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Synthesis of Allylboranes via Cu(I)-Catalyzed B–H Insertion of Vinyldiazoacetates into Phosphine–Borane Adducts

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Cite This: https://dx.doi.org/10.1021/acs.orglett.9b04619 **Read Online** ACCESS III Metrics & More [DI Article Recommendations s Supporting Information ⊕ PBu₃ ⊣ BH₂ ABSTRACT: Cu(I) catalysts enable C-B bond formation via 5 mol % Cu(MeCN)₄PF NaBArE direct insertion of vinyldiazoacetates into B-H bonds of borane-⊕ H Bu₃P−B−F 6 mol % BOX ligand phosphine Lewis adducts to form phosphine-protected allylboranes CHCl₃, 60 °C, 30 min CO₂R under mild conditions. The resulting allylborane-phosphine Lewis up to 92% viela

directly without the need for removal of the phosphine. The allylation reaction proceeds with high diastereoselectivity and yields 5,6disubstituted dihydropyranones after treatment with an appropriate acid.

A llylboranes and allylboronic acid derivatives are widely used in synthetic chemistry, most notably in asymmetric allylation of carbonyl compounds,¹ or as coupling partners in Suzuki–Miyaura reactions.² The common methods to obtain allylboranes and allylboronates³ include hydroboration of dienes (Figure 1a),⁴ carboboration of alkynes,⁵ transition

adducts can be used in the diastereoselective allylation of aldehydes



Figure 1. Representative syntheses of allylboranes and allylboronates.

metal catalyzed conjugate additions and allylic substitution using boron nucleophiles (Figures 1b and 1c),⁶ and addition of allylmetal reagents to the suitable borate esters and derivatives.⁷ Some of these methods suffer from selectivity issues or limited reaction scopes, leaving space for improvements in the synthesis of allylboranes. Although allylboration reactions proceed with excellent control of diastereoselectivity and enantioselectivity when chiral allylboranes are used,^{1c-e} the difficulties in preparing structurally complex allylboranes have prevented their use in more advanced settings such as coupling of structurally complex fragments in total synthesis.

Transition metal catalyzed insertions of carbenoids derived from diazo compounds into B-H bonds has been recently reported as an effective strategy to form C–B bonds.⁸ These reactions can be rendered enantioselective and produce chiral benzylic boranes when chiral ligands are used in combination with rhodium and copper metal catalysts.^{8,9} A visible lightinduced, transition-metal-free method for carbene insertion into B–H bonds has recently been employed in the synthesis of α -alkoxyorganoboronate esters.¹⁰ Synthesis of allylboranes via insertion of metal carbenoids into B-H bonds, however, remains elusive. Here, we report that Cu(I) catalysts enable construction of C-B bonds to form phosphine-protected allylboranes via direct insertion of vinyldiazoacetates into B-H bonds of borane-phosphine Lewis adducts under mild conditions (Figure 1d). We also demonstrate that allylborane-phosphine Lewis adducts can be used directly in diastereoselective allylation of aldehydes without the need for removal of the phosphine.

Catalytic insertions of metal carbenoids into X–H bonds (X = C, O, N, B, P, etc.) typically employ aryldiazoacetates that readily form a metal carbenoid in the presence of various metal catalyst such as Rh(II), Cu(I), Cu(II), or Ag(I).¹¹ Vinyl-diazoacetates have also been used to generate metal carbenoids in catalytic processes such as cyclopropanations,¹² oxidative rearrangements,¹³ or X–H insertion reactions.¹⁴ These reactions give valuable products, but they also highlight the difficulties in handling and selectively transforming the vinyldiazoacetates. The choice of catalyst is of great importance in such reactions: the chemoselectivity depends

