Malaysian Journal of Analytical Sciences (MJAS)
Published by Malaysian Analytical Sciences Society
ANALIS
Persatuan Sains Analisis Malaysia

SYNTHESIS AND CHARACTERIZATION OF PALLADIUM(II) SCHIFF BASE COMPLEXES DERIVED FROM PHENYLAMINES AND THEIR CATALYTIC ACTIVITY FOR STILLE REACTION

(Sintesis dan Pencirian Palladium(II) Bes-Schiff Kompleks Dari Fenilamina dan Aktiviti Sebagai Pemangkin untuk Tindak Balas Stille)

Nur Nabihah Muzammil[1], Mohd Tajudin Mohd Ali[1], and Amalina Mohd Tajuddin[1,2*]

[1]*Faculty of Applied Sciences, Universiti Teknologi MARA, Shah Alam, 40450 Shah Alam, Selangor, Malaysia*
[2]*Atta-ur-Rahman Institute for Natural Product Discovery (AuRIns), Universiti Teknologi MARA Puncak Alam, 42300, Bandar Puncak Alam, Selangor, Malaysia*

[*]*Corresponding author: amalina9487@uitm.edu.my*

Received: 10 September 2023; Accepted: 25 February 2024; Published: 29 April 2024

Abstract

Schiff bases with hydrogen and methyl at the *para*-position of phenylamines (B1H and B1Me) and their palladium(II) complexes, (PdB1H and PdB1Me) were synthesized and characterized by elemental analysis, FTIR, ^{1}H and ^{13}C NMR, magnetic susceptibility measurement and UV-Visible spectroscopy. The shifting of the ν(C=N) and ν(C-O) bands to lower frequencies in FTIR indicated that the complexation to palladium(II) occurred through these moieties. All complexes are diamagnetic and displayed low molar conductivity in acetonitrile, indicating non-electrolytic behaviour. B1H and B1Me are structurally characterized using single crystal X-ray diffraction. B1H and B1Me crystallize in orthorhombic space group $P2_12_12_1$, with a = 6.0879(2) Å, b = 9.1847(2) Å, and c = 21.0661(5) Å and a = 5.9051(9) Å, b = 9.2389(13) Å, and c = 23.297(3) Å, respectively. The palladium(II) complexes were screened for their catalytic activity in the Stille reaction. The reaction was monitored by measuring the % conversion of iodobenzene using GC-FID, where the reaction conditions used were 1.0 mmol% catalyst loading in the presence of triethylamine as the base and DMSO as the solvent at 80°C within 6 hours of the reaction time. It was observed that PdB1Me displayed the best catalytic performance for the Stille reaction, as indicated by the 80% conversion of iodobenzene.

Keywords: catalysis, palladium(II), Schiff base, Stille reaction, single crystal X-ray crystallography

Abstrak

Bes-Schiff dengan hidrogen dan metil pada fenilamina posisi *para* (B1H dan B1Me), dan kompleks paladium(II) (PdB1H dan PdB1Me), telah disintesis dan dicirikan oleh analisis unsur, FTIR, ^{1}H dan ^{13}C NMR, ukuran kerentanan magnet dan sinar ultraungu spektroskopi. Peralihan jalur ν(C=N) dan ν(C-O) kepada frekuensi yang lebih rendah dalam FTIR menunjukkan bahawa kompleksasi kepada paladium(II) berlaku melalui gugusan ini. Semua kompleks adalah diamagnet dan menunjukkan kekonduksian molar rendah dalam asetonitril yang menunjukkan tingkah laku bukan elektrolitik. B1H dan B1Me dicirikan secara struktur menggunakan pembelauan sinar-X kristal tunggal. B1H dan B1Me menghablur dalam ortorombik dengan kumpulan ruang $P2_12_12_1$, dengan a = 6.0879(2) Å, b = 9.1847(2) Å, dan c = 21.0661(5) Å dan a = 5.9051(9) Å, b = 9.2389(13) Å, dan c = 23.297(3) Å. Kompleks paladium(II) telah disaring untuk aktiviti pemangkinnya dalam tindak balas Stille. Tindak balas dipantau dengan mengukur % penukaran iodobenzena menggunakan GC-FID, di mana keadaan tindak balas yang digunakan ialah 1.0 mmol%

pemangkin dengan kehadiran trietilamina sebagai bes dan DMSO sebagai pelarut pada 80°C selama 6 jam. PdB1Me memaparkan prestasi pemangkin terbaik di kalangan kompleks untuk tindak balas Stille, ditunjukkan oleh penukaran 80% iodobenzena.

