

## Synthesis of Substituted Biarylmethanol *via* Ferrocenyloxime Palladacycles Catalyzed Suzuki Reaction of Chlorophenylmethanol in Water

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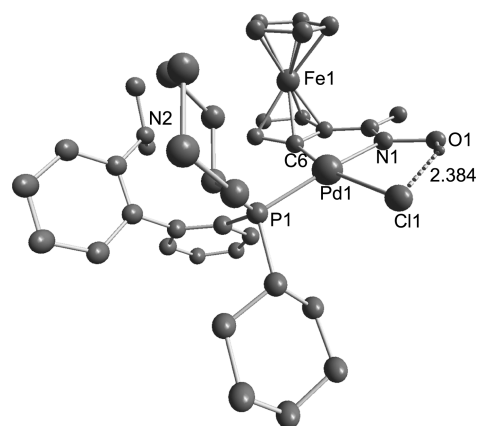
**Key Words :** Biarylmethanol, Palladacycle, Suzuki reaction, Aryl chloride, Water

Alcohols are of great importance as cheap and readily available organic materials for the preparation of many pharmaceutical products and fine chemicals.<sup>1,2</sup> In addition, alcohols as electrophiles instead of alkyl halides in C-C (N) bonds forming reactions are particularly attractive because the process produces only water as byproduct.<sup>3</sup> In spite of the existence of many synthetic routes, there are limited reports involving biarylmethanols.<sup>4</sup> The palladium-catalyzed Suzuki reaction is an extremely efficient method for the construction of biaryl compounds.<sup>5</sup> Among various Pd catalysts, palladacycles are one of the most active catalysts for the coupling reactions.<sup>6</sup> From the economical and environmental points of view, employing readily accessible aryl chlorides and the use of water as a solvent in this reaction offer many advantages.<sup>7</sup> However, there have been a few reports which demonstrated that palladacycles catalyzed Suzuki reaction of limited aryl chlorides in water.<sup>8</sup> To our knowledge, only two accounts has been reported concerning the Suzuki of 4-chlorophenylmethanol in organic solvent.<sup>9</sup>

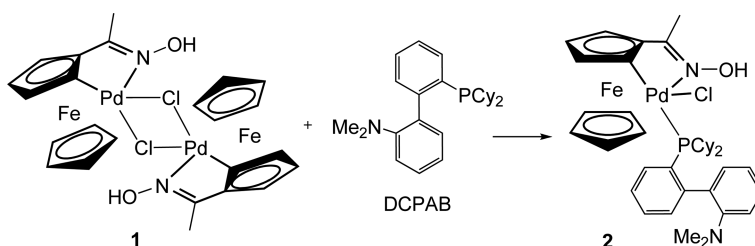
Recently, we have developed a cationic cyclopalladated ferrocenylpyrimidine catalyzed Suzuki reaction of aryl halides and 4-(hydroxymethyl)phenylboronic acid for the synthesis of substituted biarylmethanols in water.<sup>10</sup> However, substrates are limited to the aryl bromides and activated aryl chlorides. To date, there is no report that investigates the Suzuki reaction of chlorophenylmethanol in water. As a continuation of our interest in the synthesis of biarylmethanols, we have prepared a new phosphine adduct of palladacycle **2** from the reaction of cyclopalladated ferrocenyloxime dimer **1** with commercially available 2-dicyclohexylphosphanyl-2'-(*N,N*-dimethylamino) biphenyl (DCPAB) (Scheme 1) and developed a practical method for the synthesis of biarylmethanols by

the Suzuki reaction of chlorophenylmethanol and aryl-boronic acids in water.

