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Performance of SCS Palladium Pincer Complexes in Borylation of Allylic Alcohols. Control of the Regioselectivity in the One-Pot Borylation−Allylation Process

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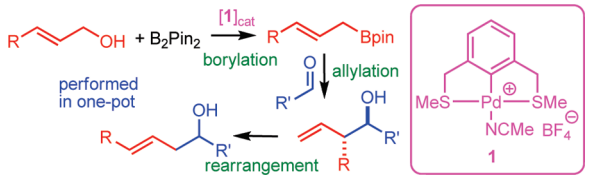
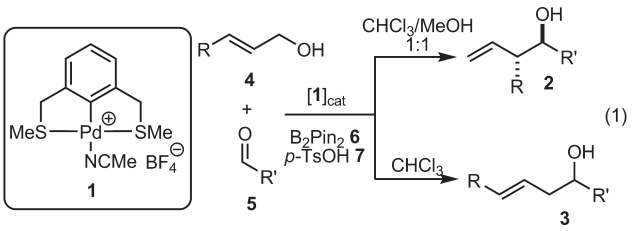


One-pot borylation−allylation reactions of aldehydes and allylic alcohols were performed under various reaction conditions. The borylation of allylic alcohols was performed using a very efficient SCS palladium pincer-complex catalyst. The regioselectivity of the allylation depends on the applied solvent. The reaction in $CHCl_3$ gave the linear allylic product; however, when MeOH was added to the reaction mixture, the branched allylic product was formed.

Allylboronates are very attractive reagents for selective carbon−carbon bond formation reactions.[1−6] One of the most widely employed area is allylation of aldehydes and other electrophilic reagents.[7−13] As a consequence, several new procedures have appeared recently on the synthesis of allylboronates, reactions with electrophiles, and even on rearrangement possibilities of the reaction products.[7,13−21]

In the recent years, we have published[13,19−21] a series of papers on application of pincer-complex catalysis[22−24] for synthesis of allylboronates from diboronate reagents and various allylic precursors. In one of the most useful versions of these reactions, allylic alcohols are used as precursors for synthesis of functionalized allylboronates.[13,21] These processes are also suitable for the design of one-pot procedures[13,25−27] in which the in situ generated allylboronates are reacted with electrophiles.



(1)

One of the simplest one-pot procedures is based on the reaction of aldehydes with allylboronates generated from allylic alcohols.[13,25−27] We have now found that SCS pincer-complex **1** has some very attractive features in these reactions (eq 1). This complex displayed a very high activity for borylation in different solvents, such as DMSO, $CHCl_3$, and MeOH. When the one-pot borylation−allylation was performed in a MeOH/$CHCl_3$ (1:1) mixture, the usual branched allylic product **2** resulted; however, when the reaction was conducted in neat $CHCl_3$, selective formation of the corresponding linear product, **3** was observed (eq 1 and Table 1).

Under the optimized conditions (Table 1), the one-pot reactions were completely selective to formation of a certain allylic regioisomer. Thus, when the reaction mixture contained MeOH (entries 1 and 4), the branched allylic products **2a**,**c** were formed exclusively with high stereoselectivity. We obtained the same selectivity in our previously reported[13,25−27] one-pot processes conducted with pincer-complex catalysts in DMSO/MeOH mixtures. However, when the reaction was performed in neat $CHCl_3$, the linear products **3a**−**e** were

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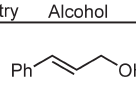
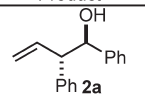
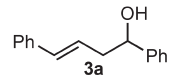
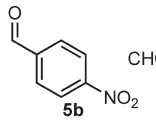
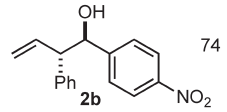
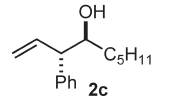
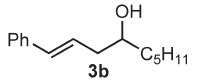
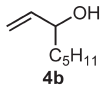
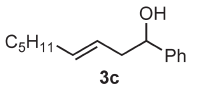
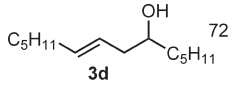
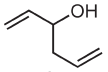
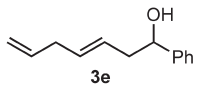
TABLE 1. One-Pot Allylation of Aldehydes with Allylic Alcohols under Various Conditions[a]

