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Preparation of anionic phosphine ligands in situ for the palladium-catalyzed Buchwald/Hartwig amination reactions of aryl halides

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ABSTRACT

2-Phenylindenyl phosphine ligand can be changed into anionic phosphine ligand in situ and utilized in the palladium-catalyzed Buchwald/Hartwig amination reactions in DME, providing good to excellent yields of amination products from aryl chlorides, bromides and iodides. ^{31}P NMR studies show that the resonance for the anionic phosphine appeared between those of the (2-phenylindenyl)-dicyclohexyl phosphonium salt and (2-phenylindenyl)dicyclohexylphosphine. The calculated results were consistent with the experimental results.

1. Introduction

Palladium-catalyzed C–N bond-forming reactions have evolved into a highly versatile and synthetically attractive technique for targeting pharmaceutically useful intermediates [1,2]. Since the discovery of the first catalytic amination method [3–5], efforts have been made to increase the reaction efficacy [6–8]. Notable ligands, such as ^{t}Bu$_3$P [9,10], Beller and co-workers' PAP [11], Buchwald and co-workers' biaryl phosphines [12,13], Hartwig and co-workers' Q-Phos and CyPF-^{t}Bu [14,15], Verkade and co-workers' amino phosphine [16,17], and Kwong and co-workers' 2-indolylaryl phosphine [18] (Fig. 1) provide excellent catalytic activity for the cross-coupling of aryl halides (especially aryl chlorides) or aryl mesylate/arenesulfonate.

Although a variety of ligands have been introduced, the rapid assembly of structurally diverse ligand systems via simple synthetic methods is still important for the development of versatile catalysts for numerous applications of coupling reactions. Recently, Plenio's group reported a number of closely related phosphine ligands which all have the same basic cyclopentadienyl core, as found in cyclopentadienyl [19], indenyl [19] or fluorenyl phosphines [20], whose Pd-complexes are excellent catalysts for various cross coupling reactions. The sulfonated derivatives of fluorenyl phosphines form water soluble Pd-complexes [21–26], which showed unprecedented catalytic activity in the cross coupling of heterocyclic substrates in aqueous reaction media (Fig. 2). In this paper, we describe the development of 2-phenylindenyl dicyclohexylphosphine as a novel ligand to activate the palladium catalyst for the Buchwald/Hartwig amination reaction.

2. Results and discussion

2.1. Synthesis of (2-phenylindenyl)dicyclohexylphosphonium tetrafluoroborate (2)

2-Phenylindenyl phosphine ligand can be easily prepared from commercially available indene and phenyl iodide (Scheme 1). 2-Phenylindene was prepared according to the reported procedure [27], then the straightforward deprotonation of 2-phenylindene by ^{n}BuLi and trapping of the lithiated intermediate by Cy$_2$PCl afforded the corresponding phosphine **1** in good yields [28]. This was isolated as its phosphonium salt **2** for easier storage and handling [10].

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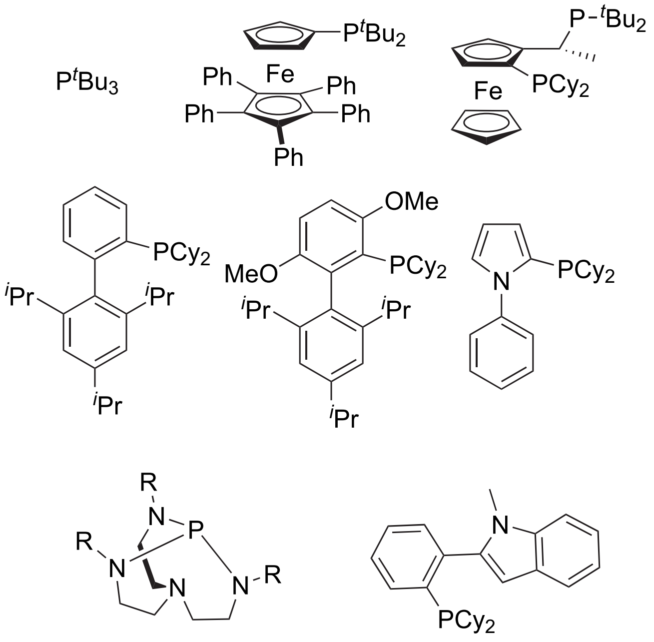


Fig. 1. Recent developments on effective phosphine ligands.

