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# Air-stable and highly efficient indenyl-derived phosphine ligand: Application to Buchwald–Hartwig amination reactions

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#### 1. Introduction

Aromatic amines and their derivatives are of significant importance in organic chemistry and play an important role as intermediates for agrochemicals, pharmaceuticals, conducting polymers, and dyes in the chemical industry. Palladium-catalyzed C-N bondforming 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 <sup>t</sup>Bu<sub>3</sub>P [9,10], indenyl-phosphines and related phosphines based on CH-acidic cyclopentadiene derivatives [11-13], Beller and co-workers' PAP [14], Buchwald and co-workers' biaryl phosphines [15-18], Hartwig and co-workers' Q-Phos and CyPF-<sup>t</sup>Bu [19,20], Verkade and coworkers' amino phosphine [21,22], and Kwong and co-workers' 2indolylaryl phosphine [23] (Scheme 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

#### ABSTRACT

2-Mesitylindenyl phosphine ligand (1) and [(2-mesitylindenyl)dicyclohexyl-phosphine]PdCl<sub>2</sub> (2) have been synthesized and fully characterized by NMR and elemental analysis, as well as by X-ray crystallography for 2. A Highly active catalyst system derived from a palladium precatalyst and bulky 2mesitylindenyl phosphine ligand (1) for the Buchwald–Hartwig amination reaction of aryl halides with primary and secondary amines has been developed. This method allows for the preparation of a wide variety of amines in moderate to excellent yields and displays a high level of activity for the coupling of aryl chlorides as well as hindered aryl bromides.

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methods is still important for the development of versatile catalysts for numerous applications of coupling reactions. We were interested in exploring indenyl-derived phosphine [24]. In this paper, we describe the development of 2-mesitylindenyl 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-mesitylindenyl)dicyclohexylphosphine (1)

(2-Mesitylindenyl) phosphine ligand can be easily prepared from 2-bromoindene and mesityl bromide (Scheme 2). 2-Mesitylindene was prepared according to the reported procedure [25], then the straightforward deprotonation of 2-mesitylindene by <sup>n</sup>BuLi and trapping of the lithiated intermediate by Cy<sub>2</sub>PCl afforded the corresponding phosphine **1** in high yields. It is interesting that this ligand is air-stable maybe because its structure is similar to that of Buchwald's ligand [26].

2.2. Synthesis and X-ray crystal structure of [(2-mesitylindenyl) dicyclohexyl-phosphine]PdCl<sub>2</sub> (2)

[(2-Mesitylindenyl)dicyclohexylphosphine]PdCl<sub>2</sub> (**2**) was prepared by stirring 2 equiv of **1** with  $PdCl_2(CH_3CN)_2$  in  $CH_2Cl_2$ .



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Scheme 1. Recent developments on effective phosphine ligands.



Scheme 2. Synthesis of 1 and 2.

This complex is also indefinitely stable in air and in solution. The molecular structure of 2 has been confirmed by single-crystal X-ray diffraction (Fig. 1). Selected bond lengths and angles are listed in Table 1. Complex 2 possesses a trans square planar geometry, including a center of inversion about the palladium atom, in which the sterically demanding 2-mesitylindene portion of the ligands are pointed away from the crowded palladium center, the bond distance Pd(1)-P(1) is 2.3514 Å, the C(10)-C(14) bond is in the single bond range (distance between C(10)-C(14) is 1.513 Å) [26], while the bond distance C(10)-C(11) is 1.358 Å, which is in the range of a carbon–carbon double bond [26]. The distance of P(1) to the indene plane is 0.0425 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 2.8287 Å, the dihedral angle between the aryl ring and the indenyl plane is 88.2°, which is similar to that of [2-(dicyclohexvlphosphino)-2',6'-dimethoxyl-1,1'-biphenyl]PdCl<sub>2</sub> [27]. It is important to note that 2 is not an intermediate in the catalytic cycle



Fig. 1. ORTEP drawing of 2. Thermal ellipsoids are drawn at the 30% probability level, hydrogen atoms and solvent molecules are omitted for clarity.

Tai	ble
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Selected bond lengths (Å) and angles (°) for compound 2.

C(11)-P(1)	1.8280(19)	C(10)-C(14)	1.513(3)
C(10)-C(11)	1.358(2)	Pd(1) - P(1)	2.3514(5)
Cl(1)-Pd(1)-Cl(1)#1	180.00(2)	C(20)-C(19)-P(1)	113.85(13)
Cl(1) - Pd(1) - P(1)	90.024(17)	C(11) - P(1) - Pd(1)	114.78(6)
C(10)-C(11)-P(1)	132.25(15)	C(19) - P(1) - Pd(1)	108.44(6)
C(12)-C(11)-P(1)	119.67(13)	C(25) - P(1) - Pd(1)	115.00(6)
C(24)-C(19)-P(1)	110.62(13)		

but a possible precursor to the active catalyst when  $PdCl_2(CH_3CN)_2$  is used as the precatalyst.

