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Suzuki and Heck coupling reactions mediated by palladium complexes bearing *trans*-spanning diphosphines

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Abstract

Palladium complexes of three *trans*-spanning diphosphines are examined for effecting C–C coupling reactions. Ten aryl halides of varying electron density were screened in Suzuki coupling reactions with phenylboronic acid and in Heck reactions with styrene. The results are discussed in terms of the unique flexibility and shape of the *meta*-terphenyl backbone upon which the diphosphine ligand is built.

Keywords: Bite angle; Diphosphine; Suzuki reaction; Heck reaction; C–C coupling

1. Introduction

Palladium-catalyzed carbon–carbon and carbon–nitrogen bond forming reactions are among the most powerful methodologies available to organic chemists [1–3]. Extensive ongoing efforts are thus focused on investigating catalysts with unique ligands to study the effect of ligand electronic and structural properties on reactivity, selectivity, and stereo/regiocontrol of products.

A great deal of interest has recently been devoted to catalysts bearing chelating diphosphines, particularly 'wide bite angle' or '*trans*-spanning' diphosphines (i.e., Xantphos-type ligands, Fig. 1) [4–7]. In addition to steric (i.e., cone angle [8–11]) and electronic (i.e., basicity [12–19]) parameters typically used to describe monophosphines, chelating phosphines may also be described by their bite angle (P-M-P angle) [4]. The bite angle has been shown to exert a pronounced influence on a variety of catalytic processes [4–7]. The contribution of steric versus electronic factors to these effects broadly defined as bite angle effects has been reviewed recently [7].

Studies specifically concerned with bite angle effects on Pd- or Ni-mediated C–C coupling reactions suggest that an increase in bite angle slows the rate of oxidative addition [20,21] and conversely enhances the rate of reductive elimination [22–24]. In one study, a 46-fold difference was observed in the rate of reductive elimination of ethane from a Ni(II) center upon variation of the bite angle of the supporting diphosphine [24]. An overall enhancement of the reaction rate in the coupling of aryl halides with arylboronic acids employing diphosphines of increasing bite angle was also noted [25].

In addition to the bite angle, the flexibility of the scaffold linking the two phosphine units also influences the behaviour of a diphosphine in a catalytic cycle. A flexible scaffold, for example, may allow the ligand to acquire a broader range of angles over the course of the

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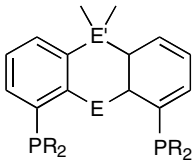


Fig. 1. Xantphos-type phosphine ligands.

catalytic cycle to accommodate necessary intermediates and transition states [20,22,24].

We recently reported a novel diphosphine built upon a *meta*-terphenyl scaffold (Fig. 2, compound **1**) [26]. X-ray structural analysis of its complex with $PdCl_2$ (**4**) displays a *trans*-spanning binding mode and bite angle of 172.97(7)°. While some other bidentate ligands with large bite angles have been employed in catalysis, the *meta*-terphenyl-based diphosphines have a unique three dimensional shape that may be desirable for tuning catalytic processes. Indeed, these properties have made *meta*-terphenyl containing ligands effective for isolating metal complexes designed to mimic the active site in enzymes, highly active naturally occurring catalysts [27–29].

In this report we present the new terphenyl-based diphosphine ligands **2** and **3** along with corresponding complexes **5** and **6**, and report on the catalytic activity of **4–6** (Fig. 2) in representative C–C coupling reactions involving aryl halides. Specifically, 10 representative aryl halides were employed in coupling reactions with phenylboronic acid (Suzuki) or styrene (Heck).

2. Experimental

All manipulations were carried out in a dry box under an atmosphere of N_2. Anhydrous acetone and dichloromethane were purchased from Acros and used as received, all other solvents were distilled from sodium benzophenone ketyl prior to use. The known materials 2,2″-bis-bromomethyl-*m*-terphenyl [30], **1** [26], and **4** [26] were prepared as previously reported. NMR spectra were recorded on a Varian Gemini instrument operating at 300 MHz for proton and 121.5 MHz for phosphorus. Proton and phosphorus spectra are referenced to residual solvent signals and 85% phosphoric acid, respectively. GC-MS analysis of product mixtures [31] was carried out on a Hewlett–Packard 5890 Series II gas chromatograph coupled to a Hewlett–Packard 5971A mass spectroscopic detector.

