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Ruthenium-Catalyzed One-Pot Synthesis of (*E*)-(2-Arylvinyl)boronates through an Isomerization/Cross-Metathesis Sequence from Allyl-Substituted Aromatics

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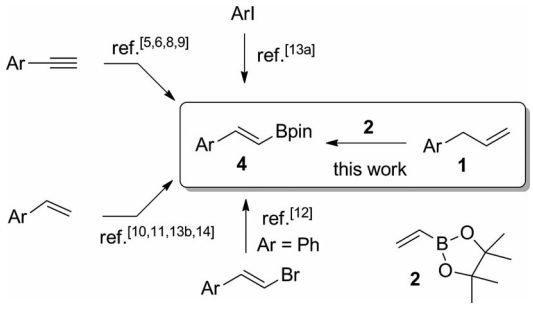
We described the efficient preparation of (*E*)-(2-arylvinyl)-boronates from allylbenzene derivatives on the basis of an isomerization/cross-metathesis sequence catalyzed by a modified Hoveyda–Grubbs catalyst. The implementation of the experimental procedure was simple and compatible with a large variety of substrates. This methodology provides a new chemical transformation not described to date. Allyl-substituted aromatics can thus be converted into diversely functionalized compounds, such as (*E*)-stilbene derivatives or (*E*)-vinyl azides, in only two steps.

Introduction

The synthetic versatility of alkenylboronates has made them key intermediates in organic synthesis.[1] Besides their aptitude to react efficiently in transition-metal-catalyzed cross-coupling reactions for C–C bond formation,[2] it has been shown that alkenylboronates can also be coupled stereospecifically to organic heteronucleophiles in the presence of copper catalysts.[3] In addition to their wide applications in organic chemistry as anionic or cationic building blocks, (*E*)-styrylboronates **4** constitute an interesting family of bioactive compounds, in particular because of their neuroprotective activities.[4]

This class of compounds can be synthesized from the corresponding terminal alkynes by uncatalyzed[5] or metal-catalyzed[6] hydroboration through a *syn*-addition approach of the boron reagents (Scheme 1).[7] The single boronation reaction of alkynes with bis(pinacolato)diboron (B_2pin_2) was also performed by using copper[8] or palladium[9] species as catalysts. Other alternative procedures have been developed from terminal alkenes, first by the rhodium-catalyzed dehydrogenative borylation of vinylarenes with dioxaborolane hydrides.[10] Thereafter, different reports have dealt with the use of B_2pin_2 and transition-metal catalysts to overcome the unwanted hydrogenation and/or hydroboration of the terminal double bond.[11] Borylation of styryl bromide with a less expensive and more atom economical boron source, pinacol borane (HBpin), was developed by using a $PdCl_2(CH_3CN)_2$/SPhos system [SPhos = 2-(dicyclohexylphosphino)-2′,6′-dimethoxybiphenyl].[12]



Scheme 1. Access routes to (*E*)-(2-arylvinyl)boronates.

Several other catalytic methods employing pinacol or 2-methyl-2,4-pentanediol ethylene boronic esters have been also reported.[13]Among these, cross-metathesis (CM) is probably one of the most elegant strategies, as it allows the production of alkenylboronates starting from styrenes with a high selectivity in favor of the (*E*) isomers in the case of ruthenium catalysts.[14,15]

Using this approach, we recently developed a route to new alkenylboronates from allyl-substituted aromatics.[16] During this work, we observed that modified Hoveyda–Grubbs catalyst $M7_1$-SIPr[17] induced the formation of by-product **4**, which may have resulted from the isomerization of starting material **1** followed by cross-metathesis with vinylboronate pinacol ester **2** (Scheme 1). This result likely implies the in situ formation of a ruthenium hydride species by partial decomposition of the catalyst[18] given the absence of an additive in the implementation.[19] To the best of our knowledge,[20] only one example of such a domino isomer-