Kata kunci: pemangkinan, paladium(II), Bes-Schiff, tindak balas Stille, kristalografi sinar-X kristal tunggal

Introduction

The Schiff base complexes have received great attention in recent years because of their highly considered as stereochemical models in coordination chemistry, their easy mode of preparation and their structural diversity [1]. Schiff bases and their complexes have a wide range of applications such as bioactive agents [2], catalysts [3], medicines [4], and anti-corrosion agents [5]. Schiff base complexes play a key role in various homogeneous catalytic reactions and the activity of these complexes changes with the types of ligands, coordination sites and metal ions [6].

Schiff bases that are derived from amines and carbonyl compounds belong to a class of ligands involved in the coordination of metal ions via azomethine nitrogen [7]. Phenylamines, through the condensation reaction with various types of organic reagents such as aldehydes, ketones, thiosemicarbazides, carbazides, and similar compounds, are able to form flexible ligand systems that are able to coordinate with a variety of metals [8]. Schiff bases derived from phenylamines and their respective transition metal complexes exhibit potential catalytic activity [9].

Palladium is considered a very important metal from a coordination point of view and it plays a key role as a catalyst in a variety of catalytic activities by varying the ligand environment [10]. Palladium complexes are excellent and versatile catalysts in many important chemical reactions such as carbon-carbon coupling, alkylation, carbonylation, and oxidation [11]. Palladium(II) Schiff base complexes are mostly used to catalyse these couplings reactions as they offer high product yields, high regioselectivity, high stereoselectivity, and compatibility with many functional moieties.

The palladium-catalysed Stille cross-coupling reaction represents one of the most important transformations in constructing carbon-carbon bonds in organic synthesis [12, 13]. Generally, the combination of palladium catalysts with various phosphine ligands is commonly used in the Stille reaction [14]. However, phosphine ligands are air-sensitive and expensive, which places significant limits on their synthetic applications [15].

This paper aims to report the synthesis and characterization of bidentate Schiff base ligands with hydrogen and methyl at the *para*-position of phenylamines with *o*-vanillin and their palladium(II) complexes. Scheme 1 shows the synthesis of the Schiff base ligands and their palladium(II) complexes. The molecular structures of all compounds were elucidated through elemental analysis, magnetic susceptibility, molar conductivity, FTIR, ^{1}H NMR, ^{13}C NMR, UV−Visible analysis, and single crystal X-ray diffraction. The palladium(II) complexes were screened for their catalytic activity in the Stille reaction.

Materials and Methods

Materials and physical measurements

All chemicals and solvents received from commercial suppliers were used. Microanalyses for C, H and N were conducted on Thermo Finnigan Flash Elemental Analyzer 2000. Melting point determination was carried out in glass capillary tubes using Buchii-B454. The IR spectra were obtained using Perkin Elmer 1750X FTIR spectrophotometer (4000-400 cm^{-1}) with samples prepared as KBr discs. Proton (^{1}H) and carbon (^{13}C) NMR (600 MHz) spectra were recorded on Bruker Varian spectrometer in $CDCl_3$ and reported in ppm (δ) relative to TMS, used as the internal reference. The UV-Vis spectra were obtained on Perkin Lambda 35 in chloroform ($CHCl_3$) 1×10^{-4} M solution at room temperature, in the range of 200-800 nm, using a quartz cuvette. Magnetic measurements were carried out using the Guoy method with distilled water as a calibrant on the Sherwood Auto Magnetic Susceptibility Balance. A single crystal X-ray diffraction investigation was carried out on Bruker D8 QUEST CCD diffractometer with PHOTON III 7 detector, using graphite-monochromated Mo-Kα radiation (λ = 0.71073 Å) at 298 K. The collected frames were processed with the software SAINT. Structures were solved by the direct methods and refined by full-matrix least-squares on F^2 using the SHELXTL software package. Mercury [16] software was utilized for the graphical representation of the X-ray results. Gas chromatographic analyses were performed on Agilent 7890A Network GC System using ethyl laurate as standard. The column used is HP-5 capillary columns with a temperature increment of 15°C per minute, and the final temperature was 300°C.