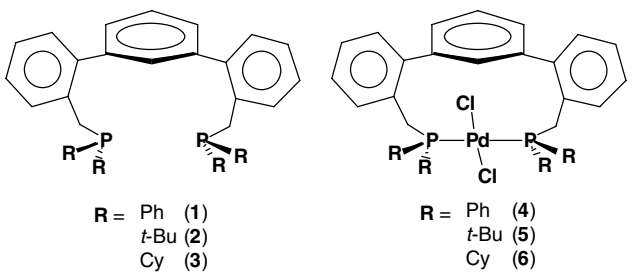


Fig. 2. Ligands and complexes used in this study.

2.1. Preparation of 1,3-bis(2-(di-t-butylphosphinomethyl)-phenyl)benzene (2)

A solution of 2,2″-bis-bromomethyl-*m*-terphenyl (1.00 g, 2.40 mmol) in acetone (15 mL) was added to 0.77 g (5.29 mmol) of di-*t*-butylphosphine, and the resultant solution refluxed for 4 h under nitrogen. After cooling to room temperature, 1.18 g (12.0 mmol) NaOAc was added and the mixture was stirred for 3 h at room temperature. The mixture was filtered and upon standing a white crystalline solid formed. The solid was collected by filtration and rinsed with a small amount of cold acetone and with *n*-pentane, and dried in vacuo (0.827 g, 63.1%). ^{1}H NMR ($CDCl_3$): δ 1.00 (d, 36H, J = 11 Hz), 2.93 (d, 4H, J = 4 Hz), 7.19 (m, 4H), 7.27–7.35 (m, 5H), 7.47 (t, 1H, J = 7 Hz), 7.80 (d, 2H, J = 7 Hz); ^{31}P NMR ($CDCl_3$): δ 40.6.

2.2. Preparation of 1,3-bis(2-(dicyclohexylphosphinomethyl)-phenyl)benzene (3)

A solution of 2,2″-bis-bromomethyl-*m*-terphenyl (1.80 g, 4.33 mmol) and dicyclohexylphosphine (1.89 g, 9.52 mm mmol) in acetone (15 mL) was refluxed for 45 min under nitrogen. After cooling to room temperature, as much acetone as possible was removed via cannula, and the remaining wet white solid was rinsed with an additional 5 mL of acetone. A solution of sodium carbonate (2.8 g in 20 mL water) was added, upon which a large amount of CO_2 was evolved and a sticky solid formed on the walls of the flask. The mixture was stirred at 80 °C for 30 min, then cooled to room temperature and as much water as possible was removed via cannula. The crude solid was rinsed with an additional aliquot of water, and dried in vacuo. The resultant light brown solid was determined to be ~95% pure (^{1}H NMR) and was used without further purification (2.04 g, 72.3%). ^{1}H NMR (C_6D_6): δ 0.95–1.25 (m, 20H), 1.35–1.90 (m, 24H), 2.96 (s, 4H), 7.05–7.30 (m, 4H), 7.30–7.45 (m, 5H), 7.58–7.72 (m, 3H); ^{31}P NMR (C_6D_6): δ 4.8.

2.3. Preparation of 5

A solution of 55 mg (0.10 mmol) of 1,3-bis(2-(di-*t*-butylylphosphinomethyl)phenyl)benzene and 32 mg (0.10 mmol) of $PdCl_2(NCPh)_2$ in CH_2Cl_2 (3 mL) was allowed to stand overnight. Removal of volatiles in vacuo yielded **5** (70 mg, 96%). Analytically pure material was produced by slow precipitation by diffusion of *n*-pentane into a saturated dichloromethane solution of **5**. ^{1}H

NMR ($CDCl_3$): δ 1.43 (m, 36H), 3.11 (m, 4H), 7.42–7.52 (m, 6H), 7.57–7.70 (m, 6H); ^{31}P NMR ($CDCl_3$): δ 59.7. Calc'd for $C_{36}H_{52}Cl_2P_2Pd$: C, 59.72; H, 7.24. Found: C, 58.97; H, 7.12.

2.4. Preparation of 6

A solution of 100 mg (0.15 mmol) of 1,3-bis(2-(dicyclohexylphosphinomethyl)phenyl)benzene and 59 mg (0.15 mmol) of $PdCl_2(NCPh)_2$ in CH_2Cl_2 (6 mL) was stirred for 30 min. Removal of volatiles in vacuo yielded **6** (110 mg, 90%). This material was used without further purification. ^1H NMR ($CDCl_3$): δ 1.00–1.35 (m, 20 H), 1.50–1.95 (m, 24H), 2.00–2.40 (br m, 2H), 3.50–3.75 (br m, 2H), 7.20–7.70 (m, 7H), 8.70–8.87 (br m, 1H); ^{31}P NMR ($CDCl_3$): δ 28.3.

