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Supporting information

Unusual Coordination of Triazolyl-Pyridine Ligand in a Pd(II) Complex: Applications in Suzuki-Miyaura Coupling Reaction

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Instrumentation methods

Nuclear magnetic resonance spectra were recorded on a JEOL ECS-400 spectrometer operating at 400 MHz for ¹H NMR and 101 MHz for ¹³C{¹H}NMR respectively. Tetramethyl silane (TMS) (0.00 ppm) was used as an reference internal solvent to record ¹H and ¹³C{¹H}NMR spectra for all the compounds. During analysis of 1H NMR spectra proton peak for CDCl3 was fixed at 7.246 ppm and the carbon peak was fixed at 77.0 ppm. ¹H NMR patterns of chemical shifts were characterized in parts per million (ppm). The terms singlet (s), doublet (d), double of doublet (dd), triplet (t), and multiplet (m) were used to describe peak splitting patterns. The coupling constant (*J*) values are given in Hertz (Hz). The Xevo G2-SQ-Tof (Waters, USA) was used to examine high-resolution electron impact mass spectra (HR-EIMS), which are compatible with ACQUITY UPLC® and nano ACQUITY UPLC® systems. The melting point of Ligand and complex were determined on an analog melting point apparatus.

NMR spectral data of 2, 3, 4 and 1a.

Spectroscopic data of di(prop-2-yn-1-yl) phthalate [dpp] (2)

¹H NMR (400 MHz, Chloroform-*d*) δ 7.72-7.70 (m, 2H), 7.52-7.50 (m, 2H), 4.85 (d, *J* = 2.4 Hz, 4H), 2.47 (t, *J* = 2.4 Hz, 2H); ¹³C NMR (101 MHz, Chloroform-*d*) δ 166.63, 131.62, 131.25, 129.27, 77.29,75.56, 53.28. HRMS (ES) m/z calcd for C₁₄H₁₀O₄ ([M+H]⁺) 243.0652; found 243.0654.

Spectroscopic data of bis((1-(pyridin-2-yl)-1H-1,2,3-triazol-5-yl) methyl) phthalate [bptmp] (3)

Mp: 180-183 °C. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.67 (s, 2H), 8.42 (s, 2H), 8.10 (d, J = 8.0 Hz, 2H), 7.86-7.82 (m, 2H), 7.69 (dd, J = 5.6, 3.2 Hz, 2H), 7.48-7.46 (m, 2H), 7.27 (dd, J = 7.6, 4.8 Hz, 2H), 5.48 (s, 4H); ¹³C NMR (101 MHz, Chloroform-*d*) δ 167.27, 149.03, 148.69, 143.02, 139.23, 131.65, 131.46, 129.26, 123.82, 121.59, 113.93, 58.89. HRMS (ES) m/z calcd for C₂₄H₁₈N₈O₄ ([M+H]⁺) 483.1524; found 483.1526. IR ν_{max} (cm⁻¹): 3132 (w, C_{sp2}-H stretch.), 3085 (w, N-N stretch.), 2958 (w, C_{sp3}-H stretch.), 1721 (s, C=O stretch.), 1581(m, C=C_{sp2} stretch.), 1477 (m, C_{sp3}-H bend.), 1280 (s, C-N stretch.), 1115 (m, C-C_{sp3} stretch.), 783 (s, C=C_{sp2} bend.) ^{1,2}.

Spectroscopic data of [PdCl₂(bptmp)] (4)

Mp: 250-253 °C. ¹H NMR (400 MHz, DMSO- d_6) δ 8.92 (s, 2H), 8.58 (d, J = 4.8 Hz, 2H), 8.11 (s, 4H), 7.83 – 7.78 (m, 2H), 7.72-7.69 (m, 2H), 7.57-7.53 (m, 2H), 5.47 (s, 4H); ¹³C NMR (101 MHz, DMSO- d_6) δ 167.02, 149.46, 144.20, 142.98, 140.63, 132.43, 131.53, 129.43, 124.95, 122.44, 114.21, 58.88. HRMS (ES) m/z calcd for C₂₄H₁₈N₈O₄ ([M+H]⁺) 658.9936; found 658.9934. IR v_{max} (cm⁻¹): 3073 (w, N-N stretch.), 1722 (s, C=O stretch.), 1118 (m, C-C_{sp3} stretch.), 503 (w, Pd-N stretch.), 423 (w, Pd-Cl stretch.)³.

