Silver Oxide Mediated Palladium-Catalyzed Cross-Coupling Reaction of Cyclopropylboronic Acids with Allylic Bromides

Han Chen and Min-Zhi Deng*

Laboratory of Organometallic Chemistry, Shanghai Institute of Organic Chemistry, Academia Sinica, 354 Fenglin Lu, Shanghai 200032, China

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The structure of cyclopropane ring is present in many natural products, and the versatility of cyclopropanes and their derivatives as building blocks in organic synthesis has attracted increasing interest in recent years.[1] The diverse methods directed toward constructing the cyclopropane moiety have been amply demonstrated in the literature.[2] Among them, cyclopropylmetal compounds are very useful reagents for the preparation of various cyclopropane derivatives. Generally, they can be prepared by the metalation of halocyclopropyl derivatives with metal or organometallic compounds such as lithium, butylithium,[3] or direct cyclopropanation of some 1- alkenylmetal compounds.[4] However, the synthetic approaches to halocyclopropanes are quiet inefficient,[5] and the obtaining of some vinylmetallic compounds is very troublesome.[6] Recently, the cyclopropylboronic acids attracted the increasing interests of chemists, because they are easily available by the stereodefined cyclopropanation of the corresponding alkenyl boronic acids (esters),[7] which are readily prepared by the hydroboration of alkynes.[8] Moreover, the cyclopropylboronic acids are very stable to air and easily purified by recrystallization from water. Thus, the expansion of the chemical transformations of cyclopropylboronic acid is still one of targets to which organic chemists pay attention.

The Suzuki−Miyaura coupling reactions are very important transformations of organoboranes for the construction of new carbon−carbon bonds because of their many advantages. [9] In our previous papers, we have reported the palladium-catalyzed cross coupling reactions of cyclopropylboronic acids with aryl halides,[10] heteroaromatic halides,[11] and bromoacrylates,[12] and investigated the Suzuki-type reactions of chiral cyclopropylboronic acids.[13] Other groups also reported this type of the reaction under the different conditions.[14] For instance, Marsden reported the palladium-catalyzed cross-coupling of the cyclopropylboronate esters as partners with aryl halides, in the presence of potassium *tert*-butoxide,[14a] and Charette also reported the cross-coupling reaction of cyclopropylboronate esters with iodocyclopropanes.[14b] Recently, Oshima reported the generation of the cyclopropylzinc compounds and their reactions with allyl bromides in the presence of CuCN·2LiCl, giving allyl-substituted cyclopropanes.[15] However, the present synthetic approach to cyclopropylzinc is rather lengthy compared to cyclopropylboronic acids from alkynes. To expand the catalytic cross-coupling reactions of cyclopropylboronic acids, we first studied the cross-coupling reactions of cyclopropylboronic acids with allyl bromides and herein wish to report the preliminary experiment results.

Considering the cyclopropyl group has some sp^2 character,[16] we initially examined the possibility of the reaction of cyclopropylboronic acid with allylic bromide under the coupling condition of 1-alkenylboranes with the allyl bromides, which was reported by Suzuki long ago,[17] but the reaction cannot take place (Table 1, entry 1). In our previous study of the coupling of the cyclopropylboronic acids with aryl bromides or bromoacrylates, we found that $K_3PO_4 \cdot 3H_2O$ as the base was effective,[10−13] but in the case of allylic bromide, the desired product was not detected (Table 1, entry 2). Chartte reported that K^t-OBu in DME was a good combination for the coupling reaction of cyclopropylboronate ester with iodocyclopropanes;[14b] however, this condition did not lead to the

* To whom correspondence should be addressed. Fax: 86-21-64166128.

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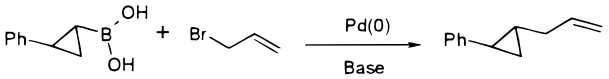
Table 1. Effect of Base and Solvent on the Cross-Coupling Reaction[a]



entry	conditions	yield[b] (%)
1	THF, KOH, reflux, 3% $Pd(PPh_3)_4$	0[c]
2	toluene, $K_3PO_4 \cdot 3H_2O$, 100 °C, 3% $Pd(PPh_3)_4$	0[c]
3	DME, Bu^tOK, 3% $Pd(PPh_3)_4$	
4	DME, CsF, 3% $Pd(PPh_3)_4$	0[c]
5	dioxane, TlOH, 3% $Pd(PPh_3)_4$	trace
6	toluene, Ag_2CO_3, 3% $Pd(PPh_3)_4$	trace
7	toluene, Tl_2CO_3, 3% $Pd(PPh_3)_4$	51
8	toluene, Ag_2O, 3% $Pd(PPh_3)_4$	47
9	dioxane, Ag_2O, 3% $Pd_2(dba)_3$, $CHCl_3$, 30%$AsPh_3$	30
10	toluene, Ag_2O, KOH, 3%$PdCl_2$(dppf)	54[d]
11	dioxane, Ag_2O, KOH, 3%$PdCl_2$(dppf)	64[d]