2.5. General procedure for Suzuki coupling reactions

A 5 mL vial equipped with a stirbar was charged with the requisite aryl halide (0.30 mmol), phenylboronic acid (0.45 mmol), Cs_2CO_3 (0.90 mmol), and 1 mol% of either **4**, **5** or **6**. The components were then taken up in 2 mL 1,4-dioxane and heated at 100 °C under N_2 for 25 h. A small aliquot of the reaction mixture was diluted in diethyl ether for direct GC-MS analysis.

2.6. General procedure for Heck coupling reactions

A 5 mL vial equipped with a stirbar was charged with the requisite aryl halide (0.32 mmol), styrene (0.45 mmol), K_2CO_3 (0.64 mmol) and 1 mol% of either **4**, **5** or **6**. The components were then taken up in 2 mL DMF and heated at 120 °C under N_2 for 25 h. A small aliquot of the reaction mixture was diluted in diethyl ether for direct GC-MS analysis.

3. Results and discussion

While ligand **1** [26] was prepared by reaction of diphenylphosphide with 2,2″-bis-bromomethyl-*m*-terphenyl [30], ligands **2** and **3** were most readily prepared by dehydrobromination of the phosphonium salt obtained upon heating of the requisite dialkylphosphine with 2,2″-bis-bromomethyl-*m*-terphenyl in acetone (Eq. 1). The $PdCl_2$ complexes **5** and **6** were prepared in excellent yields by reaction of the diphosphine with $PdCl_2(NCPh)_2$ [32] in dichloromethane at room temperature in the same manner used to prepare **4**. The *trans*-spanning binding mode for ligands **2** and **3** in complexes **5** and **6** was assigned based on the observation that these ligands display a shift of the ^{31}P NMR resonance upon binding to palladium very similar to that observed for **1** upon formation of **4** (19–24 ppm). While the lack of crystallographic data for **5** and **6** prevents an absolute assignment of the ligand binding modes, it should be noted that attempts to model **4**–**6** (using semi-empirical computational methods) with conventional (90° L–Pd–L bond angles) *cis*-binding modes failed due to excessive ring strain and steric clashes.

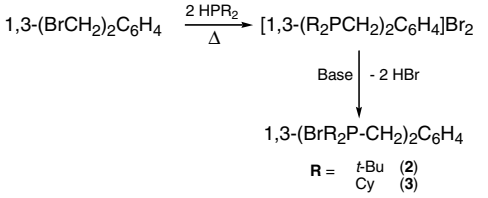
Complexes **4**–**6** thus provide a starting point for comparison of the effect of differing steric and electronic effects provided by phenyl (**4**), *t*-butyl (**5**), or cyclohexyl (**6**) substituents in C–C coupling reactions promoted by this new class of diphosphines. All three catalysts proved to be effective catalysts for Suzuki coupling reactions (Table 1). Compound **4**, in particular, was highly effective in producing the desired biaryls in quantitative yield when aryl bromides or iodides were employed. Quantitative conversions are achieved whether electron donating (entry 13, Table 1) or withdrawing (entry 22, Table 1) groups are employed, and even for more sterically hindered systems (entries 10 and 22, Table 1). Compound **5** gives rather modest yields for most substrates, with depressed yields for more hindered aryl halides (entries 17, 19, and 21, Table 1). The lower overall activity and particular lack of utility of **5** with bulkier substrates may be due to greater crowding of the active site in **5** compared to that in **4**. Unfortunately, the coupling of aryl chlorides was inefficient by either catalyst under the conditions selected, although entries 1 and

Table 1
Suzuki couplings of aryl halides with phenylboronic acid[a]

Entry	Catalyst, 1 (mol%)	ArX	Yield[b] (%)
1	**4**	PhCl	~5
2	**5**	PhCl	0
3	**6**	PhI	0
4	**4**	PhBr	100
5	**5**	PhBr	71
6	**4**	4-*i*-propyliodobenzene	100
7	**5**	4-*i*-propyliodobenzene	65
8	**4**	2-bromotoluene	100
9	**5**	2-bromotoluene	61
10	**4**	Pentamethylbromobenzene	100
11	**5**	Pentamethylbromobenzene	75
12	**6**	Pentamethylbromobenzene	100
13	**4**	2-bromoanisole	100
14	**5**	2-bromoanisole	100
15	**6**	2-bromoanisole	100
16	**4**	1-bromonaphthalene	100
17	**5**	1-bromonaphthalene	42
18	**4**	2-iodochlorobenzene	95[c]
19	**5**	2-iodochlorobenzene	59
20	**4**	3-iodo-*o*-xylene	100
21	**5**	3-iodo-*o*-xylene	56
22	**4**	Pentafluorobromobenzene	100
23	**5**	Pentafluorobromobenzene	6
24	**6**	Pentafluorobromobenzene	24