### 2.3. Pd complex of (2-mesitylindenyl)dicyclohexylphosphine in the Buchwald–Hartwig amination reaction

With this 2-mesitylindenyl 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 <sup>t</sup>BuONa was used. Of the solvents surveyed (toluene and DME), DME provided the best product yield (Table 2, entries 1–6). Screening of commonly used Pd sources indicated that Pd(dba)<sub>2</sub> and PdCl<sub>2</sub>(CH<sub>3</sub>CN)<sub>2</sub> were suitable sources and Pd(dba)<sub>2</sub> showed the highest catalytic ability for the chlorobenzene coupling reaction (Table 2, entries 4 and 7). Complex **2** catalyzed amination with a moderate efficiency, similar to the conventional Pd complexes (Table 2, entry 6).

The scope of this reaction was then investigated under optimized conditions  $[Pd(dba)_2 (1 \mod \%), ligand (2 \mod \%), ^lBuONa (1.4 equiv), 120 °C]$ , and the results are summarized in Table 3. In most cases, the different aryl chloride reacted with aniline leading to the corresponding products in good to excellent yields. For example, the reaction of 4-chloroacetophenone with aniline gave rise to the corresponding secondary amine in 66% yield (Table 3, entry 2), while the reaction of 4-chloroanisole or 2-chlorobenzonitrile with aniline afforded secondary amines in 51% or 16% yield, respectively (Table 3, entries 3 and 4). The reaction of 1, 2-dichlorobenzene with aniline afforded 48% of the diamine product (Table 3, entry 5), the analog process with 2-chloropyrimidine occurred in moderate yield (Table 3, entry 6). In addition, sterically hindered diphenylamine was converted to the tertiary amine smoothly (Table 3, entry 8).

Besides aryl amine, the scope of the Pd/(2-mesitylindenyl) dicyclohexylphosphine catalytic system can be extended to

#### Table 2

Screen of Pd sources and solvents for the coupling of phenyl chloride and aniline.<sup>a</sup>



Entry	[Pd]	Solvent	<i>T</i> (°C)	Time [h]	Yield (%) <sup>b</sup>
1	Pd(dba) <sub>2</sub>	Toluene	120	24	86
2	$Pd(OAc)_2$	Toluene	120	24	59
3	PdCl <sub>2</sub> (CH <sub>3</sub> CN) <sub>2</sub>	Toluene	120	24	12
4	Pd(dba) <sub>2</sub>	DME	120	24	91
5	$Pd(OAc)_2$	DME	120	24	87
6	2	DME	120	24	84
7	$PdCl_2(CH_3CN)_2$	DME	120	24	90
8	Pd(dba) <sub>2</sub>	DME	120	4	76
9	$Pd(OAc)_2$	DME	120	4	63
10	$PdCl_2(CH_3CN)_2$	DME	120	4	27

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

Palladium-catalyzed amination of aryl chloride and aniline.<sup>a</sup>

Entry	ArCl	Amine	Product	Time [h]	Yield [%] <sup>b</sup>
1	CI	NH <sub>2</sub>	N N	24	89
2	O H <sub>3</sub> C	NH <sub>2</sub>	OCC H H <sub>3</sub> C	24	66
3	H <sub>3</sub> CO	NH <sub>2</sub>	H <sub>3</sub> CO	24	51
4	CI	NH <sub>2</sub>		24	16
5	CI	NH <sub>2</sub>		24	48
6		NH <sub>2</sub>		24	44
7		NH <sub>2</sub>		24	10
8	CI	HNN C		24	37

<sup>a</sup> ArCl (1 mmol), amine (1.2 mmol), <sup>t</sup>BuONa (1.4 mmol), DME (4.0 mL), at 120 °C under N<sub>2</sub> for the indicated time.

<sup>b</sup> Yield of isolated product.

heterocyclic amine. For example, the amination of chlorobenzene with morpholine in DME gave the coupled product in 56% yield (Table 4, entry 1), while the reactions of morpholine with 2-chlorotoluene, 4-chlorobenzotrifluoride or 2-chlorobenzonitrile afforded the corresponding products in 27–95% yields (Table 4, entries 2–4). The reaction of morpholine with 2-chloropyridine in 24 h gave the desired product in 86% yield (Table 4, entry 5).