2.2. X-ray crystal structure of (2-phenylindenyl) dicyclohexylphosphonium tetra-fluoroborate (2)

The molecular structure of **2** has been confirmed by single-crystal X-ray diffraction (Fig. 3). Selected bond lengths and angles are listed in Table 1. In the solid state, the C(7)–C(15) bond is in the single bond range (distance between C(7)–C(15) is 1.501 Å) [29], while the bond distance C(7)–C(8) is 1.364 Å, which is in the range of a carbon–carbon double bond [29]. The distance of P(1) to the indene plane is 0.1338 Å, so the P(1) atom is almost in the same plane as the indene, while the distance of P(1) to the aryl ring is 2.1824 Å, which is similar to that of 2-(dicyclohexylphosphino)-4′-*N*,*N*-(dimethylamino)-1,1′-biphenyl [30]. The angle between the aryl ring and the indenyl plane is 52.4°, the angle between the aryl ring and the C(8)–P(1)–H(1A) plane is 58.5°. Finally, the P(1)–H(1A) bond is directed toward the aryl ring, which is similar to the structure of biaryl phosphines. The phosphorus lone pair is directed toward the aryl ring [30] and, as a result, the **2**/Pd catalyst can activate the C–Cl bond.

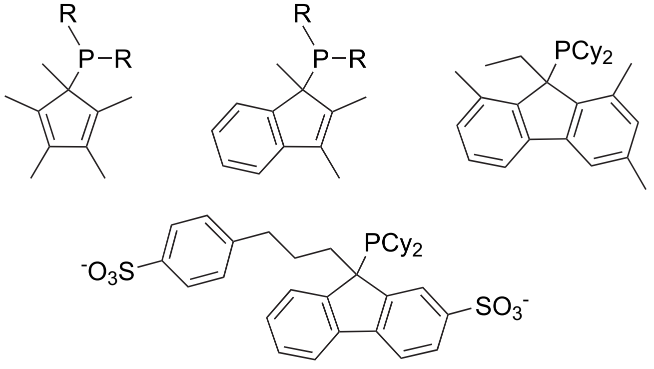
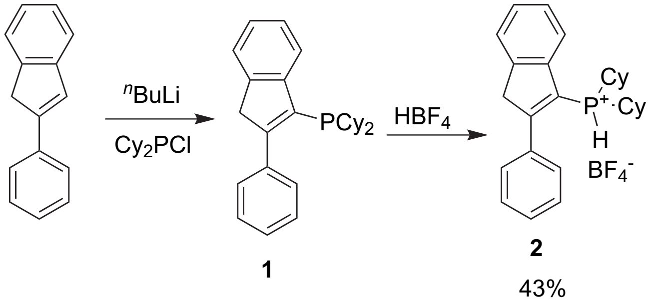


Fig. 2. Phosphine ligands containing cyclopentadienyl core.



Scheme 1. Preparation of 2-phenylindene phosphonium salt **2**.

2.3. Pd complex of (2-phenylindenyl)dicyclohexylphosphine in the Buchwald/Hartwig reaction

With this 2-phenylindenyl phosphine ligand, the feasibility of promoting C–N couplings of aryl halide was investigated. Chlorobenzene and aniline were used as the prototypical substrates in our reaction (Table 2). In this work the base was not screened and ^{t}BuONa was used. Of the solvents surveyed (toluene and DME), DME provided the best product yield (Table 2, entries 4–6). Screening of commonly used Pd sources indicated that Pd(dba)$_2$ and PdCl$_2$(CH$_3$CN)$_2$ were suitable sources and Pd(dba)$_2$ showed the highest catalytic ability for the chlorobenzene coupling reaction (Table 2, entries 4, 6–8).