[a] All the reactions were carried out using a mixture of the *trans*-2-phenylcyclopropylboronic acid (1.0 mmol), allyl bromide (2.0 mmol), and base (2 equiv) in 4 mL of solvent, for 16 h, at 80 °C (except for entry 1) under nitrogen atmosphere. [b] Isolated yields. [c] The unreacted *trans*-2-phenylcyclopropylboronic acid was recovered. [d] A similar yield of coupling product was also obtained using $Pd(PPh_3)_4$ as the catalyst.

desired product in our reaction (Table 1, entry 3), and using CsF as base was also ineffective (entry 4). Failure to react is probably due to slow transmetalation between the cyclopropylboronic acids and π-allylpalladium species because of the low nucleophilicity of cyclopropyl group on the boron. Since the base is essential for the success of Suzuki-type couplings, we then turned our attention to study the effect of other bases. Kishi in his work toward the synthesis of palytoxin showed that Ag_2O and TlOH provided dramatic rate enhancement of some vinylboronic acids couplings.[18] Other groups also reported their accelerating affects in the Suzuki coupling reaction.[19] So we examined the reaction of *trans*-2-phenylcyclopropylboronic acid with allylic bromide using many metallic bases, such as Ag_2O,Ag_2CO_3,Tl_2CO_3, and TlOH.

Using the 10% aqueous TlOH as the base provided traces of the product (entry 5), and employing Ag_2CO_3 in the reaction (entry 6) did not work either. Fortunately, we found that the coupling reaction occurred when Ag_2O was employed as an activator (entry 8). Gillmann found that adding $AsPh_3$ increased the product yields in the silver oxide-assisted palladium-catalyzed cross coupling of methyl 2-halo-2,3-butadienoates with arylboronic acids,[19b] but it gave no improvements in our case (entry 9). After further screening, the combination of Ag_2O and KOH as the base gave the best results while using more polar solvent dioxane (entries 10 and 11). Tl_2CO_3 can also accelerate the coupling of cyclopropylboronic acid with allyl bromide (entry 7).

Considering the high toxicity of Tl_2CO_3, the conditions in entry 11 were used as the optimized condition to investigate the cross-coupling reactions of various *trans*-cyclopropylboronic acids with allyl bromides. The results are shown in Table 2.

It is shown in Table 2 that good yields of the products can be obtained in the reaction of allyl bromides with cyclopropylboronic acids in the presence of Ag_2O suspended in dioxane. Surprisingly, the desired coupling product formation was not observed, when the methyl 4-bromocrotonate was used as the partner (entry 10). The methyl 4-bromocrotonate disappeared extremely rapidly within 10 min (monitored by TLC), but none of the desired product was formed. The ^1H NMR and 2D-^1H NMR spectra of the products in Table 2 showed that the configuration of the cyclopropyl group of organoboron partner was retained, as in the other Suzuki-type reactions. Table 2 also illustrated that the reaction was substrate-controlled regioselective to give only one isomer of coupling product, as determined by ^1H NMR.

In conclusion, we have first demonstrated the cross-coupling reaction of cyclopropyl boronic acids with allyl bromides, and provided a novel convenient stereocontrolled synthetic approach to stereodefined allyl-substituted cyclopropanes.

Experimental Section

Starting Materials. Cinnamyl bromide, 4-bromo-2-methyl-2-butene, and allyl bromide were commercially available and used after distillation. 3-Bromocyclohexene and methyl 4-bromocrotonate were prepared from bromination reaction using NBS in CCl_4 as reported in the literature.[20] The following palladium catalysts were prepared by reported methods: $Pd(PPh_3)_4$,[21] $PdCl_2$(dppf),[22] $Pd_2(dba)_3 \cdot CHCl_3$.[23] $Pd(OAc)_2$ was purchased from Aldrich. The starting racemic cyclopropylboronic acid were readily prepared by using a modified Simmon−Smith reaction as reported.[7,9] ^1H NMR spectra were recorded using $CDCl_3$ or CD_3COCD_3 solutions with tetramethylsilane as internal standard.