Synthesis of ligands

The Schiff base ligands were synthesized by condensation reactions between *o*-vanillin and phenylamine derivatives using a 1:1 molar ratio (Scheme 1) in hot ethanol, under reflux for 3 hours. The mixture was allowed to cool slowly to room temperature. Upon cooling, orange precipitate formed. The precipitate was filtered off, washed with ice-cold ethanol, and air-dried at room temperature. The crystals were obtained through slow evaporation of the filtrate solutions.

Synthesis of palladium(II) complexes

The ligands and palladium(II) acetate were dissolved in hot ethanol in a round-bottom flask using a 2:1 molar ratio. The mixture was stirred and refluxed for 6 hours. The mixture was allowed to cool slowly to room temperature. Upon cooling, brown precipitate formed. The precipitate was filtered off, washed with ice-cold ethanol, and air-dried at room temperature.

General procedure for the Stille reaction

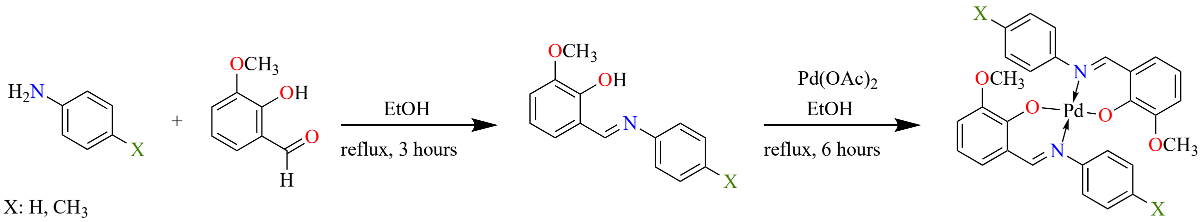
The general procedure (Scheme 2) is as follows: Iodobenzene (1 mmol, 0.2 g), tributylphenyltin (1.5 mmol, 0.55 g), triethylamine (Et_3N, 2.0 mmol, 0.20 g), palladium(II) complex (1 mmol%) (PdB1H, PdB1Me) and dimethylsulfoxide (DMSO, 7 mL) were mixed in Radley's 12-placed reaction carousel and refluxed at 80 °C for 6 hours. The reaction was monitored regularly using GC-FID.

Results and Discussion

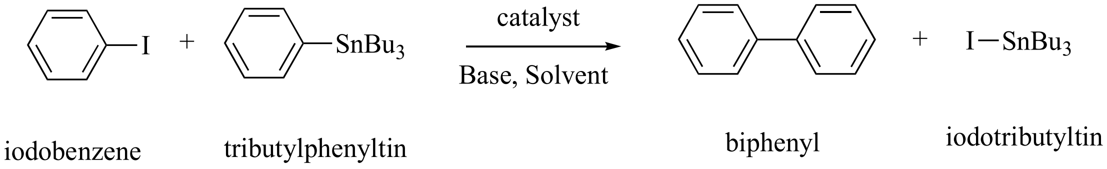
Elemental analysis

The physicochemical data of the compounds are presented in Table 1. According to the elemental analysis of C, H, and N for the compounds, the experimental values were in accordance with the calculated values of the proposed structure. The melting points of the parent ligands were typically lower than those of the complexes, which is likely due to the increased molecular sizes and the presence of stronger dative covalent bonds between C-N-Pd and ionic bonds between C-O-Pd [16].

Molar conductivity measurement for complexes revealed that the non-conductivity nature of the solution at room temperature indicating the absence of any electrolytes [17]. These values show that there are no ions existing in the outer-sphere coordination of the complexes. As expected, the magnetic susceptibility measurements showed diamagnetic behaviour reflected in the zero effective magnetic moment, $\mu_{eff} = 0$, in all complexes consistent with the 0 unpaired electrons of Pd(II) d^8 species having square planar geometry [18].



