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Synthesis and application of ortho-palladated complex of (4-phenylbenzoylmethylene)triphenylphosphorane as a highly active catalyst in the Suzuki cross-coupling reaction

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ABSTRACT

The ortho-metallated complex [Pd(μ-Cl){κ_2(*C*,*C*)-[$C_6H_4(PPh_2CHC(O)C_6H_4$-Ph-4)}]$_2$ (**2a**) was prepared by refluxing of $Pd(OAc)_2$ and {$(Ph)_3PCHCOC_6H_4$-Ph-4} (PhBPPY) of in CH_2Cl_2 followed by addition of an excess of KCl in MeOH Complex (**2a**) reacts with triphenylphosphine to give the mononuclear derivative [Pd(Cl)($PhC_6H_4COCHPPh_2C_6H_4$)(PPh_3)] (**3a**) whose crystal structure has been determined by single crystal X-ray diffraction. The Suzuki reactions of aryl bromides and chlorides of varying electron density using complex (**3a**) as an efficient catalyst were performed, giving the cross-coupling products in good to excellent yields.

1. Introduction

Palladium-catalyzed Suzuki cross-coupling reactions of aryl halides with aryl boronic acids have recently emerged as an extremely efficient and important tool in organic synthesis, as well as in a variety of industrial processes [1–12]. The key advantages of the Suzuki coupling compared to other coupling methods [13–15] are the mild reaction condition and the commercial availability of the diverse boronic acids that are environmentally safer than the other organometallic reagents [16,17]. In addition, the handling and removal of boron-containing byproducts is easy when compared to other organometallic reagents, especially in a large-scale synthesis [18].

As part of our ongoing study aimed on the synthesis of new palladium catalysts, the ortho-palladated complex of (4-phenylbenzoylmethylene)triphenylphosphorane [Pd(Cl)($PhC_6H_4COCHPPh_2C_6H_4$)(PPh_3)] (**3a**) has been prepared and its structure elucidated by single crystal X-ray analysis. The complex demonstrated to be an efficient catalyst in Suzuki cross-coupling reactions of phenyl boronic acid with aryl halides, resulting in good to excellent yields of the desired products.

2. Results and discussion

The synthesis pathway starting from the (4-phenylbenzoylmethylene)triphenylphosphorane (PhBPPY) (**1a**) and leading to the formation of the dimeric complex (**2a**) and, finally, to the triphenylphosphine mononuclear adduct (**3a**) is shown in Scheme 1. The reaction of an equimolar amount of $Pd(OAc)_2$ (OAc = acetate) with PhBPPY in refluxing CH_2Cl_2 and further reaction of the acetate intermediate with excess NaCl in methanol gives a insoluble solid, whose stoichiometry corresponds to the ortho-metallated complexes [Pd(μ-Cl){C,C-[$C_6H_4(PPh_2CHC(O)C_6H_4$-4-Ph)}]$_2$ (**2a**). As already reported for similar compounds [19–22], (**2a**) reacts with triphenylphosphine to give the mononuclear palladium (II) derivative [Pd(Cl)($PhC_6H_4COCHPPh_2C_6H_4$)(PPh_3)] (**3a**).

2.1. Structure analysis of the catalyst

In the ortho-palladated complex (**3a**) (Fig. 1), one phenyl ring of the triphenylphosphine group of the PhBPPY ligand has undergone a C–H bond activation process, leading to the *endo* metallation of the phosphorus ylide. The palladium(II) atom is coordinated by a ylidic carbon atom (C1), a metallated carbon atom (C15), a chloride anion (Cl1) and a phosphorus atom (P2) of a PPh_3 ligand in a distorted square-planar geometry. The extent of the distortion from the regular geometry may be evinced from the values of the