To further demonstrate the general applicability of our catalyst system, we studied the palladium-catalyzed Buchwald–Hartwig aminations with primary alkylamine (Table 5). Again, we adopted the optimized reaction conditions to the amination of octylamine with various aryl chlorides. Chlorobenzene, 2chlorotoluene, 4-chlorobenzotrifluoride, and 2-chlorobenzonitrile reacted smoothly in the amination with octylamine (Table 5,

Palladium-catalyzed amination of aryl chloride and morpholine.<sup>a</sup>



<sup>a</sup> ArX (1 mmol), amine (1.2 mmol), <sup>t</sup>BuONa (1.4 mmol), DME (4.0 mL), at 120 °C under N<sub>2</sub> for the indicated time.

<sup>b</sup> Yield of isolated product.

entries 1–4). In addition, heterocycles such as 2-chloropyrimidine, and 2-chloropyridine were aminated in moderate yield (Table 5, entries 5 and 6).

The optimized reaction conditions were also effective for the amination of sterically bulky aryl bromides with aniline, both 2-bromomesitylene and 1-bromo-2,4,6-triisopropylbenzene gave the expected products in good to excellent yield (Table 6, entries 1 and 2). We were pleased to find that the amination of octylamine with 2-bromomesitylene provided the corresponding products in moderate yield (Table 6, entry 3). Unfortunately, 1-bromo-2,4,6-triisopropylbenzene do not work under the current reaction conditions, the starting material remained, and the yield is too low to be isolated (Table 6, entry 4). In addition, for reactions of these two aryl bromides with morpholine, no tertiary amines were observed (Table 6, entries 5 and 6).

#### 3. Conclusion

In summary, the new air-stable ligand **1**, which was synthesized in high yield, generate a very active and broadly useful Pd catalyst system for Buchwald—Hartwig amination reactions. Couplings of an electronically diverse array of aryl halides with amines are realized in good to excellent yields. We have also described the facile synthesis, structural characteristics, and catalytic behavior of phosphine palladium complex **2**. Explorations of the potential applications of the new class of substituted indenyl phosphine ligands/catalysts exemplified by **1** and its corresponding complexes of type **2** in synthetically useful cross-coupling reactions are underway.

#### 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  $\times$  10 mm). Toluene and DME were distilled from sodium benzophenone ketyl

Palladium-catalyzed amination of aryl chloride and octylamine.<sup>a</sup>







<sup>b</sup> Yield of isolated product.

under nitrogen. Most commercially available amines were used as received. Some amines may require distillation depending on the conditions. <sup>t</sup>BuONa were purchased from Fluka. Silica gel (Merck, 70-230 and 230-400 mesh) was used for column chromatography. <sup>1</sup>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<sub>3</sub> ( $\delta$  7.26 ppm), or with tetramethylsilane (TMS,  $\delta$  0.00 ppm) as the internal standard. Chemical shifts ( $\delta$ ) were reported as part per million (ppm) in  $\delta$  scale downfield from TMS. <sup>13</sup>C NMR spectra were referenced to  $CDCl_3$  ( $\delta$ 77.0 ppm, the middle peak). <sup>31</sup>P NMR spectra were referenced to 85% H<sub>3</sub>PO<sub>4</sub> externally. Coupling constants (J) were reported in Hertz (Hz). Mass spectra (EI-MS) were recorded on an 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 gas chromatography (GC) or <sup>1</sup>H NMR. Compounds described in the literature were characterized by comparison of their <sup>1</sup>H. and/or <sup>13</sup>C NMR spectra to the previously reported data. 2-Mesitylindene was prepared according to the literature procedure [25].

#### 4.2. Preparation of (2-mesitylindenyl)dicyclohexylphosphine (1)

In a 100 mL flask 2-mesitylindene (1.83 g, 7.8 mmol) was dissolved in Et<sub>2</sub>O (50 mL) under an argon atmosphere. The mixture was cooled to -78 °C, and <sup>n</sup>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 12 h at ambient temperature. Then the mixture was cooled to -60 °C and Cy<sub>2</sub>PCl (1.8 g, 7.8 mmol) was added. The

mixture was warmed to room temperature and stirred for additional 12 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 water. The organic layer was then separated from the aqueous layer, dried over MgSO<sub>4</sub>, and filtered. Removal of the solvent provided the white solid, which was dissolved in 10 mL of CH<sub>2</sub>Cl<sub>2</sub>. 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 (2.76 g, 82%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.69 (d, J = 8.4 Hz, 1H), 7.48 (d, J = 8.4 Hz, 1H), 7.36–7.32 (m, 1H), 7.26-7.22 (m, 1H), 6.90 (s, 2H), 3.56 (s, 2H), 2.27 (s, 3H), 2.14 (s, 6H), 1.84–1.14 (m, 22H);  $^{13}$ C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  161.3, 147.2, 143.2, 136.7, 136.3, 135.2, 134.5, 128.0, 126.2, 124.1, 123.5, 122.2, 44.1, 34.6, 32.4, 30.9, 27.3, 27.2, 26.2, 21.1, 20.9; <sup>31</sup>P{<sup>1</sup>H} NMR (243 MHz, CDCl<sub>3</sub>):  $\delta$  –15.17; MS (EI): 430.11(M<sup>+</sup>); Anal. Calcd for C<sub>30</sub>H<sub>39</sub>P: C, 83.68, H, 9.13; Found: C, 83.60, H, 9.24.