Scheme 1. Synthesis of the Schiff base ligands and their Pd(II) complexes



Scheme 2. General reaction of Stille reaction

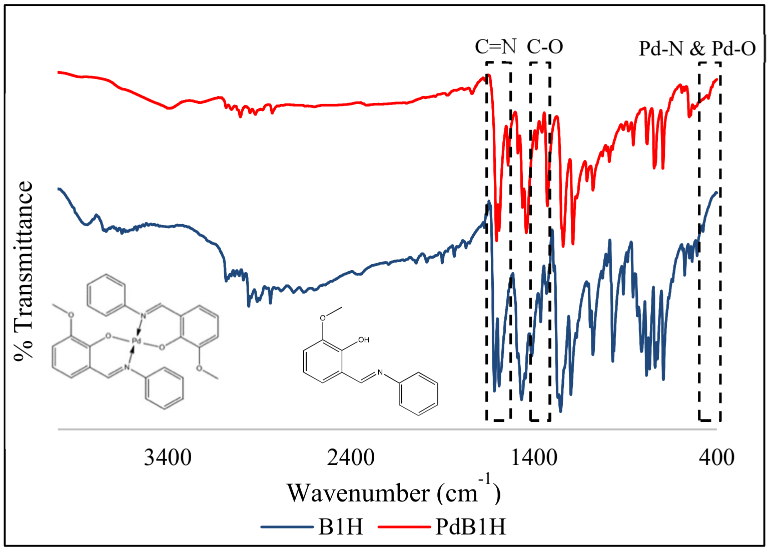
Table 1. Physicochemical data of ligands and their complexes

Compounds	**Empirical Formula**	**Colour**	**Melting Point (°C)**	**Elemental Analysis (%)**			**Magnetism**	**Molar Conductivity ($\Omega^{-1}cm^2mol^{-1}$)**
				C	**H**	**N**		
B1H	$C_{14}H_{13}NO_2$	Orange	227.26	73.99 **(73.29)**	5.77 **(5.68)**	6.16 **(6.05)**	-	-
PdB1H	$C_{28}H_{24}N_2O_4Pd$	Brown	558.93	60.17 **(59.39)**	4.33 **(4.19)**	5.01 **(4.84)**	Diamagnetic	1.275
B1Me	$C_{15}H_{15}NO_2$	Orange	241.29	74.67 **(73.79)**	6.27 **(6.24)**	5.81 **(5.71)**	-	-
PdB1Me	$C_{30}H_{28}N_2O_4Pd$	Brown	586.98	61.39 **(60.55)**	4.81 **(4.74)**	4.77 **(4.58)**	Diamagnetic	0.874

FTIR spectroscopy

The FTIR spectra of the ligands and palladium(II) complexes are shown in Figure 1. There is an absence of *v*(OH) peak in the FTIR spectra of the ligands (Figure 1). This can be attributed to the formation of hydrogen bonding between the hydrogen and nitrogen atoms in the solid state. This is supported by the discovery of an intramolecular hydrogen bond in the X-ray crystallography analysis. The strong bands around 1615-1618 cm^{-1} are assignable to the azomethine *v*(C=N) group for the ligands. This band was observed to be shifted slightly to lower frequencies of 1601– 1602 cm^{-1} in the complexes, likely due to the coordination of the imine nitrogen atom through the sharing of the N lone pair of electrons in a dative manner with the metal centre [19]. The involvement of deprotonated phenolic oxygen atoms in chelation was indicated by the shifting to lower frequencies of the *v*(C-O)$_{\text{phenolic}}$ band. The medium intensity *v*(C-O)$_{\text{phenolic}}$ band that was observed at 1253-1258 cm^{-1} in ligands shifted to lower wavenumbers of 1237-1240 cm^{-1} in complexes.

The coordination of the imine nitrogen and phenolic oxygen atoms to the metal centres was supported by the appearance of new peaks of *v*(Pd-O) and *v*(Pd-N) bands in palladium(II) complexes, which appeared in the regions of 507-572 cm^{-1} and 446-473 cm^{-1}, respectively [20].