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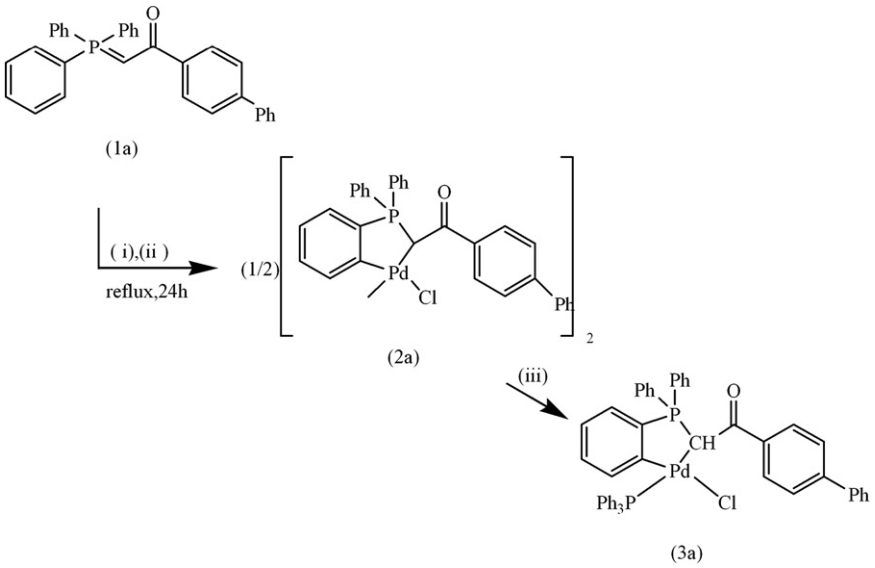


Scheme 1. (i) $Pd(OAc)_2/CH_2Cl_2$/reflux (60 °C) 24 h; (ii) NaCl/MeOH, r. t; (iii) PPh_3/CH_2Cl_2.

bond angles subtended at the metal centre (84.81(10)–177.13 (8)°; Table 1). These values are in good agreement with those observed in closely related complex [PdCl(PhBPPY)(4-picoline)] (83.5(3)–176.76(16)° [22]).

The Pd1–C15 bond distance (2.008(3) Å) involving the ortho-metallated carbon atom is not significantly different from those found in the analogous 4-picoline complex (2.002(6) Å) and in related ortho-palladated complexes (1.990(4) Å [20]; 1.9997(4) Å [23]), while the Pd1–C1 bond distance involving the ylide carbon atom (2.180(2) Å) in complex (**3a**) is longer (2.101(6) Å [22]; 2.090 (3) Å [23]). The difference observed between the Pd1–C1 and Pd1–C15 bond lengths in **3a** possibly reflecting the different *trans* effects of the phosphorus and chlorine atoms. The stabilized resonance structure for the parent ylide is destroyed due to the complex formation, thus the C1–C2 bond length (1.467(4) Å) in complex **3a** were significantly longer than the corresponding distances found in the similar uncomplexed phosphoranes (1.401(2) Å [24] and 1.407 (8) Å [25]). The P1–C1 bond length (1.777(3) Å) is identical, within experimental error, to that reported for the 4-picoline complex (1.775(6) Å [24]). The Pd1/C15/C20/P1/C1 five-membered ring metallacycle adopts an envelope conformation, with atom C1 displaced by (1.058(3) Å) from the mean plane of the remaining four atoms. The conformation of the complex molecule is stabilized by intramolecular C–H…Cl and C–H…O hydrogen bonds: C4–H4 = 0.93 Å, H4…Cl1 = 2.64 Å, C4…Cl1 = 3.518(3) Å, C4–H4…Cl1 = 158°; C28–H28 = 0.93 Å, H28…O1 = 2.40 Å, C28…O1 = 3.098(2) Å, C28–H28…O1 = 132°.

2.2. Suzuki–Miyaura coupling reactions of aryl bromides

A series of aryl and benzyl bromides (Table 2) were coupled with phenyl boronic acid with 0.01 mmol of catalyst at 60 °C under optimized reaction conditions. In the presence of electron withdrawing substituents (entries 2, 11) as well as in the case heterocyclic derivative (entry 12) the reaction afforded excellent yields of coupling products at 60 °C.

