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Synthesis and applications of a new palladacycle as a high active catalyst in the Suzuki couplings

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ABSTRACT

The reaction of biphenyl-based phosphine P(*o*-C_6H_4Me)Ph_2 (**1**) with $Pd(OAc)_2$ in toluene affords the air and water stable palladacycle (**2**) as a binuclear compound which has been characterized by multinuclear NMR spectroscopy and elemental analysis as a mixture of *cis* and *trans* isomers with relative intensity of 1:3, respectively. This palladacycle is a highly efficient catalyst precursor for the coupling of aryl boronic acids and aryl halides. Both activated and deactivated aryl bromides and chlorides are efficiently coupled in the presence of **2** to furnish the corresponding cross-coupled products in excellent yields, and a wide variety of functional groups are tolerated in aryl halides. This methodology has also been extended for the coupling of bromoarylphosphines and bromoarylphosphine oxides with aryl boronic acids for the generation of hindered corresponding products.

1. Introduction

Organopalladium complexes play an important role in carbon–carbon bond forming reactions because of their versatility, compatibility with most of functional groups and relative low toxicity [1]. Among them, palladacycles have merited special attention as their enormous supremacy in many aspects [2]. The facility of preparation and versatility for modification of their steric and electronic properties combined with their usually high air and thermal stability, infer them a variety of applications in organic synthesis, new materials, bio-organometallic chemistry, and organometallic catalysis [3–9]. Since Hermann and Beller demonstrated the extremely high catalytic activity of cyclopalladated tri-*o*-tolylphosphine in Heck reaction [10], many palladacycles have been synthesized and used as highly efficient catalysts in carbon–carbon bond forming reactions [2,10–13]. Of note is that the best results were obtained with phosphapalladacycles or with palladacycles modified with carbenes or phosphorus containing ligands. This might be rationalized by the extra stability provided by these ligands for the stabilization of the low-ligated catalytically active Pd species involved in the main catalytic cycle [14,2,15]. Also Bedford and co-workers recently investigated the possibility of using related palladated triarylphosphite complexes and found extremely high activity in Suzuki cross coupling with both electronically activated and deactivated aryl bromides [16,17].

We have already reported the synthesis of biphenyl-based phosphines by Suzuki coupling of arylboronic acids with bromoarylphosphine oxides using $Pd(dba)_2$ and PPh_3 (Scheme 1) [18]. Moreover we have recently demonstrated that biphenyl-based phosphine **1** which was made based on Buchwald's instructive and pincer works [19], has been successfully applied to the palladium catalyzed Suzuki coupling of aryl halides as well as bromoarylphosphines and bromoarylphosphine oxides, with low catalyst loading and good to excellent conversions [20]. This excellent activity in coupling reactions using palladacycles as well as our idea that the extraordinary activity of the phosphine **1** may be due to formation of palladacycle intermediate encouraged us to synthesis the corresponding palladacycle **2** from the phosphine **1** and investigate its catalytic activity in the Suzuki coupling reactions. Therefore we report herein the synthesis of palladacycle **2**, which was easily prepared from the biphenyl-based phosphine **1** on addition of $Pd(OAc)_2$ in toluene as a binuclear complex with two acetate-bridged ions (Scheme 2). To test the reactivity of the synthesized palladacycle in the Suzuki reaction, it was employed in the reaction of a series of aryl halides with arylboronic acids. This palladacycle has also been used for the coupling of bromoarylphosphines and bromoarylphosphine oxides with arylboronic acids (Scheme 3).

In order to compare the relative activity of palladacycle **2** to phosphine **1**, all coupling reactions were carried out in obtained optimized conditions for phosphine **1** [20].