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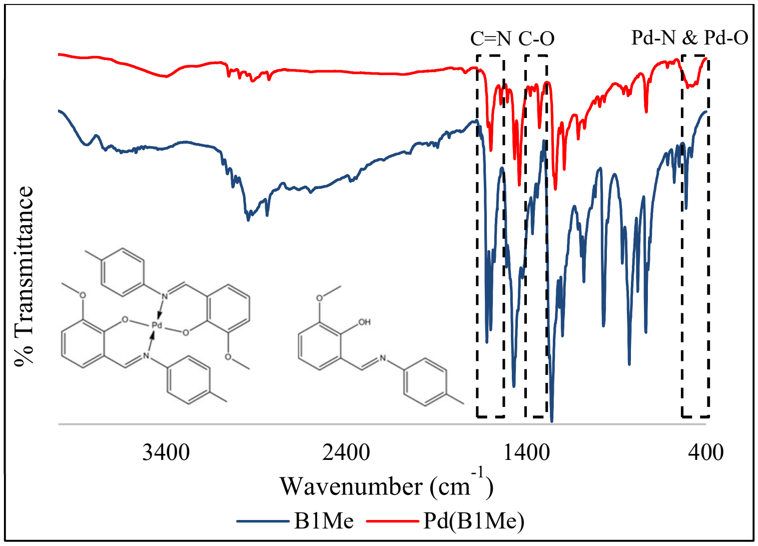
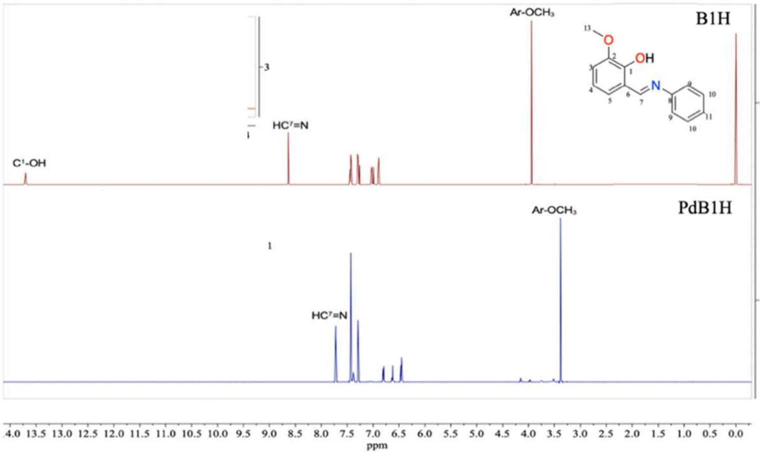


(b)

Figure 1. Infrared Spectra of (a) B1H and PdB1H and (b) B1Me and PdB1Me

Nuclear magnetic resonance (NMR) spectroscopy

The ^{1}H spectra of the ligands and palladium(II) complexes are presented in Figure 2. The ^{1}H NMR spectra showed integrations and multiplicities consistent with the proposed structures. The phenolic proton in ligands appears as a singlet in the downfield region in the range of 13.70 to 13.84 ppm [21]. Upon complexation, the peak of the phenolic proton was absent in both spectra of the complexes suggesting the deprotonation of the phenolic proton [22]. The chemical shift of the azomethine proton (-HC=N-) was shielded at the range of 7.69-7.72 ppm in the complexes compared to the ligands at 8.63 ppm, inferring coordination of the palladium(II) ion to the ligand through the azomethine nitrogen atom [23]. The metal donates the π electrons to the ligands π* orbital, adding the electron density to an antibonding molecular orbital, and this results in weakening of the C-O bond and C=N bond [24]. The spectra of the ligands and complexes show aromatic hydrogen appearing in the ranges of 6.88-7.44 and 6.41-7.32 ppm, respectively. The presence of the methyl groups attached to the structure of B1Me was confirmed by the appearance of singlet peaks at 2.38 ppm and 2.45 ppm at PdB1Me.