The coupling of 3-bromotoluene (entry 3), 2-bromobenzaldehyde (entry 4), 2-bromonaphtalene (entry 5), 3-chlorobromobenzene (entry 9), 2-chlorobromobenzene (entry 10) and 2-bromonitrobenzene (entry 13) with phenyl boronic acid resulted in good yields at 60 °C. The Suzuki coupling was also extended to C(sp^2)-C (sp^3) coupling by reacting benzyl bromide (entry 6) with phenyl boronic acid, which afforded the coupling product in excellent yields. Thus the catalyst afforded average to excellent yields of the biaryl products at 60 °C. In the literature, only a few catalysts are known for effecting the Suzuki cross-coupling reactions under mild conditions [10,26–33].

2.3. Suzuki–Miyaura coupling reactions of aryl chlorides

The cross-coupling of aryl chlorides with phenyl boronic acid in the presence of 0.01 mmol of catalyst were carried out at 60 °C using MeOH as solvent (Table 2). Electron deficient substrates such as chlorobenzene, 4-chloronitrobenzene, and benzyl chloride (entries 1, 7, and 8, respectively) coupled with phenyl boronic acid with yields of the coupling products ranging from 72 to 97%.

2.4. Comparison of aryl bromide and aryl chloride reaction

The main drawback of the Pd-mediated Suzuki cross-coupling reaction is that only aryl iodides and aryl bromides can be used efficiently. Recent progress on increasing the reaction rate of aryl chlorides permitted to overcome this problem [34–40]. The stronger C–Cl bond is in fact responsible of the slower reaction rate of aryl halides, because the oxidative addition step was suggested to be the rate determining step in cross-coupling catalytic cycles [41].

3. Experimental section

3.1. X-ray crystallography

Single crystals of (**3a**) suitable for X-ray crystallography were obtained by diffusion of n-hexane into a CH_2Cl_2 solution of the complex. Intensity data were collected at ambient temperature (294 (2) K) using graphite monochromated Mo Kα radiation ($\lambda = 0.71073$ Å) on a Bruker SMART 1000 CCD diffractometer. Data were corrected for absorption using the SABABS program [42]. All non-hydrogen atoms were refined anisotropically. All H atoms were placed in calculated positions and treated as riding on their parent atoms, with C–H = (0.93–0.98 Å), and with $U_{iso}(H) = 1.2\ U_{eq}(C)$. Crystallographic data and parameters concerning data collection and structure solution and refinement are summarized in the