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Table 1. Optimization of Reaction Conditions for B-H Insertion of Vinyldiazoacetate 7a into Borane-Lewis Base Adducts 8



	catalyst	8	cat. [mol %]	10 [mol %]	NaBArF [mol %]	7a [equiv]	$T [^{\circ}C]$	yield [%]
1	AgOTFA	8b	5.0	0	0	1.20	60	0
2	AgOTf	8b	5.0	0	0	1.20	60	0
3	AgBF ₄	8b	5.0	0	0	1.20	60	0
4	CuCl	8b	5.0	0	6.0	1.20	60	11
5	Cu(MeCN) ₄ PF ₆	8b	5.0	0	6.0	1.20	60	47
6	(CuOTf) ₂ -PhMe	8b	5.0	0	6.0	1.20	60	29
7	$Cu(OTf)_2$	8b	5.0	0	6.0	1.20	60	30
8	CuI	8b	5.0	0	6.0	1.20	60	25
9	$Rh_2(OAc)_4$	8b	1.0	0	0	1.20	60	0
10	$Rh_2(esp)_2$	8b	1.0	0	0	1.20	60	0
11	-	8b	0	0	6.0	1.20	60	0
12	Cu(MeCN) ₄ PF ₆	8a	5.0	0	6.0	1.20	60	59
13	$Cu(MeCN)_4PF_6$	8c	5.0	0	6.0	1.20	60	33
14	Cu(MeCN) ₄ PF ₆	8d	5.0	0	6.0	1.20	60	27
15	Cu(MeCN) ₄ PF ₆	8e	5.0	0	6.0	1.20	60	0
16	Cu(MeCN) ₄ PF ₆	8f	5.0	0	6.0	1.20	60	0
17	Cu(MeCN) ₄ PF ₆	8g	5.0	0	6.0	1.20	60	0
18	Cu(MeCN) ₄ PF ₆	8h	5.0	0	6.0	1.20	60	29
19	Cu(MeCN) ₄ PF ₆	8b	5.0	6.0	6.0	1.00	RT	57
20	Cu(MeCN) ₄ PF ₆	8b	10	12	12	1.00	RT	52
21	Cu(MeCN) ₄ PF ₆	8b	5.0	6.0	6.0	1.20	RT	61
22	Cu(MeCN) ₄ PF ₆	8b	5.0	6.0	6.0	1.50	RT	61
23	$Cu(MeCN)_4PF_6$	8b	5.0	6.0	6.0	3.00	RT	33
24	$Cu(MeCN)_4PF_6$	8b	2.0	2.4	2.4	1.20	RT	8
25	$Cu(MeCN)_4 PF_6$	8b	5.0	6.0	6.0	1.20	60	92

on the metal catalyst, and the selection of catalyst may be restricted to those that are stable in the presence of specific reactants. Furthermore, the possibility of dimerization of the starting vinyldiazoacetates via cyclopropanation or direct coupling can become a significant problem especially in comparison to aryldiazoacetates.¹⁵ With this in mind, we commenced our scouting study with the search for a suitable combination of metal catalyst and the borane–Lewis base adduct that would allow for B–H insertion of vinyl-diazoacetates.

After the initial screen of reaction partners and conditions, our first focused optimization attempts were carried out with diazoacetate 7a and phosphine– or amine–borane adducts 8 (Table 1). We explored Ag(I), Cu(I), Cu(II), and Rh(II) salts as catalysts known to form metal carbenoids from diazo compounds (entries 1-10).¹¹ The NaBArF additive did not catalyze the reaction itself (entry 11), but it improved the reactivity in some cases and was used consistently to ensure better solubility of the catalysts. The B–H insertion products were observed only when Cu(I) or Cu(II) salts were used. No conversion of diazo compound was observed when Ag(I)

sources were used. Rh(II) sources, however, engaged with diazoacetate but yielded complex mixtures of products, presumably due to instability of the Rh catalyst in the presence of phosphine from the phosphine-borane adduct.⁸ When Cu(MeCN)₄PF₆ was used in chloroform, the desired product was produced in 47% yield at elevated temperature. Attempts to improve the yields by the use of borane complexes with other N- and P-centered Lewis bases revealed that the B-H insertion reactions are sensitive to the steric properties and basicity of the Lewis base (entries 12-18). The borane complex with triphenylphosphine 8e did not engage in B-H insertions. When diphenylmethylphosphine-borane adduct 8d was used, the B-H product was obtained in 27% yield, and this steadily increased in the series from dialkylphenyl- (8c, 33%), tributyl- (**8b**, 47%), and trimethylphosphine (**8a**, 59%). Among amine-borane adducts, only tributylamine-borane 8h afforded the desired product in 29% yield. Further optimization was carried out with tributylphosphine-borane as the cheaper and easier to handle alternative to 8a.

