DOI: 10.1002/ejoc.200500843

Palladium-Catalyzed Suzuki–Miyaura Cross-Coupling Using Phosphinous Acids and Dialkyl(chloro)phosphane Ligands

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Keywords: Cross-coupling / Palladium / Homogeneous catalysis / Biaryls

The use of eleven palladium complexes having monomeric and μ-chloro-bridged dimeric structures and either bulky dialkyl- and diarylphosphinous acid ligands (POPd, POPd-Br, POPd1, POPd2, POPd6, POPd7, Ph1-Phoxide) or dialkyl(chloro)phosphane ligands (PXPd, PXPd2, PXPd6, PXPd7) for Suzuki–Miyaura coupling reactions has been evaluated. Screening and optimization of catalyst loading, solvent, temperature, and base showed that excellent results can be obtained with electron-deficient and electron-rich aryl iodides, bromides, and chlorides in the presence of 2.5 mol-% of palladium–phosphinous acid POPd, $(t\mathrm{Bu_2POH})_2\mathrm{PdCl_2}$, in 1,4-dioxane using cesium carbonate as base.

Introduction

Transition-metal-catalyzed cross-coupling reactions of aryl halides and boronic acids, organostannanes, organosiloxanes, organozinc, and Grignard reagents have become laboratory routine during recent years due to the development of readily available palladium complexes which exhibit high catalytic activity.[1] Especially, electron-rich and bulky phosphane ligands such as 2-(biphenyl)(dicyclohexyl)-phosphanes[2] and tri-*tert*-butylphosphane and derivatives thereof,[3] or sterically demanding *N*-heterocyclic carbene ligands[4] have been shown to afford palladium(0) species that easily undergo oxidative addition with aryl halides including non-activated chlorides thus providing excellent results in the coupling reactions mentioned above. The efficacy of palladium–phosphinous acid complexes POPd, POPd1, and POPd2 in cross-couplings including Stille, Hiyama, Sonogashira, and Heck reactions using organic solvents and water, respectively, has recently been reported by us and others.[5] General features of palladium–phosphinous acid catalysts include simple preparation from readily available phosphane oxides, RR′P(O)H, and $Pd_2(dba)_3$, $Pd(cod)Cl_2$ or $Pd(OAc)_2$, stability to air and water facilitating operation and catalyst recycling, and high catalytic activity.

Palladium- and nickel-catalyzed Suzuki–Miyaura couplings of organoboron compounds and various electrophiles such as aryl halides and triflates are among the most efficient carbon–carbon bond-forming reactions.[6] Its widespread popularity stems from distinctive advantages over other coupling methods. Typical features of Suzuki–Miyaura couplings are mild reaction conditions, facile handling and low toxicity of the reagents, commercial availability of numerous boronic acid substrates, and excellent functional group tolerance. The preparation of complex molecules exhibiting biaryl units is often accomplished through Suzuki–Miyaura coupling, which has been applied in the synthesis of many natural products and pharmacologically active compounds, such as losartan,[7] (–)-steganone,[8] dragmacidin F,[9] eupomatilone-6,[10] valsartan,[11] pulvinic acids,[12] and isoneocryptolepine.[13]

In continuation of our previous efforts to utilize palladium–phosphinous acids POPd, POPd1, and POPd2 in C–C, C–N, and C–S bond formation, we have employed 11 palladium complexes bearing either bulky phosphinous acid groups (POPd, POPd-Br, POPd1, POPd2, POPd6, POPd7, Ph1-Phoxide) or dialkyl(chloro)phosphane ligands (PXPd, PXPd2, PXPd6, PXPd7) in Suzuki–Miyaura coupling of aryl iodides, bromides, or chlorides and organoboronic acids (Figure 1 and Figure 2). The catalysts studied comprise monomeric and μ-chloro-bridged dimeric structures exhibiting phosphane ligands with *tert*-butyl, cyclohexyl, and phenyl substituents. Similar to all mononuclear complexes, the dinuclear palladium–phosphinous acids POPd1, POPd7, and Ph1-Phoxide bear two phosphane ligands at each palladium center, whereas POPd2, POPd6 and their dialkyl(chloro)phosphane analogs PXPd2 and PXPd6 have a phosphane ligand/Pd ratio of 1:1.

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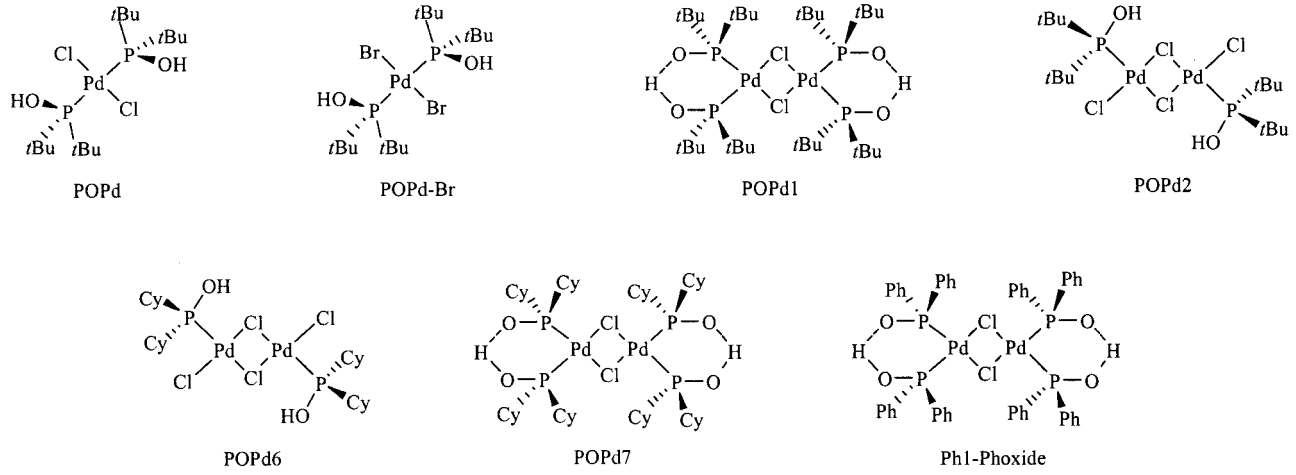


Figure 1. Structures of palladium–phosphinous acids.

