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Suzuki-Miyaura Cross-Coupling Reaction Based on Novel Palladium(II) Diphenylphosphine Derivative Catalyst

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Abstract
The synthesis and characterization of palladium complexes $((CH_3)_3SiC_6H_4PPh_2)_2PdCl_2$ **4a**, and $((CH_3)_3SiC_{12}H_8PPh_2)_2PdCl_2$ **4b,** containing phosphine $(CH_3)_3SiC_6H_4PPh_2$ **3a** and $(CH_3)_3SiC_{12}H_8PPh_2$ **3b** ligands are reported. The phosphine ligands are prepared conveniently in high yield by treatment of the corresponding 1,4-dibromoarene with one equivalent of butyl lithium, and one equivalent of $ClPPh_2$ at -78 °C under an atmosphere of argon. The palladium complexes are synthesized by the reaction of $Pd(cod)Cl_2$ with two equivalent of the above mentioned phosphine ligands. The new complexes were fully characterized by spectroscopic methods and elemental analysis. Furthermore, the use of the palladium (II) complexes of such system as pre-catalysts for the Suzuki-Miyaura coupling of some arylbromides and arylchloride with substituted phenyl boronic acid has been tested.
Keywords:1,4-dibromoarene, Palladium complex, Suzuki coupling, catalyst.

1. Introduction

There is a considerable interest in the preparation of new phosphorus ligands for the design and synthesis of transition metal complexes with good catalytic properties (Apple and Woollins, 2002; Fey, *et al*., 2008; Bhattacharrya and Woollins, 1995). Moreover, these types of complexes are directed towards medicinal and biological activity applications, especially with nickel, ruthenium, palladium and platinum. This type of complexes can also be used as highly active catalysts in different types of industrially important reactions such as hydroformylation, hydrogenation of olefins, amination, Heck reaction, and Suzuki coupling reactions. (Miyaura, and Suzuki, 1995; Suzuki, 2002 ; Wolfe and Buchwald, 1999 ; Tomori, *et al.*, 2000; Heck and Nolley, 1972). The Suzuki-Miyaura reaction is used as powerful tool for industrial synthesis. For example, natural products, pharmaceuticals and Agrochemicals (Broger *et al.*, 1998; Bellina, *et al.*, 2004; Kotha, *et al.*, 2002; Littke, *et al.*, 2000; Frisch and Beller, 2005). The interest in studying metal-organic palladium complexes concentrate mainly on the ability to employ their steric and electronic structure and their usefulness as catalysts. Moreover, phosphines serve as supporting ligands in numerous transition metal-catalyzed reactions. Therefore, in this work, we focused our interest to create phosphine ligands based on 1,4-disubstiuted arene and PPh_2Cl. The synthesized phosphine ligands are used in the synthesis of square planar palladium (II) complexes. In addition to synthesis work, the prepared complexes were applied in the Suzuki Miyaura cross-coupling reaction of different types of aryl bromides and aryl chlorides with boronic acid derivatives.

2. Experimental

2.1 General Setup, Chemicals and Instrumentation

All preparations and other operations were carried out under dry oxygen-free nitrogen or argon atmosphere following a conventional Schlenk techniques and vacuum-line manipulations. Diethyl ether and THF were purified by distillation from sodium/benzophenoneketyl and degassed before use. Ethanol was purified by distillation from magnesium. Water was prepared by ultrasonicating distilled water under vacuum and degassed. The starting materials PPh_2Cl, $ClSiMe_3$ **1a**, and **1b** respectively, were purchased from Aldrich and used without further purification. While **2a**, **2b** ,and **3a** were prepared following published procedures (Sellin, *et al.*, 2002). Purifications by flash column chromatography were carried out using Merck silica gel 60 (230e400 mesh ASTM). NMR spectra were run on Bruker Advance III 500 spectrometer; chemical shifts for ^1H and ^{13}C NMR are referenced internally to the residual protons and the ^{13}C NMR signal of the deuterated solvents. Elemental analyses (C, H, N) were carried out by using a Thermo Flash AE 1112 analyzer. Infrared spectra were recorded with a Nicolet IR200 FT-IR spectrometer using KBr pellets.