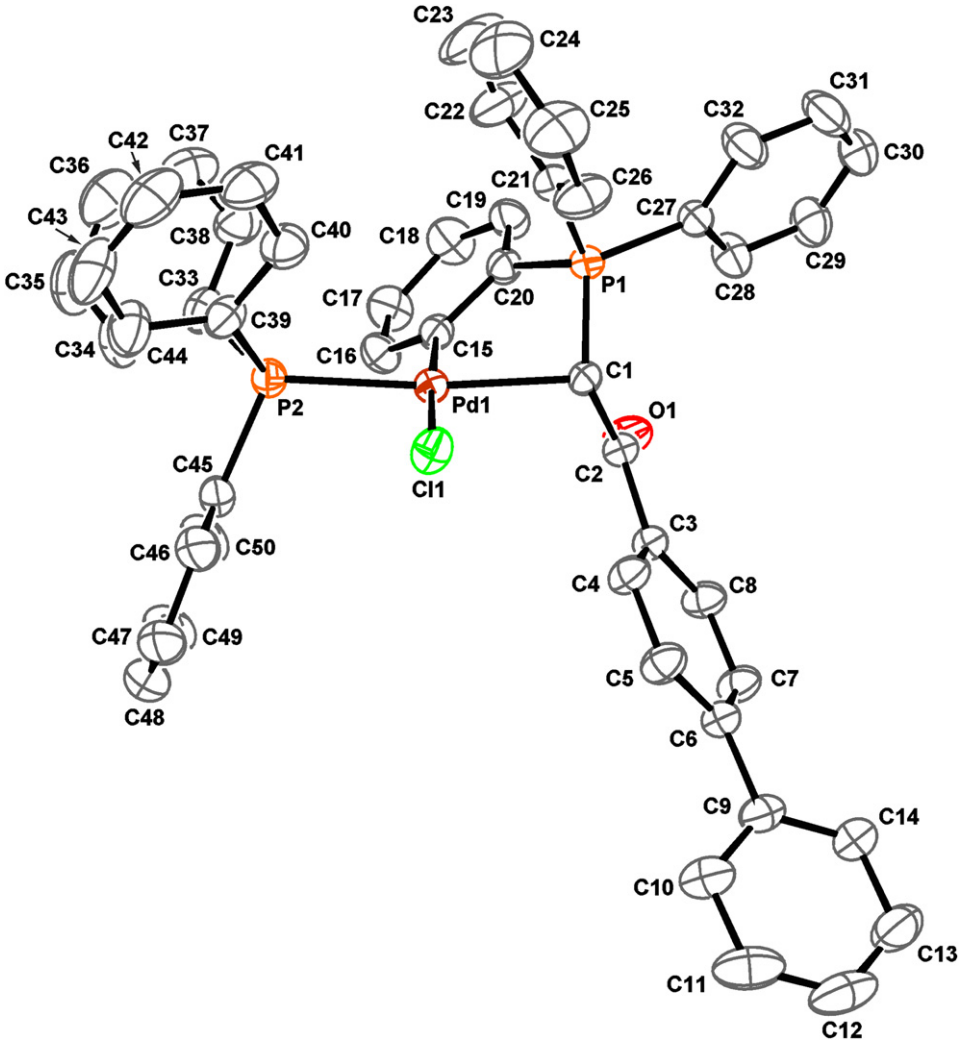


Fig. 1. The molecular structure of **3a**. Displacement ellipsoids are drawn at the 40% probability level. Hydrogen atoms are omitted for clarity.

Supplementary data, selected bond lengths (Å) and angles (°) are listed in Table 1. An ORTEP-3 [43] diagram of the complex molecule is shown in Fig. 1, a view of the unit-cell content is given in the Supplementary data.

3.2. Materials and techniques

All chemicals were purchased from Fluka and Merck companies. Gas chromatography carried out with a Shimadzu GC 14-A gas chromatograph and thin layer chromatography on precoated silica gel fluorescent 254 nm (0.2 mm) were used for monitoring the reaction progresses. ^{1}H NMR (500 MHz), ^{13}C NMR (125 MHz), and ^{31}P NMR (202 MHz) spectra were recorded in $CDCl_3$ solutions at room temperature (TMS was used as an internal standard) on a Bruker Avance spectrometer. The cross-coupling products were characterized by their ^{1}H NMR spectra and melting points.

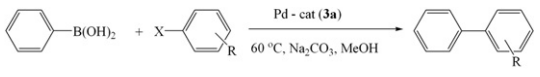
Table 1
Selected bond lengths (Å) and angles (°) for complex **2a**.

Pd(1)–P(2) 2.9316(7)	P(1)–C(1) 1.777(3)
Pd(1)–Cl(1) 2.4058(7)	P(1)–C(27) 1.810(3)
Pd(1)–C(1) 2.180(2)	P(1)–C(20) 1.782(3)
Pd(1)–C(15) 2.008(3)	P(1)–C(21) 1.800(3)
P(2)–C(39) 1.826(3)	C(1)–C(2) 1.467(4)
P(2)–C(33) 1.847(3)	C(2)–O(1) 1.226(3)
P(2)–C(45) 1.823(3)	C(2)–C(3) 1.507(4)
C(15)–Pd(1)–P(2) 95.18(8)	C(1)–Pd(1)–P(2) 173.87(7)
Cl(1)–Pd(1)–P(2) 86.97(3)	C(15)–Pd(1)–P(1) 61.15(8)
C(15)–Pd(1)–C(1) 84.81(10)	C(1)–Pd(1)–P(1) 37.13(7)
Cl(1)–Pd(1)–C(1) 92.85(7)	P(2)–Pd(1)–P(1) 138.22(2)
C(15)–Pd(1)–Cl(1) 177.13(8)	Cl(1)–Pd(1)–P(1) 115.99(2)