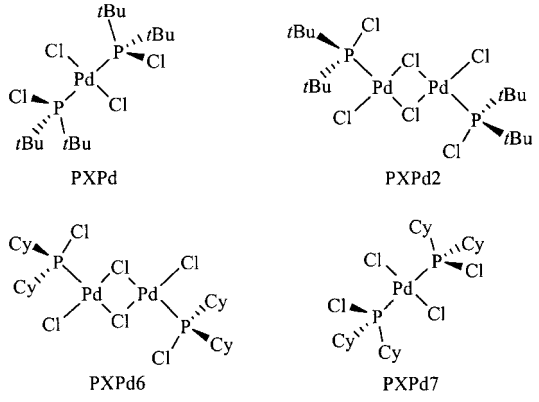
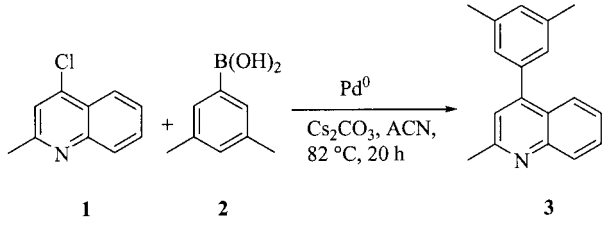


Figure 2. Structures of dialkyl(chloro)phosphane-derived palladium catalysts.

Results and Discussion

Based on our previous experience with palladium–phosphinous acid catalyzed cross-couplings, we decided to evaluate the ability of POPd, POPd-Br, POPd1, POPd2, POPd6, POPd7, Ph1-Phoxide and their chlorophosphane-derived analogs PXPd, PXPd2, PXPd6, and PXPd7 to promote the coupling reaction of 4-chloro-2-methylquinoline (**1**) and 3,5-dimethylphenylboronic acid (**2**) in acetonitrile in the presence of an inorganic base (Scheme 1). For better comparison, all reactions were conducted under identical conditions employing 10 mol-% of the catalysts and 1.5 equiv. of cesium carbonate in acetonitrile at 82 °C for 20 h. Our initial screening showed that all Pd complexes catalyze the formation of 4-(3′,5′-dimethylphenyl)-2-methylquinoline (**3**), albeit in varying yields, Table 1.

The dimeric complexes POPd2, POPd6, PXPd2, and PXPd6 which exhibit a palladium/phosphane ligand ratio of 1:1 proved to give only moderate yields, i.e. 42–55% (Table 1, Entries 3, 4, 7, and 8). Representing mono- and dinuclear structures having either two dialkylphosphinous acid or two dialkyl(chloro)phosphane ligands attached to one palladium atom, POPd, POPd7, and PXPd showed the highest catalytic activity providing **3** in 70 to 73% (Entries 1, 5, and 6). The comparison of the yields of **3** obtained with dinuclear catalysts exhibiting two phosphinous acid ligands attached to each palladium center (POPd1, POPd7, and Ph1-Phoxide) reveals that the incorporation of cyclohexyl groups into the phosphane ligands affords superior results (Entries 2, 5, and 11). By contrast, the replacement of cyclohexyl groups of monomeric palladium–dialkyl-



Scheme 1. Suzuki–Miyaura cross-coupling of 4-chloro-2-methylquinoline (**1**) and 3,5-dimethylphenylboronic acid (**2**).

Table 1. Yields of 4-(3′,5′-dimethylphenyl)-2-methylquinoline (**3**) obtained by Suzuki–Miyaura cross-coupling of 4-chloro-2-methylquinoline (**1**) and 3,5-dimethylphenylboronic acid (**2**) with various Pd catalysts.[a]

Entry	Catalyst	Yield (%)[b][c]
1	POPd (tBu$_2$POH)$_2$PdCl$_2$	73
2	POPd1 [(tBu$_2$POH)(tBu$_2$PO)(μ-Cl)Pd]$_2$	50
3	POPd2 [(tBu$_2$POH)(μ-Cl)PdCl]$_2$	42
4	POPd6 [(Cy$_2$POH)(μ-Cl)PdCl]$_2$	55
5	POPd7 [(Cy$_2$POH)(Cy$_2$PO)(μ-Cl)Pd]$_2$	70
6	PXPd (tBu$_2$PCl)$_2$PdCl$_2$	70
7	PXPd2 [(tBu$_2$PCl)(μ-Cl)PdCl]$_2$	55
8	PXPd6 [(Cy$_2$PCl)(μ-Cl)PdCl]$_2$	55
9	PXPd7 (Cy$_2$PCl)$_2$PdCl$_2$	52
10	POPd-Br (tBu$_2$POH)$_2$PdBr$_2$	63
11	Ph1-Phoxide [(Ph$_2$POH)(Ph$_2$PO)(μ-Cl)Pd]$_2$	55

[a] All reactions were carried out with 100 mg of **1**, 1.2 equiv. of **2**, 10 mol-% Pd catalyst, and 1.5 equiv. of Cs_2CO_3 in 3 mL of ACN at 82 °C for 20 h. [b] Isolated yields of 4-(3′,5′-dimethylphenyl)-2-methylquinoline (**3**). [c] No product was obtained in the absence of Cs_2CO_3.