The scope of this reaction was then investigated under optimized conditions [Pd(dba)$_2$ (1 mol%), ligand (2 mol%), ^{t}BuONa (1.4 equiv), 120 °C], and the results are summarized in Table 3. In most cases, the aryl chloride reacted with different amines leading to the corresponding products in good to excellent yields. For example, the reaction of chlorobenzene with octyl amine gave rise to the corresponding secondary amine in 63% yield (Table 3, entry 1), while the reaction of 4-chlorotoluene or 2-chlorotoluene with octyl amine afforded secondary amines in 55% or 36% yield, respectively (Table 3, entries 2 and 4). Excellent yield was observed when chlorobenzene or 2-chlorotoluene reacted with cyclic amine

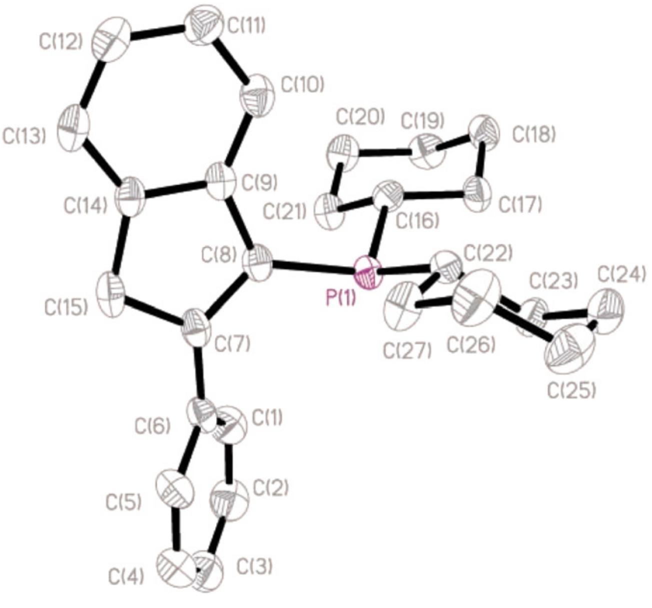


Fig. 3. Molecular structure of **2**, H atoms and PF$_6^-$ anion are omitted for clarity.

Table 1
Selected bond distances (Å) and angles (°) for compound **2**.

C(7)–C(8)	1.364(4)	C(16)–P(1)	1.804(3)
C(7)–C(15)	1.501(5)	C(22)–P(1)	1.811(3)
C(8)–P(1)	1.781(3)	C(1)–C(7)	1.469(5)
C(8)–C(7)–C(1)	129.4(3)	C(8)–C(7)–C(15)	108.7(3)
C(7)–C(8)–P(1)	126.9(3)	C(8)–P(1)–C(16)	109.77(15)
C(9)–C(8)–P(1)	122.8(2)	C(8)–P(1)–C(22)	110.46(15)

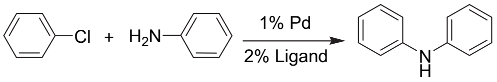
(Table 3, entries 5 and 6). Similar results were obtained when aryl amine was used as a replacement for morpholine (Table 3, entries 7–13).

Besides aryl chloride, the scope of the Pd/(2-phenylindenyl) dicyclohexylphosphine catalytic system can be extended to heteroaryl halide. For example, the amination of 2-chloropyridine with morpholine in DME gave the coupled product in 94% yield, while the reaction of 2-chloropyridine with octyl amime, aniline or *ortho*-toluidine afforded the corresponding products in moderate yield (Table 4, entries 1, 3, 4). Reactions of heteroaryl bromide with amines were faster than reactions of heteroaryl chloride. For instance, morpholine reacted with 2-bromopyridine in 12 h to give the desired product in 97% yield (Table 4, entry 6). Octyl amine, aniline and *ortho*-toluidine formed the coupled products in acceptable yield in 12 h (Table 4, entries 5, 7, 8).

The reaction conditions optimized for the amination of aryl chlorides were also effective for the amination of aryl bromides. Generally, the rates for amination of aryl bromide were faster than those for amination of aryl chlorides. For instance, phenyl bromide was readily aminated with the Pd(dba)$_2$/**2** catalyst system, and the reactions were completed in 5 h (Table 5, entries 1–4). We also found that it was possible to efficiently couple sterically hindered 2-bromomesitylene with a variety of amines (Table 5, entries 5, 7 and 8).