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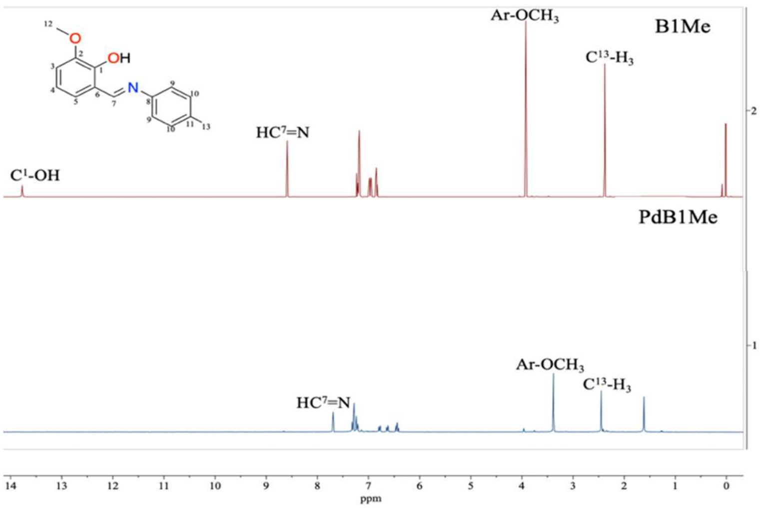
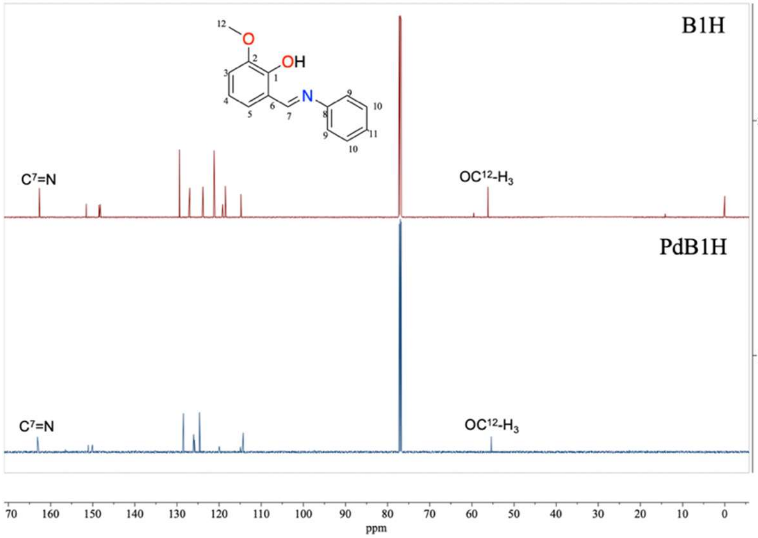


(b)

Figure 2. ^{1}H NMR spectra of (a) B1H and PdB1H and (b) B1Me and PdB1Me

The ^{13}C NMR spectra of the ligands and palladium(II) complexes in Figure 3, respectively recorded signals of sharp peaks representing the chemically non-equivalent carbon atoms in the compound. The phenolic carbon for the ligands was found in the range of 148.5-151.5 ppm and upon complexation, they appeared in the downfield region for complexes. The shifting indicates the establishment of new bond between metal centre and phenolic oxygen. The presence of the methyl group was detected at 22.7 and 19.6 ppm for B1Me and PdB1Me, respectively.



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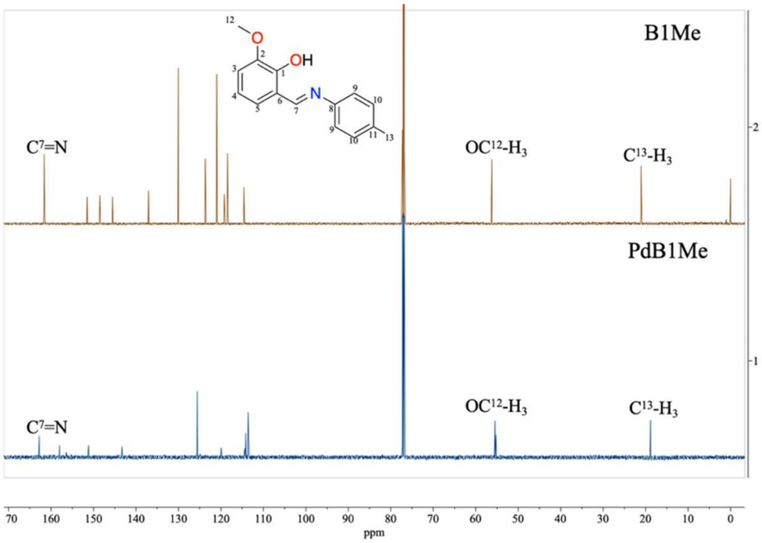


(b)