(chloro)phosphanes by bulky *tert*-butyl groups increased the yields of **3** from 52 to 70% (Entries 6 and 9). Results obtained with POPd and POPd-Br showed that the replacement of chloro ligands attached to the palladium center by bromide reduces the catalytic activity by 10% (Entries 1 and 10). All reactions gave the biaryl **3** and recovered starting materials but no side-products after work-up suggesting that cross-coupling results can be further improved by optimization of the reaction conditions, such as solvent, temperature, and base.

We then screened the most promising catalysts POPd, POPd7, and PXPd in various solvents reducing the catalyst loading to 5 mol-% to further optimize the reaction conditions and to better differentiate between the activity of these Pd complexes (Table 2). Employing POPd in refluxing THF, toluene, 2-propanol, ethyl acetate, dimethylformamide, and 1,4-dioxane showed that the latter gives superior results, which may be attributed to the enhanced solubility of cesium carbonate in this solvent (Entries 1–7). Under these conditions, monomeric POPd and PXPd exhibiting di-*tert*-butylphosphinous acid and di-*tert*-butyl(chloro)-phosphane ligands gave **3** in 93 and 86% yield while dimeric POPd7 bearing dicyclohexylphosphinous acids provided the coupling product in only 72% (Entries 7–9).

Table 2. Formation of 4-(3′,5′-dimethylphenyl)-2-methylquinoline (**3**) using POPd, PXPd, and PXPd7 in different solvents.[a]

Entry	Catalyst	Solvent	*T* (°C)	Yield[b] (%)
1	POPd	THF	66	25
2	POPd	toluene	110	66
3	POPd	2-propanol	82	50
4	POPd	EtOAc	80	86
5	POPd	DMF	145	75
6	POPd	ACN	82	73
7	POPd	1,4-dioxane	100	93
8	PXPd	1,4-dioxane	100	86
9	POPd7	1,4-dioxane	100	72

[a] All reactions were carried out with 100 mg of aryl chloride **1**, 1.1 equiv. of boronic acid **2**, 5 mol-% Pd catalyst, and 1.5 equiv. of Cs_2CO_3 in 3 mL solvent for 20 h. [b] Isolated yields.

Although excellent results were also observed in ethyl acetate at 80 °C (Table 2, Entry 4) we decided to continue our optimization efforts evaluating the effect of base with PXPd in 1,4-dioxane, Table 3. Screening of *t*BuOK, Cy_2NMe, and various inorganic bases showed that cesium carbonate affords superior results while high yields of the cross-coupling product **3** can also be obtained in the presence of potassium carbonate and potassium phosphate (Entries 2, 5, and 8). Variation of PXPd loading using Cs_2CO_3 as base in 1,4-dioxane proved that the catalyst amount can be reduced to 2.5 mol-% without compromising yields (compare Entries 8–11).

Table 3. Screening of the PXPd-catalyzed Suzuki–Miyaura formation of **3** in the presence of different bases and optimization of catalyst loading.[a]

Entry	Base	Catalyst loading [mol-%]	Yield [%][b]
1	*t*BuOK	5	63
2	K_3PO_4	5	80
3	NaOAc	5	66
4	NaOH	5	66
5	K_2CO_3	5	77
6	Na_2CO_3	5	18
7	Cy_2NMe	5	30
8	Cs_2CO_3	5	86
9	Cs_2CO_3	10	86
10	Cs_2CO_3	2.5	88
11	Cs_2CO_3	1.0	66

[a] All reactions were carried out with 100 mg of **1**, 1.1 equiv. of **2**, and 1.5 equiv. of base in 3 mL of 1,4-dioxane at 100 °C for 20 h. [b] Isolated yields.

Having optimized the catalyst choice, solvent, base, and catalyst loading, using quinolyl chloride **1** and boronic acid **2**, we employed a variety of aryl chlorides in the POPd-catalyzed Suzuki–Miyaura cross-coupling reactions using 1.5 equiv. of Cs_2CO_3 and 2.5 mol-% of the catalyst in 1,4-dioxane, Table 4. We were pleased to find that coupling of **1** and arylboronic acids **2**, **4**, and **6** affords the corresponding 4-aryl-2-methylquinolines **3**, **5**, and **7** in 93 to 98% (Entries 1–3). The formation of *trans*-alkene **9** from **1** and vinyl-

Table 4. POPd-catalyzed Suzuki–Miyaura cross-coupling using aryl chlorides.[a]

