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An Easily Prepared Tetraphosphine and Its Use in the Palladium-Catalyzed Suzuki–Miyaura Coupling of Aryl Chlorides

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Abstract An air-stable tetraphosphine *N*,*N*,*N*′,*N*′-tetra(diphenylphosphinomethyl)-benzene-1,3-diamine (**L3**) was easily prepared in two steps from triphenylphosphine, which in combination with $[Pd(\eta^3\text{-}C_3H_5)Cl]_2$ affords an efficient catalyst for Suzuki–Miyaura coupling of activated chloroarenes. Even at high temperature of 130 °C, this catalyst exhibits good stability and longevity, and could allow a high turnover number of 680,000 to be reached.

Keywords Suzuki–Miyaura coupling · Palladium · Tetraphosphine · Aryl chlorides

1 Introduction

The palladium-catalyzed Suzuki–Miyaura cross-coupling reaction, which is one of the most efficient methods for the synthesis of biaryl compounds, is particularly popular in the pharmaceutical and chemical industries [1–5]. For the applications, the use of cheaper chloroaryls is important, but their reluctance to perform the oxidative addition to palladium makes the coupling procedure difficult [6, 7]. In recent years, a great deal of attention has been focused on the exploration of catalytic systems that could improve efficiency of the reactions of aryl chlorides [8–14], but the coupling conducted at low catalyst loadings was less involved and is still an ongoing field [15–18].

NHC-based ligand could allow high turnover numbers (TONs) to be reached [19, 20]; Buchwald's monodentate phosphines are indubitable in terms of reactivity and efficiency [21–26]. Improved catalytic activities were detected from systems that associate palladium to strong σ-electron-donating ligands. Bedford and coworkers proposed a concept of catalyst longevity [27]. They attributed the spectacular TONs they observed using palladacycle not to a high activity of the catalyst, but more to an outstanding catalyst longevity, consequently the stability of the catalytic species. This study inspired chemists to develop ligands which could stabilize palladium species efficiently. A tetradentate phosphine Tedicyp [28–32] by Doucet and Santelli displayed remarkable TONs, which was regarded as a breakthrough in this area. This discovery prompted the examination of various carbocyclic [33, 34], ferrocenyl-based [35–37] and cavity-shaped [38, 39] multidentate phosphines in low catalyst loading Suzuki couplings. A cyclodextrin-tetraphosphine hybrid coined α-Cytep [39] displayed exceptionally high TONs. The authors associated this property with its ability to super-stabilize the catalytic species over a very long time via multiple binding to the metal. Multidentate phosphines shed light on the development of ultra-low loading catalytic processes.

However, the long synthetic route of multidentate phosphines results in the limitation on their applications [28, 33–39]. In addition, due to the sensitivity to air, these ligands were always protected and stored as their borane complexes, and required deboronation before use in the catalytic reaction [28, 39]. Although the Suzuki coupling using multidentate phosphines has been much investigated, the studies on the reaction of aryl chlorides were less involved [29, 35, 39].

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In this paper, we aim to synthesize an air-stable tetraphosphine, which could be easily prepared from commercially available reactants, expecting to develop an effective catalytic system for the low palladium-loading Suzuki–Miyaura coupling of aryl chlorides.

2 Results and Discussion

We have reported the results in Suzuki–Miyaura coupling based on tetraphosphine *N*,*N*,*N*′,*N*′-tetra(diphenylphosphinomethyl)-1,2-ethylenediamine (**L1**) (Fig. 1) [40, 41], which exhibits a good stabilization to the active palladium species. Aryl and heteroaryl bromides coupled with phenylboronic acid efficiently at low catalyst loadings, and a high TON of 750,000 could be achieved. Unfortunately, the catalyst system consisting of Pd/**L1** is not active for aryl chlorides.

Previous studies have demonstrated that in the catalytic cycle the oxidative addition step is favored by electron-rich ligands. On the other hand, the rate of reductive elimination is usually increased by the coordination of bulky ligands to the metal center. Therefore, those ligands combination of both electronic and steric properties are of benefit for the Suzuki reaction of non-activated substrates. Lacking of sufficient steric hindrance in **L1**, which lies in the flexibility of the 1,2-ethylenediamine backbone probably leads to the unsatisfactory results for the couplings of chloroarenes.