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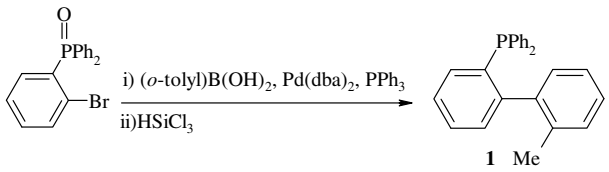
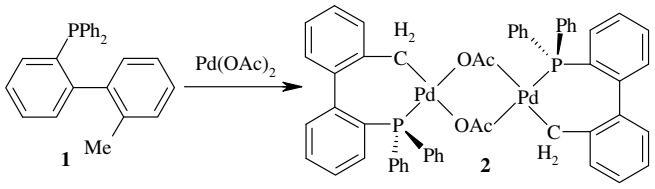
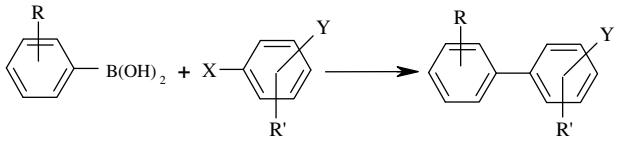


Scheme 1. The synthesis route for preparation of biphenyl-based phosphine **1**.



Scheme 2. The synthesis route for preparation of palladacycle **2**.



R = H, Me; R' = H, Me, Br, COMe, COPh, OMe, Ph, NO_2;
X = Cl, Br; Y = H, PPh_2, $OPPh_2$

Scheme 3. The Suzuki cross coupling reaction.

2. Results and discussion

2.1. Synthesis and characterization of the palladacycle *2*

The palladacycle **2** was easily obtained on 85% yield as white powder from the reaction 2-diphenylphosphino-2′-methylbiphenyl, phosphine **1**, with $Pd(OAc)_2$ in toluene via coordination of palladium followed by C–H activation which was as a binuclear compound with two acetate ion bridge (Scheme 2). Structure of this palladacycle has been ascertained by means of elemental analysis and multi-nuclear NMR spectroscopy as a mixture of *cis* and *trans* isomers with relative intensity 1:3 and 1 mole ether of crystallization. The NMR data are similar to another acetate-bridged palladacycle, which is previously prepared by Herrmann on addition of P(*o*-tolyl)$_3$ to $Pd(OAc)_2$ [21].

2.2. Suzuki coupling reactions of aryl halides

To test the reactivity and the catalytic properties of the synthesized palladacycle, it was first employed in the Suzuki coupling reaction of a series of aryl halides with phenylboronic acid and *o*-tolylboronic acid. The obtained results are presented in Table 1. This table shows that palladacycle **2** is a high reactive catalyst, which can catalyze the Suzuki cross coupling of even extremely electron-rich aryl halides such as 3-chloroanisole (Table 1, entry 12). As expected, the catalytic activity depends on type of the halide. Similar conversions were obtained with high activate substituted aryl bromides and chlorides (e.g. Table 1, entries 2, 10). However, usually less reactive aryl chlorides resulted poorer conversion and yields compared to the corresponding aryl bromides (Table 1, entries 1, 9). The reactivity increases with increasing the reactivity of aryl chloride; reaches to 100% in the case of high reactive functional groups such as acyl (Table 1, entry 10).

In the case of less-bulky aryl halides, the rates and conversions were independent to steric effects of the studied boronic acids. For

Table 1
Suzuki coupling of aryl halides using palladacycle **2**[a]