*2.2 Synthesis of Diphenyl(4-trimethylsilylbiphenyl)phosphine (**3a**)* .

Compound **2a** (0.95 g, 3.11 mmol) was dissolved in dry dichloromethane (20 mL) and cooled to -78 °C. *n*-Butyl lithium (2.03 mL, 1.6 M n-hexane, 3.15 mmol, 1 equivelant.) was added drop wise and the reaction mixture was stirred for 35 min at this temperature. After dropwise addition of chlorodiphenyl phosphine (0.70 g, 2.11 mmol);

the reaction mixture was allowed to attain ambient temperature within 2.25 hours. After addition of MeOH (~1 mL) in order to eliminate unreacted *n*-BuLi, the mixture was filtered and then all volatiles were removed in vacuum and the residue was dissolved in dichloromethane (15 mL). At ca. -60°, the precipitation of a white powder was started. After stirring for 3 hour at ambient temperature, the solvent was removed using a membrane-pump vacuum, and then a portion of diethyl ether (10 mL) was added. The mixture was quenched by the addition of 8 ml of degassed water, and then extracted with diethyl ether (2 X 15 mL) under Argon. The combined organic extracts were dried with $MgSO_4$. Afterward, the amount of solvent was reduced in vacuum to 4 mL and *n*-hexane (15 mL) was added. The supernatant layer was removed and the precipitate was dissolved in dichloromethane (10 mL) and chromatographed on silica gel using THF as eluent. After drying in vacuum, product **3a** was obtained as colorless oil. The resulting colorless oil was recrystallized from ethanol to yield a white crystals of **3a** (Yield : 63%)**.** ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.86 (d), 7.61-7.55 (m), 7.44 (d), 7.33 – 7.36 (m), 7.29 (d), 0.74 (s, $SiMe_3$) . $^{31}P\{^{1}H\}$ NMR (C_6D_6): δ .26.85. $^{13}C\{^{1}H\}$ NMR (126 MHz, C_6D_6): δ 141, 138.2, 138, 137.6, 136.4, 135.9, 129.5, 128.1, 128.4,126.9, -.59 (s, CH_3Si). IR (KBr): 3052 (m), 1430 (s, P-C). Elemental analysis (%) Calcd. for $C_{27}H_{27}PSi$ (410.562.466 g·mol^{-1}): C 78.99, H 6.63; found C 79.49, H 7.01.

*2.3 Synthesis of Diphenyl(4-trimethylsilylphenyl) phosphine (**3b**) (*Sellin, *et al.*, 2002*)*
Following the synthesis procedure described above, Compound **2b** (0.88 g, 3.8 mmol) was dissolved in dry chloromethane (20 mL) and cooled to -78 °C. Butyl lithium (2.48 mL, 1.6 M n-hexane, 3.8 mmol, 1 equiv.) was added dropwise and the reaction mixture was stirred for 35 min at -78 °C. After dropwise addition of chlorodiphenyl phosphine (0.84 g, 2.53 mmol); the reaction mixture was allowed to attain the ambient temperature within 2 hours. After addition of methanol (~1 mL) in order to eliminate unreacted *n*-BuLi, the mixture was filtered and then all volatiles were removed in vacuum and the residue was dissolved in dichloromethane (15 mL). At -55°C, the precipitation of a white powder was started. After stirring for 3 hours at ambient temperature, the solvent was removed in membrane-pump vacuum, and then a portion of diethyl ether (10 mL) was added. The mixture was quenched by the addition of 8 ml of degassed water, and then extracted with diethyl ether (2 X 15 mL) under Argon. The combined organic extracts were dried with Na_2SO_4. Afterward, the amount of solvent was reduced in vacuum to 4 mL and *n*-hexane (15 mL) was added. The supernatant layer was removed and the precipitate was dissolved in dichloromethane (10 mL) and chromatographed on silica gel using THF as eluent. After drying in vacuum, product **3b** was obtained as colorless oil. The resulting colorless oil was recrystallized from ethanol to yield a white crystals of **3b** (Yield : 69%)**.** ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.62 (m), 7.45 (d), 7.38 – 7.34 (m), 7.31 (m), 0.79 (s, $SiMe_3$).$^{31}P\{^{1}H\}$ NMR (C_6D_6): δ -4.95.$^{13}C\{^{1}H\}$ NMR (126 MHz, C_6D_6): δ 143, 139.2, 135.7, 133.6, 132.9, 132.3, 129.2, 128.9, 128.7, -1.42 (s, CH_3Si). IR (KBr): 3049 (m), 1434 (s, P-C). Elemental analysis (%) Calcd. for $C_{21}H_{23}PSi$ (334.466 g·mol^{-1}): C 75.41, H 6.93; found C 75.54, H 7.10.