To prove the hypothesis, we synthesized a new tetraphosphine *N*,*N*,*N*′,*N*′-tetra(diphenylphosphinomethyl)-cyclohexane-1,2-diamine (**L2**) (Fig. 1) and tested its efficacy for the palladium-catalyzed Suzuki reaction of aryl chlorides [42]. Under a relatively mild reaction condition (60 °C), aryl and heteroaryl chlorides bearing electron-withdrawing substituents could be successfully transformed to the desired products in excellent yields with 0.1 mol% Pd, and a yield of 80 % was given for the coupling of 3-chloroanisole. This ligand seems to possess a fine balance of electronic and steric properties. However, the increased electron density at phosphorus caused by the cyclohexane moiety in **L2** makes this tetraphosphine air-sensitive. Consequently, it is unfavorable to use in microscale catalytic experiments, and isolating oxygen for storage is required.

Aiming to prepare a stable phosphine ligand which could perform satisfactory results for the Suzuki coupling of aryl chlorides, we replace the core structure of **L1** with a rigid moiety 1,3-phenyldiamine to obtain a new tetraphosphine *N*,*N*,*N*′,*N*′-tetra(diphenylphosphinomethyl)-benzene-1,3-diamine (**L3**) (Fig. 1). Based upon the procedure reported previously [42, 43], we modified the reaction conditions to provide the target ligand in a simple protocol with an overall yield of 73 % (Scheme 1). **L3** is stable in air and consequently, is very easy to store and handle.

We employed the coupling of 4-chloroacetophenone with phenylboronic acid as the test case to gauge the performance of our new ligand and to optimize the reaction parameters (Table 1). Dimethylacetamide (DMAc) and K_3PO_4 were adopted as solvent and base respectively for good results. 98 % conversion of 4-chloroacetophenone was observed using 0.05 mol% $[Pd(\eta^3\text{-}C_3H_5)Cl]_2$ and 0.1 mol% **L3** at 130 °C for 20 h (Table 1, entry 9).

In the absence of ligand, only a trace amount (2 %) of biaryl adduct was detected under the same reaction conditions (Table 2, entry 1), and Pd black appeared rapidly. It is suggested that the addition of **L3** highly improved the stability of the active palladium species, and the decomposition of the catalyst could be avoided even at the relatively high temperature of 130 °C.

For the further evaluation of the importance of tetradentate phosphine **L3** on the complex, we compared the rate of the reactions based on PPh_3 and electronically similar diphosphines bearing diphenylphosphinomethyl group: 1,2-bis(diphenylphosphino)ethane (dppe), 1,4-bis(diphenylphosphino)butane (dppb), 2,2′-bis(diphenylphosphinomethyl)-1,1′-biphenyl (BISBI) and 1,2-bis(diphenylphosphinomethyl)benzene (BDPX). This comparison was carried out with 4-chloroacetophenone as the test substrate (Table 2, entries 5–21). When 0.1 mol% Pd/PPh_3 was used as catalyst, only 4 %

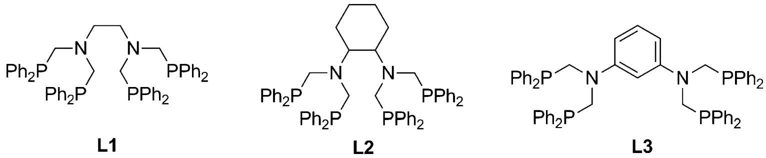
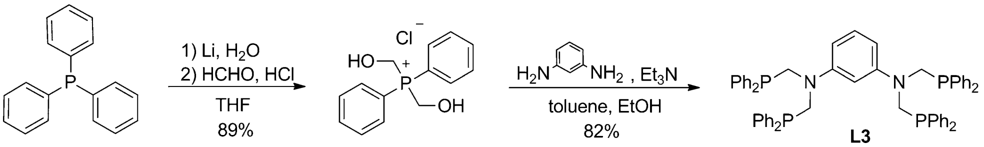
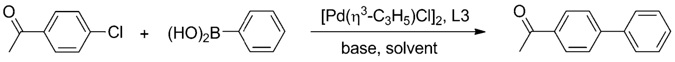


Fig. 1 Tetraphosphines we have reported for the Suzuki coupling



Scheme 1 Synthesis of *N*,*N*,*N*′,*N*′-tetra(diphenylphosphinomethyl)-benzene-1,3-diamine (**L3**)

Table 1 The effect of solvent and base on the Suzuki cross-coupling reaction

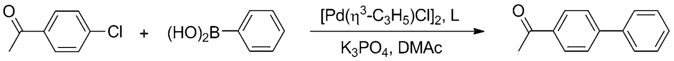


Entry	Solvent	Base	Yield (%)
1	2-Ethoxyethanol	K_2CO_3	41
2	Xylene	K_2CO_3	39
3	DMF	K_2CO_3	35
4	DMAc	K_2CO_3	62
5	DMAc	Cs_2CO_3	71
6	DMAc	NaOH	83
7	DMAc	KOH	76
8	DMAc	K_3PO_4	87
9	DMAc	K_3PO_4	98[a]