Figure 3. ^{13}C NMR spectra of (a) B1H and PdB1H and (b) B1Me and PdB1Me

UV-visible spectroscopy

The UV−vis spectroscopy spectra and data of the ligands and palladium(II) complexes are shown in Figure 4 and Table 2. The electronic transitions result in three main characteristic absorption peaks, i.e., π−π*(C=C), π−π*(C=N), and n−π*(C=N). The π−π*(C=C) peak of the ligand was observed at 210-229 nm, and the peak shifted to a bathochromic shift in the complexes, appearing at 220-253 nm for palladium(II) complexes. Besides, the π−π*(C=N) peak at 271-279 nm of the ligands underwent a bathochromic shift upon complexation, appearing at 301-305 nm. It arises due to the coordination of the lone pair of electrons with the metal ion [25]. The n−π* transitions of imine in the complexes were not found in the complexes, possibly due to the stabilization of the electron pair on the nitrogen atom upon the formation of the metal-nitrogen bond [26]. Upon complexation, there is the appearance of ligand to metal charge transfer at 420-421 nm, which suggests the square planar geometry around the Pd(II) centre [27]. Thus, it could be said that the magnetic susceptibility measurements for the PdB1H and PdB1Me, which show diamagnetic behaviour with square planar geometry, are complimentary to the UV-visible data obtained in Table 2.

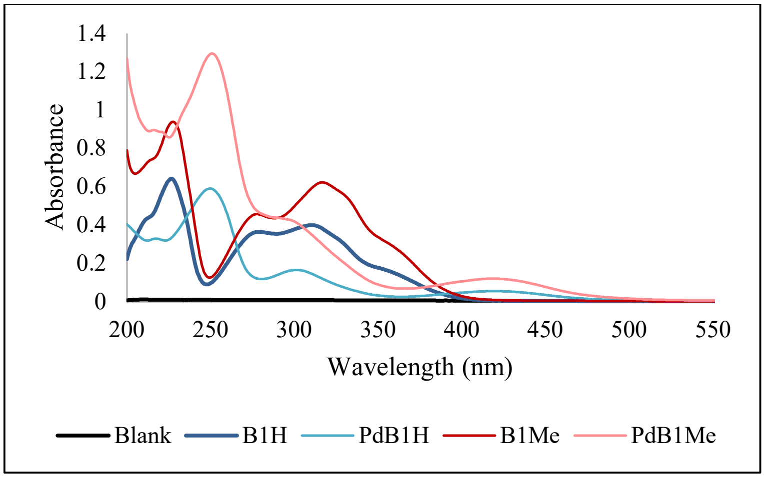


Figure 4. UV-visible spectra of ligands and complexes

Table 2. UV-Visible Data of Ligands and Their Pd(II) Complexes

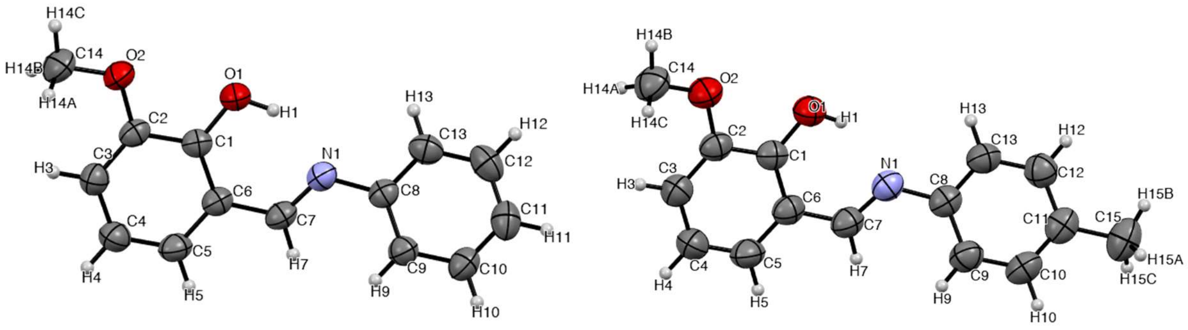
Compounds	**$\pi \rightarrow \pi^*$ (C=C)**	**$\pi \rightarrow \pi^*$ (C=N)**	**$n \rightarrow \pi^*$**	**Ligand to Metal Charge Transfer**
B1H	210 (1386), 225 (2108)	271 (1102)	310 (1331)	-
PdB1H	220 (1080), 253 (1918)	305 (539)	-	421 (183)
B1Me	213 (2441), 229 (3104)	279 (1517)	317 (2074)	-
PdB1Me	220 (2953), 253 (4274)	301 (1379)	-	420 (400)