Synthesis of 2-azidopyridine (1a)

Compound 1a was synthesised according to literature procedure ⁴.



Fig. S1 ¹H NMR spectrum of 2 in CDCl₃ (400 MHz)



Fig. S3 EI mass spectrum of 2



Fig. S4 ¹H NMR spectrum of 3 in CDCl₃ (400 MHz)



Fig. S5 ¹³C NMR spectrum of 3 in CDCl₃ (101 MHz)



Fig. S6 EI mass spectrum of 3



Fig. S7 FT-IR spectrum of bptmp 3

EuroEA Elemental Analyser

AutoRun Name	:	2022-43 (189)
Date of Analysis	:	17 Dec 2022
Time of Analysis	:	15:13:38
Analysed By	:	EVR
Signed By	:	EVR
Operator Group	:	GRP1
Configuration	:	CHNS
Sample Name	:	bptmp (Ligand)A
Sample Position #	:	10
Туре	:	Smp
Sample Weight	:	1.122 (mg)
Calibration type	:	Linear

24

1

1

1000

Instrument Parameters

Carrier (kPa)	Purge (ml/min)	Oxygen (ml)	Delta P O2 (kPa) Sampling Delay (s)	Run Time (s)	Front (°C)	Rear (°C)	Oven (°C)
100	80	15	35	8	500	980	Off	100

Chromatogram



Results

Element	RT (s)	Area	Area %	Element %
Nitrogen	39	557,563	11.423	24.181
Carbon	86	4,019,868	82.353	61.148
Hydrogen	200	303,825	6.224	3.016
Sulphur	н	-	-	(2 7)

Fig. S8 C,H,N analysis of bptmp 3



Fig. S9 ¹H NMR spectrum of 4 in DMSO-d6 (400 MHz)





Fig. S10 ¹³C NMR spectrum of 4 in DMSO-d6 (101 MHz)

Fig. S11 EI mass spectrum of 4



Fig. S12 FT-IR Spectrum of PdCl₂(bptmp) 4

EuroEA Elemental Analyser								EURO EA
AutoRun N	lame	:	2022-43 (189	9)				-
Date of An	alysis	:	17 Dec 2022	Î.				
Time of An	alysis	:	15:13:38				_	
Analysed I	Зу	:	EVR					
Signed By		:	EVR					
Operator C	Group	:	GRP1					
Configurat	ion	:	CHNS					
Sample Na	me	:	PdCl2(bptmp) B				
Sample Po	sition #	:	11					
Туре		:	Smp					
Sample We	eight	:	1.668 (mg)					
Calibration	n type	:	Linear					
Instrume	ent Paramet	ers						
Carrier (kPa)	Purge (ml/min)	Oxygen (ml)	Delta P O2 (kPa)	Sampling Delay (s)	Run Time (s)	Front (°C)	Rear (°C)	Oven (°C)
100	80	15	35	8	500	980	Off	100

Chromatogram



Fig. S13 FT-IR Spectrum of PdCl₂(bptmp) 4

Crystallographic Information for catalyst 4.

Single-crystal suitable for X-ray diffraction study for **4** was obtained from a mixed solvents of chloroform-acetonitrile (10:1) at room temperature. The crystals were chosen from the mother liquor, immersed in paratone oil, mounted on the tip of a glass fiber, and cemented using epoxy resin. Bruker APEX-III photon diffractometer was used to collect the single crystal data at 100

K. Bruker software package SAINT was used for data reduction, whereas SADABS was used for the absorption corrections and additional systematic error corrections. SHELXT was used for structure solving and refined by SHELXL-2019. In SHELX program suite, latest X-seed version 4.20 was used as graphical interface. Both the disordered carbonyl groups are model with fractional occupancy. Riding model was used to locate all the hydrogen atoms. The crystal contains severely disordered and partially occupied chloroform molecule. Hence, it was squeezed using the program PLATON and the squeeze data is appended in the .CIF file.