Reaction conditions: 4-chloroacetophenone 1 mmol, phenylboronic acid 1.5 mmol, base 2 mmol, solvent 2 mL, **L3**/$[Pd(\eta^3\text{-}C_3H_5)Cl]_2 = 2/1$, $[Pd(\eta^3\text{-}C_3H_5)Cl]_2$ 0.0005 mmol, **L3** 0.001 mmol, temperature 130 °C, 20 h, analyzed by GC, average of two runs

[a] K_3PO_4 2.5 mmol

Table 2 The effect of phosphine ligands on the Suzuki cross-coupling reaction



Entry	Ligand	Pd loading (mol%)	Yield (%)
1	–	0.1	2
2	**L1**	0.1	43
3	**L2**	0.1	99
4		0.01	13
5	**L3**	0.1	98
6		0.01	40
7		0.01	98[a]
8	PPh_3	0.1	4[c]
9		0.1	5[a,b]
10	dppe	0.1	31
11		0.1	34[a]
12		0.01	5
13		0.01	6[a]
14	dppb	0.1	36
15		0.1	40[a]
16		0.01	5
17		0.01	5[a]
18	BISBI	0.1	18
19		0.1	21[a]
20	BDPX	0.1	21
21		0.1	25[a]

Reaction conditions: 4-chloroacetophenone 1 mmol, phenylboronic acid 1.5 mmol, K_3PO_4 2.5 mmol, Ligand/$[Pd(\eta^3\text{-}C_3H_5)Cl]_2 = 2/1$, DMAc 2 mL, temperature 130 °C, 20 h, analyzed by GC, average of two runs

[a] Reaction time 60 h

[b] Ligand/$[Pd(\eta^3\text{-}C_3H_5)Cl]_2 = 4/1$

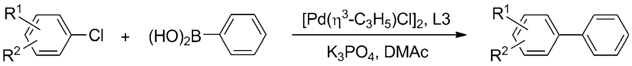
conversion was given (Table 2, entry 8). The utilization of bidentate ligands led to higher yields (Table 2, entries 10, 14, 18, 20), and the backbone structure of phosphines seems to affect the reaction rate. The diphosphines with alkyl backbones (Table 2, entries 10, 14) provided better performance than those with aryl backbones (Table 2, entries 18, 20). However, at the end of the above reactions (Table 2, entries 8, 10, 14, 18, 20) Pd black was clearly observed, and the conversions were not increased markedly even when the reaction time was extended to 60 h (Table 2, entries 9, 11, 15, 19, 21). The result using tetraphosphine **L3** compared favorably with these mono- or bidentate phosphines, and this advantage was shown more obviously when the Pd loading was decreased to 0.01 mol% (Table 2, entries 6, 7, 12, 13, 16, 17). The comparative investigation illustrates that the species formed by association of tetraphosphine **L3** and $[Pd(\eta^3\text{-}C_3H_5)Cl]_2$ appears to be more stable than the systems formed with electronically similar mono- or bidentate phosphines.

In order to discuss the effect of structure of diamine moiety in tetraphosphines on the reaction rate, the coupling of 4-chloroacetophenone with phenylboronic acid was investigated with **L1**, **L2** and **L3** under the same reaction conditions, respectively (Table 2, entries 2–7). As expected, compared to the steric hindered ligand **L2** and **L3**, **L1** was unfavorable for the coupling. With 0.1 mol% Pd, just a moderate yield of 43 % was given (Table 2, entry 2). When the catalyst loading was reduced to 0.01 mol%, the same reaction performed with **L2** and **L3** resulted in the yield of 13 % (Table 2, entry 4) and 40 % (Table 2, entry 6), respectively. **L2** associated to palladium showed a good activity in our previous report, but this tetraphosphine seems to compare unfavorably with **L3** for the reactions at low catalyst loading. **L2** is air-sensitive and in microscale experiments the probability of oxidation on phosphorus would increase, which probably led to the inefficiency for the generation of palladium complex and for the stabilization to active palladium species. The aromatic backbone in **L3** could not provide a high electron density at phosphorus to accelerate the oxidative addition process as the ligand with alkyl moieties. However, a steric factor from the rigid diamine might be beneficial for the reductive elimination step, and finally increase the reaction rate. As a result, a higher yield could be achieved by use of **L3**.