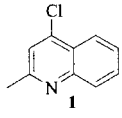
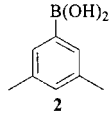
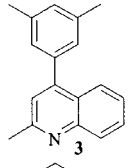
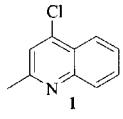
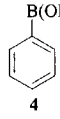
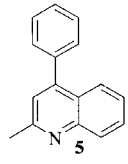
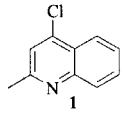
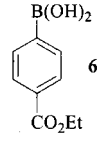
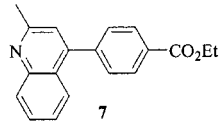
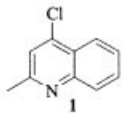
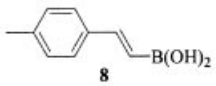
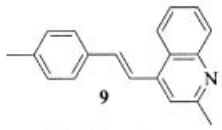
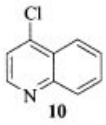
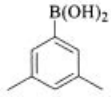
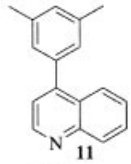
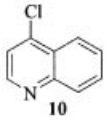
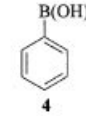
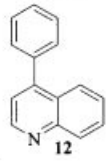
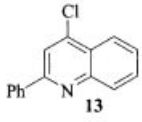
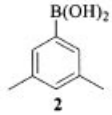
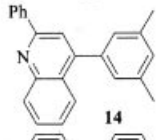
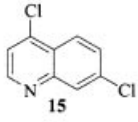
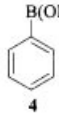
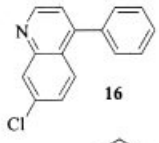
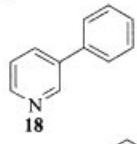
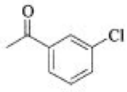
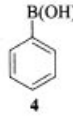
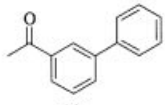
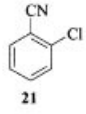
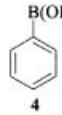
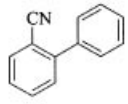
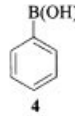
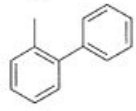
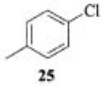
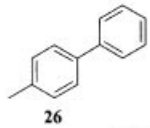
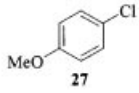
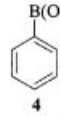
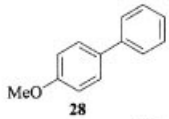
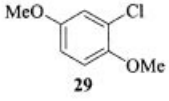
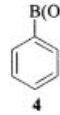
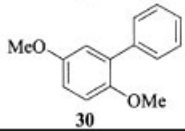
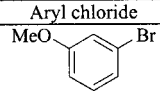
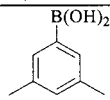
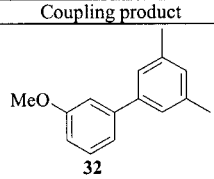
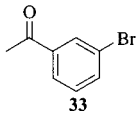
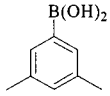
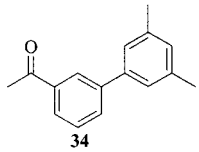
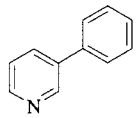
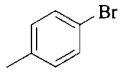
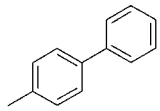
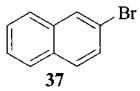
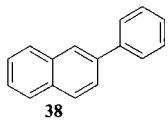
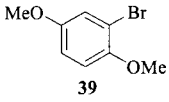
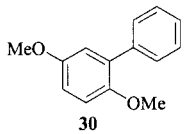
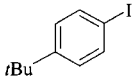
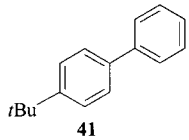
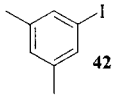
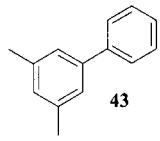
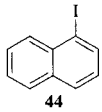
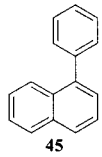
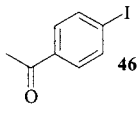
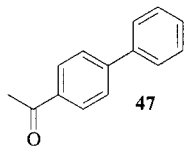
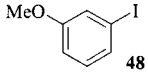
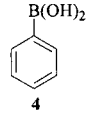
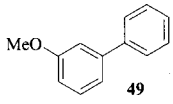
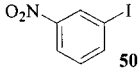
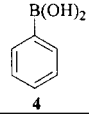
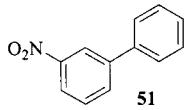
Entry	Aryl chloride	Boronic acid	Coupling product	Yield (%)[b]
1	 **1**	 **2**	 **3**	93
2	 **1**	 **4**	 **5**	98
3	 **1**	 **6**	 **7**	97

Table 4. (*continued*)

Entry	Aryl chloride	Boronic acid	Coupling product	Yield (%)[b]
4	 1	 8	 9	74
5	 10	 2	 11	86
6	 10	 4	 12	99
7	 13	 2	 14	94
8	 15	 4	 16	84
9	 17	 4	 18	65
10	 19	 4	 20	85
11	 21	 4	 22	72
12	 23	 4	 24	75
13	 25	 4	 26	72
14	 27	 4	 28	80
15	 29	 4	 30	78

[a] All reactions were carried out with 100 mg of aryl chloride, 1.1 equiv. of boronic acid, 2.5 mol-% of POPd, and 1.5 equiv. of Cs_2CO_3 in 3 mL of 1,4-dioxane at 100 °C for 20 h. [b] Isolated yields.

Table 5. POPd-catalyzed Suzuki–Miyaura cross-coupling using aryl bromides and iodides.[a]

Entry	Aryl chloride	Boronic acid	Coupling product	Yield (%)[b]
1	 **31**	 **2**	 **32**	85
2	 **33**	 **2**	 **34**	82
3	 **35**	 **4**	 **18**	69
4	 **36**	 **4**	 **26**	70
5	 **37**	 **4**	 **38**	93
6	 **39**	 **4**	 **30**	97
7	 **40**	 **4**	 **41**	93
8	 **42**	 **4**	 **43**	96
9	 **44**	 **4**	 **45**	99
10	 **46**	 **4**	 **47**	88
11	 **48**	 **4**	 **49**	91
12	 **50**	 **4**	 **51**	98

[a] All reactions were carried out with 100 mg of aryl halide, 1.1 equiv. of boronic acid, 2.5 mol-% of POPd, and 1.5 equiv. of Cs_2CO_3 in 3 mL of 1,4-dioxane at 100 °C for 20 h. [b] Isolated yields.