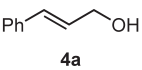
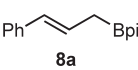
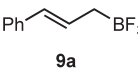
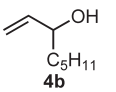
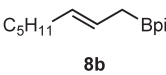
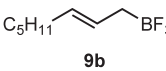
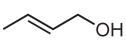
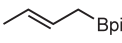
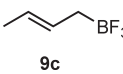
Entry	Alcohol	Aldehyde	Solv.	Time[h][b]	Product	Yield[%][c]
1	 **4a**	 **5a**	MeOH $CHCl_3$	24	 **2a**	72
2	**4a**	**5a**	$CHCl_3$	16	 **3a**	73
3	**4a**	 **5b**	$CHCl_3$	24	 **2b**	74
4[d]	**4a**	 **5c**	MeOH $CHCl_3$	24	 **2c**	78
5	**4a**	**5c**	$CHCl_3$	20	 **3b**	60
6	 **4b**	**5a**	$CHCl_3$	20	 **3c**	74
7[e]	**4b**	**5c**	$CHCl_3$	18	 **3d**	72
8	 **4c**	**5a**	$CHCl_3$	18	 **3e**	63

[a]In a typical reaction, **4** (0.15 mmol), **5** (0.18 mmol), **6** (0.18 mmol), **7** (5 mol %), and SCS catalyst **1** (5 mol %) were dissolved in the corresponding solvent and the mixture stirred at 50 °C. [b]Reaction time. [c]Isolated yield. [d]**5c** (0.30 mmol). [e]**5c** (0.45 mmol) and **7** (10 mol %).

obtained (entries 2 and 5−8). The selectivity was the same for both benzaldehyde **5a** and hexanal **5c**. However formation of the linear product from aliphatic aldehyde **5c** required somewhat longer reaction times and, in some cases, a larger excess of **5c** (entries 4 and 7) for completion than the analogous processes with benzaldehyde **5a**. Nitrobenzaldehyde (**5b**) gave only the branched product (**2b**) independently from the employed reaction conditions (entry 3). The substituents on the allylic alcohol component did not influence the outcome of the reaction. Accordingly, the described procedure can be a useful method for synthesis of certain aliphatic and aromatic homoallylic alcohols with a high level of regiocontrol.

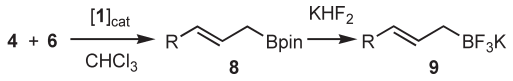
As we have reported previously,[13,21,28] the process can be interrupted at the borylation step when aldehyde is not employed. A particularly interesting finding is that use of SCS catalyst **1** does not require application of a strong acid (such as **7**) for the borylation step when bis(pinacolato) diboron **6** was employed as boronate source (eq 2). Our previous studies[13,28] have shown that the borylation using (very expensive) diboronic acid can be carried out with a broad variety of palladacycles, even in the absence of strong

TABLE 2. Borylation of Allylic Alcohols with SCS Catalyst 1[a]

Entry	Alcohol	Time[h][b]	Product	Yield[c]
1	 **4a**	16	 **8a**	79
2	**4a**		 **9a**	77
3	 **4b**	20	 **8b**	73
4	**4b**		 **9b**	72
5	 **4d**	16	 **8c**	73
6	**4d**		 **9c**	60

[a]In a typical reaction, **4** (1.0 mmol), **6** (1.2 mmol), and catalyst **1** (5 mol %) were dissolved in $CHCl_3$ and the mixture stirred at 50 °C. [b]Reaction time. [c]Isolated yield (%).

acids; however, in the case of **6**, catalytic amounts of strong acid such as **7** had to be used. Thus, by application of SCS catalyst **1**, the synthetic scope of the borylation under neutral conditions can be further broadened. We demonstrated this by applying the new, improved conditions for a couple of allylic alcohol substrates (Table 2). Thus, using SCS catalyst **1** both cinnamyl alcohol **4a** and aliphatic alcohols **4b**,**d** could be readily borylated with **6** as boronate source under neutral conditions to provide linear allylboronates **8a**−**c**. The obtained pinacolboronates **8a**−**c** could be readily converted to their trifluoroborate analogs **9a**−**c**, which are also useful synthons in palladium-catalyzed coupling with sulfonylimines[29,30] and in other reactions.[31−34]