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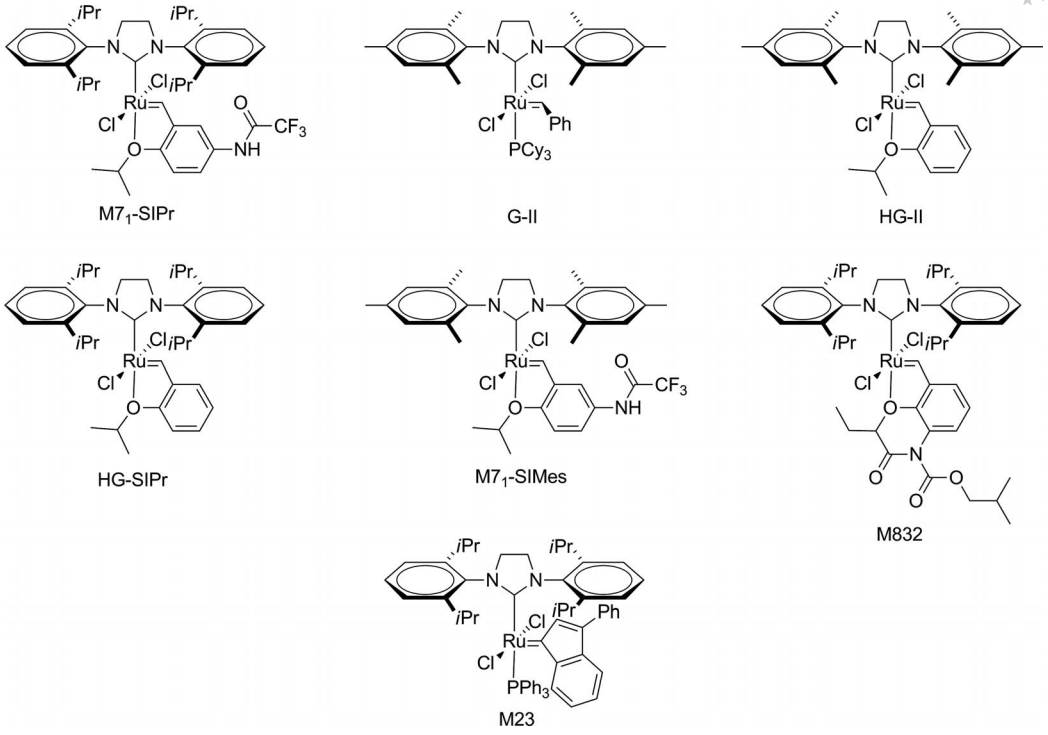


Figure 1. Ruthenium catalysts used in this study; Cy = cyclohexyl.

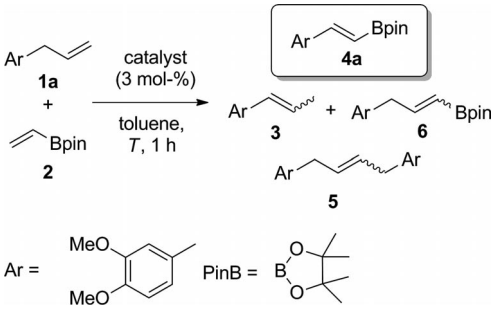
ization/cross-metathesis by using Hoveyda–Grubbs catalyst HG-II with allyltrimethylsilane as the cross partner has been reported.[21] The isomerization reaction can be followed by ring-closing metathesis (RCM), which adds an additional quantity of catalyst (tandem reaction) as described by Arisawa and van Otterlo for the synthesis of heterocycles.[22,23] Given that the stereoselective chemical transformation of allyl aromatics into (*E*)-(2-arylvinyl)boronates is novel and could be useful in organic synthesis, we searched to optimize and generalize this catalytic process. In this paper, we report our results with this goal in mind. A structure–activity relationship study was realized with different ruthenium catalysts to identify the influence of the fragments on the product distribution (Figure 1). We also show that allyl aromatics can be converted easily into diversely functionalized compounds.

