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Phosphane-Free Palladium-Catalyzed Carbonylative Suzuki Coupling Reaction of Aryl and Heteroaryl Iodides

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The carbonylative cross-coupling reaction of an arylboronic acid with an aryl iodide and a heteroaryl iodide to give unsymmetrical biaryl ketones was carried out by using $Pd(tmhd)_2/Pd(OAc)_2$ as a phosphane-free catalyst. Various aryl and heteroaryl iodides with different arylboronic acid derivatives provided good to excellent yields of the desired products under optimized reaction conditions.

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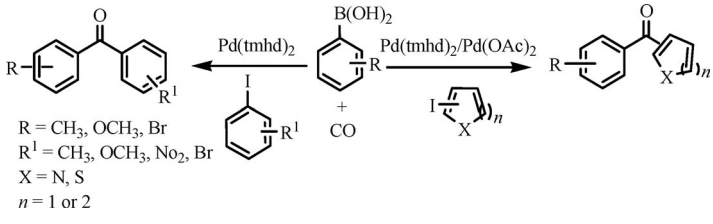
Introduction

The cross-coupling reaction is one of the most straightforward and general methods for the formation of carbon–carbon bonds.[1] In this area, the transition-metal-catalyzed, three-component, cross-coupling reaction between organometallic reagents, carbon monoxide, and organic halides is now considered as a useful tool for the synthesis of ketones. Biaryl ketones are important moieties in many biologically active molecules, natural products, and pharmaceuticals.[2] Traditionally, biaryl ketones are synthesized by the transition-metal-catalyzed, three-component, cross-coupling reaction between arylmetal reagents, carbon monoxide, and aryl electrophiles. Various arylmetal reagents like magnesium,[3] silicon,[4] aluminum,[5] and tin[6] have been reported. However, the known protocols are limited due to the formation of side products ensuing from direct coupling of the aryl groups.

Among carbonylative cross-coupling reactions, the Suzuki carbonylative coupling reaction is one of the most promising methods for direct synthesis of biaryl ketones from carbon monoxide, aryl halides, and arylboronic acids. Several groups have reported a variety of palladium-based catalytic systems such as $PdCl_2(PPh_3)_2$,[7] $PdCl_2(PPh_3)_2$/$PdCl_2$(dppf),[8] $Pd(OAc)_2$–imidazolium salts,[9] $Pd(OAc)_2$/*N*,*N*-bis(2,6-diisopropylphenyl)dihydroimidazolium chloride,[10] and so on for this reaction. Usually, the catalyst is generated in situ from a palladium source in the presence of highly efficient N/P-containing ligands. In spite of the significant advances in this area, there are very few reports that employ phosphane-free Pd complexes as catalysts. Moreover, there are a limited number of reports on this reaction using heteroaryl halides together with different phenylboronic acid derivatives.

In continuation of our previous work on phosphane-free carbonylation reactions,[11] we report here a facile protocol for the carbonylative Suzuki coupling of boronic acids with aryl and heteroaryl iodides by using $Pd(tmhd)_2/Pd(OAc)_2$ (tmhd = 2,2,6,6-tetramethyl-3,5-heptanedionate) as the catalyst (Scheme 1). Excellent yields of the desired biaryl



Scheme 1. Carbonylative Suzuki coupling reaction of aryl and heteroaryl iodides with phenylboronic acid.

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ketones were obtained by using only 2 to 2.5 mol-% of the catalyst under optimized reaction conditions.

Results and Discussion

The reaction of iodobenzene with phenylboronic acid in the presence of $Pd(tmhd)_2$ was chosen as a model reaction, and the influence of various reaction parameters such as catalysts {$Pd(OAc)_2$, palladium bis(acetylacetonate) [$Pd(acac)_2$], palladium bis(ethyl acetoacetonate) [$Pd(eaa)_2$], and $PdCl_2$} were tested (Table 1, Entries 1, 3–5). All these complexes showed a lower activity with respect to $Pd(tmhd)_2$, which provided a 96% yield of the desired product. The influence of different solvents on the carbonylative Suzuki coupling reaction was then studied (Table 1, Entries 6–10). Solvents like anisole (96%), toluene (78%), *N*,*N*-dimethylforamide (DMF, 56%), 1,4-dioxane (62%), THF (68%), and water (34%) were screened. It was observed that the reaction gave better results with the use of anisole as a solvent. An inorganic base such as K_2CO_3 led to a better yield than did the use of Et_3N (Table 1, Entry 11). The influences of temperature and CO pressure were also studied (Table 1, Entries 12 and 13), and the optimum temperature and pressure were used for further studies. Hence, the optimized reaction conditions are: $Pd(tmhd)_2$ (2 mol-%), anisole, K_2CO_3, 80 °C, CO (100 psi), 6 h.