(2)

In order to get a deeper insight in the mechanism of the presented one-pot transformations, we monitored the process with ^{1}H NMR spectroscopy (Figure 1) in $CDCl_3$. These studies showed decrease of the concentration of reactants **4a** and **5a** and an initial formation of the branched product **2a**. Interestingly, the amount of allylboronate **8a** was very low under the entire reaction sequence, indicating that the in situ formed **8a** reacted immediately with benzaldehyde **5a**. The concentration of the branched product **2a** passes a maximum value after 4 h. After approximately 2 h, the linear product **3a** appeared, and then its amount increased monotonously. Apparently, the formation of the linear product **3a** takes place on the cost of the branched product **2a**.

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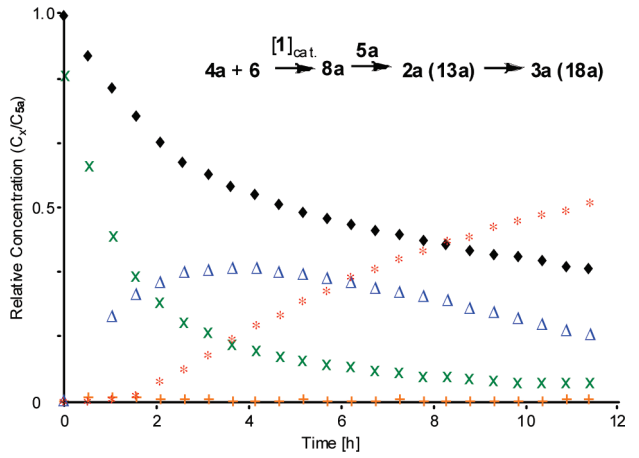

FIGURE 1. Changing of the concentration of the reaction component in the one-pot allylation of benzaldehyde (**5a**) with cinnamyl alcohol (**4a**) in $CDCl_3$. Legend: ♦, benzaldehyde **5a**; **X**, cinnamyl alcohol **4a**; +, cinnamyl boronate **8a**; Δ, branched product **2a** (or its boronic ester **13a**, see Figure 2); ∗, linear product **3a** (or its boronic ester **18a**, see Figure 2).

Based on the above findings, a reasonable mechanistic scheme can be proposed (Figure 2). Accordingly, the borylation of the allylic alcohol substrate **4** starts with activation of the hydroxy group. Our previous studies indicate[13] that this may take place by transesterification of diboron reagent **6** with the allylic alcohol **4** to give boronate ester **10**. This process may also be catalyzed by SCS complex **1**. The next step is the transfer of a boronate functionality from **6** or **10** to the palladium atom of catalyst **12a** to give complex **12b**. A similar reaction was observed[35,36] for the analogous process using hexamethylditin as the metal source. This process gives **11** in which the hydroxy group is activated as in **10**. The boronate group is then transferred from palladium complex **12b** to the activated allylic alcohol substrate **10** or **11**. This results in allylboronate product **8**. In the absence of aldehydes, this compound can be isolated from the reaction mixture (eq 2, Table 2). In the presence of aldehyde **5**, a highly stereoselective allylation occurs affording homoallylic boronate ester **13**. In the presence of methanol (or water), this boronate ester undergoes solvolysis to provide the branched allylic product of the reaction (Table 1, entries 1 and 4). When the reaction is conducted in neat $CHCl_3$ (or $CDCl_3$, Figure 1), aldehyde **5** and **13** may form acetal **14**. According to the studies[37−39] by Ramachandran, Nokami, and Loh, this process takes place particularly easily with boronate esters in the presence of Lewis acid catalysts. Under the applied conditions, both SCS complex **1** and toluenesulfonic acid **7** could serve as Lewis acid catalyst.