Although more reactive, aryl iodides usually provide lower yields than their bromide counterparts in such reactions [16,31,32], the reason being that the iodide is inhibiting the reaction by binding to a Pd(II) intermediate and forming a Pd ate complex [33]. Only a few groups have reported an efficient procedure for the coupling of aryl iodides with amines using phosphine ligands [15,34,35]. Recently, Buchwald's group reported that reactions were smothered when DME was the solvent [33], while we found that the Pd(dba)$_2$/(2-phenylindenyl)dicyclohexylphosphine catalyst system, in combination with DME as the solvent, allowed phenyl iodide to couple successfully with amines in 5 h (Table 6).

Table 2
Screen of Pd Sources and solvents for the coupling of phenyl chloride and aniline.[a]



Entry	Pd source	Solvent	T (°C)	Time [h]	Yield[b] (%)
1	Pd(dba)$_2$	Toluene	120	12	92
2	Pd(OAc)$_2$	Toluene	120	12	11
3	PdCl$_2$(CH$_3$CN)$_2$	Toluene	120	12	65
4	Pd(dba)$_2$	DME	120	2	98
5	Pd(OAc)$_2$	DME	120	2	21
6	PdCl$_2$(CH$_3$CN)$_2$	DME	120	2	98
7	Pd(dba)$_2$	DME	80	1	51
8	PdCl$_2$(CH$_3$CN)$_2$	DME	80	1	34

[a] ArCl (1 mmol), aniline (1.2 mmol), Pd (1 mol%), ligand (2 mol%), ^{t}BuONa (1.4 mmol), solvent (4 mL).
[b] GC yields.

The reasons for the particular efficiency of **2** as a ligand are not clear to us. However, according to the previously reported biaryl phosphine ligands [36], and the well-documented deprotonation of indene by base [19], we suggest two reasons: (1) the structure of **2** is similar to that of the reported biaryl phosphines; (2) the reaction of **2** with ^{t}BuONa in DME can generate the anionic ligand in situ, and the strong electron-donor ability of the indene anion may increase the electron density at the phosphorus and improve the reaction rate as a result. To provide support for this finding, we undertook a series of ^{31}P NMR studies. When one equivalent of ^{t}BuONa was added to a solution of phosphonium salt in DME, the resonance for the phosphonium salt disappeared, and a new signal appeared (δ −17.17 ppm), corresponding to phosphine. When 4.0 equiv ^{t}BuONa was added to the phosphonium salt in DME it gave a red solution immediately, the resonance for the phosphonium salt disappeared, and another new signal appeared (δ −13.15 ppm), corresponding to anionic phosphine. The results from the calculations were consistent with the experimental results, since the resonance for the anionic phosphine appeared between that of the phosphonium salt **2** and **1** (Scheme 2, Table 7). Work is in progress in our laboratory with the aim of demonstrating the anionic effect of this ligand on the catalytic activity.

3. Conclusion

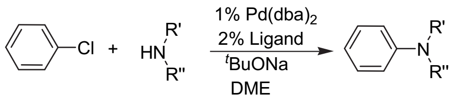
In summary, we synthesized 2-phenylindenyl phosphine ligand. This ligand can be changed into anionic phosphine ligand in situ and utilized in the palladium-catalyzed Buchwald/Hartwig amination reactions in DME, providing good to excellent yields of amination products from aryl chlorides, bromides and iodides. ^{31}P NMR studies show that the resonance for the anionic phosphine appeared between those of the (2-phenylindenyl)-dicyclohexyl phosphonium salt and (2-phenylindenyl)dicyclohexylphosphine. The calculated results were consistent with the experimental results.