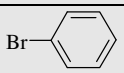
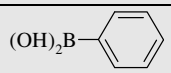
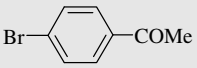
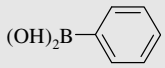
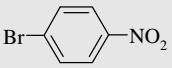
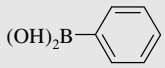
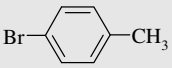
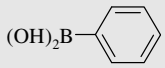
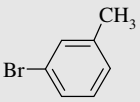
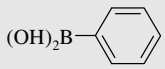
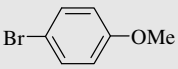
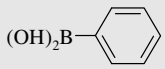
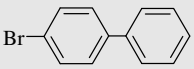
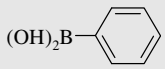
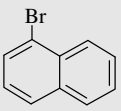
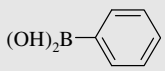
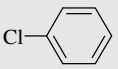
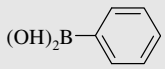
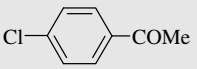
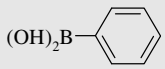
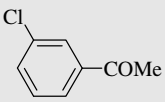
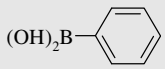
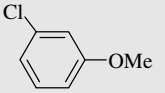
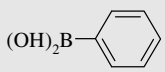
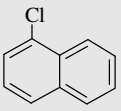
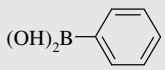
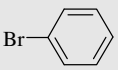
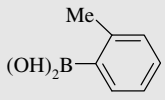
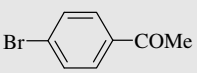
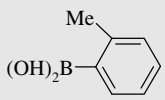
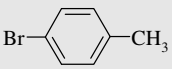
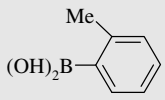
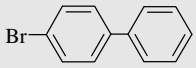
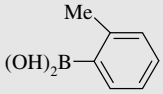
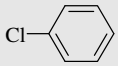
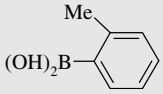
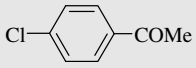
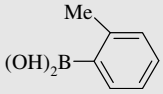
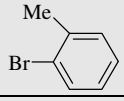
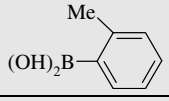
Entry	Aryl halide	Arylboronic acid	Yield[b] (%)	10^{-3} Ton
1			100	100
2			100	100
3			75	75
4			100	100
5			65	65
6			100	100
7			90	90
8			60	60
9			65	65
10			100	100
11			100	100
12			95	95
13			90	90
14			100	100
15			100	100
16			100	100

Table 1 (*continued*)

Entry	Aryl halide	Arylboronic acid	Yield[b] (%)	10^{-3} Ton
17			85	85
18			60	60
19			85	85
20			25	25

[a] *Reaction conditions:* 1.0 mmol aryl halide, 1.5 mmol $ArB(OH)_2$, 0.001 mol% palladacycle, 2.0 mmol K_3PO_4, 5 mL toluene, 1 mL water, 100 °C, 1 h (6 h for aryl chlorides).
[b] Isolated yield.

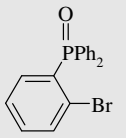
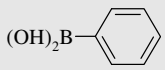
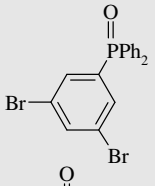
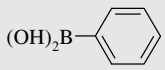
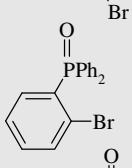
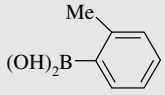
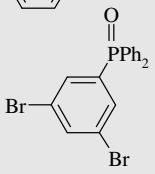
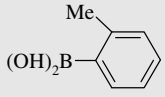
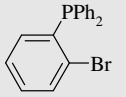
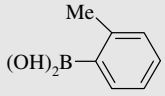
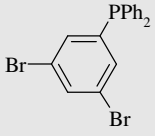
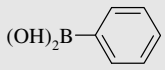
ortho-substituted aryl halides such as 2-bromotoluene (Table 1, entry 20) however, the *ortho* effects of two *ortho* methyls on the *o*-tolylboronic acid and 2-bromotoluene led to a remarkable decrease on the conversion. This is probable due to increasing the contribution of transmetallation step in the rate of reaction.

2.3. Suzuki coupling reactions of bromoarylphosphine oxides and bromoarylphosphines

We have extended the experimental protocol for the preparation of biphenyl-based phosphines and biphenyl-based phosphine oxides. Table 2 summarizes the results of the Suzuki cross coupling of bromoarylphosphine oxides and bromoarylphosphines with two arylboronic acids. Monobromophosphine oxide was coupled with 1.2 equiv. of arylboronic acids (Table 2, entries 1, 3) to give the corresponding biphenylphosphine oxides. Dibromophosphine oxide was coupled with 2.4 equiv. of arylboronic acids (Table 2, entries 2, 4) to give the corresponding terphenylphosphine oxides as a sole product without biphenyl byproduct which was expected to be formed. This method is also efficient for coupling arylboronic acids with haloarylphosphines instead of their oxides.