Compound **14** may readily be converted to 2-oxonia intermediate **15**.[37] Intermediate **15** readily undergoes[37] [3,3]-sigmatropic rearrangement to **16**, which then gives the rearranged acetal **17**. Linear acetal **17** is thermodynamically more stable than the branched acetal **14**, which is probably the driving force of the rearrangement. Decomposition of **17** to **18** recovers the aldehyde component **5**, which may catalyze the rearrangement of **13** to **18**. Hydrolysis of **18** leads to formation of the linear product **3** (Table 1, entries 2 and 5−8).

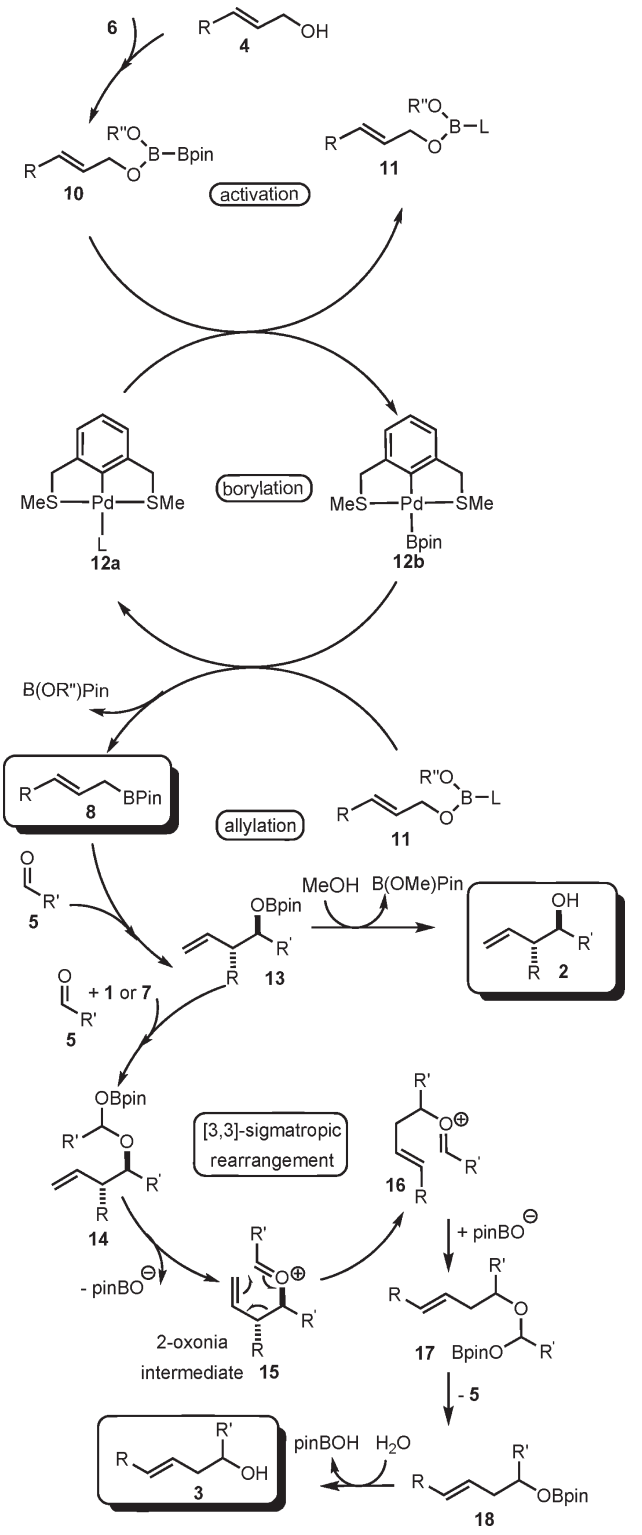

FIGURE 2. Proposed mechanism of the one-pot borylation−allylation reaction under different reaction conditions.

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The rearrangement readily proceeds with simple aromatic and aliphatic aldehydes **5a** and **5c**, respectively; however, when nitrobenzaldehyde **5b** was used, only the branched product **2b** could be obtained (Table 1, entry 3). Formation of acetal **14** is probably encumbered by the electron-withdrawing nitro group in **5b**.

Experimental Section

All reactions were performed in freshly distilled solvents under ambient atmosphere. The palladium pincer-complex **1** was prepared according to the literature procedure.[40] All other chemicals were obtained from commercial sources and used as received. The detailed experimental procedures and full characterization of all products are given in the Supporting Information.

