOH (0.4 mmol), THF (0.2 M). ‘

[b] Determined by ^{1}H NMR analysis.

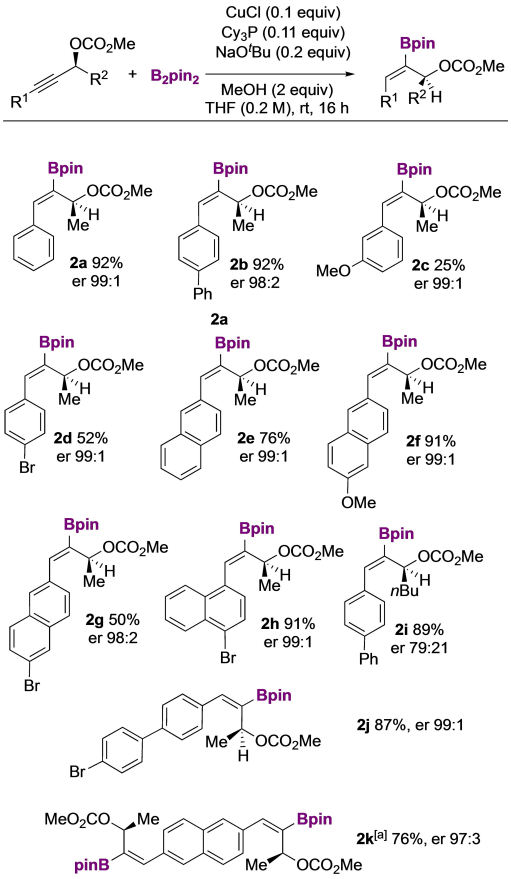
[c] Isolated yield by chromatography.

[d] Complex formed between CuCl and IMesCl prepared using a known procedure.[19]

[e] Isolated yield by precipitation.

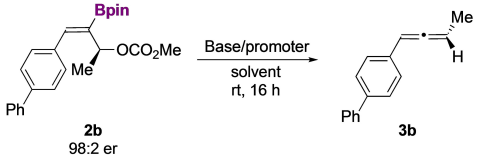
Using this protocol, different propargylic carbonates were borylated (Scheme 4). In all cases, purification by column chromatography was avoided to prevent allene formation.

At this stage we focused our attention on the 1,2-elimination step. Using compound **2b**, we tested different bases or promoters looking not only for good yields but also for high chirality transfer. Potassium and sodium carbonates gave very low conversions in toluene (Table 2, entries 1–2). Switching to MeOH allene **3b** was formed in high yield but low enantiospecifity (Table 2, entry 3). Stronger bases, like sodium methoxide and sodium *tert*-butoxide, gave allene **3b** in 63 and 59% isolated yield but moderate enantiomeric ratio (Table 2, entries 4 and 5). Lithium and potassium *tert*-butoxides did not improve the results (Table 2, entries 6 and 7). We decided then to check a fluoride source as elimination promoter, as reported previously by Konoike and Araki for related acetoxy alkenyl stannanes.[10] We were please to find that using TBAF, **3b** was obtained in 73% yield and 91:9 enantiomeric ratio (Table 2, entry 8). Decreasing the equivalents of TBAF, as well as increasing the concentration was not



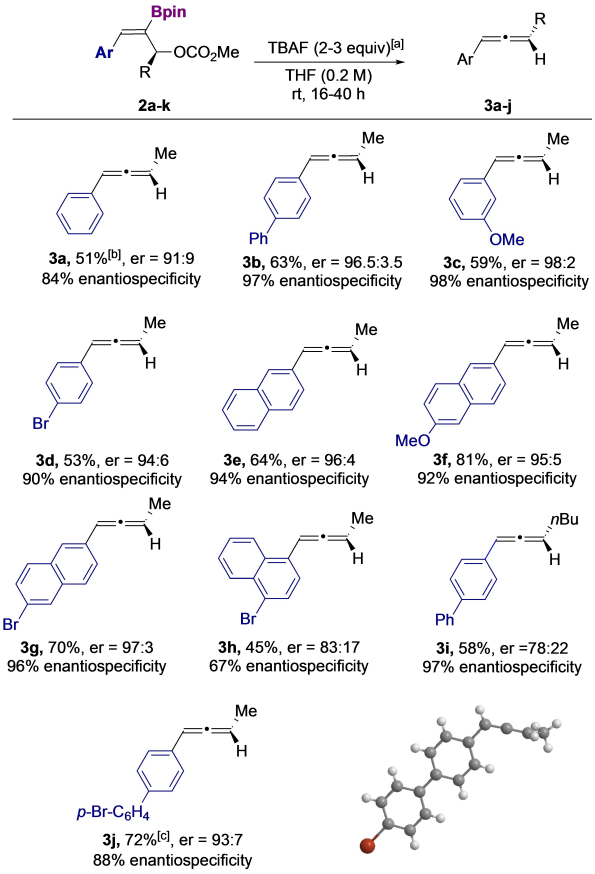
Scheme 4. Synthesis of vinyl boronates from propargylic derivatives.[a] B_2pin_2 (2.6 equiv.), NaO*t*Bu (0.4 equiv.), Cy_3P (0.22 equiv.).