*2.4 Synthesis of complex (Diphenyl(4-trimethylsilylbiphenyl)phosphine)$_2$PdCl$_2$ (**4a**).*
To a dichloromethane solution (10 mL) containing two equivalents of **3a** (0.500 g, 1.22 mmol); one equivalent of $PdCl_2$(Cod) (0.060g , 0.61 mmol) in dichloromethane (5 ml) was added dropwise at room temperature. The resulting mixture was stirred for 4.5 hours at room temperature. After removal of all volatiles using a membrane-pump vacuum, the crude product was purified by column chromatography on Silica using a mixture of n-hexane-diethyl ether (ratio 1:1, v:v), which was then dried under high vacuum to obtain the product **4a** in 84 % yield as a pale-yellow powder M.p.: 179-182°C. Elemental analysis (%) for [$C_{54}H_{54}Cl_2P_2Si_2Pd$] (998.450 g·mol^{-1}): C 64.96, H 5.45; found C 64.23, H 5.33. ^{1}H NMR (500 MHz, $CDCl_3$, 25 °C) δ 7.69 – 7.63 (m), 7.57 – 7.54 (m), 7.4 – 7.36 (m), 3.74 – 3.55 (m), 0.19 (s, $SiMe_3$)).. $^{13}C\{^{1}H\}$ NMR (126 MHz, C_6D_6): δ 140, 134.2, 136, 133.6, 133.4, 130.9, 1297, 127.1, 123.4,122.9, 0.04 (s, CH_3Si).. $^{31}P\{^{1}H\}$ NMR (202.5 MHz, $CDCl_3$, 25 °C): δ = 65.34 ppm. IR (KBr): ν = 369 (Pd-Cl), 851, 1433 (P-Ph), 3056 (C-H), 1566 cm^{-1}.