Fig. S14 Molecular structure and atom numbering scheme of compound **4**. The thermal ellipsoids are drawn at the 30% probability level. Hydrogen atoms are omitted for clarity. Selected bond lengths [pm] and angles [°]:



General procedure for the synthesis of C-C cross coupling of variety of aryl halides (5A-Q) with different type of phenyl boronic acids (6a-c). A mixture of different type of aryl halides (5A-Q) (0.5 mmol) with different phenyl boronic acid (6a-c) (0.55 mmol) in the presence of catalyst 4 (0.1 mol%) and K₂CO₃ (1mmol) was charged in a reaction tube at a temperature of 50°C for 1h in EtOH/H₂O combination. After the completion of the reaction, the reaction mixture was cooled and the work-up was done with H₂O (15 mL) and ethyl acetate (3×20 mL). The combined organic layers were dried over anhydrous Na₂SO₄, filtered, and concentrated under reduced pressure to get the crude residue which was further purified through column chromatography on silica gel (230-400 mesh) using hexane: ethyl acetate (99:1 ratio) as eluting solvent to afford the C-C coupling product **7Aa-7Oa**, **7Fb-7Pb** and **7Qc-7Mc** in 68-98 % yield.



Scheme 1. Synthesis of C-C cross coupling product using different type of aryl halides (5A-Q) with various phenyl boronic acid (6a-c)

Characterization data of C-C cross coupling product 7Aa-7Oa,7Fb-7Pb and 7Qc-7Mc.

[1,1'-biphenyl]-2-carbaldehyde (7Aa)

¹H NMR (400 MHz, Chloroform-*d*) δ 9.90 (s, 1H), 7.94 (d, J = 8.0 Hz, 1H), 7.55 (td, J = 7.6, 1.6 Hz, 1H), 7.43 – 7.28 (m, 7H); ¹³C NMR (101 MHz, Chloroform-*d*) δ 192.61, 146.06, 137.79, 133.74, 133.67, 130.86, 130.18, 128.51, 128.20, 127.86, 127.63. **7Aa** was more confirmed by ref⁵.

1-([1,1'-biphenyl]-4-yl) ethan-1-one (7Ba)

¹H NMR (400 MHz, Chloroform-*d*) δ 7.94 (d, J = 8.4 Hz, 2H), 7.59 (d, J = 8.4 Hz, 2H), 7.53 (d, J = 6.8 Hz, 2H), 7.40-7.36 (m, 2H), 7.32 (d, J = 7.6 Hz, 1H), 2.54 (s, 3H); ¹³C NMR (101 MHz, Chloroform-*d*) δ 197.89, 145.84, 139.91, 135.88, 129.03, 129.00, 128.31, 127.34, 127.29, 26.76. **7Ba** was more confirmed by ref⁶.

2-phenylpyridine (7Ca)

¹H NMR (400 MHz, Chloroform-*d*) δ 8.64 (d, *J* = 5.2 Hz, 1H), 7.93 (d, *J* = 7.6 Hz, 2H), 7.74-7.67 (m, 2H), 7.44 – 7.33 (m, 3H), 7.21-7.18 (m, 1H); ¹³C NMR (101 MHz, Chloroform-*d*) δ 155.73, 147.51, 137.08, 132.73, 128.56, 127.94, 126.13, 121.42, 120.19. **7Ca** was more confirmed by ref⁷. 1-phenylnaphthalene (7Da)

¹H NMR (400 MHz, Chloroform-*d*) δ 7.82-7.78 (m, 2H), 7.75 (d, *J* = 8.4 Hz, 1H), 7.43 – 7.29 (m, 9H); ¹³C NMR (101 MHz, Chloroform-*d*) δ 140.85, 140.35, 133.88, 131.70, 130.18, 128.36, 127.73, 127.34, 127.03, 126.12, 125.87, 125.49. **7Da** was more confirmed by ref⁷.