[a] Reaction conditions: catalyst (1 mol%), aryl halide (0.30 mmol), phenylboronic acid (0.45 mmol), and Cs_2CO_3 (0.90 mmol) at 100 °C in 1,4-dioxane under N_2 for 25 h.
[b] GC yields, ±2%.
[c] ~5% of *o*-terphenyl was also observed.

18 indicate that there is slight activity with **4**. Coupling of phenylboronic acid with four of the aryl halides was also attempted using **6** as the catalyst (entries 3, 12, 15, and 24, Table 1). Catalyst **6** was, however, also inefficient in the coupling of chlorobenzene (entry 3) and was found to be inferior to **4** for the coupling of aryl bromides in some cases (entries 15 and 24), again possibly due to the greater steric encumbrance of cyclohexyl versus phenyl substituents.



(1)

Based on the quantitative couplings of aryl bromides in reactions employing **4**, the coupling of bromobenzene with phenylboronic acid (entry 4) was selected for further investigations. This reaction was repeated with lower catalyst loading, and was found to proceed quantitatively with 0.1 mol% or even 0.01 mol% of **4**, corresponding to a turnover number of at least 10,000 in this reaction.

Heck coupling reactions (Table 2) were also successfully carried out by both **4** and **5**. Once again, no marked differences for electron rich or electron deficient aryl halides were observed. As was observed for Suzuki coupling, when sterically hindered aryl halides are employed **4** gives better yields than **5**. In this case, however, the impact is even more pronounced (compare entries 10–11 and 22–23, Table 2). It is also of note that while **5** produces only *trans*-stilbenes in all cases, mixtures are typically observed when **4** is employed.

Surprisingly, complex **6** proved very ineffective in any of the Heck couplings in which it was employed (entries 3, 12, 15, and 24, Table 2). This finding was especially puzzling in light of the generally greater efficiency of **6** compared to that of **5** in Suzuki couplings (compare entries 11 and 12 or 23 and 24, Table 1). Based on a simple steric argument, the efficiency of **6** may be expected to be higher than that of **5** (as observed in Suzuki reactions) due to the smaller cone angle presumably present at a dicyclohexylphosphino center versus a di-*t*-butylphosphino center. Since the results of Suzuki coupling with **6** indicate that oxidative addition of the aryl halide to the Pd center is occurring, the disparity in efficiency might involve a subsequent step. Another possibility is that relative reactivities may be related to the extent to which one of the phosphine arms dissociates to form monophosphine complexes under the experimental conditions.

If both phosphorus atoms in **4**–**6** remain coordinated during catalysis, then discussion of how readily each complex can change from a *trans*-spanning mode (inefficient for reductive elimination/oxidative addition at a single metal center) to a *cis*-binding mode or, more likely, a wide-bite angle mode is appropriate. The ability of bidentate phosphines to undergo such transformations has been previously analyzed in terms of their "natural bite angles" [33,34]. In such models, a bidentate phosphine is characterized by a natural bite angle that reflects the inherently most stable P–M–P bond angle for a select M–P bond distance (usually determined by molecular mechanics in the absence of other ligands within the M coordination sphere). Possible backbone motions accompanying such angular changes for ligands **1**–**3** are depicted in Fig. 3. As the P–M–P bond angles move away from 180° in either direction, there is a point at which the R groups on the phosphorus atoms begin to clash (depicted as semi-circles in Fig. 3). For the *tert*-butyl containing **2**, this effect may be amplified with respect to smaller phe-

Table 2
Heck couplings of aryl halides with styrene[a]