The Cross-Coupling Reaction of Allyl Bromides with Cyclopropylboronic Acids for Preparation of *trans*-1-Allyl-2-phenylcyclopropane (3f) Is Representative. To a solution of allyl bromide (2.0 mmol, 242 mg) in dioxane (4 mL), *trans*-2-phenylcyclopropylboronic acid (1.0 mmol, 162 mg), and Ag_2O (2 mmol, 463 mg) were added KOH (2 mmol, 112 mg) and $PdCl_2$(dppf) (0.03 mmol, 22 mg) under a nitrogen atmosphere. The reaction mixture was stirred at 80 °C for 24 h, then cooled to room temperature and diluted with petroleum ether, washed with saturated brine, and dried over magnesium sulfate. The solvents were removed in vacuo and the residue purified by column chromatography, eluting with petroleum ether (60−90 °C) to yield *trans*-1-allyl-2-phenylcyclopropane (**3f**) as a colorless liquid (112 mg, 71%).

***trans*-1-Allyl-2-phenylcycloprane (3f):** ^1H NMR δ ppm 7.05−7.27 (m, 5H), 5.91 (m,1H), 5.10 (dd, J = 17, 1.6 Hz, 1H), 4.99 (dd, J = 10.2, 1.6 Hz, 1H), 2.15 (t, J = 6.5 Hz, 2H), 1.66 (ddd, J = 4.2, 4.8, 9.0 Hz, H_a, 1H), 1.14 (m, H_b, 1H), 0.94 (ddd, J = 9.0, 5.0, 5.2 Hz, H_c, 1H), 0.83 (ddd, J = 8.6, 5.2, 4.8 Hz, H_d, 1H); IR(neat) 912 cm^{-1}; MS *m/e* 158 (M^+, 8.86), 117 (100). In the 2D-^1H NMR, the proton H_a(1.66 ppm) showed strong NOE interaction with proton H_d (0.83 ppm) but very weak NOE interaction with H_c (0.94 ppm) and H_b (1.14 ppm). Anal. Calcd for $C_{12}H_{14}$: C, 91.08; H, 8.92. Found: C, 90.86; H, 9.01.

The following compounds were prepared similarly using the conditions listed in Table 2.

***trans*-1-((*E*)-3-Phenyl-2-propenyl)-2-butylcyclopropane (3a):** yield 82%; colorless liquid; ^1H NMR δ 7.18−7.39 (m, 5H), 6.44 (d, J = 16 Hz, 1H), 6.30 (dt, J = 16, 6.4 Hz, 1H), 2.15 (t, J = 6.5 Hz, 2H), 1.31−1.4 (m, 4H), 1.24−1.29 (m, 2H), 0.91 (t, J = 7 Hz, 3H), 0.55 (m, 2H), 0.30 (m, 2H); IR (neat) 964 cm^{-1}; MS *m/e* 214 (M^+, 4.19), 117 (100), 129 (80), 104 (61.26). Anal. Calcd for $C_{16}H_{22}$: C, 89.65; H, 10.72. Found: C, 89.49; H, 10.47.

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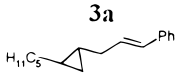
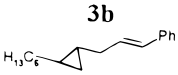
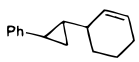
Table 2. Silver Oxide-Assisted Palladium-Catalyzed Cross-Coupling of Allyl Bromides with Cyclopropylboronic Acids[a]

Entry	boronic acids	Allyl bromides	Products[b]	Time	Yields %[c]
1	**1a**	Cinnamyl bromide (1.0 mmol)	 **3a**	2h	82
2	**1b**	Cinnamyl bromide (1.0 mmol)	 **3b**	2h	89
3	**1c**	Cinnamyl bromide (1.0 mmol)	 **3d**	2h	80
5	**1c**	Allyl bromide (2.0 mmol)	 **3e**	24h	89
6	**1d**	Allyl bromide (2.0 mmol)	 **3f**	24h	71
7	**1d**	4-Bromo-2-methyl-2-butene (2.0 mmol)	 **3g**	24h	62
8	**1c**	4-Bromo-2-methyl-2-butene (2.0 mmol)	 **3h**	24h	80
9	**1d**	3-Bromocyclohexene (2.0 mmol)	 **3i**	24h	40[(d)]
10	**1d**	Methyl 4-bromocrotonate (2.0 mmol)	0	10min	0

[a] All the reactions were carried out using a mixture of cyclopropylboronic acids (1.0 mmol) and allyl bromides, 2 equiv of Ag_2O, 2 equiv of KOH (based on boronic acids), and 3 mol % $PdCl_2$(dppf) in 4 mL of dioxane at 80 °C under a nitrogen atmosphere. [b] All the products were identified by ^{1}H NMR, IR, and mass spectral and elemental analysis. [c] Isolated yield. [d] The reaction was carried out at 60 °C.