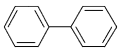
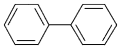
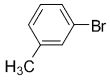
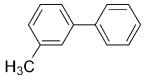
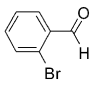
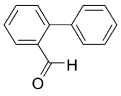
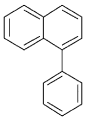
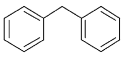
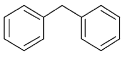
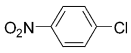
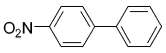
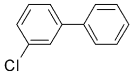
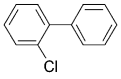
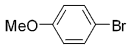
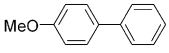
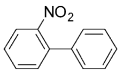
3.3. General procedure for the synthesis of the Pd complex (*3a*): *[Pd(Cl)(PhC$_6$H$_4$COCHPPh$_2$C$_6$H$_4$)(PPh$_3$)]*

To a solution of $Pd(OAc)_2$ (0.116 g, 0.52 mmol) in CH_2Cl_2 (15 ml) (4-phenylbenzoylmethyl)triphenylphosphorane (PhBPPY) (0.237 g, 0.52 mmol) (**1a**) was added. The mixture was refluxed for 24 h at 60 °C. The mixture was then evaporated to dryness and MeOH was added to the solid and the green suspension treated with an excess of NaCl (0.111 g, 1.034 mmol) at room temperature. A yellow suspension immediately produced and stirring was maintained for 12 h at room temperature, after which the precipitate was filtered, washed with Et_2O (5 ml) and water (10 ml) and dried in vacuum to obtain $[Pd(\mu\text{-}Cl)\{C,C\text{-}[C_6H_4(PPh_2CHC\text{-}(O)C_6H_4\text{-}4\text{-}X)\}]_2$ (**2a**).To a suspension of (**2a**) (0.1194 g, 0.1 mmol) in CH_2Cl_2 (15 mL) was added PPh_3 (0.0524 g, 0.2 mmol). The initial yellow suspension gradually dissolved after stirring for 8 h at room temperature. The resulting solution was filtered over a Celite© pad to remove any residual

Table 2
Reactants and products of the Suzuki cross-coupling reactions.[a]



R = H, Me, CHO, Ph, OMe, NO_2, Cl
X = Br, Cl

Entry	Aryl halide	Product	Yield (%)[b]
1			97
2			100
3			78
4			91
5			84
6			100
7			72
8			77
9			85
10			84
11			100
12			96
13			81

[a] Reaction conditions: aryl halide (0.5 mmol), $PhB(OH)_2$ (0.75 mmol), [Pd(Cl)(PhBPPY)(PPh_3)] (0.01 mmol), Na_2CO_3 (1.5 mmol), MeOH (6 ml), Reflux 60 °C, 40 min.
[b] GC yields, ±2%.

insoluble solid. The clear solution obtained and was evaporated to dryness, and the treatment of the residue with Et_2O (30 mL) gave [Pd(Cl)(PhBPPY)(PPh_3)] (**3a**) as a yellow solid.