boronic acid **8** was observed to proceed with 74% yields (Entry 4). As expected, POPd-catalyzed cross-coupling of arylboronic acids **2** and **4** with quinolyl chlorides **10**, **13**, and **15** gave high yields and excellent regioselectivity providing 7-chloro-4-phenylquinoline (**16**) in 84% (Entries 5–8). Under the same conditions, biphenyls **20** and **22** were produced in 72 and 85% yield. The moderate yield of volatile 3-phenylpyridine (**18**) obtained by the reaction of 3-chloropyridine (**17**) with **4** can be partially attributed to losses during work-up, which also explains why coupling with 3-bromopyridine (**27**) gave similar results (compare Table 4, Entry 9 and Table 5, Entry 3). The synthesis of quinoline derivatives continues to be an active area of heterocyclic chemistry.[14] The preparation of **3**, **5**, **7**, **9**, **11**, **12**, **14**, and **16** in 74–99% yield is therefore noteworthy since 4-substituted quinoline derivatives have been reported to display pronounced biological activities including antimalarial activity.[15] Good results were also obtained with non-activated and deactivated aryl chlorides. Coupling of boronic acid **4** with 2-chlorotoluene (**23**), 4-chlorotoluene (**25**) and electron-rich 4-chloroanisole (**27**), and 1-chloro-2,5-dimethoxybenzene (**29**), gave biaryls **24**, **26**, **28**, and **30** in 72–80% yield (Entries 12–15). The presence of one *ortho*-cyano or methyl group in **21** and **23**, respectively, does not significantly impede POPd-catalyzed cross-coupling with **4**. However, we found that the reaction proceeds slowly when two *ortho*-methyl groups are present, i.e. the reaction of phenylboronic acid and 1-chloro-2,6-dimethylbenzene showed less than 50% conversion after 20 h.

In general, coupling of aryl bromides with arylboronic acids **2** and **4** was found to give excellent yields. Biaryls **30**, **32**, **34**, and **38** were obtained in 82 to 97% (Table 5, Entries 1, 2, 5, and 6). The reduced yield of 4-methylbiphenyl (**26**), is mostly a consequence of difficulties observed during the chromatographic purification and removal of the starting material **36** (Entry 4). Employing POPd in Suzuki–Miyaura cross-coupling of aryl iodides gave a variety of biphenyls in excellent yields. Using phenylboronic acid **4**, we produced biaryls **41**, **43**, **45**, **47**, **49**, and **51** in 88 to 99%. Based on GC/MS analysis, POPd-catalyzed cross-coupling of boronic acids and aryl bromides or iodides generally proceeds quantitatively, but isolated yields are sometimes limited due to difficulties during the separation of reaction products from small amounts of remaining starting materials exhibiting similar chromatographic behavior.

Conclusion

We have previously shown that palladium–phosphinous acid complexes afford high yields in Stille, Hiyama, Sonogashira, and Heck reactions. These catalysts are readily available, recyclable, stable to air and water and therefore easy to handle.[5g] Screening of the efficacy of 11 palladium complexes with monomeric and μ-chloro-bridged dimeric structures exhibiting either bulky dialkyl- and diarylphosphinous acid groups (POPd, POPd-Br, POPd1, POPd2, POPd6, POPd7, Ph1-Phoxide) or dialkyl(chloro)phosphane ligands (PXPd, PXPd2, PXPd6, PXPd7) suggested, that these catalysts can also be successfully employed in Suzuki–Miyaura coupling reactions. Best results were obtained with POPd and PXPd in 1,4-dioxane and ethyl acetate using cesium carbonate as base. Coupling of a range of electron-deficient and electron-rich aryl iodides, bromides, or chlorides and various organoboronic acids in the presence of 2.5 mol-% of POPd gave the corresponding biaryl products in good to high yields.

Experimental Section

General Procedure: A solution of aryl halide (200 mg), boronic acid (1.1 equiv.), potassium carbonate (1.5 equiv.), and POPd (2.5 mol-%) in 3 mL of 1,4-dioxane was stirred at 100 °C for 20 h. The reaction mixture was cooled to room temperature, quenched with water and extracted with dichloromethane. The combined organic layers were dried with $MgSO_4$ and the solvents were removed under vacuum. The residue was purified by flash chromatography on silica gel as indicated below. NMR spectra were obtained with a Varian FT-NMR spectrometer at 300 MHz (^{1}H NMR) and 75 MHz (^{13}C NMR) using $CDCl_3$ as the solvent. Chemical shifts are reported in ppm relative to TMS.

4-(3′,5′-Dimethylphenyl)-2-methylquinoline (3): Purification by chromatography on silica gel using hexanes/diethyl ether/triethylamine, 100:100:1, gave **3** (260 mg, 93%) as a yellow oil. ^{1}H NMR: δ = 2.36 (s, 6 H), 2.74 (s, 3 H), 7.06 (s, 3 H), 7.16 (s, 1 H), 7.38 (ddd, J = 1.2 Hz, 6.9 Hz, 8.2 Hz 1 H), 7.63 (ddd, J = 1.2 Hz, 6.9 Hz, 8.4 Hz 1 H), 7.87 (dd, J = 1.2 Hz, 8.4 Hz, 1 H), 8.08 (dd, J = 1.2 Hz, 8.2 Hz, 1 H) ppm. ^{13}C NMR: δ = 21.6, 25.6, 122.3, 125.6, 125.9, 126.1, 127.6, 129.2, 129.5, 130.2, 138.3, 148.4, 148.8, 158.5 ppm. $C_{18}H_{17}N$ (247.14): calcd. C 87.41, H 6.93, N 5.66; found C 87.18, H 7.32, N 5.59.

2-Methyl-4-phenylquinoline (5):[5g] Purification by chromatography on silica gel using hexanes/diethyl ether/triethylamine, 100:100:1, affords compound **5** (243 mg, 98%) as an orange oil. ^{1}H NMR: δ = 2.78 (s, 3 H), 7.21 (s, 1 H), 7.41 (dd, J = 7.3 Hz, 8.1 Hz, 1 H), 7.45–7.58 (m, 5 H), 7.68 (dd, J = 7.3 Hz, 8.1 Hz, 1 H), 7.85 (d, J = 8.3 Hz, 1 H), 8.12 (d, J = 7.6 Hz, 1 H) ppm. ^{13}C NMR: δ = 25.7, 122.5, 125.3, 125.9, 126.0, 128.6, 128.8, 129.3, 129.5, 129.7, 138.4, 148.7, 158.7 ppm.