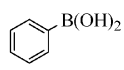
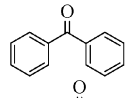
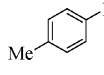
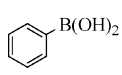
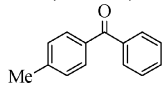
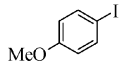
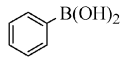
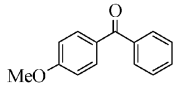
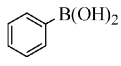
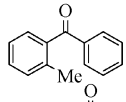
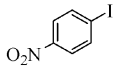
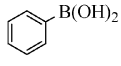
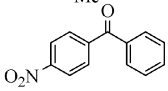
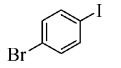
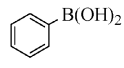
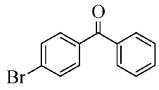
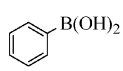
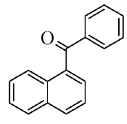
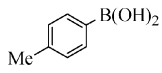
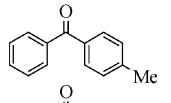
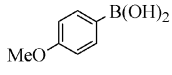
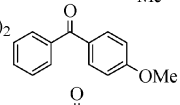
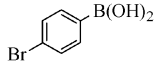
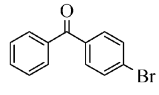
Table 1. Effect of reaction parameters on the carbonylative Suzuki coupling reaction of iodobenzene with phenylboronic acid.[a]

Entry	Catalyst	Solvent	Yield [%][b]
	Effect of catalyst		
1	$Pd(OAc)_2$	anisole	84
2	$Pd(tmhd)_2$	anisole	96
3	$Pd(acac)_2$	anisole	87
4	$Pd(eaa)_2$	anisole	82
5	$PdCl_2$	anisole	79
	Effect of solvent		
6	$Pd(tmhd)_2$	toluene	78
7	$Pd(tmhd)_2$	DMF	56
8	$Pd(tmhd)_2$	water	34
9	$Pd(tmhd)_2$	THF	68
10	$Pd(tmhd)_2$	dioxane	62
11[c]	$Pd(tmhd)_2$	anisole	76
12[d]	$Pd(tmhd)_2$	anisole	65
13[e]	$Pd(tmhd)_2$	anisole	74

[a] Reaction conditions: iodobenzene (1 mmol), phenylboronic acid (1.2 mmol), catalyst (2 mol-%), K_2CO_3 (3 mmol), solvent (10 mL), 6 h, 80 °C, CO (100 psi). [b] GC yield. [c] Et_3N was used as the base instead of K_2CO_3. [d] T = 50 °C. [e] p(CO) = 50 psi.

The optimized reaction conditions were then applied to the carbonylative Suzuki coupling reaction of various aryl iodides with different phenylboronic acid derivatives (Table 2, Entries 1–10). Various electron-donating and electron-withdrawing groups such as -CH_3, -OCH_3, -NO_2, and -Br on both aryl iodides and phenylboronic acids were well tolerated to give the desired ketone in good to excellent yield. Phenylboronic acid was found to react smoothly with iodobenzene, providing 96% yield of benzophenone. Electron-donating groups such as -CH_3 and -OCH_3 on the iodoaryl partner gave excellent results. The reaction of sterically hindered *o*-iodotoluene with phenylboronic acid also provided good yield of the desired ketone under the optimized reaction conditions (Table 2, Entry 4). The reaction also worked well with less-reactive *p*-nitroiodobenzene, providing 72% yield of *p*-nitrobenzophenone (Table 2, Entry 5). Aryl bromides were not reactive under the carbonylation reaction conditions, and thus, *p*-bromoiodobenzene could be converted into a bromophenyl ketone selectively (Table 2, Entry 6). The carbonylation of bulky 1-iodonaphthalene with phenylboronic acid yielded 88% yield of benzoyl naphthalene (Table 2, Entry 7). We then investigated the effect of substituents on the phenylboronic acid, and both electron-donating and electron-withdrawing groups were well tolerated under the reaction conditions. Electron-donating groups like -CH_3 and -OCH_3 on the boronic acid gave 84% yield of the corresponding ketones (Table 2, En-

Table 2. Carbonylative Suzuki coupling reaction of aryl iodides with various boronic acids.[a]

Entry	Aryl iodide	Boronic acid	Product	Yield [%][b]
1				96
2				93
3				90
4				78
5				72
6				78
7				88
8				84
9				84
10				70

[a] Reaction conditions: aryl iodide (1 mmol), arylboronic acid (1.1 mmol), $Pd(tmhd)_2$ (2 mol-%), K_2CO_3 (3 mmol), anisole (10 mL), 6 h, 80 °C, CO (100 psi). [b] Isolated yields.