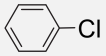
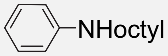
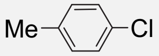
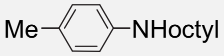
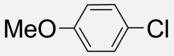
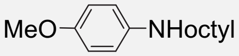
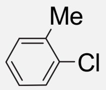
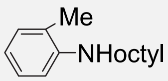
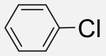
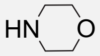
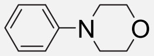
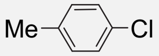
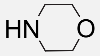
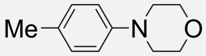
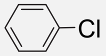
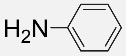
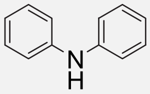
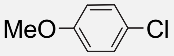
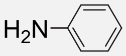
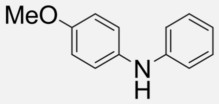
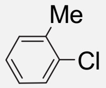
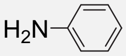
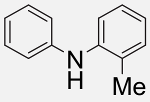
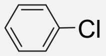
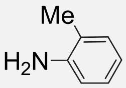
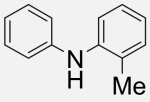
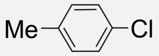
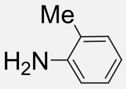
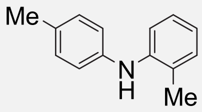
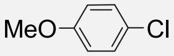
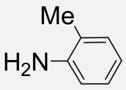
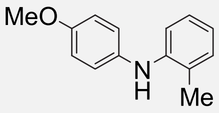
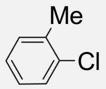
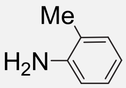
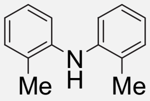
4. Experimental section

4.1. General considerations

Unless otherwise noted, all reagents were purchased from commercial suppliers and used without purification. All Buchwald/Hartwig amination reactions were performed in resealable screw cap Schlenk flask (approx. 20 mL volume) in the presence of Teflon-coated magnetic stirrer bar (3 mm × 10 mm). Toluene and DME were distilled from sodium benzophenone ketyl under nitrogen. Most commercially available amines were used as received. Some amines may require distillation depending on the conditions. ^{t}BuONa were purchased from Fluka. Silica gel (Merk, 70–230 and 230–400 mesh) was used for column chromatography. ^{1}H NMR spectra were recorded on a Mercury-Plus (400 MHz or 600 MHz) spectrometer. Spectra were referenced internally to the residual proton resonance in CDCl$_3$ (δ 7.26 ppm), or with tetramethylsilane (TMS, δ 0.00 ppm) as the internal standard. Chemical shifts (δ) were reported as part per million (ppm) in δ scale downfield from TMS. ^{13}C NMR spectra were referenced to CDCl$_3$ (δ 77.0 ppm, the middle peak). ^{31}P NMR spectra were referenced to 85% H$_3$PO$_4$ externally. Coupling constants (J) were reported in Hertz (Hz). Mass spectra (EI-MS) were recorded on a HP 5989B Mass Spectrometer. The products described in GC yield were accorded to the authentic samples/dodecane calibration standard from Agilent 6890 GC system. All yields reported refer to isolated yield of compounds estimated to be greater than 95% purity as determined by capillary

Table 3
Palladium-catalyzed amination of aryl chloride.[a]



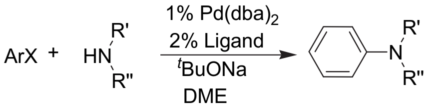
Entry	ArCl	Amine	Product	Time [h]	Yield[b] [%]
1		OctylNH_2		24	63
2		OctylNH_2		24	55
3		OctylNH_2		24	< 5
4		OctylNH_2		24	36
5				24	93
6				24	93
7				24	96
8				15	90
9				24	91
10				24	91
11				24	92
12				15	91
13				24	92

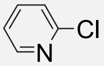
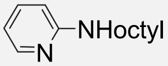
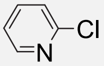
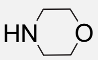
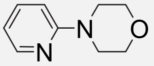
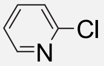
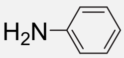
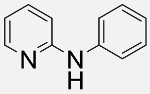
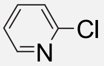
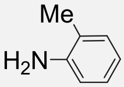
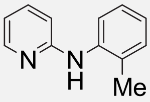
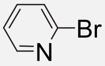
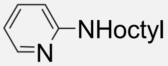
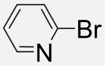
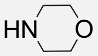
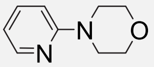
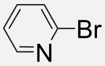
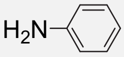
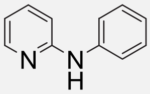
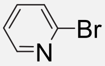
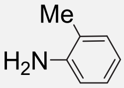
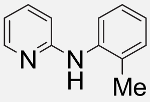
[a] ArCl (1 mmol), amine (1.2 mmol), ^{t}BuONa (1.4 mmol), DME (4.0 mL), at 120 °C under N_2 for the indicated time.