2-methyl-3-phenylquinoline (7Ea)

¹H NMR (400 MHz, Chloroform-*d*) δ 8.05 (dd, J = 20.4, 8.4 Hz, 2H), 7.88 – 7.84 (m, 2H), 7.60-7.57 (m, 2H), 7.38 (t, J = 7.6 Hz, 2H), 7.40 – 7.27 (m, 1H), 7.21 (d, J = 7.6 Hz, 1H), 2.70 (s, 3H); ¹³C NMR (101 MHz, Chloroform-*d*) δ 158.73, 145.85, 140.14, 139.07, 137.69, 129.93, 129.08, 128.03, 127.87, 127.45, 126.81, 125.30, 122.61, 24.70. **7Ea** was more confirmed by ref⁸.

4-nitro-1,1'-biphenyl (7Fa)

¹H NMR (400 MHz, Chloroform-*d*) δ 8.19 (d, *J* = 8.8 Hz, 2H), 7.63 (d, *J* = 8.8 Hz, 2H), 7.54 – 7.51 (m, 2H), 7.42 – 7.33 (m, 3H); ¹³C NMR (101 MHz, Chloroform-*d*) δ 147.69, 147.13, 138.82, 129.25, 129.02, 127.87, 127.47, 124.19. **7Fa** was more confirmed by ref ⁹. 2-phenylpyrazine (**7Ga**)

¹H NMR (400 MHz, Chloroform-*d*) δ 8.93 (d, J = 1.6 Hz, 1H), 8.52 (dd, J = 2.4, 1.6 Hz, 1H), 8.40 (d, J = 2.4 Hz, 1H), 7.93 – 7.90 (m, 2H), 7.43 – 7.35 (m, 3H); ¹³C NMR (101 MHz, Chloroform-*d*) δ 152.85, 144.24, 142.92, 142.22, 136.34, 129.99, 129.11, 126.99. **7Ga** was more confirmed by ref ¹⁰.

4-phenylisoquinoline (7Ha)

¹H NMR (400 MHz, Chloroform-*d*) δ 9.24 (s, 1H), 8.43 (s, 1H), 8.01 (d, *J* = 7.2 Hz, 1H), 7.87 (d, *J* = 8.8 Hz, 1H), 7.66 – 7.58 (m, 2H), 7.49 – 7.40 (m, 5H); ¹³C was found in good agreement with the ref ¹¹.

4-fluoro-1,1'-biphenyl (7Ia)

¹H NMR (400 MHz, Chloroform-*d*) δ 7.46 – 7.24 (m, 7H), 7.04-6.99 (m, 2H); ¹³C NMR (101 MHz, Chloroform-*d*) δ 162.40 (J_{C-F} = 244.8 Hz), 140.18, 137.26, 128.76, 128.62 (J_{C-F} = 8.2 Hz), 127.20, 126.96, 115.54 (J_{C-F} = 21.2 Hz). **7Ia** was more confirmed by ref ¹².

3,5-bis(trifluoromethyl)-1,1'-biphenyl (7Ja)

¹H NMR (400 MHz, Chloroform-*d*) δ 7.18 (s, 2H), 7.04 (s, 1H), 6.77 – 6.75 (m, 2H), 6.69 – 6.59 (m, 3H); ¹³C NMR (100 MHz, Chloroform-*d*) δ 143.40, 138.31, 132.20 (J_{C-F} = 33 Hz), 129.34, 128.94, 127.29, 124.87, 122.16, 120.97 (J_{C-F} = 4.0 Hz).¹³C was more confirmed by ref ¹³

[1,1'-biphenyl]-4-carbonitrile (7Ka)

¹H NMR (400 MHz, Chloroform-*d*) δ 7.69-7.62 (m, 4H), 7.56 – 7.53 (m, 2H), 7.46 – 7.36 (m, 3H); ¹³C NMR (101 MHz, Chloroform-*d*) δ 145.75, 139.25, 132.69, 129.21, 128.76, 127.82, 127.32, 119.06, 110.97. **7Ka** was more confirmed by ref ¹².