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tries 8 and 9), whereas *p*-bromophenylboronic acid gave 70% yield of the desired product under the optimized reaction conditions (Table 2, Entry 10).

For generality, we extended our protocol to heteroaryl iodides such as 3-iodopyridine, 2-iodopyridine, and 2-iodothiophene (Table 3, Entries 1–9). The reaction of 3-iodopyridine with phenylboronic acid was optimized with respect to different parameters; the optimized reaction conditions are: $Pd(tmhd)_2$ (2.5 mol-%), toluene, K_2CO_3, 100 °C, CO (200 psi), 12 h.

Table 3. Carbonylative Suzuki coupling reaction of heteroaryl iodides with various boronic acids.[a]

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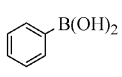
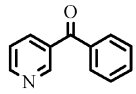
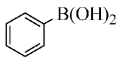
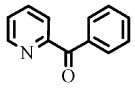
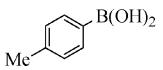
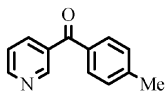
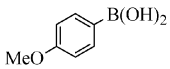
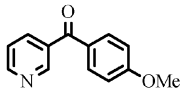
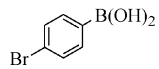
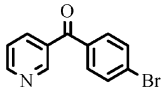
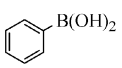
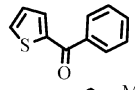
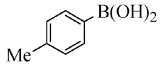
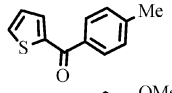
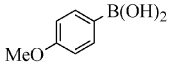
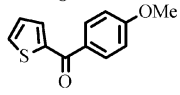
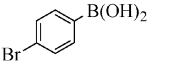
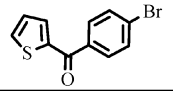
1				90
2				86
3				92
4				84
5				78
6				93
7				88
8				82
9				76

[a] Reaction conditions: aryl iodide (1 mmol), arylboronic acid (1.2 mmol), $Pd(tmhd)_2$ (2.5 mol-%), K_2CO_3 (3 mmol), toluene (10 mL), 12 h, 100 °C, CO (200 psi). [b] Yield based on GC and GC–MS. [c] $Pd(OAc)_2$ was used as catalyst, 10 h.

The coupling reaction of 3-iodopyridine and 2-iodopyridine with phenylboronic acid in the presence of carbon monoxide gave 90 and 86% yield, respectively, of the desired products. The effect of substituents on the boronic acid partner with heteroaryl iodides was then explored. Electron-donating group such as $-CH_3$ and $-OCH_3$ on the boronic acid gave 92 and 84% yield, respectively, of the expected heteroaryl ketones (Table 3, Entries 3 and 4). The reaction of 3-iodopyridine with *p*-bromophenylboronic acid gave a satisfactory yield of the desired product (Table 3, Entry 5).

For the reaction of 2-iodothiophene with phenylboronic acid, a comparatively similar yield of the desired product was obtained with $Pd(tmhd)_2$ and $Pd(OAc)_2$ as catalysts in 10 h, so the subsequent reactions were carried out with $Pd(OAc)_2$ as a catalyst. The reaction of 2-iodothiophene with phenylboronic acid by using $Pd(OAc)_2$ as a catalyst gave 93% of benzoyl thiophene (Table 3, Entry 6). Further, the effect of various groups on the boronic acid partner demonstrated that electron-donating groups ($-CH_3$ and $-OCH_3$) and electron-withdrawing groups (Br) gave good to excellent yields (76–88%) of the desired products under the optimized reaction conditions (Table 3, Entries 7–9).

Conclusions

In conclusion, we have developed an efficient protocol for the carbonylative Suzuki coupling of aryl and heteroaryl iodides with various boronic acids by using phosphane-free palladium complexes viz. $Pd(tmhd)_2/Pd(OAc)_2$ (2–2.5 mol-%) as a catalyst. The reaction was optimized with respect to various reaction parameters and enables carbonylative coupling reactions of various electron-rich, electron-deficient, and sterically hindered iodides with different boronic acids, affording excellent yields of the desired products; thus, the methodology has a broad application in this field.