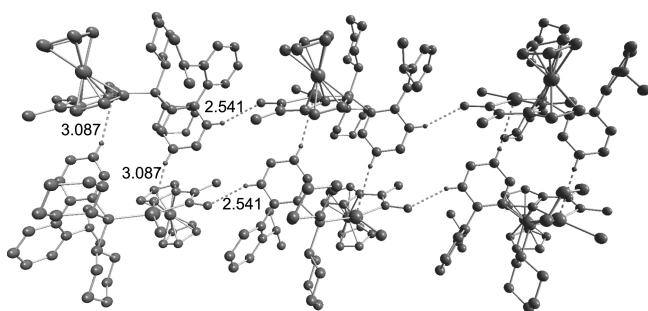
Complex **2** is air- and moisture-stable, both in solid state and in solution. It was fully characterized by elemental analysis, IR, NMR and ESI-MS. In comparison with the corresponding frequencies of the free ferrocenyloxime,<sup>11</sup> the C=N absorptions of **2** has been shifted to low values (1599 cm<sup>-1</sup>), indicating that the N atom is coordinated to palladium. The <sup>1</sup>H NMR spectrum of **2** exhibits more signals than expected, and the <sup>31</sup>P NMR appears two singlets at  $\delta$  65.54 and 67.15 ppm, suggesting the existence of isomers in solution. A similar phenomenon was also found in the <sup>1</sup>H NMR



**Figure 1.** Molecular structure of complex **2**. Non-hydrogen bonding H atoms are omitted for clarity. Selected bond lengths (Å) and angles (°): Pd(1)–C(6) 1.991(4), Pd(1)–N(1) 2.114(3), Pd(1)–P(1) 2.2599(10), Pd(1)–Cl(1) 2.4067(11) and C(6)–Pd(1)–N(1) 79.10(14), C(6)–Pd(1)–P(1) 95.00(11), P(1)–Pd(1)–Cl(1) 97.00(4), N(1)–Pd(1)–Cl(1) 89.52(10).



**Scheme 1.** Synthesis of **2**.



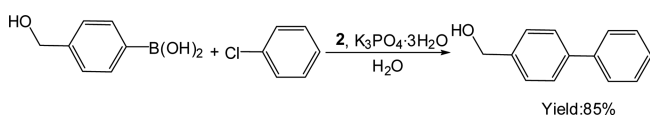
**Figure 2.** Two-dimensional network structure of complex **2** formed by C–H···Pd and C–H···Cl hydrogen bonds. Non-hydrogen bonding H atoms are omitted for clarity.

spectra of related cyclopalladated complexes.<sup>12</sup> Additionally, its detailed structure has been determined by single-crystal X-ray diffraction.

The molecule of **2** together with selected bond distances and angles is shown in Figure 1. It adopts a *trans* configuration of the coordinated DCPAB to the imino nitrogen. The bicyclic system formed by the palladacycle and the C<sub>5</sub>H<sub>3</sub> moiety is approximately coplanar (dihedral angle of 6.4°). The Pd–P (2.2599(10) Å) bond length of **2** is longer than that of the corresponding PPh<sub>3</sub>-palladacycle (2.249(3) Å) possibly due to the steric bulk of the DCPAB ligand,<sup>10</sup> it is similar to those of the related DCPAB-palladacycles (2.2592(12)–2.2679(15) Å).<sup>12c,13</sup> In the crystal of **2** there exist three types of hydrogen bonds.<sup>14</sup> One intramolecular hydrogen bond is between the chlorine atom and the O–H group of oxime (Cl···H = 2.384 Å). The other intermolecular hydrogen bond is O···H–C (O···H = 2.541 Å). It is worthy of notice that there also exist C–H···Pd hydrogen bond<sup>12c,15</sup> (Pd–H = 3.087 Å), which link the molecules into a 2D network structure (Figure 2).