1,1':2',1"-terphenyl (7La)

¹H NMR (400 MHz, Chloroform-*d*) δ 7.34 (d, *J* = 1.2 Hz, 4H), 7.13-7.10 (m, 6H), 7.07 – 7.04 (m, 4H); ¹³C NMR (101 MHz, Chloroform-*d*) δ 141.57, 140.63, 130.68, 129.97, 127.93, 127.55, 126.52. **7La** was more confirmed by ref ¹⁴. 4-methoxy-1,1'-biphenyl (**7Ma**)

¹H NMR (400 MHz, Chloroform-*d*) δ 7.49 – 7.45 (m, 4H), 7.36-7.32 (m, 2H), 7.25 – 7.18 (m, 1H), 6.91 (d, *J* = 8.4 Hz, 2H), 3.78 (s, 3H); ¹³C was found in good agreement with the ref ¹⁵.

4-methyl-1,1'-biphenyl (7Na)

¹H NMR (400 MHz, Chloroform-*d*) δ 7.50 (d, *J* = 7.2 Hz, 2H), 7.41 (d, *J* = 8.0 Hz, 2H), 7.37-7.32 (m, 2H), 7.26 – 7.21 (m, 1H), 7.16 (d, *J* = 8.0 Hz, 2H), 2.31 (s, 3H); ¹³C was found in good agreement with the ref ¹⁵.

4-(trifluoromethoxy)-1,1'-biphenyl (7Oa)

¹H NMR (400 MHz, Chloroform-*d*) δ 7.53 – 7.46 (m, 4H), 7.39 – 7.34 (m, 2H), 7.31 – 7.26 (m, 1H), 7.20 (d, *J* = 8.0 Hz, 2H); ¹³C was found in good agreement with the ref ¹⁶.

4-methoxy-4'-nitro-1,1'-biphenyl (7Fb)

¹H NMR (400 MHz, Chloroform-*d*) δ 8.19 (d, *J* = 8.8 Hz, 2H), 7.61 (d, *J* = 8.8 Hz, 2H), 7.50 (d, *J* = 8.8 Hz, 2H), 6.94 (d, *J* = 8.8 Hz, 2H), 3.80 (s, 3H); ¹³C was found in good agreement with the ref ¹⁷.

4'-methoxy-[1,1'-biphenyl]-4-carbonitrile (7Kb)

¹H NMR (400 MHz, Chloroform-*d*) δ 7.61 – 7.54 (m, 4H), 7.45 (d, *J* = 8.8 Hz, 2H), 6.92 (d, *J* = 8.4 Hz, 2H), 3.78 (s, 3H); ¹³C was found in good agreement with the ref ¹⁸.

4,4'-dimethoxy-1,1'-biphenyl (7Mb)

¹H NMR (400 MHz, Chloroform-*d*) δ 7.40 (d, *J* = 8.8 Hz, 4H), 6.88 (d, *J* = 8.8 Hz, 4H), 3.76 (s, 6H); ¹³C was found in good agreement with the ref ¹⁹.

4-methoxy-4'-methyl-1,1'-biphenyl (7Nb)

¹H NMR (400 MHz, Chloroform-*d*) δ 7.44 (d, *J* = 8.8 Hz, 2H), 7.38 (d, *J* = 8.4 Hz, 2H), 7.18 – 7.14 (m, 2H), 6.89 (d, *J* = 8.8 Hz, 2H), 3.77 (s, 3H), 2.31 (s, 3H); ¹³C was found in good agreement with the ref ²⁰.

1-(4'-methoxy-[1,1'-biphenyl]-4-yl) ethan-1-one (7Pb)

¹H NMR (400 MHz, Chloroform-*d*) δ 7.92 (d, *J* = 8.4 Hz, 2H), 7.53 (dd, *J* = 25.6, 8.4 Hz, 4H), 6.92 (d, *J* = 8.8 Hz, 2H), 3.78 (s, 3H), 2.54 (s, 3H); ¹³C was found in good agreement with the ref ²⁰.