Experimental Section

Materials and Methods: All chemicals were obtained from Lancaster (Alfa-Aesar) and used as is. Optimized yields were based on GC (Chemito 1000) and GC–MS (Shimadzu QP 2010) analysis. All the products were characterized by ^{13}C NMR (Varian 100 MHz), ^1H NMR (Varian 400 MHz), GC–MS (Shimadzu QP 2010), and FTIR (Perkin–Elmer).

General Procedure for the Carbonylative Suzuki Reaction of Aryl Iodides: To a 100-mL autoclave vessel was added the aryl iodide (1.0 mmol), phenylboronic acid (1.1 mmol), $Pd(tmhd)_2$ (2 mol-%), anisole (10 mL), and K_2CO_3 (3 mmol). The mixture was stirred for 10 min, CO (100 psi) was then introduced, and the reaction mixture was heated at 80 °C for 6 h. After completion of the reaction, the reaction mixture was cooled to room temperature and the solvent was removed under reduced pressure. The residue obtained was purified by column chromatography (silica gel, 60–120 mesh; petroleum ether/ethyl acetate, 60:80) to afford the desired carbonylated product. All the prepared compounds are known and compared with authentic samples and confirmed by GC–MS, ^1H and ^{13}C NMR, and FTIR analysis. Purity of the compounds was determined by GC–MS analysis.

General Procedure for the Carbonylative Suzuki Reaction of Heteroaryl Iodides: To a 100-mL autoclave vessel was added the heteroaryl iodide (1.0 mmol), phenylboronic acid (1.2 mmol), $Pd(tmhd)_2$/$Pd(OAc)_2$ (2.5 mol-%), toluene (10 mL), and K_2CO_3 (3 mmol). The mixture was stirred for 10 min, CO (200 psi) was then introduced, and the reaction mixture was heated at 100 °C for 10 h. After completion of the reaction, the reaction mixture was cooled to room temperature and the solvent was removed under reduced pressure. The residue obtained was purified by column chromatography (silica gel, 60–120 mesh; petroleum ether/ethyl acetate, 60:80) to afford

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the desired carbonylated product. All the prepared compounds were confirmed by GC–MS, ^{1}H and ^{13}C NMR, and FTIR analysis. Purity of the compounds was determined by GC–MS analysis.

Phenyl(pyridin-3-yl)methanone (Table 3, Entry 1): Solid. Yield: 90% (164 mg). ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C): δ = 9.03 (d, J = 1.2 Hz, 1 H), 8.85–8.86 (dd, J = 4.8, 1.8 Hz, 1 H), 8.15–8.18 (dd, J = 7.9, 1.8 Hz, 1 H), 7.80 (d, J = 7.9 Hz, 1 H), 7.36–7.79 (m, 4 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 194.6, 152.0, 150.2, 138.0, 133.7, 133.6, 130.1, 129.0, 128.4, 123.9 ppm. GC–MS (EI, 70 eV): *m*/*z* (%) = 183 (100) [M^+], 105 (80), 77 (95), 51 (45). IR (KBr): $\tilde{\nu}$ = 1663 cm^{-1}.

Phenyl(pyridin-2-yl)methanone (Table 3, Entry 2): Solid. Yield: 86% (157 mg): GC–MS (EI, 70 eV): *m*/*z* (%) = 183 (40) [M^+], 155 (80), 105 (65), 77 (100). R (KBr): $\tilde{\nu}$ = 1660 cm^{-1}.

(4-Methylphenyl)(pyridin-3-yl)methanone (Table 3, Entry 3): Solid. Yield: 92% (183 mg). ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C): δ = 8.97 (s, 1 H), 8.80 (d, J = 3.4 Hz, 1 H), 8.11 (d, J = 1.4 Hz, 1 H), 7.73–7.71 (d, J = 7.8 Hz, 2 H), 7.43–7.46 (dd, J = 4.87, 4.87 Hz, 1 H), 7.30–7.32 (d, J = 7.8 Hz, 2 H), 2.45 (s, 3 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 194.5, 152.5, 150.7, 144.3, 137.3, 134.0, 133.7, 130.3, 129.4, 123.5, 115.4, 21.8· ppm. GC–MS (EI, 70 eV): *m*/*z* (%) = 197 (50) [M^+], 182 (50), 119 (100), 91 (50). IR (KBr): $\tilde{\nu}$ = 1656 cm^{-1}.