Results and Discussion

The effects of varying the catalyst on the isomerization/cross-metathesis sequence were explored by using 4-allyl-1,2-dimethoxybenzene (**1a**) as the model substrate (Table 1). This compound was chosen for the optimization of the sequential reaction owing to its high propensity to undergo double-bond isomerization in the presence of ruthenium–carbene catalysts.[24] The reported results were obtained by using pinacol vinylboronate[25] (**2**, 2 equiv.) in toluene, and starting material **1a** was completely consumed in all studied catalytic processes. The increase in temperature in the presence of M7$_1$-SIPr was a determinant factor that favored the formation of the cross-metathesis product resulting from the isomerization reaction (Table 1, entries 1–3).[26] Heating at reflux was necessary to produce the (*E*) isomer of **4a** stereoselectively in 62% yield after purification.[27,28] From these results, it appears that the cross-metathesis was the slow kinetic step in the sequential reaction. Under these experimental conditions, the unusual ambivalent behavior of the M7$_1$-SIPr catalyst is highlighted if we compare with more conventional ruthenium olefin metathesis catalysts such as G-II and HG-II (Table 1, entries 4 and 5). With these two catalysts, there was no formation of desired product **4a**, although the isomerization reaction took place. In the case of HG-II, only isomerized product **3** was observed.[29]

To investigate if the amino carbonyl group present in M7$_1$-SIPr was essential for its catalytic activity, we tried another Hoveyda–Grubbs-type complex, that is, HG-SIPr (Table 1, entry 6). A slight drop in the conversion to **4a** was observed, which indicated that this function was not crucial for the efficiency of the catalyst, unlike the bulkiness of the N-heterocyclic carbene ligand. This was confirmed by the use of the M7$_1$-SIMes catalyst, which showed a significantly lower efficiency for the production of **4a** compared to M7$_1$-SIPr (Table 1, entry 7). A possible explanation could be the increased thermal stability of the catalyst, thanks to the bulky SIPr ligand.[30] By heating at reflux, the supposed formation of a ruthenium hydride species is thus limited while safeguarding a certain quantity of the catalyst for an efficient cross-metathesis reaction.[31] Other catalysts, such as M832[17b] and M23, were investigated with less success (Table 1, entries 8 and 9). In particular, the replacement of the benzylidene ligand by an indenylidene ligand (see M23) had a detrimental effect on the "one-pot" sequential reaction, and a mixture of **3** and **6** was produced, the latter of

Table 1. Optimization of the sequential process with **1a** by using different ruthenium catalysts.[a]



Entry	Catalyst	*T* [°C]	Ratio **3/4a/5/6**[c]	Yield of **4a** [%][d]
1	$M7_1$-SIPr	50[b]	1:2.5:0:0	60
2	$M7_1$-SIPr	70[b]	1:3.4:0:0	72
3	$M7_1$-SIPr	reflux	1:5.9:0:0	84 (62)[e]
4	G-II	reflux	1:0:0:1.7	n.d.
5	HG-II	reflux	1:0:0:0	n.d.
6	HG-SIPr	reflux	1:4:0:0	78
7	$M7_1$-SIMes	reflux	1:1.7:0:0	n.d.
8	M832	reflux	1.7:1:0:0	n.d.
9	M23	reflux	1.4:0:0:1	n.d.

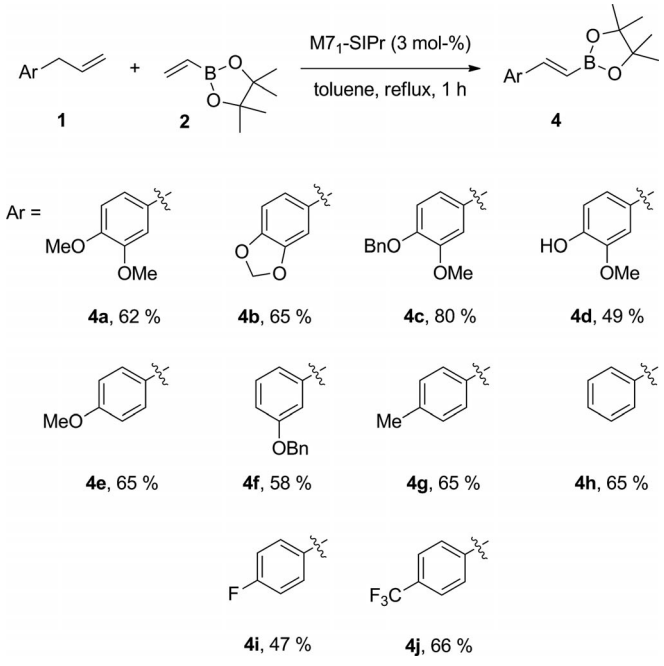
[a] 0.2 M in toluene. [b] The catalyst in solution was heated for 5 min at reflux before the addition of **1a** and **2** at the indicated temperature. [c] Determined by ^{1}H NMR spectroscopy by analysis of the product mixtures prior to purification. Only the (*E*) isomer of **4a** was observed. [d] Determined with 3-fluoro-4-nitrotoluene as an internal standard; n.d.: not determined. [e] Yield of the product after isolation by flash chromatography on silica gel.