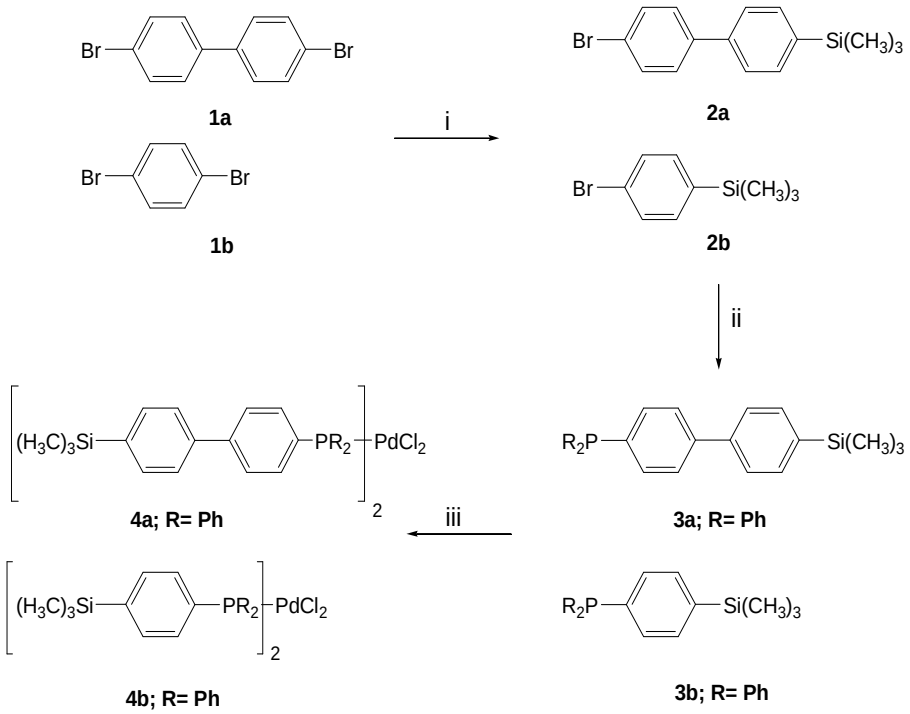
*2.5 Synthesis of complex (Diphenyl(4-trimethylsilylphenyl)phosphine)$_2$PdCl$_2$ (**4b**) (*Sellin, *et al.*, 2002*).*
To a dichloromethane solution (10 mL) containing two equivalents of $Ph_2PC_6H_4SiMe_3$, **3b** (0.85 g, 2.47 mmol); one equivalent of $PdCl_2$(Cod) (0.120g , 1.23 mmol) in dichloromethane (5 mL) was added dropwise at room temperature. The dark yellow suspension was stirred for 3.5 hours. After removal of all volatiles in membrane-pump vacuum, the crude product was purified by column chromatography on Silica using a mixture of n-hexane-diethyl ether (ratio 1:1, v:v), which was then dried under high vacuum to obtain the product **4b** in 74 % yield as yellow powder M.p.: 169-171°C. Elemental analysis (%) for [$C_{42}H_{46}Cl_2P_2Si_2Pd$] (846.258 g·mol^{-1}): C 59.61 , H 5.48 ; found C 58.98, H 5.39. ^{1}H NMR (500 MHz, $CDCl_3$, 25 °C) δ 7.47 – 7.39 (m), 7.30 – 7.25 (m), 7.22 – 7.19 (m), 7.08 (d, *J* = 7.4 Hz), 6.82 (t, *J* = 7.3 Hz), 0.86 (s, $SiMe_3$).^{13}C NMR (126 MHz, $CDCl_3$, 25 °C) δ 143.78 , 141.15, 132.99 (s), 132.83 (s), 129.70, 128.58, 127.28, 126.77, 123.56, 120.73, 1.23.$^{31}P\{^{1}H\}$ NMR (202.5 MHz, $CDCl_3$, 25 °C): δ = 22.5 ppm. IR (KBr): ν = 364 (Pd-Cl), 842, 1429 (P-Ph), 1262, 3059 (C–H)cm^{-1}. ESI-TOF MS: calcd (m/z) for [$C_{42}H_{46}Cl_2P_2Si_2Pd$ +H]$^+$(100%) 847.01 found 848.21 [$C_{42}H_{46}Cl_2P_2Si_2Pd$ +H]$^+$.

2.6 General procedure for the Suzuki-Miyaura coupling

An oven-dried Schlenk flask was evacuated and back-filled with argon and charged with aryl halides (2.9 mmol), phenylboronic acid (3.75 mmol), Na_2CO_3 (3 equv.) and 0.5 mmol of acetylferrocene. The components were dissolved in 9 mL of 1,4-dioxane at 90°C. After addition of an appropriate catalyst (**4a** or **4b**), the reaction mixture was heated at 90°C with continuously stirring for the given time. After 2.5, 5, 10, 15, 30, 45, 60, 90, 120, 150, 180, 210, 240, 300, and 360 min., samples (~1 mL each) were taken for characterization. The solvent was evaporated and the residue was chromatographed on silica gel with diethyl ether (hexane or dichloromethane) as eluent, and all volatiles were evaporated under reduced pressure. The conversions were monitored by ^{1}H NMR spectroscopy.