1-(2'-nitro-[1,1'-biphenyl]-4-yl) ethan-1-one (7Qc)

¹H NMR (400 MHz, Chloroform-*d*) δ 7.95 (d, J = 8.4 Hz, 2H), 7.86 (dd, J = 8.0, 1.2 Hz, 1H), 7.61-7.57(m, 1H), 7.49-7.45 (m, 1H), 7.38 – 7.33 (m, 3H), 2.57 (s, 3H); ¹³C was found in good agreement with the ref ²¹.

1,1'-([1,1'-biphenyl]-4,4'-diyl) bis(ethan-1-one) (7Pc)

¹H NMR (400 MHz, Chloroform-*d*) δ 8.00 (d, *J* = 8.4 Hz, 4H), 7.66 (d, *J* = 8.4 Hz, 4H), 2.59 (s, 6H); ¹³C was found in good agreement with the ref ²⁰.

1-(4'-methoxy-[1,1'-biphenyl]-4-yl) ethan-1-one (7Mc)

Since the structure of compound 7Mc is same as 7Pb, so ¹H NMR of 7Mc is matched with 7Pb and it found in good agreement with ref ²⁰.

4'-fluoro-2-methoxy-1,1'-biphenyl (7Rd)

¹H NMR (400 MHz, Chloroform-*d*) δ 7.57 – 7.53 (m, 2H), 7.40 – 7.33 (m, 2H), 7.17 – 7.02 (m, 4H), 3.85 (s, 3H); ¹³C NMR (101 MHz, Chloroform-*d*) δ 162.15 (J_{*C*-*F*} = 245 Hz), 156.51, 134.58 (J_{*C*-*F*} = 3 Hz), 131.28 (J_{*C*-*F*} = 8 Hz), 130.91, 129.78, 128.90, 121.03, 115.01 (J_{*C*-*F*} = 22 Hz), 111.36, 55.62. **7Rd** was more confirmed by ref ²².



Fig. S15 ¹H NMR spectrum of 7Aa in CDCl₃ (400 MHz)





Fig. S17 ¹H NMR spectrum of 7Ba in CDCl₃ (400 MHz)



Fig. S18¹³C NMR spectrum of 7Ba in CDCl₃ (101 MHz)



Fig. S19 ¹H NMR spectrum of 7Ca in CDCl₃ (400 MHz)



Fig. S20 ¹³C NMR spectrum of 7Ca in CDCl₃ (101 MHz)





Fig. S22 ¹³C NMR spectrum of 7Da in CDCl₃ (101 MHz)



Fig. S23 ¹H NMR spectrum of 7Ea in CDCl₃ (400 MHz)





Fig. S24 ¹³C NMR spectrum of 7Ea in CDCl₃ (101 MHz)







Fig. S26¹³C NMR spectrum of 7Fa in CDCl₃ (101 MHz)







Fig. S28 ¹³C NMR spectrum of 7Ga in CDCl₃ (101 MHz)

Fig. S30 ¹H NMR spectrum of 7Ia in CDCl₃ (400 MHz)





Fig. S32 ¹H NMR spectrum of 7Ja in CDCl₃ (400 MHz)





Fig. S36 ¹H NMR spectrum of 7La in CDCl₃ (400 MHz)





Fig. S38 ¹H NMR spectrum of 7Ma in CDCl₃ (400 MHz)



Fig. S40 ¹H NMR spectrum of 7Oa in CDCl₃ (400 MHz)

Fig. S42 ¹H NMR spectrum of 7Kb in CDCl₃ (400 MHz)





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Fig. S44 ¹H NMR spectrum of 7Nb in CDCl₃ (400 MHz)



Fig. S46 ¹H NMR spectrum of 7Qc in CDCl₃ (400 MHz)

Fig. S48 ¹H NMR spectrum of 7Rd in CDCl₃ (400 MHz)



Fig. S49 ¹³C NMR spectrum of 7Rd in CDCl₃ (101 MHz)

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