Based on our previous experiments,<sup>9</sup> we performed the coupling of 4-hydroxymethylphenylboronic acid with chlorobenzene to evaluate the effectiveness of the new palladacycle in the Suzuki coupling of aryl chlorides in water under same conditions (Scheme 2). Using 0.5 mol % of catalyst in the presence of 3 equivalents of K<sub>3</sub>PO<sub>4</sub>·3H<sub>2</sub>O as base at 100 °C provided coupled product in a good yield (85%). To develop efficient methodologies for the synthesis of substituted biarylmethanol, we explored the Suzuki coupling of chlorobenzene containing hydroxymethyl with varying arylboronic acids in water.

Initially, the coupling of 4-chlorophenylmethanol with phenyl boronic acid was selected as the model reaction to probe the optimal reaction conditions using 0.5 mol % of **2** at 100 °C for 24 h, and the results are shown in Table 1. Among the eight different bases tested, Cs<sub>2</sub>CO<sub>3</sub> was found to



**Scheme 2.** Suzuki coupling of 4-hydroxymethylphenylboronic acid with chlorobenzene catalyzed by **2**.

**Table 1.** Influence of base and catalyst on the Suzuki coupling of 4-chlorophenylmethanol with phenyl boronic acid<sup>a</sup>

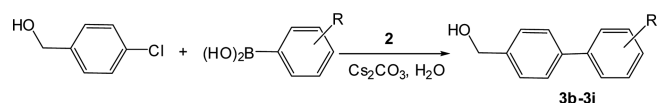
Entry	Catalyst (mol %)	Base	Yield (%) <sup>b</sup>
1	<b>2</b> (0.5)	K <sub>3</sub> PO <sub>4</sub> ·3H <sub>2</sub> O	82
2	<b>2</b> (0.5)	Na <sub>2</sub> CO <sub>3</sub>	65
3	<b>2</b> (0.5)	K <sub>2</sub> CO <sub>3</sub>	87
4	<b>2</b> (0.5)	Cs <sub>2</sub> CO <sub>3</sub>	92
5	<b>2</b> (0.5)	NaOH	51
6	<b>2</b> (0.5)	KOH	64
7	<b>2</b> (0.5)	KOAc	43
8	<b>1</b> (0.25)	Cs <sub>2</sub> CO <sub>3</sub>	trace
9	Pd(OAc) <sub>2</sub> /DCPAB (0.5/1)	Cs <sub>2</sub> CO <sub>3</sub>	trace
10	Pd(PPh <sub>3</sub> ) <sub>4</sub> /DCPAB (0.5/1)	Cs <sub>2</sub> CO <sub>3</sub>	trace
11	<b>1</b> /NBu <sub>4</sub> Br (0.5/100)	Cs <sub>2</sub> CO <sub>3</sub>	12
12	<b>1</b> /DCPAB (0.5/1)	Cs <sub>2</sub> CO <sub>3</sub>	56

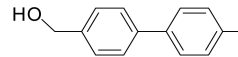
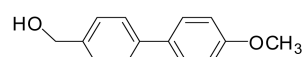
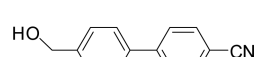
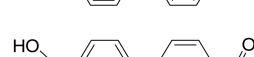
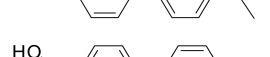
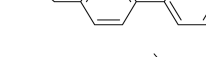
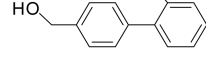
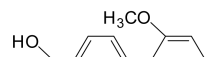
<sup>a</sup>Reaction conditions: 4-chlorophenylmethanol (1.0 mmol), PhB(OH)<sub>2</sub> (1.5 mmol), base (2.0 mmol), water (5 mL), 100 °C, 24 h. <sup>b</sup>Isolated yields.

give the best result (92%, entry 4), K<sub>3</sub>PO<sub>4</sub>·3H<sub>2</sub>O and K<sub>2</sub>CO<sub>3</sub> also afforded good yields (82% and 87%, entries 1 and 3). The dimer **1**, Pd(OAc)<sub>2</sub> and Pd(PPh<sub>3</sub>)<sub>4</sub> were almost inactive under the same reaction conditions (entries 8–10). Furthermore, the addition of tetrabutylammonium bromide (TBAB) as a phase transfer agent,<sup>16</sup> did not give a good result (entry 11). A moderate yield (56%, entry 12) was obtained by the addition of DCPAB suggesting that DCPAB participated in the catalytic cycles.<sup>12c,13</sup>