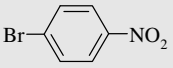
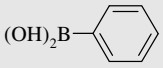
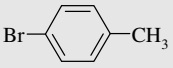
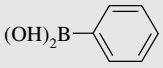
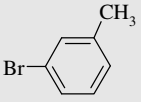
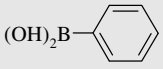
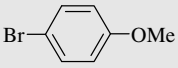
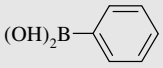
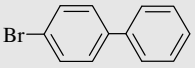
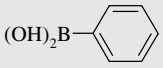
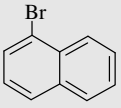
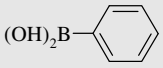
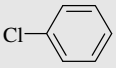
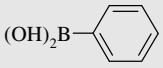
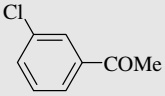
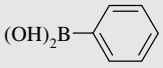
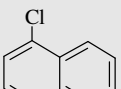
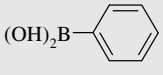
2-Diphenylphosphinobromobenzene, $[PPh_2(o\text{-}C_6H_4Br)]$ (Table 2, entry 5) and 5-diphenylphosphino-1,3-dibromobenzene, $[PPh_2(C_6H_3Br_2)]$, (Table 2, entry 6) were coupled with phenylboronic acid to give **1** and terphenylphosphine in 75 and 70% yield, respectively according to ^{31}P NMR. This method negates a

Table 2
Suzuki coupling of bromoarylphosphine oxides and bromoaryl-phosphines using palladacylce 2[a]

Entry	Substrate	Arylboronic acid	Yield[b] (%)	Ton
1			85	4250
2			82	4100
3			65	3250
4			40	2000
5			75	3750
6			70	3500

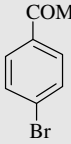
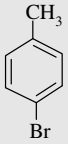
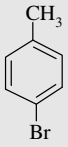
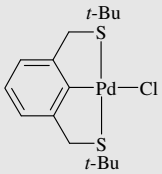
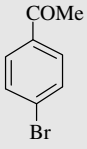
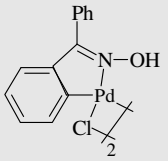
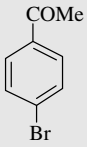
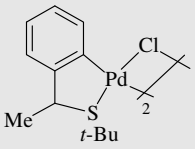
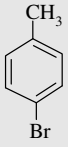
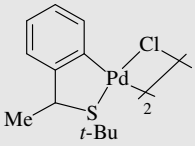
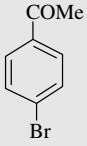
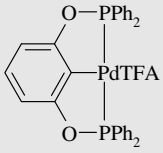
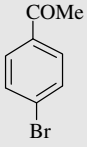
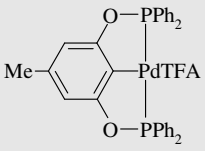
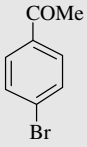
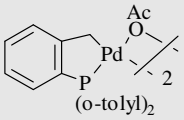
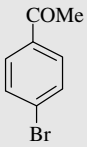
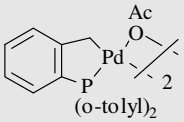
[a] *Reaction conditions:* 1.0 mmol phosphine oxide (or phosphine), 1.2 mmol $ArB(OH)_2$ (2.4 mmol for entries 2, 4, and 6), 0.02 mol% palladacycle **5**, 2.0 mmol K_3PO_4, 5 mL toluene, 1 mL water, 5 h.
[b] Isolated yield according to ^{31}P NMR, not optimized.