Ethyl 4-(2′-Methyl-4′-quinolyl)benzoate (7):[16] Purification by chromatography on silica gel using diethyl ether/triethylamine, 100:1, provided a colorless oil of **7** (319 mg, 97%). ^{1}H NMR: δ = 1.44 (t, J = 7.1 Hz, 3 H), 2.78 (s, 3 H), 4.44 (q, J = 7.1 Hz, 2 H), 7.22 (s, 1 H), 7.42 (dd, J = 7.3 Hz, 8.3 Hz, 1 H), 7.55 (d, J = 8.1 Hz, 2 H), 7.69 (dd, J = 7.2 Hz, 7.6 Hz, 1 H), 7.76 (d, J = 8.3 Hz, 1 H), 8.09 (d, J = 8.3 Hz, 1 H), 8.19 (d, J = 8.1 Hz, 2 H) ppm. ^{13}C NMR: δ = 14.6, 25.6, 61.4, 122.3, 125.5, 126.3, 129.4, 129.7, 129.8, 130.0, 130.6, 142.9, 147.6, 148.3, 158.5, 166.7 ppm.

(*E*)-1-(4′-Methylphenyl)-2-(2′-methyl-4′-quinolyl)ethene (9): Purification by chromatography on silica gel using hexanes/diethyl ether/triethylamine, 200:100:1, gave **9** (217 mg, 74%) as a viscous colorless oil. ^{1}H NMR: δ = 2.39 (s, 3 H), 2.76 (s, 3 H), 7.21 (d, J = 7.7 Hz, 2 H), 7.27 (d, J = 15.4 Hz, 2 H), 7.42 (s, 1 H), 7.44–7.52 (m, 3 H), 7.63–7.72 (m, 2 H), 8.09 (dd, J = 6.4 Hz, 6.9 Hz, 1 H) ppm. ^{13}C NMR: δ = 21.6, 25.7, 117.9, 122.1, 123.5, 125.8, 127.3, 129.5, 129.8, 134.1, 134.9, 139.0, 143.3, 158.8 ppm. $C_{19}H_{17}N$ (259.14): calcd. C 87.99, H 6.61, N 5.40; found C 88.26, H 6.58, N 5.39.

4-(3′,5′-Dimethylphenyl)quinoline (11): Purification by chromatography on silica gel using hexanes/diethyl ether/triethylamine, 100:100:1, gave **11** (246 mg, 86%) as a yellow oil. ^{1}H NMR: δ = 2.40 (s, 6 H), 7.10 (s, 3 H), 7.28 (d, J = 3.4 Hz, 1 H), 7.45 (dd, J = 7.5 Hz, 8.5 Hz, 1 H), 7.70 (dd, J = 6.5 Hz, 7.5 Hz, 1 H), 7.95 (d, J = 8.5 Hz, 1 H), 8.21 (d, J = 6.5 Hz, 1 H), 8.92 (d, J = 3.4 Hz, 1 H) ppm. ^{13}CNMR: δ = 21.6, 121.5, 126.3, 126.7, 127.2, 127.6, 129.5, 130.1, 130.3, 138.2, 138.4, 149.1, 149.2, 150.2 ppm. $C_{17}H_{15}N$ (233.12): calcd. C 87.52, H 6.48, N 6.00; found C 87.03, H 6.56, N 5.98.

4-Phenylquinoline (12):[5g] Purification by chromatography on silica gel using hexanes/diethyl ether/triethylamine, 100:100:1, afforded **12** (249 mg, 99%) as an orange oil. ^{1}H NMR: δ = 7.25 (d, J = 4.4 Hz, 1 H), 7.38–7.50 (m, 6 H), 7.67 (dd, J = 7.3 Hz, 7.3 Hz, 1 H), 7.89 (d, J = 8.3 Hz, 1 H), 8.21 (d, J = 8.3 Hz, 1 H), 8.91 (d, J = 4.4 Hz, 1 H) ppm. ^{13}C NMR: δ = 121.6, 126.1, 126.9, 127.0, 128.7, 128.8, 129.6, 129.8, 130.1, 138.2, 148.7, 148.9, 150.2 ppm.

4-(3′,5′-Dimethylphenyl)-2-phenylquinoline (14): Purification by chromatography on silica gel using hexanes/diethyl ether/triethylamine, 800:100:1, provided **14** (243 mg, 94%) as a colorless oil. ^{1}H NMR/δ = 2.37 (s, 6 H), 7.08 (s, 1 H), 7.12 (s, 2 H), 7.35–7.51 (m, 4 H), 7.65 (ddd, J = 1.5 Hz, 7.5 Hz, 7.7 Hz, 1 H), 7.77 (s, 1 H), 7.89 (d, J = 8.4 Hz, 1 H), 8.14–8.19 (m, 2 H), 8.21 (d, J = 7.8 Hz, 1 H) ppm. ^{13}C NMR: δ = 21.7, 119.5, 125.2, 126.1, 126.2, 126.5, 127.7, 127.9, 129.2, 129.6, 129.8, 130.3, 130.5, 138.7, 140.1, 149.2, 149.8, 157.1 ppm. $C_{23}H_{19}N$ (309.15): calcd. C 89.28, H 6.19, N 4.53; found C 89.18, H 6.13, N 4.52.

7-Chloro-4-phenylquinoline (16):[5g] Purification by chromatography on silica gel using hexanes/diethyl ether/triethylamine, 100:100:1, gave **16** (204 mg, 84%) as a colorless oil. ^{1}H NMR: δ = 7.29 (d, J = 4.4 Hz, 1 H), 7.40–7.55 (m, 6 H), 7.91 (d, J = 8.9 Hz, 1 H), 8.09 (d, J = 1.9 Hz, 1 H), 8.98 (d, J = 4.4 Hz, 1 H) ppm. ^{13}C NMR: δ = 121.7, 125.4, 127.6, 127.8, 128.9, 129.0, 129.7, 135.4, 137.7, 148.8, 149.3, 151.2 ppm.