3. Results and Discussion

The reaction of **2a** and **2b** with two equivalents of diphenylchlorophosphine in anhydrous dichloromethane followed by removal of the solvent in vacuum which produces the ligads **3a** and **3b** that obtained in good yields (81% and 78%, respectively) Scheme 1. Diphenyl(4-trimethylsilylarene) phosphines **3a** and **3b** are accessible by various methods starting from cheap and commercially available 1,4 dibromoarenes **1a**, and **1b**, respectively. The preparation is by the route depicted in **Error! Reference source not found.**. Preparation of **1a** and/or **1b** by n-BuLi in diethyl ether at -78 °C and after gradual increase of temperature, the resulting solution was cooled at -78 °C and then a solution of chlorotrimethylsilane in diethyl ether was added. The reaction mixture was allowed to warm to room temperature and stirred for 90 min. The conversion of **2a** to Diphenyl(4-trimethylsilylarene) phosphines **3a** and **3b**, respectively, proceeds smoothly without any limitations. Samples taken after 20 and 40 min reaction time showed an incomplete reaction. The sample taken after 75 min revealed an almost complete reaction. After quenching with water, samples were analyzed by ^{1}H, ^{31}P, and ^{13}C{^{1}H}-NMR spectra. After addition of methanol in order to eliminate the unreacted n-Butyllithium, the reaction mixture was filtered and the filtrate was concentrated under vacuum. The resulting residue was extracted again with n-hexane, filtered under argon and the solvent was removed from the filtrate to give a yellow oil. The crude products can be further purified by means of recrystallization from cold ethanol, which was found to be pure by NMR measurements. The new phosphines ligand **3a** and known phosphine **3b** can be isolated as an analytically pure colorless and yellow solid material, respectively. Both are soluble in common organic solvents including dichloromethane, tetrahydrofuran and chloroform, while in diethyl ether and n-hexane **3a** and **3b** are not soluble. NMR and IR results of the ligands **3a** and **3b** were found to be in good agreement with those reported in literature for related system (Sellin, *et al.*, 2002).



Scheme 1: General synthetic strategy of phosphine **3a** and **3b** thier related complexes **4a** and **4b**, respectively **:** i)^{n}BuLi, -78 °C; $ClSiMe_3$. ii)^{n}BuLi, -78 °C; $ClPPh_2$ and iii) $Pd(cod)Cl_2$.

In order to use the of phosphines **3a** and **3b** as ligands, they were reacted with $Pd(cod)Cl_2$ Scheme 1. The phosphine ligands **3a** and **3b** were reacted with an equimolar amounts of $[Pd(cod)Cl_2]$ to produce the corresponding palladium complexes **4a** and **4b** in dichloromethane at room temperature in good yields (83% (**4a**), and 74% (**4b**)). The two palladium complexes **4a** and **4b** are thermally stable and can be handled in air. Palladium compounds **4a** and **4b**, were characterized by elemental analysis, IR and NMR (^{1}H,$^{13}C\{^{1}H\}$, $^{31}P\{^{1}H\}$) spectroscopy as well as mass spectroscopy to confirme the appropriate molecular structures. The $^{31}P\{^{1}H\}$-NMR spectra of the complexes **4a** and **4b** show singlets at 65.34 and 22.5 ppm, respectively, indicating that the gene (COD) has been replaced by the monodentate phosphine **3a** and **3b**, respectively. In addition, both palladium complexes exhibit no NMR signals broadening at low temperatures. The IR spectrum of **4a**, and **4b** show characteristic bands at 3056 cm^{-1} (**4a**), and 3059 cm^{-1} (**4b**) due to (C–H) stretching, 1433 cm^{-1} (**4a**), and 1429 cm^{-1} (**4b**) due to (P–Ph) stretching. Elemental analyses of palladium complexes **4a** and **4b** are found to be consistent with the suggested molecular formulas as outlined in Scheme 1.