***trans*-1-((*E*)-3-Phenyl-2-propenyl)-2-pentylcyclopropane (3b):** yield 89%; colorless liquid; ^{1}H NMR δ 7.17−7.37 (m, 5H), 6.43 (d, J = 16 Hz, 1H), 6.27 (dt, J = 16, 6.5 Hz, 1H), 2.15 (t, J = 6.4 Hz, 2H), 1.27−1.44 (m, 8H), 0.89 (t, J = 6.6 Hz, 3H), 0.54 (m, 2H), 0.29 (m, 2H); IR (neat) 964 cm^{-1}; MS *m/e* 228 (M^+, 2.98), 117 (100), 129 (77), 104 (69). Anal. Calcd for $C_{17}H_{24}$: C, 89.32; H, 10.68. Found: C, 89.54; H, 10.64.

***trans*-1-((*E*)-3-Phenyl-2-propenyl)-2-hexylcyclopropane (3d):** yield 80%; colorless liquid; ^{1}H NMR δ 7.15−7.39 (m, 5H), 6.44 (d, J = 16 Hz, 1H), 6.33 (dt, J = 16, 6.5 Hz, 1H), 2.14 (t, J = 6.5 Hz, 2H), 1.26−1.41 (m, 10H), 0.85 (t, J = 6.8 Hz, 3H), 0.55 (m, 2H), 0.27 (m, 2H); IR (neat) 964 cm^{-1}; MS *m/e* 242 (M^+, 5.7), 117 (100), 129 (82), 104 (50). Anal. Calcd for $C_{18}H_{26}$: C, 89.19; H, 10.81. Found: C, 89.11; H, 10.69.

***trans*-1-Allyl-2- hexylcyclopropane (3e):** yield 89%; colorless liquid; ^{1}H NMR δ 5.85 (m, 1H), 5.02 (dd, J = 16, 1.7 Hz, 1H), 4.90 (dd, J = 10.2, 1.7 Hz, 1H), 1.93 (t, J = 5.5 Hz, 2H), 1.27−1.38 (m, 10H), 0.86 (t, J = 6.4 Hz, 3H), 0.46 (m, 2H), 0.20 (m, 2H); IR (neat) 911 cm^{-1}; MS *m/e* 166 (M^+, 4.2), 67 (100), 129 (82), 109 (31). Anal. Calcd. for $C_{12}H_{22}$: C, 86.77; H, 13.23. Found: C, 86.70; H, 13.61.

***trans*-1-(3-Methyl-2-butenyl)-2-phenylcyclopropane (3g):** yield 62%; colorless liquid, ^{1}H NMR δ 7.05−7.35 (m, 5H), 5.26 (t, J = 5.8 Hz, 1H), 2.13 (t, J = 5.8 Hz, 2H), 1.74 (s, 3H), 1.65−1.70 (m, 1H, overlapped with CH_3), 1.65 (s, 3H), 1.10 (m, 1H), 0.0.79−0.92 (m, 1H + 1H); IR (neat) 1610 cm^{-1}; MS *m/e* 186 (M^+, 2.7), 117 (100), 82 (59). Anal. Calcd for $C_{14}H_{18}$: C, 90.26; H, 9.74. Found: C, 90.11; H, 9.40.

***trans*-1-(3-Methyl-2-butenyl)-2-hexylcyclopropane (3h):** yield 80%; colorless liquid; ^{1}H NMR δ 5.20 (t, J = 5.7 Hz, 1H), 1.91 (t, J = 5.8 Hz, 2H), 1.70 (s, 3H), 1.59(s, 3H), 1.26−1.41 (m, 10H), 0.88 (t, J = 6.5 Hz, 3H), 0.42 (m, 2H), 0.17 (m, 2H); IR (neat) 1021 cm^{-1}; MS *m/e* 194 (M^+, 3.8), 69 (100), 82 (64). Anal. Calcd for $C_{14}H_{26}$: C, 86.52; H, 13.48. Found: C, 86.75; H, 13.20.

***trans*-1-(3-Cyclohexenyl)-2-phenylcyclopropane (3i):** yield 40%; colorless liquid; ^{1}H NMR δ 7.06−7.26 (m, 5H), 5.72 (m, 2H), 1.98 (m, 1H), 1.70−1.82 (m, 2H), 1.54−1.68 (m, 4H + 1H), 1.06 (m, 1H), 0.83−0.94 (m, 1H + 1H); IR (neat) 1678 cm^{-1}; MS *m/e* 198 (M^+, 3.8), 94 (100), 117 (63), 82 (59). Anal. Calcd for $C_{15}H_{18}$: C, 90.85; H, 9.15. Found: C,90.51; H, 9.20.

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