The reactions at lower temperature suffered from low conversion. Although this improved at high temperature, the vields remained similar. We hypothesized that the low conversion was a consequence of the catalyst deactivation. This was corroborated by the change in the color of the reaction mixture which always coincided with the halt in the turnover. Since the addition of catalyst could not effect continued transformation, we hypothesized that the reaction product acts as an inhibitor. This hypothesis was tested in a series of reactions where increasing amounts of 9a were added together with the starting materials 7a and 8b. The conversion rates and yields were inversely proportional to the amount of 9a added to the reaction mixture suggesting that the active catalyst was being sequestered by the reaction products. In an attempt to stabilize the active catalyst, we used the BOX ligand 10 that is commonly employed in Cu(I) and Cu(II) catalyzed reactions. The optimized protocol, which included mixing of the precatalyst with ligand 10 prior to the reaction, proved instrumental in improving the yields to the levels above 90% (Table 1, entries 19–25).

Having a better understanding of the optimal reaction conditions, the focus was shifted to testing the reaction scope with a set of substituted vinyldiazoacetates. The vinyldiazoacetates were prepared via Regitz diazotransfer to the β , γ -unsaturated esters using *p*-ABSA and DBU as a base.¹⁶ The β , γ -unsaturated esters were, in turn, prepared via Pd-catalyzed CO-insertion into cinnamyl alcohol derivatives¹⁷ or via the Wittig olefination of aldehydes with (2-carboxyethyl)triphenylphosphonium bromide¹⁸ followed by a simple esterification protocol.¹⁹

Both aryl and alkyl substituted vinyldiazoacetates produced the desired B-H insertion products (Scheme 1). The yields for the alkyl substituted allylboranes (9d-9f) were markedly lower than those for the corresponding aryl substituted products (9a-9c and 9g-9w), which may be a consequence of the lower stability of the carbenoid intermediates in the synthesis of the former.²⁰ The aryl substituted vinyldiazoacetates reacted well to produce the B-H insertion products in good yields (46%-92%). Similar reactivity was seen for electron-rich (9g-9i) and electron-poor substrates (9p-9r) with slightly better yields observed with the latter. Further, substitution in the ortho position of the aryl substituent led to lowered yields in electron-rich substrates (9i). Aryl halogenides, including those suitable for further functionalization, were well tolerated with yields between 62% and 72% (9j-9n). Thiophenyl- (9s) or furanyl-substituted substrates (9t) were also well tolerated in these reactions as were the vinvldiazoacetates with bulkier (9u and 9w) and activated esters (9v). Finally, in addition to the vinyldiazoacetates, corresponding ketones undergo B-H insertion reactions although with lower yields (9x). The moderate yields observed with some starting materials could be a consequence of the competitive intramolecular cyclization of the vinyldiazoacetates to produce pyrazoles.²¹

After establishing that *E*-substituted vinyldiazoacetates produce the *E*-1,2-disubstituted allylboranes, we explored the possibility of preparing trisubstituted and *Z*-disubstituted allylboranes using a similar protocol (Scheme 2). We expected that the competing intramolecular cyclization of vinyldiazoacetate would be slower when an additional substituent is present at C4 of the vinyldiazoacetate, but we were also concerned that the allylic strain in the metal carbenoids derived from such substrates may hamper B-H insertion. When that substituent was a methyl group, the corresponding allylborane **9aa** was, pleasingly, isolated with a yield of 90%