3.1 The Suzuki coupling reaction

As mentioned above, phosphine derivatives coordinated to certain types of metal ions have been found to be excellent in catalysic applications. A preliminary experiment palladium complexes **4a** and **4b** were tested as catalysts in the Suzuki-Miyaura cross coupling of some aryl halides with phenylboronic acid derivatives, which is one of the most efficient methods for carbon-carbon bond formation. The ability of palladium complexes **4a** or **4b** to promote Suzuki-Miyaura cross section reaction with time has been performed. A series of reactions using different amounts of palladium complex **4a** or **4b**, were tested. A positive order with respect to phosphine was observed. Acetyl ferrocene as internal standard was added to the appropriate reaction solution to estimate the conversion rate. The later was monitored by ^{1}H NMR spectroscopy (Jakob, *et al.*, 2008 and Lang, *et al.*, 2012).

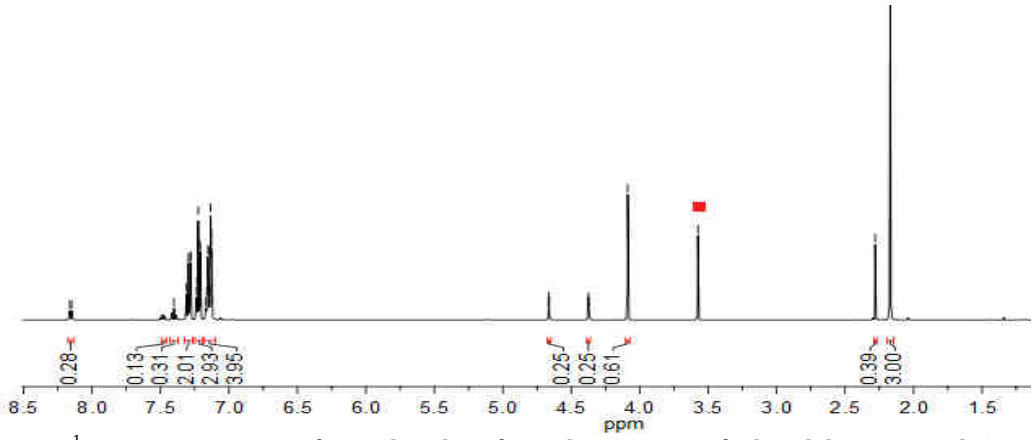
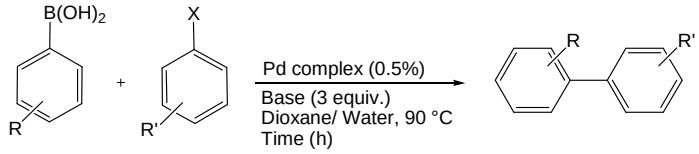
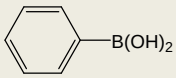
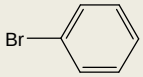


Figure 1. ^{1}H NMR spectrum of sample taken from the reaction of phenyl boronic acid (240 min, 100% conversion) with 2-bromo toluene using palladium complex **4a,** acetyl ferrocene as internal standard and 1,4 dioxane as solvent.(■).

The conversions that monitored by ^{1}H NMR spectroscopy are based on the appearance of aryl halide derivatives. It was found that palladium complex **4a** and **4b** promoted the cross-coupling of different substrates listed in Table 1 with good yield. Palladium complex **4b** was more active as catalyst than **4a**. This can be observed by high percent of conversion after a short time. All catalytic reactions were inspected after 2.5, 5, 10, 15, 30, 45, 60, 90, 120, 150, 180, 210, 240, 300, and 360 min. The catalytic results are collected in Table 1. At room temperature no appreciable formation of cross coupling product was observed. When the temperature was increased to 90 °C good conversions were obtained. Different solvents were tested for imploying of Suzuki Miyaura cross coupling in catalysis. It was observed that the reaction performed in 1,4-dioxane and/or 1,4-dioxane:water (ratio 2:1, v:v) as a solvent at 90 °C in the presence of sodium carbonate as base was found to be the best. For comparison, we initially tested the catalytic activity of the complex **4a** and **4b** for the coupling of 4-bromoacetophenone with phenylboronic acid. Under the determined reaction conditions, bromobenzene, 4-bromoacetophenone, 4-bromoanisole, 2-bromoanisol, 4-bromotoluene, and 4-chloroacetophenone are reacted with boronic acid derivatives to obtain good yields (Table 1). Higher conversion rates were observed with complex **4a** when activated aryl bromides were used as substrates. Figures 1 and 2 show that quantitative conversions are obtained from the coupling reactions of p-bromoacetophenone with phenyl boronic acid using complexes **4a** and **4b**, respectively.