[b] Yield of isolated product.

Table 4

Palladium-catalyzed amination of heteroaryl halide.[a]



Entry	ArX	Amine	Product	Time [h]	Yield[b] (%)
1		OctylNH_2		24	66
2				24	94
3				24	67
4				24	57
5		OctylNH_2		12	66
6				12	97
7				12	55
8				12	64

[a] ArX (1 mmol), amine (1.2 mmol), ^{t}BuONa (1.4 mmol), DME (4.0 mL), at 120 °C under N_2 for the indicated time.

[b] Yield of isolated product.

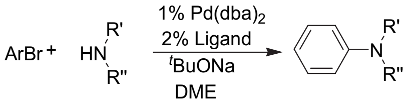
gas chromatography (GC) or ^{1}H NMR. Compounds described in the literature were characterized by comparison of their ^{1}H, and/or ^{13}C NMR spectra to the previously reported data.

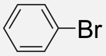
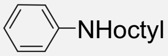
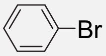
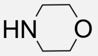
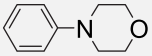
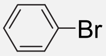
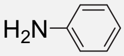
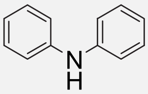
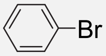
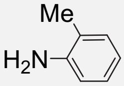
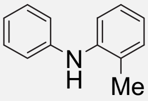
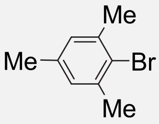
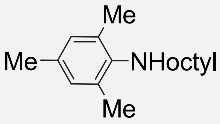
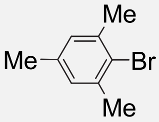
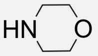
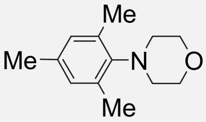
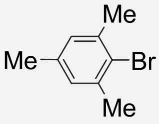
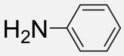
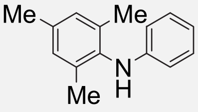
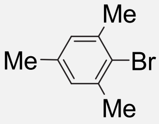
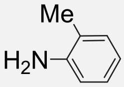
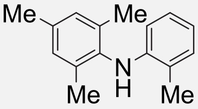
4.2. Preparation of new phosphonium salt **2**

2-Phenylindene was prepared according to the method of Nifant'ev et al [27].

(2-Phenylindenyl)dicyclohexylphosphonium tetra-fluoroborate (**2**): In a 100 mL flask 2-phenylindene (1.5 g, 7.8 mmol) was dissolved in Et_2O (50 mL) under an argon atmosphere. The mixture was cooled to −78 °C, and ^{n}BuLi (3.2 mL, 2.5 M solution in hexane, 8.0 mmol) was added. The solution was stirred for 30 min at −78 °C and then for 3 h at ambient temperature. Then the mixture was cooled to −60 °C and Cy_2PCl (1.8 g, 7.8 mmol) was added. The mixture was warmed to room temperature and stirred for additional 2 h. and the LiCl that formed was removed by filtration over a pad of Celite under Schlenk conditions. The resulting filtrate was treated dropwise with HBF_4 (48 wt% aqueous solution; 13.0 mL, 72 mmol). The organic layer was then separated from the aqueous layer, dried over $MgSO_4$, and filtered. Removal of the solvent provided the white solid, which was dissolved in 10 mL of CH_2Cl_2. After filtration the clear filtrate was dropped into hexane (100 mL, vigorously stirred). The white precipitate that formed was separated via suction filtration. Removal of the volatiles in vacuo afforded **2** as a white solid (1.6 g, 43%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.21–1.87 (m, H), 2.08 (br, 2H), 2.91 (br, 2H), 4.13 (s, 2H), 6.54 (d, $^1J = 484$ Hz, 1H), 7.31–7.66 (m, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.8, 25.6, 25.7, 25.9, 27.5, 28.1, 28.9, 29.3, 121.1, 124.6, 127.3, 128.0, 128.3, 130.0, 130.4, 133.5, 141.4, 141.5, 170.1; ^{31}P NMR (243 MHz, $CDCl_3$): δ 11.49(d, J (P, H) = 484.0 Hz); MS (EI): 388 ($[M-HBF_4]^+$, 100). Anal. Calcd for $C_{27}H_{34}BF_4P$: C, 68.08, H, 7.19; found: C, 67.96, H, 7.39.