which resulted from the cross-metathesis of starting material **1a**.

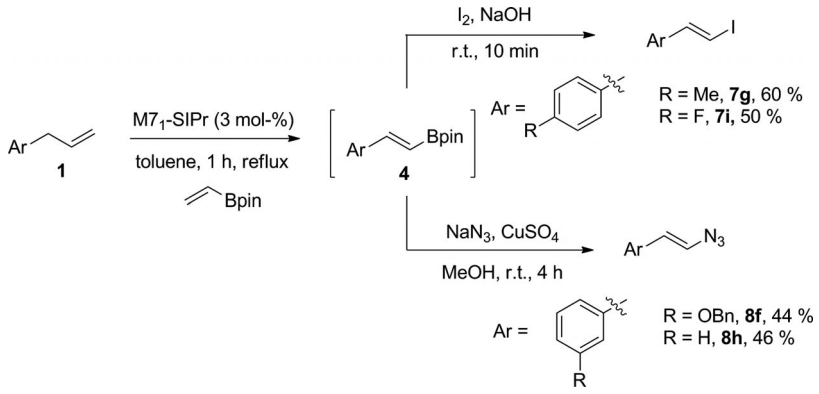
With these results in hand, we then envisaged the extension of this optimized catalytic process to various allyl aromatics. In all cases, alkenylboronates **4** were obtained as a single (*E*) stereoisomer with modest to good yields ranging from 47 to 80%. The presence of different electron-rich and electron-deficient substituents on the aromatic core had no apparent effect on the successful progress of this sequential reaction, and **4d** was obtained with a nonprotected phenolic group (Scheme 2).

The usefulness of this chemical transformation was emphasized by proceeding to replace the boron substituent by various functional groups. (*E*)-Vinyl iodides **7g** and **7i** were prepared by a two-step synthesis from the corresponding allyl aromatics (Scheme 3). After evaporation of toluene, the crude mixture of the one-pot isomerization/cross-metathesis reaction was treated directly with an iodine solution under basic conditions (I_2, NaOH)[32] to afford the desired compounds in 60 and 50% yield, respectively, over two steps. The boron substituent can be also replaced by an azide group under mild conditions as reported in the literature.[33] Using this catalytic procedure (NaN_3, $CuSO_4$), (*E*)-alkenyl azides **8f** and **8h** were obtained in good yields from the corresponding allyl aromatics.