Table 1. Results of the efficiency of palladium complexes 4a and 4b to facilitate Suzuki cross-coupling between aryl halides with phenylboronic acid derivatives [a]



Entry	Phenylboronic acid derivatives	Aryl halides derivatives	Catalyst	Time	Yield (%)[b]
1			4a	7h	61
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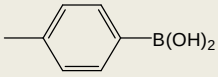
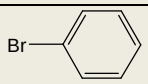
2			4a	9h	60
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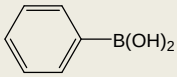
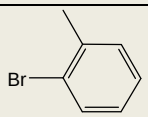
3			4a	4h	58
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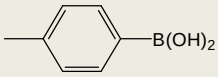
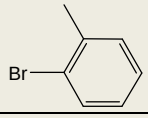
4			4a	7h	53
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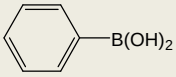
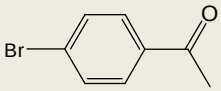
5			4a	3.5h	88
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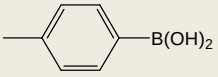
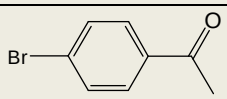
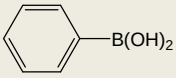
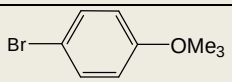
6			4a	4h	78
			4b	4h	82
7			4a	5h	71
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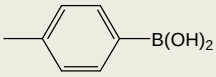
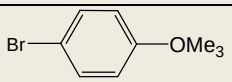
8			4a	6h	67
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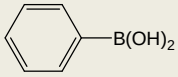
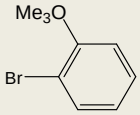
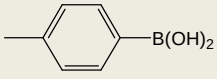
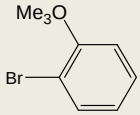
9			4a	6 h	62
			4b	6.5h	65
10			4a	7h	57
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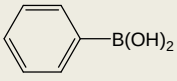
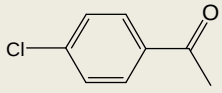
11			4a	6h	77
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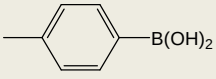
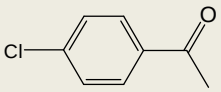
12			4a	7h	69
			4b	8h	73

[a]Reaction conditions: 3.75 mmol $B(OH)_2$-C_6H_4-R, 2. mmol X-C_6H_4-R, 3.0 mmol K_2CO_3. 1.0 % Pd complex, 1,4-dioxane-water (ratio 2:1, v:v) (10 mL·mmol^{-1}). [b]Purity percent of compounds was obtained by NMR and yields are based on the appearance of aryl halide derivatives, all reactions were monitored by NMR.