M.p. 146 °C; Yield (0.1014 g, 59%); Anal. Calc for $C_{50}H_{39}OP_2PdCl$: C, 65.86; H, 4.27; Found. C, 65.39; H, 4.0; IR (KBr, cm^{-1}): ν (C=O): 1619. ^1H NMR (500 MHz, $CDCl_3$, ppm); δ = 5.5 (s, 1H, CHP), 6.5 (s, 2H, C_6H_4 ^3J(H–H) = 3 Hz), 6.90 (m, 1H, C_6H_4, ^3J(H–H) = 4 Hz), 7.19 (m, 6H, H_m, PPh_3), 7.32 (m, 6H, H_m, PPh_2 + H_m, C_6H_5), 7.44 (m, 6H, H_p, C_6H_5 + PPh_2 + PPh_3), 7.6 (m, 6H, H_o, PPh_3), 7.66 (m, 2H, H_m, C_6H_4CO), 7.88 (m, 2H, H_o, C_6H_5), 8.02 (m, 2H, H_o, PPh_2), 8.29 (m, 2H, H_o, PPh_2), 8.31 (d, 2H, H_o, C_6H_4CO, $^3J_{HH}$ = 7.1 Hz). $^{31}P\{^1H\}$ NMR ($CDCl_3$): δ = 15.06 (s, CHP), 31.86 (s, Pd-PPh_3). $^{13}C\{^1H\}$ NMR: δ = 40.47 (dd, CHP, $^1J_{PC}$ = 63.13 Hz, $^2J_{PC}$ = 21 Hz), 123.72 (d, C_1, $^1J_{PC}$ = 13.20 Hz), $C_{aromatic}$ {δ = 126.71, 127.46, 128.12 (d, C_o PPh_2 or C_o PPh_3, $^2J_{PC}$ = 10.19 Hz), 129.08 (d, C_m PPh_2, $^3J_{PC}$ = 6.03 Hz), 129.68 (d, C_i PPh_2, $^2J_{PC}$ = 11.82 Hz), 130.02, 130.53, 130.66, 131.72, 132.84, 134.64, 138.06, 139.2, 140.8, 144.2}, δ = 133.31 (d, C_3, $^3J_{PC}$ = 9.31 Hz), δ = 135.16 (d, C_i PPh_3, $^2J_{PC}$ = 11.82 Hz) [22].

3.4. General experimental procedure for Suzuki cross-coupling reactions

A mixture of an aryl halide (0.5 mmol), phenyl boronic acid (0.75 mmol), [Pd(Cl)(PhBPPY)(PPh_3)] (0.01 mmol), Na_2CO_3 (1.5 mmol), and MeOH (6 ml) was heated to 60 °C for 40 min under reflux. The reaction mixture was then cooled to room temperature and the solvent was removed under reduced pressure. The combined organic extracts were washed with brine and dried over $MgSO_4$. The solvent was evaporated and a crude product was obtained. A small aliquot of the reaction mixture was diluted in MeOH for direct GC analysis.

The aryl halides employed in the Suzuki cross-coupling reactions with phenyl boronic acid and the coupling products are listed in Table 2.

3.5. Characterization of the products of Suzuki cross-coupling reactions

3.5.1. Biphenyl

A white solid, M.p. 70.5–72.0 °C; IR (KBr): υ: 3037, 1569, 1480, 1429, 729, 697; ^1H NMR (300 MHz, $CDCl_3$, ppm): δ = 7.35–7.37 (m, 2H, ArH), 7.4–7.5 (m, 4H, ArH), 7.6–7.6 (m, 4H, ArH) [44].

3.5.2. 3-Methylbiphenyl

M.p. 30–34 °C; ^1H NMR (300 MHz, $CDCl_3$, ppm): δ = 7.7 (m, 2H), 7.5 (m, 6H), 7.3 (m, 1H), 2.5 (s, 3H) [45].

3.5.3. Biphenyl-2-carboxaldehyde

A white solid, M.p. 69–74 °C; ^1H NMR (300 MHz, $CDCl_3$, ppm): δ = 10.01 (d, ^4J(H–H) = 0.9 Hz, 1H, CHO), 8.05 (dd, ^3J(H–H) = 7.5, ^4J(H–H) = 1.2 Hz, 1H, Ar), 7.7 (dt, ^3J(H–H) = 7.5, ^4J(H–H) = 1.2 Hz, 1H, Ar), 7.55–7.39 (m, 7H, Ar) [46].

3.5.4. 1-Phenyl-naphthalene

M.p. 91–96 °C; IR (KBr): υ: 3056, 1592, 1493, 1395, 802, 779, 761, 703, 570. ^1H NMR (300 MHz, $CDCl_3$, ppm): δ = 7.90–7.8 (m, 3H), 7.51–7.36 (m, 9H). ^{13}C NMR (75 MHz, $CDCl_3$, ppm): δ = 140.9, 140.4, 134.0, 131.8, 130.2, 128.4, 127.7, 127.3, 127.0, 126.1, 125.9, 125.5 [47].