With the optimized conditions in hand, the coupling reactions of 4-chlorophenylmethanol with a variety of electronically and structurally diverse aryl boronic acids were investigated (Table 2). Similarly, the more reactive 4-methylphenylboronic acid and 4-methoxyphenylboronic acid gave the products **3b** and **3c** in excellent yields (entries 1–2). The aryl boronic acids containing electron-withdrawing groups reacted with 4-chlorophenylmethanol to give **3d–3f** respectively in good yields (84–88%, entries 3–5). The coupling of hindered aryl boronic acids proceeded efficiently to form the expected products **3g–3i** in moderate yields (67–75%, entries 6–8), which shows that steric factors have an influence on these reactions.

In contrast to Suzuki coupling of 4-halophenylmethanol, only two accounts has been reported concerning the Suzuki of 2-bromophenylmethanol in organic solvent.<sup>17</sup> In the following experiments, the Suzuki coupling of 2-chlorophenylmethanol with aryl boronic acids was investigated (Table 3). Under the same condition, complex **2** showed high catalytic activity, the 2-arylphenylmethanols were isolated in moderate to good yields (75–90%, entries 1–5). In cases of *ortho*-aryl boronic acids, using 1 mol % of **2** afforded the products in good yields (entries 6–7). Electron-withdrawing substrates reacted to give the corresponding products **4h–4j** in moderate yields (73–77%, entries 8–10).

**Table 2.** Suzuki coupling of 4-chlorophenylmethanol with aryl boronic acids catalyzed by **2**<sup>a</sup>


Entry	R	Product	Yield (%) <sup>b</sup>
1	4-CH <sub>3</sub>	 <b>3b</b>	91
2	4-OCH <sub>3</sub>	 <b>3c</b>	93
3	4-CN	 <b>3d</b>	88
4	4-COCH <sub>3</sub>	 <b>3e</b>	85
5	4-NO <sub>2</sub>	 <b>3f</b>	84
6	2-CH <sub>3</sub>	 <b>3g</b>	72
7	2-OCH <sub>3</sub>	 <b>3h</b>	75
8	2-COCH <sub>3</sub>	 <b>3i</b>	67

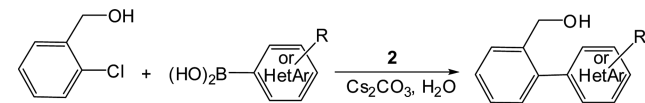
<sup>a</sup>Reaction conditions: 4-chlorophenylmethanol (1.0 mmol), aryl boronic acids (1.5 mmol), Cs<sub>2</sub>CO<sub>3</sub> (2.0 mmol), water (5 mL), 100 °C, 24 h.  
<sup>b</sup>Isolated yields.

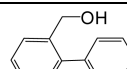
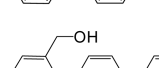
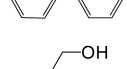
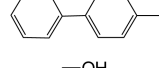
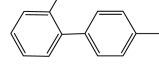
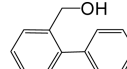
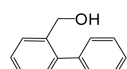
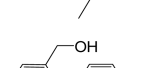
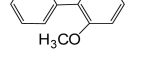
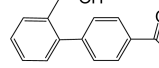
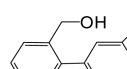
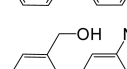
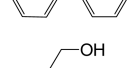
This protocol was found also to proceed successfully with heteroarylboronic acids, furnishing good yields (80-89%, entries 11-13).