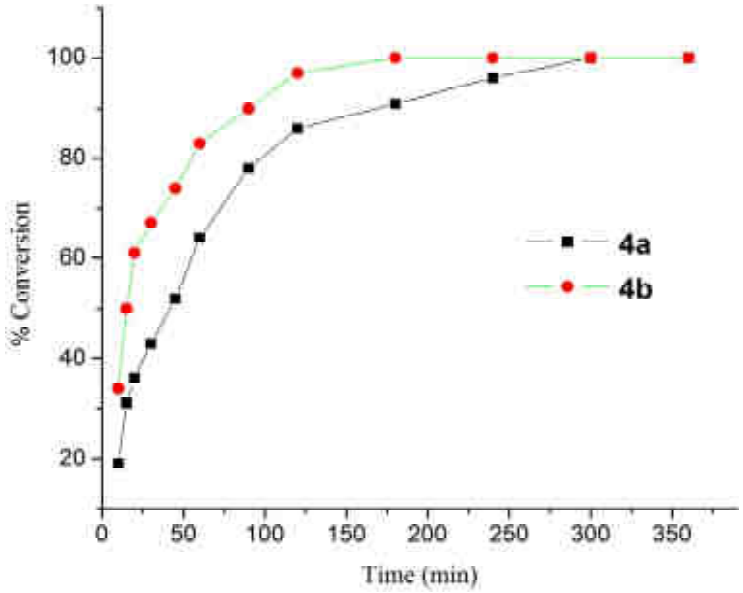


Figure 2: Reaction profile for the coupling of p-bromoacetophenone (2.90 mmol) with phenyl boronic acid (3.75 mmol) to give 4-acetyl biphenyl using **4a** and **4b** (0.5 mol%) in the presence of sodium carbonate (8.5 mmol) in 1,4-dioxane at 90 °C.

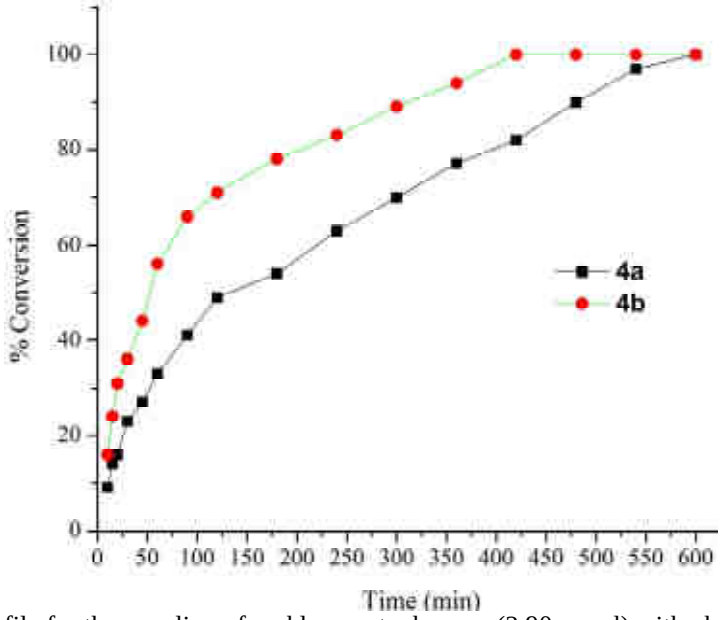


Figure 3: Reaction profile for the coupling of p-chloroacetophenone (2.90 mmol) with phenyl boronic acid (3.75 mmol) to give 4-acetyl biphenyl using **4a** and **4b** (0.5 mol%) in the presence of sodium carbonate (8.5 mmol) in 1,4-dioxane at 90 °C.

The reaction of activated p-chloroacetophenone with phenyl boronic acid required, as expected, longer conversion time (Figure 3). In comparison with the biphenyl ligand : Buchwald ligand (Wolfe, *et al.*, 1999; Walker, *et al.*, 2004) has significantly lower conversions using 0.5 mole% palladium. Moreover, compared with the trialkyl phosphino ligands by Fu and Beller, (2000) and Littke, *et al.*, (2000) showed lower conversion under the applied reaction conditions. Future work will examine the ability of these complexes to evaluate the catalytic activity of such system.

4. Conclusion

Two palladium complexes have been synthesized with phosphine ligands based on diphenyl (4-trimethylsilylarene) phosphines **3a** and **3b**. The structures of all isolated complexes were ensured by NMR, IR, and elemental analysis. Palladium complexes exhibit no NMR signals broadening at low temperatures. It was found that palladium complexes of such system inhanced a homogeneous catalysis that can be employed in Suzuki-Miyaura cross coupling of activated and deactivated aryl bromides with good yields.

Acknowledgement

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