Table 5
Palladium-catalyzed amination of aryl bromide.[a]



Entry	ArBr	Amine	Product	Time [h]	Yield[b] (%)
1		OctylNH$_2$		5	65
2				5	85
3				5	99
4				5	99
5		OctylNH$_2$		24	64
6				24	< 5
7				24	78
8				24	95

[a] ArBr (1 mmol), amine (1.2 mmol), ^{t}BuONa (1.4 mmol), Pd(dba)$_2$/ligand (1:2 ratio, mol% as indicated), DME (4.0 mL), at 120 °C under N$_2$ for the indicated time.

[b] Yield of isolated product.

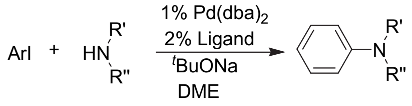
4.3. General procedures for reaction condition screenings

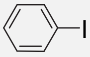
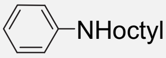
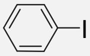
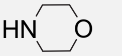
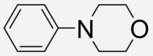
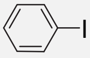
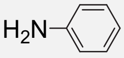
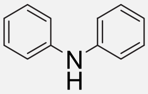
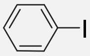
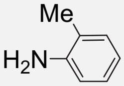
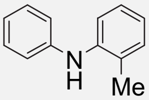
Pd source (0.010 mmol), phosphonium salt **2** (0.020 mmol) and ^{t}BuONa (1.4 mmol) were loaded into a Schlenk tube equipped with a Teflon-coated magnetic stir bar. The tube was evacuated and flushed with nitrogen for three times. Chlorobenzene (1.0 mmol), aniline (1.2 mmol) and solvent (4.0 mL) were loaded into the tube. The tube was then placed into a preheated oil bath (120 °C or 80 °C) and stirred for the time period as indicated in Table 2. After completion of reaction, the reaction tube was allowed to cool to room temperature. Ethyl acetate (10 ml), dodecane (200 μL, internal standard) and water were added. The organic layer was subjected to GC analysis. The GC yield obtained was previously calibrated by authentic sample/dodecane calibration curve purified by column chromatography (hexane/ethyl acetate as eluent) to afford the desired product.

4.4. General procedures for palladium-catalyzed amination of aryl halides

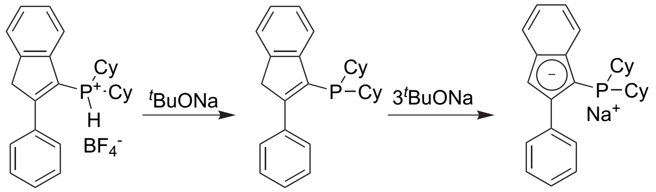
Pd(dba)$_2$/**2**-catalyzed amination of aryl halides: an oven-dried Schlenk tube equipped with a magnetic stirring bar was charged with Pd(dba)$_2$ (1 mol%), ligand **2** (2 mol%) and ^{t}BuONa (1.4 mmol). The flask was capped with a rubber septum, evacuated, and then flushed with argon. This cycle was repeated three times. Amine (1.2 mmol), aryl halide (1.0 mmol) and DME (4 mL) were then successively added by syringe. The tube was stirred at room temperature for several minutes and then placed into a preheated

Table 6
Palladium-catalyzed amination of aryl iodide.[a]



Entry	ArI	Amine	Product	Yield[b] (%)
1		OctylNH$_2$		55
2				88
3				96
4				98

[a] ArI (1 mmol), amine (1.2 mmol), ^{t}BuONa (1.4 mmol), DME (4.0 mL), at 120 °C under N$_2$ for 5 h.
[b] Yield of isolated product.