General Procedure A: Allylation of Aldehydes (Table 1). The corresponding allylic alcohol **4** (0.15 mmol) was dissolved in chloroform (0.4 mL) or a mixture of methanol and chloroform (0.2 mL/0.2 mL) (see Table 1), followed by addition of bis(pinacolato)diboron (**6**) (0.18 mmol), pincer complex **1** (0.0075 mmol, 5 mol %), *p*-toluenesulfonic acid (**7**) (0.0075 mmol, 5 mol %), and aldehyde **5** (0.18 mmol). This reaction mixture was then stirred at 50 °C for the allotted times listed in Table 1. After evaporation of the solvent, the products **2a**−**c** and **3a**−**e** were purified by silica gel chromatography.

3-Phenyl-1-nonen-4-ol (2c). Compound **2c** was prepared in a mixture of methanol and chloroform (0.2 mL/0.2 mL) according to general procedure A, except that 0.30 mmol of aldehyde **5c** was used. Product **2c** was isolated in 78% yield (26.0 mg) using pentane/diethyl ether (5:1 ratio) as eluent for silica gel chromatography. The NMR data obtained for **2c** are in agreement with previously reported literature values:[13] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35−7.29 (m, 2H), 7.25−7.18 (m, 3H), 6.13 (ddd, J = 9.0, 10.3, 17.0 Hz, 1H), 5.23 (d, J = 10.3 Hz, 1H), 5.20 (d, J = 17.0 Hz, 1H), 3.79 (dt, J = 3.8, 7.4 Hz, 1H), 3.25 (dd, J = 7.4, 9.0 Hz, 1H), 1.71 (br s, 1H), 1.52−1.14 (m, 8H), 0.85 (t, J = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 141.7, 138.4, 128.7, 128.0, 126.6, 117.8, 74.0, 57.4, 34.4, 31.8, 25.4, 22.6, 14.0; HRMS (pos. ESI) m/z calcd for $C_{15}H_{22}NaO$ $[M + Na]^+$ 241.1563, found 241.1558.

(*E*)-8-Tetradecen-6-ol (3d). Compound **3d** was prepared in chloroform (0.4 mL) according to general procedure A, except that 0.45 mmol of aldehyde **5c** and 10 mol % of *p*-toluenesulfonic acid (**7**) was used. Product **3d** was isolated in 72% yield (23.0 mg) using CH_2Cl_2 as eluent for silica gel chromatography: ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.54 (td, J = 6.7, 15.2 Hz, 1H), 5.45−5.36 (m, 1H), 3.61−3.54 (m, 1H), 2.27−2.20 (m, 1H), 2.10−1.98 (m, 3H), 1.57 (br s, 1H), 1.48−1.23 (m, 14H), 0.89 (t, J = 6.9 Hz, 3H), 0.88 (t, J = 6.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 134.8, 125.8, 70.9, 40.7, 36.7, 32.6, 31.9, 31.4, 29.2, 25.4, 22.6, 22.5, 14.0; HRMS (pos ESI) m/z calcd for $C_{14}H_{28}NaO$ $[M + Na]^+$ 235.2032, found 235.2035.

General Procedure B: Preparation of Pinacolboronates 8a−c and Potassium Trifluoroborates 9a−c (Table 2). The corresponding allylic alcohol **4** (1.00 mmol) was dissolved in chloroform (2.0 mL) followed by addition of bis(pinacolato)diboron (**6**) (1.20 mmol) and pincer-complex **1** (0.05 mmol, 5 mol %). This reaction mixture was then stirred at 50 °C for the allotted times listed in Table 2. Thereafter, 2.0 mL of pentane was added followed by flash chromatography using pentane/diethyl ether (95:5 ratio) as eluent to yield analytically pure allylboronates **8a**−**c**. **Potassium Trifluoroborates from Pinacolboronates.** To the purified allylboronates was added 6.0 equiv of KHF_2 in water/methanol (2.0 mL/2.0 mL), and this mixture was stirred at room temperature for 2 h. Thereafter, the precipitate was separated, and the filtrate containing the crude potassium trifluoroborates was evaporated. The remaining solid was extracted with acetone and filtered through cotton. Subsequently, the solvent was evaporated, and potassium trifluoroborates **9a**−**c** were recrystallized from acetone/diethyl ether.