3-Phenylpyridine (18):[5h] Purification by chromatography on silica gel using hexanes/diethyl ether/triethylamine, 100:200:1, gave **18** (178 mg, 65% from **17**) as a colorless oil. ^{1}H NMR: δ = 7.33–7.51 m, 4 H), 7.56–7.61 (m, 2 H), 7.83–7.88 (m, 1 H), 8.59 (dd, J = 1.5 Hz, 4.9 Hz, 1 H), 8.86 (d, J = 2.2 Hz, 1 H) ppm. ^{13}C NMR: δ = 123.8, 127.4, 128.4, 129.3, 134.7, 136.9, 138.0, 148.5, 148.6 ppm.

3-Phenylacetophenone (20):[5h] Purification by chromatography on silica gel using hexanes/diethyl ether/triethylamine, 200:100:1, provided **20** (217 mg, 85% from **19**) as a oil. ^{1}H NMR: δ = 2.67 (s, 3 H), 7.39–7.56 (m, 4 H), 7.65 (d, J = 7.3 Hz, 2 H), 7.91 (J = 7.6 Hz, 1 H), 7.96 (d, J = 7.8 Hz, 1 H), 8.23 (s, 1 H) ppm. ^{13}C NMR: δ = 27.0, 127.2, 127.4, 127.5, 128.1, 129.1, 129.4, 131.9, 137.9, 140.0, 141.9, 198.3 ppm.

2-Cyanobiphenyl (22):[5h] Purification by chromatography on silica gel using hexanes/diethyl ether/triethylamine, 400:100:1, gave **22** (207 mg, 72%) as an orange oil. ^{1}H NMR: δ = 7.37–7.63 (m, 8 H), 7.72 (d, J = 8.4 Hz, 1 H) ppm. ^{13}C NMR: δ = 111.5, 118.9, 127.9, 129.0, 129.1, 130.4, 133.2, 134.0, 138.3, 145.7 ppm.

2-Methylbiphenyl (24):[17] Purification by chromatography on silica gel using hexanes/diethyl ether, 5:1, gave **24** (200 mg, 75%) as a colorless oil. ^{1}H NMR: δ = 2.27 (s, 3 H), 7.21–7.25 (m, 4 H), 7.27–7.34 (m, 3 H), 7.36–7.42 (m, 2 H) ppm. ^{13}C NMR: δ = 20.8, 126.1, 127.1, 127.5, 128.4, 129.1, 129.5, 130.1, 130.7, 135.7, 142.3 ppm.

4-Methylbiphenyl (26):[5h] Purification by chromatography on silica gel using hexanes/diethyl ether/triethylamine, 8000:100:1, gave **26** (138 mg, 70% from **36**) as a colorless oil. ^{1}H NMR: δ = 2.37 (s, 3 H), 7.22 (dd, J = 1.2 Hz, 8.1 Hz 2 H), 7.30 (d, J = 8.4 Hz, 1 H), 7.39 (dd, J = 8.1 Hz, 8.1 Hz, 2 H), 7.47 (d, J = 8.1 Hz, 2 H), 7.56 (dd, J = 2.5 Hz, 8.4 Hz, 2 H) ppm. ^{13}C NMR: δ = 21.5, 127.4, 129.2, 129.9, 131.5, 131.6, 137.6, 138.4, 141.8 ppm.

4-Methoxybiphenyl (28):[18] Purification by chromatography on silica gel using hexanes/diethyl ether, 16:1, gave **28** (207 mg, 80%) as a colorless oil. ^{1}H NMR: δ = 3.95 (s, 3 H), 6.94 (dd, J = 2.2 Hz, 6.6 Hz, 2 H), 7.23–7.29 (m, 1 H), 7.34–7.41 (m, 2 H), 7.47–7.55 (m, 4 H) ppm. ^{13}C NMR: δ = 55.6, 114.6, 127.0, 127.1, 128.5, 129.1, 134.1, 141.2, 159.5 ppm.

4-Methoxy-2-phenylanisole (30):[19] Purification by chromatography on silica gel using hexanes/diethyl ether/triethylamine, 800:100:1, afforded **30** (192 mg, 97% from **39**) as a colorless oil. ^{1}H NMR: δ = 3.88 (s, 3 H), 3.94 (s, 3 H), 6.65–6.71 (m, 2 H), 7.34–7.41 (m, 2 H), 7.51 (dd, J = 7.5 Hz, 7.5 Hz, 2 H), 7.63 (d, J = 7.3 Hz, 2 H) ppm. ^{13}C NMR: δ = 55.7, 55.8, 99.3, 104.9, 124.0, 126.8, 128.3, 129.8, 131.6, 138.8, 157.5, 160.3 ppm.

3-(3′,5′-Dimethylphenyl)anisole (32):[20] Purification by chromatography on silica gel using hexanes/diethyl ether/triethylamine, 700:50:1, afforded **32** (194 mg, 85%) as a yellow oil. ^{1}H NMR: δ = 2.33 (s, 6 H), 3.78 (s, 3 H), 6.83 (d, J = 8.1 Hz, 1 H), 6.94 (s, 1 H), 7.07–7.14 (m, 2 H), 7.17(s, 2 H), 7.24 (dd, J = 8.1 Hz, 8.1 Hz, 1 H) ppm. ^{13}C NMR: δ = 21.8, 55.6, 113.0, 113.3, 120.1, 125.5, 129.5, 130.0, 138.6, 141.6, 143.4, 160.3 ppm.