Subsequently, we screened reactions of various aryl chlorides with phenylboronic acid, and all the results are illustrated in Table 3.

First, we investigated the Suzuki reaction of several *para* substituted aryl chlorides. A strong substituent effect on the reaction rate was unfolded. Electron-withdrawing groups in the aryl chlorides support the process. In the presence of 0.01–0.1 mol% Pd/**L3** catalyst, the couplings of 4-chloroacetophenone, 4-chloronitrobenzene, 4-chlorobenzotrifluoride, 4-chlorobenzonitrile and 4-chlorobenzaldehyde with phenylboronic acid proceeded to provide the corresponding biaryls in good results (Table 3, entries 3, 7, 11, 13, 17). On the other hand, electron-donating chloroaryls was unfavorable. With 4-chloroanisole, a TON of 18 was obtained (Table 3, entry 1), which exhibited a similar efficacy in comparison to the reported Pd/tetraphosphine catalyst systems [29, 35].

Table 3 Suzuki coupling of aryl chlorides with phenylboronic acid based on Pd/**L3**



Entry	R^1	R^2	Ratio substrate/catalyst	Yield (%)
1	4-MeO	H	100	18
2	4-MeCO	H	1,000	95
3	4-MeCO	H	10,000	96^{a}
4	2-MeCO	H	1,000	44
5	2-MeCO	H	1,000	93^{a}
6	4-NO_2	H	1,000	90
7	4-NO_2	H	10,000	90^{a}
8	2-NO_2	H	1,000	43
9	2-NO_2	H	1,000	93^{a}
10	4-CF_3	H	1,000	98^{b}
11	4-CF_3	H	10,000	$95^{a,b}$
12	4-CN	H	1,000	64^{b}
13	4-CN	H	1,000	$91^{b,c}$
14	2-CN	H	1,000	34^{b}
15	2-CN	H	1,000	$87^{a,b}$
16	4-CHO	H	1,000	61^{b}
17	4-CHO	H	1,000	$88^{b,c}$
18	2-CHO	H	1,000	90^{b}
19	2-NO_2	4-CF_3	10,000	99^{b}
20	2-NO_2	4-CF_3	100,000	87^{b}
21	2-NO_2	4-CF_3	1,000,000	$41^{a,b}$
22	2-NO_2	4-CF_3	1,000,000	$60^{b,d}$
23	2-NO_2	4-CF_3	1,000,000	$68^{b,e}$

Reaction conditions: aryl chloride 1 mmol, phenylboronic acid 1.5 mmol, K_3PO_4 2.5 mmol, **L3**/$[Pd(\eta^3\text{-}C_3H_5)Cl]_2 = 2/1$, DMAc 2 mL, temperature 130 °C, 20 h, isolated yield, average of two runs

[a] Reaction time 60 h

[b] K_2CO_3 as base (2.5 mmol)

[c] Reaction time 40 h

[d] Reaction time 120 h

[e] Reaction time 200 h

Some activated substrates bearing functional groups were sensitive to the strong basicity of K_3PO_4, which resulted in the formation of undesired products. Thus K_2CO_3 was used instead (Table 3, entries 10–23).

Next, we studied the influence of an *ortho* substituent on the chloroaryls, and an obvious steric effect was observed on the reaction rate. 2-Chloroacetophenone, 2-chloronitrobenzene and 2-chlorobenzonitrile led to much lower yields than the corresponding *para* substituted substrates (Table 3, entries 4, 8, 14). However, 2-chlorobenzaldehyde was an exception (Table 3, entry 18), which was more beneficial than 4-chlorobenzaldehyde for the coupling reaction under the same conditions.

We tried to determine the limitation of the catalyst loading. The very activated substrate 2-chloro-5-(trifluoromethyl)nitrobenzene could be coupled with phenylboronic acid with a Pd loading as low as 0.0001 mol%. After 60 h, 41 % conversion was detected (Table 3, entry 21). When the reaction time extended to 200 h, the desired biaryls was given with a high TON of 680,000 (Table 3, entry 23), and the yield did not increase any more even when the reaction time was further prolonged. The results indicated that the Pd/**L3** catalyst system exhibits a good longevity even at a high temperature, which is probably attributed to the tetraphosphine **L3** providing the active palladium species with good stabilization. It is also demonstrated that multidentate ligand had advantages for the exploration of low loading catalyst systems.