### 4.3. Preparation of [(2-mesitylindenyl)dicyclohexyl-phosphine] PdCl<sub>2</sub> (**2**)

To a stirred solution of PdCl<sub>2</sub>(CH<sub>3</sub>CN)<sub>2</sub> in dry CH<sub>2</sub>Cl<sub>2</sub> was added **1** (100 mg, 0.23 mmol) at room temperature. The reaction mixture was stirred for 1 h, and then the volatiles were removed, leaving **2** as a bright yellow solid (113 mg, 95%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.32 (b, 4H), 7.19 (b, 4H), 6.81 (b, 4H), 3.53 (s, 4H), 2.22–1.07 (m, 62H); <sup>13</sup>C NMR (100 MHz, CD<sub>2</sub>Cl<sub>3</sub>)  $\delta$  156.2, 147.0, 141.6, 136.9, 134.8, 134.0, 127.5, 125.4, 124.9, 124.2, 122.3, 46.5, 29.5, 27.0, 26.0, 22.6, 21.8, 20.4; <sup>31</sup>P{<sup>1</sup>H} NMR (243 MHz, CDCl<sub>3</sub>):  $\delta$  20.34; Anal. Calcd for C<sub>60</sub>H<sub>78</sub>Cl<sub>2</sub>P<sub>2</sub>Pd: C, 69.39, H, 7.57; Found: C, 69.52, H, 7.60.

Palladium-catalyzed amination of aryl bromide.<sup>a</sup>





<sup>a</sup> ArBr (1 mmol), amine (1.2 mmol), <sup>t</sup>BuONa (1.4 mmol), DME (4.0 mL), at 120 °C under N<sub>2</sub>.

<sup>b</sup> Yield of isolated product.

#### 4.4. General procedures for reaction condition screenings

Pd source (0.010 mmol), phosphine ligand (1) (8.6 mg, 0.020 mmol) and <sup>t</sup>BuONa (134 mg, 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 (112 mg, 1.0 mmol), aniline (111 mg, 1.2 mmol) and solvent (4.0 mL) were loaded into the tube. The tube was then placed into a preheated oil bath 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 (170 mg, 1.0 mmol, 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.

## 4.5. General procedures for palladium-catalyzed amination of aryl halides

Pd(dba)<sub>2</sub>/**1**-catalyzed amination of aryl halides: An oven-dried Schlenk tube equipped with a magnetic stirring bar was charged

Table 7	
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Crystal data and structure refinements for 2.

Empirical formula	$C_{60}H_{78}CI_2P_2Pd$
Formula weight	1038.46
Temperature (K)	298(2)
Crystal system	Monoclinic
Space group	P2(1)/n
a (Å)	12.6537(9)
b (Å)	16.6557(12)
c (Å)	12.9308(9)
α (°)	90
β (°)	97.1040(10)
γ (°)	90
Volume (Å <sup>3</sup> )	2704.3(3)
Z, $D_{\text{calcd}}$ (mg m <sup>-3</sup> )	4, 1.275
Absorption coefficient (mm <sup>-1</sup> )	0.538
F(000)	1096
Crystal size (mm <sup>3</sup> )	$0.16 \times 0.12 \times 0.10$
$\theta$ range (°)	2.00-28.34
Reflections collected	33025
Independent reflections	6689 [ $R_{int} = 0.0482$ ]
Completeness to $\theta$ (%)	99.1
Maximum and minimum transmission	0.9482 and 0.9189
Data/restraints/parameters	6689/0/298
Goodness-of-fit on F <sup>2</sup>	0.947
Final $R_1$ and $wR_2$ indices $[I > 2\sigma(I)]$	0.0333, 0.0829
$R_1$ and $wR_2$ indices (all data)	0.0445, 0.0860

with Pd(dba)<sub>2</sub> (5.8 mg, 0.010 mmol), ligand **1** (8.6 mg, 0.020 mmol) and <sup>t</sup>BuONa (134 mg, 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 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 the reaction mixture was adsorbed onto silica gel, and then purified by column chromatography (hexane/ethyl acetate as eluent) to afford the desired product.

#### 4.6. 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 areadetector diffractometer with graphite-monochromated Mo K $\alpha$  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

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#### Appendix. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jorganchem.2012.02.007.

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