3-(3′,5′-Dimethylphenyl)acetophenone (34): Purification by chromatography on silica gel using hexanes/diethyl ether/triethylamine, 500:50:1, afforded **34** (186 mg, 85%) as a colorless oil. ^{1}H NMR: δ = 2.44 (s, 6 H), 2.69 (s, 3 H), 7.05–7.09 (m, 1 H), 7.26–7.30 (m, 2 H), 7.55 (ddd, J = 0.6 Hz, 7.7 Hz, 7.7 Hz, 1 H), 7.81 (ddd, J = 1.1 Hz, 1.9 Hz, 7.7 Hz, 1 H), 7.94 (ddd, J = 1.1 Hz, 1.7 Hz, 7.7 Hz, 1 H), 8.22 (dd, J = 1.7 Hz, 1.9 Hz, 1 H) ppm. ^{13}C NMR: δ = 21.7, 27.0, 125.4, 127.2, 127.3, 129.2, 129.7, 132.0, 137.8, 138.7, 140.4, 142.2, 198.4 ppm. $C_{16}H_{16}O$ (224.12): calcd. C 85.68, H 7.19; found C 85.12, H 7.12.

2-Phenylnaphthalene (38):[5h] Purification by chromatography on silica gel using hexanes/diethyl ether/triethylamine, 8000:300:1, gave **38** (184 mg, 93%) as a colorless oil. ^{1}H NMR: δ = 7.38–7.46 (m, 1 H), 7.47–7.57 (m, 4 H), 7.74–7.81 (m, 3 H), 7.86–7.92 (m, 3 H), 8.08 (s, 1 H) ppm. ^{13}C NMR: δ = 126.0, 126.3, 126.4, 126.7, 127.6, 128.1, 128.7, 128.9, 129.2, 129.3, 133.1, 134.2, 140.0, 141.5 ppm.

4-*tert*-Butylbiphenyl (41):[21] Purification by chromatography on silica gel using hexanes/diethyl ether/triethylamine, 8000:100:1, gave **41** (150 mg, 93%) as a colorless oil. ^{1}H NMR: δ = 1.35 (s, 9 H), 7.25–7.31 (m, 1 H), 7.35–7.47 (m, 4 H), 7.49–7.59 (m, 4 H) ppm. ^{13}C NMR: δ = 31.8, 126.1, 127.2, 127.4, 129.2, 139.2, 140.1, 150.1 ppm.

3,5-Dimethylbiphenyl (43):[22] Purification by chromatography on silica gel using hexanes/diethyl ether/triethylamine, 2000:100:1, provided **43** (150 mg, 96%) as a yellow oil. ^{1}H NMR: δ = 2.58 (s, 6 H), 7.19 (s, 1 H), 7.42 (s, 2 H), 7.51 (d, J = 7.5 Hz, 1 H), 7.61 (dd, J = 7.5, 7.7 Hz, 2 H), 7.79 (d, J = 7.7 Hz, 2 H) ppm. ^{13}C NMR: δ = 21.9, 125.6, 127.5, 127.6, 129.1, 129.3, 135.5, 138.6, 142.0, 142.1 ppm.

1-Phenylnaphthalene (45):[21] Purification by chromatography on silica gel using hexanes/diethyl ether/triethylamine, 8000:100:1, gave **45** (160 mg, 99%) as a yellow oil. ^{1}H NMR: δ = 7.26–7.45 (m, 9 H), 7.79–7.86 (m, 3 H) ppm. ^{13}C NMR: δ = 125.8, 126.1, 126.4, 127.3, 127.5, 127.6, 128.0, 128.6, 128.9, 129.1, 130.5, 132.0, 134.2, 140.6, 141.1 ppm.

4-Phenylacetophenone (47):[22] Purification by chromatography on silica gel using hexanes/diethyl ether/triethylamine, 700:50:1, gave **47** (140 mg, 88%) as a colorless oil. ^{1}H NMR: δ = 2.65 (s, 3 H), 7.42–7.53 (m, 3 H), 7.64–7.72 (m, 4 H), 8.06 (d, J = 6.6 Hz, 2 H) ppm. ^{13}C NMR: δ = 26.9, 127.5, 127.5, 128.5, 129.2, 129.3, 136.1, 140.0, 146.0, 198.0 ppm.

3-Phenylanisole (49):[5g] Purification by chromatography on silica gel using hexanes/diethyl ether/triethylamine, 8000:100:1, gave **49** (142 mg, 91%) as a yellow oil. ^{1}H NMR: δ = 3.95 (s, 3 H), 7.02 (dd, J = 2.7 Hz, 8.4 Hz, 1 H), 7.16–7.26 (m, 2 H), 7.35–7.51 (m, 2 H), 7.52–7.68 (m, 2 H), 7.74 (d, J = 8.1 Hz, 2 H) ppm. ^{13}C NMR: δ = 55.6, 113.0, 113.3, 120.0, 127.6, 127.8, 129.1, 130.1, 141.5, 143.0, 160.2 ppm.

3-Nitrobiphenyl (51):[23] Purification by chromatography on silica gel using hexanes/diethyl ether/triethylamine, 1000:500:1, provided **51** (156 mg, 98%) as a yellow oil. ^{1}H NMR: δ = 7.41–7.51 (m, 3 H), 7.56–7.64 (m, 3 H), 7.91 (d, J = 7.6 Hz 1 H), 8.19 (d, J = 8.2 Hz 1 H), 8.44 (s, 1 H) ppm. ^{13}C NMR: δ = 122.2, 122.3, 127.4, 127.5, 128.8, 129.4, 130.0, 133.3, 138.5, 143.0 ppm.

Acknowledgments

We gratefully acknowledge financial support for this work from NIH (R01 AI060792). We thank Combiphos Catalysts, Inc., New Jersey (www.combiphos.com) for the catalysts.

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Received: October 28, 2005
Published Online: February 6, 2006