### Experimental

**Materials and Measurement.** The chloride-bridged palladacyclic dimer **1** was prepared according to published procedures.<sup>18</sup> All other chemicals were used as purchased. Elemental analyses were determined with a Thermo Flash EA 1112 elemental analyzer. IR spectra were collected on a Bruker VECTOR22 spectrophotometer using KBr pellets. NMR spectra were recorded on a Bruker DPX-400 spectrometer in CDCl<sub>3</sub> with TMS as an internal standard. Mass spectra were measured on a LC-MSD-Trap-XCT instrument. Crystallographic data were collected on a Bruker SMART APEX-II CCD diffractometer. CCDC reference number 625798 for **2**. The data can be obtained free of charge from The Cambridge Crystallographic Data Centre via [www.ccdc.cam.ac.uk/data\\_request/cif](http://www.ccdc.cam.ac.uk/data_request/cif).

**[PdCl<sub>2</sub>[(η<sup>5</sup>-C<sub>5</sub>H<sub>5</sub>)]Fe[(η<sup>5</sup>-C<sub>5</sub>H<sub>5</sub>)-C(CH<sub>3</sub>)=NOH](DCPAB)] (**2**):** A solution of **1** (0.1 mmol) and DCPAB (0.20 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (10 mL) was stirred at room temperature for 30 min. The product was separated by passing through a short

**Table 3.** Suzuki coupling of 2-chlorophenylmethanol with aryl boronic acids catalyzed by **2**<sup>a</sup>


Entry	R (or HetAr)	Product	Yield (%) <sup>b</sup>
1	CH <sub>3</sub>	 <b>4a</b>	86
2	4-C <sub>6</sub> H <sub>5</sub>	 <b>4b</b>	75
3	4-CH <sub>3</sub>	 <b>4c</b>	88
4	4-C <sub>2</sub> H <sub>5</sub>	 <b>4d</b>	87
5	4-OCH <sub>3</sub>	 <b>4e</b>	90
6 <sup>c</sup>	2-CH <sub>3</sub>	 <b>4f</b>	81
7 <sup>c</sup>	2-OCH <sub>3</sub>	 <b>4g</b>	83
8	4-COCH <sub>3</sub>	 <b>4h</b>	73
9	3-COCH <sub>3</sub>	 <b>4i</b>	77
10	3-NO <sub>2</sub>	 <b>4j</b>	74
11	pyridin-4-yl	 <b>4k</b>	89
12	pyridin-3-yl	 <b>4l</b>	85
13	furan-2-yl	 <b>4m</b>	80

<sup>a</sup>Reaction conditions: 2-chlorophenylmethanol (1.0 mmol), aryl boronic acids (1.5 mmol), Cs<sub>2</sub>CO<sub>3</sub> (2.0 mmol), water (5 mL), 100 °C, 24 h.  
<sup>b</sup>Isolated yields. <sup>c</sup>Catalyst **2** (1 mol %).

silicagel column with CH<sub>2</sub>Cl<sub>2</sub>/ethyl acetate (1:1, v/v) as eluent. The second band was collected and afforded complex **2**. Red solid, 89% yield. Found: C, 58.92; H, 6.10; N 3.92. Calc. for C<sub>38</sub>H<sub>48</sub>ClFeN<sub>2</sub>OPPd: C, 58.70; H, 6.22; N, 3.73%. IR (KBr, cm<sup>-1</sup>): 2925, 2851, 1599 (C=N), 1447, 1315, 1264, 1106, 1052, 1002, 808, 752. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ

9.89 (1H, brs, OH), 7.98 (1H, m, ArH), 7.21-7.37 (5H, m, ArH), 7.04-7.09 (2H, m, ArH), 4.24 (1H, s, C<sub>5</sub>H<sub>3</sub>), 4.07-4.14 (6H, m, C<sub>5</sub>H<sub>5</sub>+C<sub>5</sub>H<sub>3</sub>), 3.92 (1H, s, C<sub>5</sub>H<sub>3</sub>), 2.56 (3H, s, CH<sub>3</sub>), 2.51 (3H, s, CH<sub>3</sub>), 2.17 (3H, s, CH<sub>3</sub>), 2.35 (2H, m, PCy<sub>2</sub>), 1.76-1.06 (20H, m, Cy). <sup>31</sup>P{<sup>1</sup>H}NMR (162 MHz, CDCl<sub>3</sub>) δ 67.15, 65.54. MS-ESI<sup>+</sup> [*m/z*]: 741.2 (M<sup>+</sup>-Cl).