(*E*)-2-(3-Phenyl-2-propenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (8a). Compound **8a** was prepared according to general procedure B. Compound **8a** was isolated in 79% yield (192.0 mg) using pentane/diethyl ether (95:5 ratio) as eluent for silica gel chromatography. The NMR data obtained for **8a** are in agreement with previously reported literature values:[28] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35−7.14 (m, 5H), 6.37 (d, J = 15.8 Hz, 1H), 6.29 (td, J = 7.1, 15.8 Hz, 1H), 1.88 (d, J = 7.1 Hz, 2H), 1.26 (s, 12H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 138.2, 130.2, 128.3, 126.5, 126.3, 125.8, 83.4, 24.8; ^{11}B NMR (128 MHz, $CDCl_3$) δ 32.9; HRMS (pos ESI) m/z calcd for $C_{15}H_{21}BNaO_2$ $[M + Na]^+$ 267.1530, found 267.1527.

(*E*)-2-(2-Octenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (8b). Compound **8b** was prepared according to general procedure B. Compound **8b** was isolated in 73% yield (174.3 mg) using pentane/diethyl ether (95:5 ratio) as eluent for silica gel chromatography: ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.46−5.33 (m, 2H), 1.98−1.92 (m, 2H), 1.62 (d, J = 6.4 Hz, 2H), 1.34−1.20 (m, 6H), 1.24 (s, 12H), 0.86 (t, J = 6.8 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 131.0, 124.6, 83.1, 32.7, 31.3, 29.3, 24.7, 22.5, 14.1; ^{11}B NMR (161 MHz, $CDCl_3$) δ 33.0; HRMS (pos ESI) m/z calcd for $C_{14}H_{27}BNaO_2$ $[M + Na]^+$ 261.1999, found 261.1991.

Potassium (*E*)-Trifluoro(3-phenyl-2-propenyl)borate (9a). Compound **9a** was prepared according to general procedure B. **9a** was isolated in 77% yield (171.9 mg) calculated from allylic alcohol **4a**. The NMR data obtained for **9a** are in agreement with previously reported literature values:[21] ^{1}H NMR (400 MHz, acetone-d_6) δ 7.26 (d, J = 7.9 Hz, 2H), 7.19 (t, J = 7.9 Hz, 2H), 7.02 (t, J = 7.9 Hz, 1H), 6.51 (td, J = 7.8 Hz, 15.8 Hz, 1H), 6.08 (d, J = 15.8 Hz, 1H), 1.25 (br s, 2H); ^{13}C NMR (101 MHz, acetone-d_6) δ 140.8, 136.4, 129.0, 126.4, 126.0, 125.9; ^{11}B NMR (128 MHz, acetone-d_6) δ 4.5; ^{19}F NMR (377 MHz, acetone-d_6) δ −139.9; HRMS (neg. ESI) m/z calcd for $C_9H_9BF_3$ $[M - K]^-$ 185.0757, found 185.0754.

Monitoring the One-Pot Transformation of Cinnamyl Alcohol (4a) by ^{1}H NMR Spectroscopy (Figure 1). In an NMR tube, cinnamyl alcohol **4a** (0.15 mmol) was dissolved in $CDCl_3$ (0.4 mL) followed by addition of bis(pinacolato)diboron **6** (0.18 mmol), *p*-toluenesulfonic acid (**7**) (5 mol %), aldehyde **5a** (0.18 mmol), and palladium catalyst **1** (5 mol %). The reaction was conducted in the NMR tube at 50 °C for 12 h. The progress of the reaction was monitored using ^{1}H NMR spectroscopy (400 MHz) by measuring the integrals for the corresponding peaks of **2a**, **3a**, **4a**, **5a**, and **8a**. Due to the different relaxation times and partial overlap of certain peaks, the estimated error of this measuring method is about 10−15%.[41]

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Supporting Information Available: Detailed experimental procedures; characterization and NMR spectra of the products. This material is available free of charge via the Internet at http://pubs.acs.org.

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