Scheme 2. Reaction of **2** with ^{t}BuONa.

oil bath (120 °C) for the time period as indicated in tables. After completion of reaction as judged by GC analysis, the reaction tube was allowed to cool to room temperature and adsorbed onto silica gel, and then purified by column chromatography (hexane/ethyl acetate as eluent) to afford the desired product.

4.5. X-ray diffraction studies

Crystals of **2** for X-ray diffraction were obtained by recrystallization of the pure product from dichloromethane/hexane layers. Crystallographic data was collected on a Bruker SMART CCD area-detector diffractometer with graphite-monochromated Mo Kα radiation ($\lambda = 0.71073$ Å). Diffraction measurements were made at room temperature. An absorption correction by SADABS was applied to the intensity data. The structures were solved by Patterson method. The remaining non-hydrogen atoms were determined from the successive difference Fourier syntheses. All non-hydrogen atoms were refined anisotropically except those mentioned otherwise. The hydrogen atoms were generated geometrically and refined with isotropic thermal parameters. The structures were refined on F^2 by full-matrix least-squares methods using the SHELXTL-97 program package [37]. The crystal data and structural refinements details are listed in Table 8. CCDC reference number is 740759.

Table 7
GIAO calculation of absolute nuclear shielding (in ppm) and chemical shifts (δ, ppm) of ^{31}P NMR.

Entry	H$_3$PO$_4$	**1**	**2**	Anionic phosphine
σHF (δ)[a]	346.2 (0)	337.3 (−8.9)	370.2 (24)	364.2 (18)
σDFT (δ)[a]	288.5 (0)	272.0 (−16.5)	292.7 (4.2)	285.8 (−2.7)

[a] δ (mol) = σ(H$_3$PO$_4$) − σ(mol), relative to H$_3$PO$_4$ ($\delta = 0.0$).

Table 8
Crystal data and structure refinements for **2**.

Empirical formula	C$_{27}$H$_{34}$BF$_4$P
Formula weight	476.32
Temperature (K)	298(2)
Crystal system	Monoclinic
Space group	P2(1)
a (Å)	9.7260(5)
b (Å)	19.8110(8)
c (Å)	15.5020(6)
α (°)	90
β (°)	123.837(2)
γ (°)	90
Volume (Å^3)	2481.04(19)
Z, D_{calc} (mg m^{-3})	4, 1.275
Absorption coefficient (mm^{-1})	0.154
F(000)	1008
Crystal size (mm^3)	0.20 × 0.10 × 0.10
θ range (°)	1.89–25.00
Reflections collected	11209
Independent reflections	3989 [$R_{\mathrm{int}} = 0.0847$]
Completeness to θ (%)	91.10
Maximum and minimum transmission	0.9848 and 0.9699
Data/restraints/parameters	3989/51/338
Goodness-of-fit on F^2	0.811
Final R_1 and wR_2 indices [$I > 2\sigma(I)$]	0.0603, 0.0820
R_1 and wR_2 indices (all data)	0.1348, 0.0988

4.6. Calculation

In this work calculation for ^{31}P NMR chemical shifts have been carried out using the Gaussian 03 package. Structures have been fully optimized at the DFT of Becke's 3-parameter hybrid method using the correlation function of Lee, Yang, and Parr (B3LYP) 15 at 6-31+G(d) level. Calculations of absolute shielding constants have been performed using the gauge-including atomic orbitals (GIAO) perturbation method with HF and DFT methods at 6-311+G(2d, p) level. Theoretical ^{31}P chemical shifts were calculated using absolute shielding constants and reference to H$_3$PO$_4$ ($\delta = 0.0$).

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Appendix. Supplementary material

Characterization data for all coupling products, Crystallographic information files (CIF) for **2**. This material is available free of charge via supplementary data. CCDC 740759 also contain crystallographic data for **2**. This material can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jorganchem.2010.04.005

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