Table 2. Optimization of the 1,2-elimination. Screening of bases and solvents.[a]



Entry	Base/promoter/ solvent	Yield (%)	Er[b]	Es (%)
1	K_2CO_3/Toluene	–	–	–
2	Na_2CO_3/Toluene	–	–	–
3	K_2CO_3/MeOH	70	70:30	42
4	NaOMe/Toluene	63	76:24	54
5	NaO^{t}Bu/Toluene	59	78:22	58
6	LiO^{t}Bu/Toluene	76	74:26	50
7	KO^{t}Bu/Toluene	53	59:41	19
8[c]	TBAF/Toluene	73	91:9	85
9[c]	TBAF/Toluene[d]	56	90:10	83
10[c]	TBAF/CH_2Cl_2[d]	74	83:17	66
11[c]	TBAF/*n*-Hexane[d]	51	89:11	81
12[c]	**TBAF/THF[e]**	**63**	**96.5:3.5**	**97**
13[c]	TBAF/THF[f]	57	97:3	89

[a] Elimination conditions: **2b** (0.1 mmol), base (0.5 mmol), solvent (0.025 M).
[b] Er were obtained by chiral HPLC.
[c] TBAF was used as a 1 M THF solution.
[d] 2.0 equiv. of TBAF solution were used. Solvent was employed in a 0.1 M concentration.
[e] 2.0 equiv. of TBAF solution were used. Solvent was employed in a 0.2 M concentration.
[f] 5.0 equiv. of TBAF solution were used. Enantiospecificity (es) = ee-product/ee-starting material×100%.



Scheme 5. Scope of the 1,2-elimination. [a] Reaction conditions: Table 2 entry 12. [b] Yield determined by ^{1}H NMR analysis using 1,4-diacetylbenzene as internal standard and THF-d^8 as solvent. Enantiospecificity (es) = ee-product/ee-starting material×100%.

beneficial (Table 2, entry 9). Next, we looked for the most suitable solvent for this elimination (Table 2, entries 10–12). Dichloromethane and *n*-hexane were also adequate solvents but the best balance of yield and enantiospecificity was found in THF that afforded **3b** in 63% yield with 97% enantiospecifity (Table 2, entry 12). Increasing the amount of TBAF did not improve the yield (Table 2, entry 13).

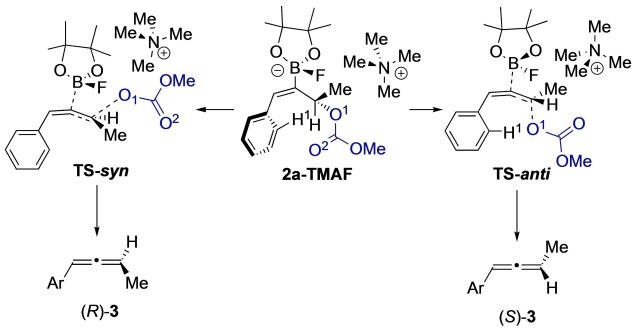
At this point we studied the 1,2-*anti*-elimination process with different vinyl boronates (Scheme 5). Under the optimized conditions (Table 2, entry 12) allene **3a** was obtained with a disappointing 25% isolated yield due to its volatility, but with high enantiomeric ratio (91:9). Using THF-d^8 and 1,4-diacetylbenzene as internal standard the ^{1}H NMR yield of **3a** increased to 51%. Next, we studied the effect of different substituents at the phenyl ring of the vinyl boronate. Thus allylic carbonate **2c**, with a *meta*-methoxy group, gave allene **3c** in 59% yield and excellent stereocontrol. Boronate **2d**, with a bromide at the *para*- position, afforded allene **3d** in moderate yield (53%) and good enantiomeric ratio. Moreover, naphthyl derivative **2e** produced allene **3e** with excellent enantiospecificity. Different substitutions in the napththyl ring were tolerated. Vinyl boronate **2f**, with a methoxy group at carbon 6 of the naphthyl moiety afforded allene **3f** in 81% yield and 92% es. Similarly, allene **3g** with a bromide substituent at carbon 6 was obtained with excellent stereocontrol from vinyl boronate **2g**. Finally, vinyl boronate **2h**, with a bromide substituent at carbon 4, afforded allene **3h** in moderate yield and enantiospecificity. Next, we studied the elimination of a vinyl boronate **2i**, with an *n*-butyl chain at the carbonate position, that provided allene **3i** with moderate yield and high levels of chirality transfer.