Entry	Catalyst, 1 (mol%)	ArX	Yield[b] (%)	*Cis*:*trans*
1	**4**	PhCl	0	–
2	**5**	PhCl	0	–
3	**6**	PhCl	0	–
4	**4**	PhBr	63	All trans
5	**5**	PhBr	100	All trans
6	**4**	4-*i*-propyliodobenzene	100	1:1.5
7	**5**	4-*i*-propyliodobenzene	100	All trans
8	**4**	2-bromotoluene	76	1:3
9	**5**	2-bromotoluene	100	All trans
10	**4**	pentamethylbromobenzene	100	1:5.3
11	**5**	pentamethylbromobenzene	36	All trans
12	**6**	pentamethylbromobenzene	0	–
13	**4**	2-bromoanisole	22	1:1
14	**5**	2-bromoanisole	16	All trans
15	**6**	2-bromoanisole	0	–
16	**4**	1-bromonaphthalene	98	1:5.7
17	**5**	1-bromonaphthalene	100	All trans
18	**4**	2-iodochlorobenzene	100	1:4
19	**5**	2-iodochlorobenzene	29	All trans
20	**4**	3-iodo-*o*-xylene	100	1:2.3
21	**5**	3-iodo-*o*-xylene	72	All trans
22	**4**	Pentafluorobromobenzene	67	All trans
23	**5**	Pentafluorobromobenzene	25	All trans
24	**6**	Pentafluorobromobenzene	0	–

[a] Reaction conditions: catalyst (1 mol%), aryl halide (0.32 mmol), styrene (0.45 mmol), and K_2CO_3 (0.64 mmol) at 120 °C in DMF under N_2 for 25 h.
[b] GC Yields., ±2%.

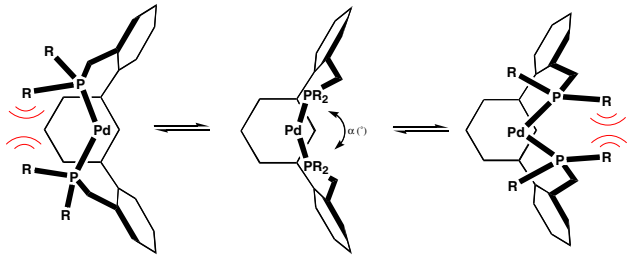


Fig. 3. Movement of P–Pd–P bond angle in Pd·**L**.

nyl-substituted **1**. The Ph_2P analogue may therefore have more ready access to smaller bond angles (towards 90°) that may be necessary for the intermediates involved in effective catalysis. NMR data for **1** suggest fluxional processes that are in accord with the motions depicted in Fig. 3 [26]. Compound **1** may thus be an example of a *trans*-spanning ligand capable of reaching a angles needed for coupling catalysis. More work is needed to support this hypothesis.

The catalytic efficiency of **4**–**6** is of particular note, as it has been observed that some chelating phosphines suppress or slow coupling reactions [1,35]. Other chelating ligands, however, have proven to be highly efficient if reaction conditions are carefully selected [36]. The bis(diphenylphosphino)ferrocene and related ligands, for example are particularly efficient [1,2,22]. Metallocene-diphosphines have an element of flexibility that allows access to an extended range of bite angles. In metallocene-diphosphines the flexibility is acquired through low-energy 'scissoring' of cyclopentadienyl units, a process believed to contribute to the efficacy of such ligands in catalysis [22].

Comparably few applications of chelating, *trans*-spanning phosphines to coupling reactions have been reported. The more rigid wide-bite angle xantphos ligands, for example, have not been extensively employed in Suzuki or Heck reactions. Heck coupling reactions employing complexes complexes having a xantphos-type ligand (Fig. 1, R = *o*-Tol; E = O, E′ = C) are highly dependent on the electron density of the aryl halide. For example, coupling of *n*-butylacrylate with *p*-(CO_2Me)C_6H_4Br (53%), 4-bromotoluene (4%), or 4-bromoanisole (~0%) demonstrate a significantly enhanced yield when electron withdrawing substituted aryl bromides are employed [37]. Studies of palladium catalyzed amination using the xantphos ligand (Fig. 1, R = Ph; E = O, E′ = C) have also been undertaken, uncovering a catalytically active complex with the xantphos ligand in a *trans*-spanning binding mode (P–Pd–P bond angle of 150.7°) [38].

4. Concluding remarks

In conclusion, the preliminary reactions screened herein indicate that diphosphines built upon a *meta*-terphenyl scaffold hold promise as catalysts for C–C bond forming reactions. The unique shape and steric properties of the ligand backbone may, in concert with variation of R groups on the phosphorus center, allow for further development of catalysts for such reactions. Current work is also underway to examine the utility of these ligands in other catalytic transformations.

Acknowledgements

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