(4-Methoxyphenyl)(pyridin-3-yl)methanone (Table 3, Entry 4): Solid. Yield: 84% (178 mg). ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C): δ = 8.81 (s, 1 H), 8.55 (d, J = 4.2 Hz, 1 H), 7.83 (d, J = 7.6 Hz, 1 H), 7.50–7.53 (d, J = 8.6 Hz, 2 H), 7.26–7.35 (dd, J = 5.13, 7.33 Hz, 1 H), 6.99–7.02 (d, J = 8.6 Hz, 2 H), 3.85 (s, 3 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 189.0, 159.7, 147.9, 147.7, 136.3, 133.9, 130.1, 128.2, 124.0, 123.5, 114.5, 55.4 ppm. GC–MS (EI, 70 eV): *m*/*z* (%) = 213 (60) [M^+], 198 (20), 135 (100). IR (KBr): $\tilde{\nu}$ = 1659 cm^{-1}.

(4-Bromophenyl)(pyridin-3-yl)methanone (Table 3, Entry 5): Solid. Yield: 78% (203 mg). ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C): δ = 9.04 (s, 1 H), 8.90 (d, J = 4.4 Hz, 1 H), 8.18 (d, J = 7.2 Hz, 1 H), 7.83–7.86 (d, J = 8.3 Hz, 2 H), 7.63–7.66 (d, J = 8.3 Hz, 2 H), 7.50–7.57 (dd, 1 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 193.0, 151.5, 149.5, 138.3, 138.2, 135.4, 135.2, 135.0, 133.2, 131.9, 128.7, 124.8 ppm. GC–MS (EI, 70 eV): *m*/*z* (%) = 261/263 (10) [M^+], 182 (100), 155 (20). IR (KBr): $\tilde{\nu}$ = 1667 cm^{-1}.

Phenyl(thien-2-yl)methanone (Table 3, Entry 6): Solid. Yield: 93% (174 mg). ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C): δ = 7.85–7.87 (m, 2 H), 7.48–7.73 (m, 5 H), 7.15–7.17 (m, 1 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 188.3, 143.7, 138.2, 135.0, 133.6, 132.4, 129.2, 129.0, 128.5, 128.0, 124.5 ppm. GC–MS (EI, 70 eV): *m*/*z* (%) = 188 (60) [M^+], 111 (100), 77 (35). IR (KBr): $\tilde{\nu}$ = 1629 cm^{-1}.

(4-Methylphenyl)(thien-2-yl)methanone (Table 3, Entry 7): Solid. Yield: 88% (177 mg). ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C): δ = 7.77–7.79 (d, J = 8.0 Hz, 2 H), 7.69–7.70 (m, 1 H), 7.63–7.64 (m, 1 H), 7.28–7.30 (d, J = 7.6 Hz, 2 H), 7.14–7.16 (m, 1 H), 2.44 (s, 3 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 188.0, 143.9, 143.2, 135.5, 134.6, 134.0, 129.5, 129.2, 128.0, 21.8 ppm. GC–MS (EI, 70 eV): *m*/*z* (%) = 202 (100) [M^+], 187 (45), 119 (90), 111 (80). IR (KBr): $\tilde{\nu}$ = 1624 cm^{-1}.

(4-Methoxyphenyl)(thien-2-yl)methanone (Table 3, Entry 8): Solid. Yield: 82% (178 mg). ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C): δ = 7.87–7.89 (d, J = 8.5 Hz, 2 H), 7.62–7.67 (m, 2 H), 7.14–7.16 (m, 1 H), 6.95–6.98 (d, J = 8.5 Hz, 2 H), 3.87 (s, 3 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 187.0, 163.2, 143.9, 134.2, 133.6, 131.8, 130.7, 127.9, 113.8, 55.6 ppm. GC–MS (EI, 70 eV): *m*/*z* (%) = 218 (80) [M^+], 187 (20), 135 (100). IR (KBr): $\tilde{\nu}$ = 1626 cm^{-1}.

(4-Bromophenyl)(thien-2-yl)methanone (Table 3, Entry 9): Solid. Yield: 76% (202 mg). ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C): δ = 7.73–7.75 (d, J = 6.95 Hz, 2 H), 7.62–7.65 (m, 2 H), 7.16–7.26 (m, 3 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 189.0, 143.3, 136.7, 134.9, 134.7, 131.8, 130.8, 128.2, 127.4 ppm. GC–MS (EI, 70 eV): *m*/*z* (%) = 266/268 (20) [M^+], 187 (20), 111 (100). IR (KBr): $\tilde{\nu}$ = 1631 cm^{-1}.

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