3 Conclusion

In conclusion, we synthesized a simple and air-stable tetraphosphine *N*,*N*,*N*′,*N*′-tetra- (diphenylphosphinomethyl)-benzene-1,3-diamine (**L3**) in two steps from PPh_3, and a catalyst system comprised of $[Pd(\eta^3\text{-}C_3H_5)Cl]_2$/**L3** for the Suzuki cross-coupling of aryl chlorides has been established. Even at high temperature of 130 °C, this tetraphosphine could efficiently stabilize the active palladium species over a long time. From the comparative investigation, this catalyst is more stable than the complexes formed with electronically similar mono- or diphosphines. The couplings of electron-deficient chloroarenes can be performed with 0.01–0.1 mol% catalyst, and a high TON of 680,000 is obtained for the reaction of the very activated substrate. Unfortunately, this Pd/tetraphosphine system is unfavorable for electron-rich aryl chloride. Further work to improve the structure of the ligand is in progress.

4 Experimental

Reactions were carried out under a nitrogen atmosphere. All chemicals were purchased from commercial suppliers. Solvents were dried over appropriate drying agents and distilled under nitrogen before use.

4.1 Synthesis of *N*,*N*,*N*′,*N*′-tetra(diphenylphosphinomethyl)-benzene-1,3-diamine (**L3**)

$[Ph_2P(CH_2OH)_2]Cl$ was prepared according to the reported methods with a yield of 89 % [44]. *m*-Phenyldiamine (216 mg, 2 mmol) in 10 mL ethanol was added to excess of triethylamine (3 mL, 20 mmol) and $[PPh_2(CH_2OH)_2]Cl$ (2.8 g, 10 mmol) in mixture solvent of 10 mL ethanol and 20 mL toluene. The solution was refluxed for 40 h. At the end of reaction, the mixture was cooled to room temperature, and the product was washed with water (3 × 10 mL), then dried over magnesium sulfate and filtered. Subsequently, the solvent was removed under vacuum and the resulting crude product was refluxed in 30 mL ethanol for 2 h. The solution was slowly cooled to room temperature. The pale yellow product was filtered, washed with ethanol (3 × 5 mL) and dried under vacuum. Yield: 1.48 g (82 %). ^{31}P NMR (162 MHz, $CDCl_3$): δ −26.88 ppm. ^1H NMR (400 MHz, DMSO): δ 7.36–7.27 (m, 40H, PPH_2), 6.89 (t, $J = 8.0$ Hz, 1H, PH^5), 6.29 (s, 1H, PH^2), 6.21 (d, $J = 8.0$ Hz, 2H, $PH^{4,6}$), 3.87 (d, $^2J_{PH} = 4.0$ Hz, 8H, NCH_2). ^{13}C NMR (101 MHz, $CDCl_3$): 148.17 (s, $C_6^{1,3}H_4N_2$), 136.64 (d, $^1J_{CP} = -16.2$ Hz, $PC_6^1H_5$), 132.23 (s, $C_6^5H_4N_2$), 132.22 (d, $^2J_{CP} = 20.2$ Hz, $PC_6^{2,6}H_5$), 128.20 (s, $PC_6^4H_5$), 127.36 (d, $^3J_{CP} = 22.2$ Hz, $PC_6^{3,5}H_5$), 104.37 (s, $C_6^{4,6}H_4N_2$), 101.67 (s, $C_6^2H_4N_2$), 53.21 (d, $^1J_{CP} = -7.1$ Hz, NCH_2P). HRMS (ESI) Calcd. for $C_{58}H_{52}N_2P_4$: 900.3081 $[M]^+$, found: 900.3076.

4.2 General Procedure of Suzuki Coupling Reactions

Tetraphosphine **L3** (9.0 mg, 0.01 mmol) and $[Pd(\eta^3\text{-}C_3H_5)Cl]_2$ (1.8 mg, 0.005 mmol) were added into a Schlenk tube equipped with a magnetic bar, and then DMAc (1.0 mL) was added. The mixture was stirred at room temperature for 1.5 h. 4-Chloroacetophenone (130 μL, 1.0 mmol), phenylboronic acid (183 mg, 1.5 mmol) and K_3PO_4 (531 mg, 2.5 mmol) were added into another Schlenk tube with a magnetic bar. The dissolved mixture of **L3**/Pd (100 μL, 0.001 mmol) was transferred to the Schlenk tube of reactants by syringe. Then, DMAc (2 mL) was added. The reaction mixture was heated at 130 °C for 20 h. At the end of the reaction, the solution was cooled to room temperature and water (5 mL) was added. The mixture solution was extracted with ethyl acetate (3 × 5 mL) and the organic layer was dried over magnesium sulfate. The dried solution was filtered and reduced to ca 1–2 mL under vacuum, then purified with silica gel chromatography to give the corresponding product with an isolated yield.

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