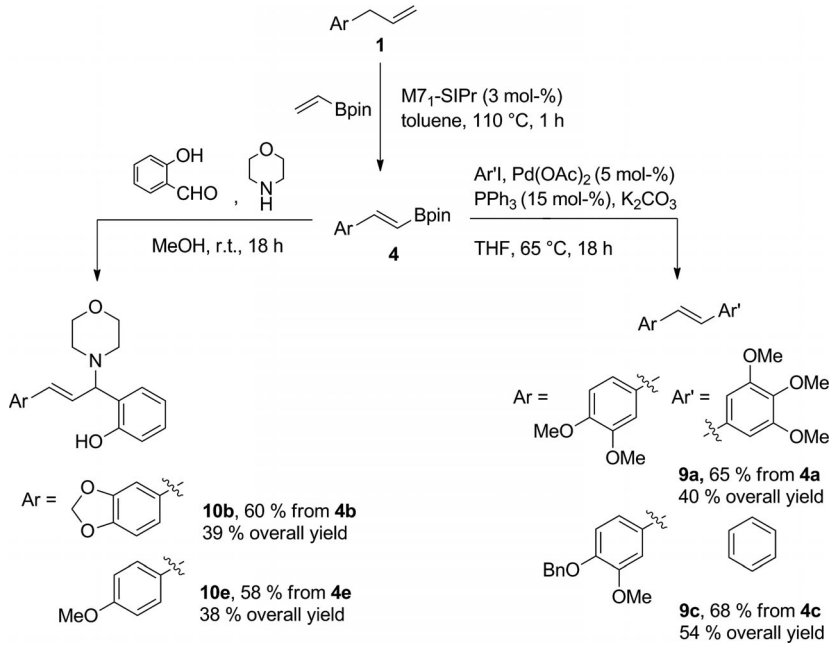
As another illustration of the synthetic interest of this methodology, we next turned our attention towards the transformation of allyl aromatics into stilbene derivatives **9** by including a Suzuki cross-coupling reaction in the synthesis (Scheme 4). For instance, we planned the synthesis of 3,5,3′,4′,5′-pentamethoxystilbene (**9a**), a methoxylated analog of resveratrol, which has been described to exert more potent inhibition of cell growth than resveratrol and other methoxylated derivatives in the human breast carcinoma cell line MCF-7.[34] Compound **9a** was thus prepared stereoselectively in 40% yield overall from **1a**. Alkenylboronates **4** can also serve as building blocks for a one-step multicomponent process such as the Petasis reaction.[35] By using salicylaldehyde and morpholine, allylamines **10b** and



Scheme 2. Extending the sequential reaction to other allyl-substituted aromatics.



Scheme 3. Synthetic transformations including the replacement of the boron substituent by functional groups.



Scheme 4. Synthetic transformations including a carbon–carbon bond-formation reaction.

10e were obtained in acceptable yields (60 and 58%, respectively) taking into account that the trivalent boron nucleophile is a pinacol boronate ester instead of a boronic acid.[36]

Conclusions

We described in this paper the stereoselective synthesis of (*E*)-(2-arylvinyl)boronates **4** through a ruthenium-catalyzed isomerization/cross-metathesis sequence from allyl-substituted aromatics. $M7_1$-SIPr is the more efficient catalyst for this process, probably owing to its thermal stability. We showed that this simple procedure is reliable with differently substituted substrates. This new chemical transformation could open attractive opportunities in organic synthesis in view of the fact that allylbenzene derivatives can be converted into diversely functionalized compounds in only two steps. Work is underway in our laboratory with the idea to transpose this strategy to other cross-metathesis partners.

Experimental Section

General Procedure for the Isomerization/Cross-Metathesis Sequential Reaction: 4,4,5,5-Tetramethyl-2-vinyl-1,3,2-dioxaborolane (**2**, 0.60 mmol) was added to a solution of **1** (0.30 mmol) in anhydrous toluene (0.2 M) under an inert atmosphere followed by $M7_1$-SIPr (3 mol-%). The resulting mixture was heated at reflux for 1 h in a preheated oil bath. Thereafter, toluene was removed under reduced pressure, and the crude product was purified by column chromatography by using the appropriate eluent to afford desired compound **4** as a single (*E*) isomer.

Supporting Information (see footnote on the first page of this article): Experimental details and copies of the NMR spectra for key intermediates **4a**–**j** and final products **7**–**10**.

Acknowledgments

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[27] The use of a preheated oil bath instead of gradual warming to reflux was crucial for good reproducibility.

[28] The loss of product during chromatography on silica was noticed as a result of the partial hydrolysis of the boronic ester.

[29] The synthesis of **4a** from **1a** was possible by using the HG-II catalyst over two steps. An isomerization reaction [HG-II (3 mol-%), toluene, 110 °C, 1 h] followed by cross-metathesis with 2 equiv. of vinylboronate **2** [HG-II (3 mol-%), CH_2Cl_2, 45 °C, 18 h] furnished the desired product in 51% overall yield compared to 62% by using $M7_1$-SIPr in one step.

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