3.5.5. Diphenylmethane

M.p. 21–24 °C; ^1H NMR (300 MHz, $CDCl_3$, ppm): δ = 3.96 (s, 2H, CH_2), 7.23–7.33 (m, 10H, ArH), ^{13}C NMR (75 MHz, $CDCl_3$, ppm): δ = 42.2, 125.1, 131, 129.3, 144 [48].

3.5.6. 4-Nitro-biphenyl

IR (KBr): υ: 3077, 1598, 1515, 1345, 1105, 854, 740, 697. ^1H NMR (300 MHz, $CDCl_3$, ppm): δ = 8.29 (d, J(H–H) = 8.7 Hz, 2H), 7.73 (d, J(H–H) = 8.7 Hz, 2H), 7.62 (dd, J(H–H) = 8.4, 1.5 Hz, 2H), 7.52–7.41 (m, 3H). ^{13}C NMR (75 MHz, $CDCl_3$, ppm): δ = 147.7, 147.2, 138.9, 129.3, 129.0, 127.9, 127.5, 124.2 [47].

3.5.7. 3-Chloro-biphenyl

A yellow solid, M.p. 44.5–47.5 °C; IR (KBr): υ: 3057, 3033, 2924, 1380, 1494, 1464, 1425, 1130, 1072, 1038, 1011, 771, 746, 697; ^1H NMR (300 MHz, $CDCl_3$, ppm): δ = 7.37–7.55 (m, 8H, ArH), 8.01 (d, 1H, ArH) [44].

3.5.8. 2-Chloro-biphenyl

A yellow solid, M.p. 32.5–33.5 °C; IR (KBr): υ: 3059, 3031, 2924, 1380, 1498, 1467, 1425, 1128, 1075, 1036, 1009, 770, 748, 699; ^1H NMR (300 MHz, $CDCl_3$, ppm): δ = 7.24–7.61 (m, 9H, ArH) [44].

3.5.9. 4-Methoxy-biphenyl

IR (KBr): υ: 3020, 1612, 1519, 1487, 1216, 909, 759. ^1H NMR (300 MHz, $CDCl_3$, ppm): δ = 7.56–7.51 (m, 4H), 7.41 (t, J(H–H) = 7.5 Hz, 2H), 7.29 (t, J(H–H) = 7.5 Hz, 1H), 6.98 (d, J(H–H) = 7.5 Hz, 2H), 3.85 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$, ppm): δ = 159.3, 140.9, 133.9, 128.9, 128.3, 126.9, 126.8, 114.3, 55.4 [47].

3.5.10. 2-Phenylpyridine

^1H NMR (500 MHz, $CDCl_3$, ppm): δ = 8.70 (d, J(H–H) = 4.5 Hz, 1H), 7.98 (d, J(H–H) = 7.5 Hz, 2H), 7.75 (m, 2H), 7.48 (t, J(H–H) = 5.0 Hz, 2H), 7.42 (d, J(H–H) = 7.5 Hz, 1H), 7.24 (d, J(H–H) = 5.5 Hz, 1H) [49].

3.5.11. 2-Nitro-biphenyl

^1H NMR (500 MHz, $CDCl_3$, ppm): δ = 7.84 (q, J(H–H) = 2.5 Hz, 1H), 7.60 (m, 1H), 7.44 (m, 5H), 7.31 (m, 2H) [49].

4. Conclusion

In this paper, the synthesis and characterization is reported of a highly active and efficient catalyst for promoting the Suzuki cross-coupling reaction of various aryl halides to produce the corresponding products in average to excellent yields. The ease of preparation of the complex, its high solubility in organic solvents, indefinite shelf life, and stability toward air make it an ideal complex for the above transformations.

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Appendix A. supplementary data

CCDC 783343 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via http://www.ccdc.cam.ac.uk/deposit or deposit@ccdc.cam.ac.uk.

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