Scheme 1. B-H Insertion Reaction Scope for Vinyldiazoacetate



Scheme 2. B—H Insertion Using Trisubstituted and Z-Configured Vinyldiazoacetates



(Scheme 2a). As this substituent becomes larger, the yield is lowered, with 34% observed for *n*-butyl substituted allylborane **9ab** and no B-H insertion product **9ac** observed when the

corresponding phenyl substituted vinyldiazoacetate was used. Substitution at C3 proved to be more problematic; the allylboranes **9ad** and **9ae** could not be obtained due to the fast cyclization of the vinyldiazoacetates to the corresponding pyrazoles. Finally, when Z-vinyldiazoacetate Z-7y was used, the only isolated product of B–H insertion, **9y**, was found to be of *E*-configuration (Scheme 2b). It could be construed that the isomerization of the Z-vinyldiazoacetate occurs readily at elevated temperature and the corresponding *E*-vinyldiazoacetate undergoes B–H insertion at a higher rate than the Zisomer.

In analogy to the previous work on Rh- and Cu-catalyzed reactions of diazo compounds, we propose that the B–H bond insertions of vinyldiazoacetates into phosphine–borane adducts also operate via transient formation of the copper carbenoid which readily inserts into B–H bonds in a concerted, asynchronous manner.^{8d,15c}

BOX ligand 10 was used to ensure the stability of the active Cu(I) catalyst in the solution. The use of enantiopure ligand 10 was expected to result in an enantioselective formation of the allylboranes. We have indeed determined that the enantiomeric ratio in the synthesis of 9k was 65:35, suggesting that these reactions could be a foundation for the development of an enantioselective catalytic method for the synthesis of chiral allylboranes (Scheme 3a).

Scheme 3. Reaction of Phosphine-Protected Allylborane to a 5,6-Disubstituted Dihydropyranone in Two Steps



The prepared allylborane-Lewis adducts with phosphines are bench-stable phosphine-protected boranes and easy to handle. Since allylboration of carbonyls using allylboranes depends on the coordination of the carbonyl oxygen to the borane, which triggers the transfer of the allyl group to the carbonyl carbon,^{1c,22} we hypothesized that the adducts with phosphines could be used in such reactions directly. It was expected that an aldehyde could displace the phosphine and make an adduct with the allylborane which would result in the transfer of allyl group to the carbonyl carbon via a chairlike sixmembered transition state that also assures good control of diastereoselectivity. To test this hypothesis, we carried out reactions of 9k with p-bromobenzyl aldehyde. Heating in toluene for 6 h was required to drive the reaction to completion. A simple isomerization/cyclization protocol under acidic conditions resulted in the synthesis of 5,6disubstituted dihydropyranone trans-11²³ which was isolated as a single diastereomer in 67% yield, without prior focused optimization of reaction conditions. Only minor loss in enantiomeric ratio was observed (e.r. 63:37 for 11, compared to 65:35 for the starting material) suggesting that this protocol is robust despite being carried out at elevated temperatures.

The 5,6-disubstituted dihydropyranones are a common motif in natural products that provides a large set of potential applications of the described method.²⁴

In summary, a simple Cu(I)-catalyzed protocol for the synthesis of allylboranes via B-H insertion of carbenoids derived from vinyldiazoacetates into borane-phosphine Lewis adducts was developed. The reactions feature a good scope for the *E*-substituted vinyldiazoacetates and can accommodate some starting materials that produce trisubstituted allylboranes. The products are isolated as bench-stable Lewis adducts with phosphines and can be directly used for diastereoselective allylation on aldehydes without the need for prior removal of the phosphine. When enantiopure BOX ligand is used, the allylboranes are isolated in enantioenriched form. This result is the foundation for our current optimization efforts toward highly enantioselective synthesis of allylboranes which will be reported in due course.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acs.orglett.9b04619.

Detailed experimental procedures, spectral data for all compounds and ¹H, ¹³C, ¹¹B, ¹⁹F, and ³¹P spectra (PDF)

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Notes

The authors declare no competing financial interest.

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