**General Procedure for the Coupling Reaction of Chlorophenylmethanol.** In a Schlenk tube, a mixture of the prescribed amount of catalyst, aryl halides (1.0 mmol), aryl boronic acids (1.5 mmol) and the selected base (2.0 mmol) in water was evacuated and charged with nitrogen. The reaction mixture was heated at 100 °C for 24 h. After being cooled, the mixture was extracted with CH<sub>2</sub>Cl<sub>2</sub> and evaporated, the resulting residue was purified by flash chromatography on silica gel using mixture of CH<sub>2</sub>Cl<sub>2</sub>/ethyl acetate (5/1) as eluent. The products **4b**, **4d** and **4j** were determined by <sup>1</sup>H and <sup>13</sup>C NMR. Other products were characterized by comparison of data with those in the literature.<sup>17,19</sup>

**4'-Phenyl-biphenyl-2-methanol (4b):** Yield 75%. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.22-8.29 (m, 3H, ArH), 7.76 (m, 2H, ArH), 7.40-7.49 (m, 3H, ArH), 7.40-7.49 (m, 4H, ArH), 7.29 (d, 1H, ArH), 4.60 (s, 2H, CH<sub>2</sub>), 1.68 (s, 1H, OH). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 141.0, 140.7, 140.1, 139.6, 138.1, 130.1, 129.7, 129.1, 128.9, 128.6, 128.4, 127.8, 127.5, 127.1, 63.3. MS-ESI<sup>+</sup> [*m/z*]: 260.1 (M<sup>+</sup>). Anal. Calc. for C<sub>19</sub>H<sub>16</sub>O: C, 87.66; H, 6.19. Found: C, 87.85; H, 6.07.

**4'-Ethyl-biphenyl-2-methanol (4d):** Yield 87%. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.52-7.55 (m, 1H, ArH), 7.23-7.39 (m, 7H, ArH), 4.61 (s, 2H, CH<sub>2</sub>), 2.70 (q, 2H, CH<sub>2</sub>), 1.71 (s, 1H, OH), 1.28 (t, 3H, CH<sub>3</sub>). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 143.3, 141.3, 138.1, 137.9, 130.2, 129.1, 128.9, 128.4, 127.8, 127.6, 62.9, 29.7, 15.6. MS-ESI<sup>+</sup> [*m/z*]: 212.1 (M<sup>+</sup>). Anal. Calc. for C<sub>15</sub>H<sub>16</sub>O: C, 84.87; H, 7.60. Found: C, 84.99; H, 7.46.

**3'-Nitro-biphenyl-2-methanol (4j):** Yield 74%. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.63-7.66 (m, 2H, ArH), 7.55-7.59 (m, 1H, ArH), 7.32-7.48 (m, 5H, ArH), 4.66 (s, 2H, CH<sub>2</sub>), 1.72 (s, 1H, OH). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 148.1, 142.3, 139.1, 137.8, 135.4, 130.0, 129.2, 129.1, 128.8, 128.2, 124.1, 122.3, 63.0. MS-ESI<sup>+</sup> [*m/z*]: 229.1 (M<sup>+</sup>). Anal. Calc. for C<sub>13</sub>H<sub>11</sub>NO<sub>3</sub>: C, 68.11; H, 4.84; N, 6.11. Found: C, 68.34; H, 4.72; N, 6.27.

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