To determine the stereochemical outcome of the 1,2-elimination, we prepared **2j** with a bromide atom at the distal phenyl ring. Under our protocol allene **3j** was obtained in 72% yield and 93:7 enantiomeric ratio. The absolute configuration was determined to be (*S*) by X-ray diffraction analysis of a single crystal.[20] From the absolute configuration of **3j**, we could determine that the stereochemical outcome of the 1,2-

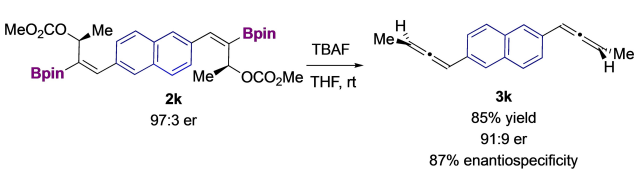
elimination was *anti* (Scheme 6). In contrast, in the presence of alkaline bases, the *syn* elimination competes with the *anti*-pathway probably due to coordination of the borate complex and the leaving group with the alkaline cation. DFT calculations were performed to better understand the preference for the *anti* versus *syn* elimination (See SI for details). A weak hydrogen bond between the aryl and carbonate groups (H_1-O_2: 3.02 Å) seems to determine the preferred conformation of the borate complex formed from **2a** and the ammonium fluoride (**2a-TMAF**). This interaction is strongly reinforced in **TS-*anti*** (H_1-O_1: 2.06 Å) whereas it does not exist in **TS-*syn***. This fact along with the higher steric interactions between the boronate moiety and the carbonate leaving group in **TS-*syn*** could justify the different stability between both transition states ($\Delta\Delta G = 2.8\ kcal \cdot mol^{-1}$) that would predict a ratio of products 99:1 in relatively good agreement with experimental results.

To enhance the scope of the protocol, we decided to examine a double 1,2-elimination on bis-boronate **2k** easily available by double borylation of **1k**. Interestingly, we obtained bis-allene **3k** in 85% yield and good stereocontrol (Scheme 7).

In summary, chiral disubstituted allenes are prepared with high enantiospecificity from readily available propargylic carbonates through a sequence that entails regioselective hydroboration to give trisubstituted (*Z*)-vinyl boronates **2** followed by *anti*-1,2-elimination using fluoride anions as promoters. This reaction occurs under mild conditions and provides an alternative tool to synthesize enantioenriched disubstituted allenes from propargylic carbonates. In general terms, our work on the synthesis of allenes from propargyl derivatives has revealed that the stereochemistry of the 1,2-elimination can be controlled by selecting the oxygenated leaving group, *syn* for epoxides or *anti* for carbonates.[17]



Scheme 6. Stereochemical outcome for the 1,2-elimination.



Scheme 7. Synthesis of bis-allene **3k**. Reaction conditions: Table 2, entry 12, TBAF (6 equiv.).

Experimental Section

A Representative Procedure for Allene Synthesis

An oven dried vial with a stir bar and (*Z*)-vinyl boronate (**2**, 0.1 mmol, 1 equiv.) was closed with a cap and it was purged by three vacuum/Ar cycles. Then, dry THF (0.5 mL, 0.2 M) and TBAF solution (1 M in THF, 0.2 mL, 2 equiv.) were added. The reaction was stirred overnight (16 h) and checked by TLC adding another equivalent of TBAF solution if starting material was remaining and stirring 24 h more. Then, it was quenched with water and the layers were separated. The aqueous phase was extracted with diethyl ether and the combined organic phases were dried over magnesium sulfate and filtered. The solvent was removed under reduced pressure and the crude mixture was purified by column chromatography on silica gel to afford pure **3**.

Acknowledgements

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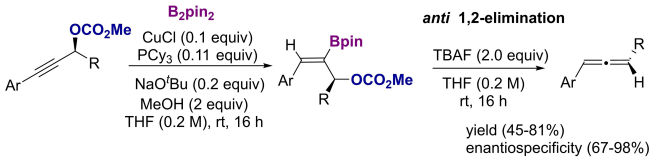
[20] CCDC 1844115 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre.

COMMUNICATIONS

Enantiospecific Synthesis of 1,3-Disubstituted Allenes from Propargylic Carbonates through a Borylation-1,2-Elimination Process



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