Table 3
Comparison of Suzuki coupling using palladacycle **2** and phosphine **1**[a]

Entry	Aryl halide	Arylboronic acid	Yield[b] (%)	
			Phosphine **1**	Palladacycle
1			20(90)	75
2			20(25) 50(70)	100
3			0	65
4			50	100
5			40	90
6			0(30)–(20)	60
7			10(50)	65
8			40(70)	95
9			20(60)	90

[a] *Reaction conditions:* 1.0 mmol aryl halide, 1.5 mmol $ArB(OH)_2$, 0.001 mol% $Pd(OAc)_2$ (or palladacycle **2** in the absence of phosphine **1**), 0.002 mol% phosphine **1**, 2.0 mmol K_3PO_4, 5 mL toluene, 1 mL water, 100 °C, 1 h (6 h for aryl chlorides using palladacycle).
[b] *Isolated yield.* The numbers in parentheses are yields after 24 h.

Table 4
Comparison of Suzuki coupling of phenylboronic acid and different aryl halides using palladacycle **2** and other palladacycles

Entry	Ar-X	Palladacycle	[Pd] (%)	Base	Solvent	*T* (°C)	Time (h)	Yield (%)	Ref.
1		**2**	0.001	K_3PO_4	Toluene	100	1	100	–
2		**2**	0.001	K_3PO_4	Toluene	100	1	100	–
3			1	K_3PO_4	DMF	130	1	69	[23]
4			0.1	K_2CO_3	Toluene	110	0.5	93	[24]
5			0.2	K_3PO_4	DMF	130	2	97	[23]
6			0.002	K_3PO_4	DMF	130	13	74	[24]
7			0.001	K_2CO_3	Toluene	130	18	59	[17]
8			0.001	K_2CO_3	Toluene	130	18	92	[17]
9			0.05	K_2CO_3	*o*-Xylene	130	20	92	[26]
10			0.001	K_2CO_3	*o*-Xylene	130	20	74	[26]

reduction step and if established generally could be an alternative and efficient method for synthesis a wide variety of phosphines.

The results show that phenylboronic acid gives higher conversion in comparison with *o*-tolylboronic acid (Table 2, entries 1–4). Same results were reported in other Suzuki coupling systems [22]. This feature shows that rate of conversion is depended to the type of arylboronic acid and so suggests that transmetallation step may be contributed in the rate of reaction.

2.4. Comparison of Suzuki coupling using palladacycle **2** and phosphine 1

In order to investigate the relative activity of the palladacycle **2** compared to the phosphine **1**, some similar Suzuki cross coupling reactions using palladacycle **2** and phosphine **1** which we have recently demonstrated [21], are listed in Table 3.

The results show that the palladacycle **2** is very reactive than phosphine **1** in the Suzuki coupling reactions. It may be due to the mechanism aspects. The Suzuki coupling for phosphine **1** system proceeds by a mechanism in which the formation of a monophosphine palladacycle intermediate contribute significantly in the rate of reaction.

2.5. Comparison of Suzuki coupling using palladacycle **2** and other palladacycles

High activity of palladacycle **2** in the Suzuki coupling reactions enforced us to compare the obtained results with palladacycle **2** and other similar reported palladacycle systems. Table 4 summarizes this comparison study.

Table 4 illustrates that the palladacycle **2** gives higher conversion in lower catalyst loading in comparison with similar systems (Table 4, entries 1–5). Also in comparatively similar system using same catalyst loading, palladacycle **2** gives higher conversion with lower reaction times (Table 4, entries 1–2, 6–8).

In conclusion we have demonstrated that this new palladacycle is a highly efficient catalyst precursor for the coupling of arylboronic acids with aryl halides and, from the standpoint of yields, it ranks with the best reported systems in the literature, especially when compared with the analogous phosphine adducts of halogen-bridged dimer palladacycles [25]. Palladacycle **2** is also efficient catalyst for the generation of biphenyl-based phosphines and biphenyl-based phosphine oxides. In addition, compared to other similar reported systems, our suggested system has some advantages such as higher activity, lower catalyst loading, lower reaction times, etc.

3. Experimental

3.1. General remarks

All reactions were performed under an atmosphere of either dry nitrogen or argon. All chemicals were purchased commercially from Fluka and/or Merck companies that were used without further purification. Solvents were treated using standard procedures and were distilled under an atmosphere of nitrogen before use. The biphenyl-based phosphine **1** was prepared according to a modification of our previously work [18].

^{1}H (400 MHz), ^{13}C (100 MHz) and ^{31}P (162 MHz) NMR spectra recorded on a Bruker Avance Spectrometer. Elemental analysis was performed using CHN Herause rapid model. Shimadzu GC 14-A and thin layer chromatography on precoated silica gel Fluorescent 254 nm (0.2 mm) on aluminum plates were used for monitoring the reactions. Conversions were determined by GC, based on bromoacetophenone. The cross coupling products were characterized by their ^{1}H NMR spectra and melting points.

3.2. Preparation of palladacycle **2**

A Schlenk tube was charged with phosphine **1** (1.0 g, 2.85 mmol) in toluene (60 mL). A solution of $Pd(OAc)_2$ (0.45 g, 2 mmol) in toluene (30 mL) was added from dropping funnel drop wise. The solution turned pale yellow immediately. The mixture was heated to 50 °C under nitrogen for 3 min and cooled quickly. The volume of mixture was reduced to a quarter *in vacuo*. A white powder was obtained on addition of hexane (60 mL) together with a few black particles which was filtered and the solvent was evaporated using rotary evaporator. The crude product was resolved in toluene (10 mL) and was purified over cellite. The solvent was evaporated and the white powder was crystallized with toluene/ether, yield was 85%.

^{1}H NMR (400 MHz, $CDCl_3$): δ 1–1.5 (m, 16, CH_3 of ether, CH_3 of acetate bridges, CH_2 coordinated to palladium), 3.48 (q, 4, CH_2 of ether), 6.5–7.8 (m, 36, aromatic protons); ^{13}C NMR (200 MHz, $CDCl_3$): 15.5, 21.4, 22.2, 25.6, 28.1, 63.4, 125.1, 125.6, 126.3, 127, 2, 127.5, 128.2, 128.9, 129.4, 132,5, 133.8, 135,2, 136,3, 138,4, 139,6, ^{31}P{1 H} NMR (162 MHz, $CDCl_3$): δ 15.40 (s), 27.69 (s) (for both *cis* and *trans* isomers with relative intensity of 1:3). Anal. Calc. for $C_{54}H_{46}O_4P_2Pd_2 \cdot C_4H_{10}O$: C, 62.87, H, 5.05, P, 5.57, Pd, 19.19. Found: C, 63.01, H, 4.95, P, 5.65, Pd, 18.94%.

3.3. General procedure for the Suzuki coupling of aryl halides

Reaction tube was charged with $PhB(OH)_2$ (1.5 mmol), K_3PO_4 (2 mmol) under a dry nitrogen atmosphere. A solution of 4-bromoacetophenone (1.0 mmol in 2 mL of freshly dried toluene) along with a solution of palladacycle **2** (0.001 mmol in 3 mL of freshly dried toluene) were added through a rubber septum. After addition of water (1 mL), the resulting mixture was heated at 100 °C for 1 h. After extraction with ether, the organic phase was dried over $MgSO_4$. The solvent was evaporated and a crude product was obtained which was characterized by its ^{1}H NMR spectrum and melting point. To isolate the product, the crude product was purified by chromatography with EtOAc/hexane (1:8).

3.4. General procedure for the coupling of bromoarylphosphines and bromophosphine oxides

Reaction tube was charged with $PhB(OH)_2$ (1.2 mmol), K_3PO_4 (2.0 mmol), $PPh_2(o\text{-}C_6H_4Br)$ or $OPPh_2(o\text{-}C_6H_4Br)$ (1.0 mmol), palladacycle **2** (0.02 mmol) in freshly dried toluene (5 mL) under a dry nitrogen atmosphere. After addition of water (1 mL), the resulting mixture was heated at 100 °C for 5 h. After cooling to room temperature, the mixture was diluted with water and extracted with chloroform (3×10 mL), the combined organic extracts were washed with brine, dried over $MgSO_4$. The solvent was evaporated and a crude product was obtained. The crude product was purified by flash chromatography (1:4 EtOAc/hexane).

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