Single X-ray crystallography

Suitable single crystals of B1H and B1Me were subjected to X-ray crystallography investigation to determine the structure. Both crystals were obtained through slow evaporation of their filtrate solutions. Crystallographic data collection parameters for the compounds are shown in Table 3. B1H and B1Me crystallize in orthorhombic space group $P2_12_12_1$, with *a* = 6.0879(2) Å, *b* = 9.1847(2) Å, and *c* = 21.0661(5) Å. and *a* = 5.9051(9) Å, *b* = 9.2389(13) Å, and *c* = 23.297(3) Å, respectively. The ORTEP diagrams of the compounds drawn at 50% probability ellipsoids are depicted in Figure 5.

Table 3. Crystallographic Data Collection Parameters for B1H and B1Me

Compounds	**B1H**	**B1Me**
CCDC number	2233459	2233467
Empirical formula	$C_{14}H_{13}NO_2$	$C_{15}H_{14}NO_2$
Formula weight	227.27	240.27
Temperature (K)	298.50	299.50
Crystal system	Orthorhombic	Orthorhombic
Space group	$P2_12_12_1$	$P2_12_12_1$
Unit cell dimensions		
a (Å)	6.0879(2) Å	5.9101(14) Å
b (Å)	9.1847(2) Å	9.235(2) Å
c (Å)	21.0661(5) Å	23.303(6) Å
Volume (Å^3)	1177.92 (5) Å^3	1271.9(5) Å^3
Z	4	4
D_{calc} (Mg/m^3)	1.282 Mg/m^3	1.255 Mg/m^3
Absorption coefficient (mm^{-1})	0.086	0.084
F (000)	480.0	512.0
Crystal size (mm^3)	0.500 × 0.320 × 0.150 mm^3	0.39 × 0.27 × 0.13 mm^3
Theta range for data collection	6.96 to 56.6	6.854 to 56.582
Index ranges	$-7 \leq h \leq 8$, $-12 \leq k \leq 12$, $-27 \leq l \leq 28$	$-7 \leq h \leq 7$, $-10 \leq k \leq 12$, $-29 \leq l \leq 30$
Reflections collected	35437	22568
Independent reflections	2933 [R_{int}= 0.0603]	3145 [R_{int}= 0.0532]
Data/restraints/parameters	2933/0/159	3145/0/165
Goodness-of-fit on F^2	0.974	1.031
Final R indices [I > 2sigma(I)]	R1= 0.0400, wR2= 0.1189	R_1 = 0.0434, wR_2 = 0.1187
R indices (all data)	R1= 0.0524, wR2= 0.1314	R_1 = 0.0583, wR_2 = 0.1296
Largest diff. peak and hole	0.15/-0.17	0.29/-0.14



B1H B1Me

Figure 5. The ORTEP diagrams of compounds with the atom labelling scheme drawn at 50% probability ellipsoids

For B1H, the two benzene rings C1-C6 and C8-C13 are each non-planar with a maximum deviation of 0.010(2) and 0.015(2) Å for atoms C1 and C8, respectively, from their mean square planes. Both planes make a dihedral angle of 30.58°. The C-O bond length ranges from 1.352(2) to 1.419(3) Å in the B1H molecule. The C-N and C=N bond lengths of B1H are 1.279(2) and 1.396(3) Å, respectively (Table 4). The bond length values correspond to the previously reported values [28].

For B1Me, the two benzene rings C1-C6 and C8-C13 are each non-planar with maximum deviation of 0.018(2) and 0.012(2) Å for atom C1 and C8, respectively from their mean square planes. Both planes make a dihedral angle of 26.84°. The bond lengths and angles of B1Me are in normal ranges and the values in Table 4 are similar to those in a previous study [29].

Table 4. Selected Bond Lengths (Å) and Bond Angles (°) of B1H and B1Me

	B1H	**B1Me**
Bond Lengths (Å)		
O1—C1	1.352(2)	1.348(2)
O1—C2	1.368(2)	1.362(2)
O2—C14	1.419(3)	1.420(3)
N1—C7	1.279(2)	1.275(3)
N1—C8	1.396(3)	1.414(2)
Bond Angles (°)		
O2—O2—C14	117.12(16)	117.03(18)
C7—N1—C8	120.64(15)	121.31(16)

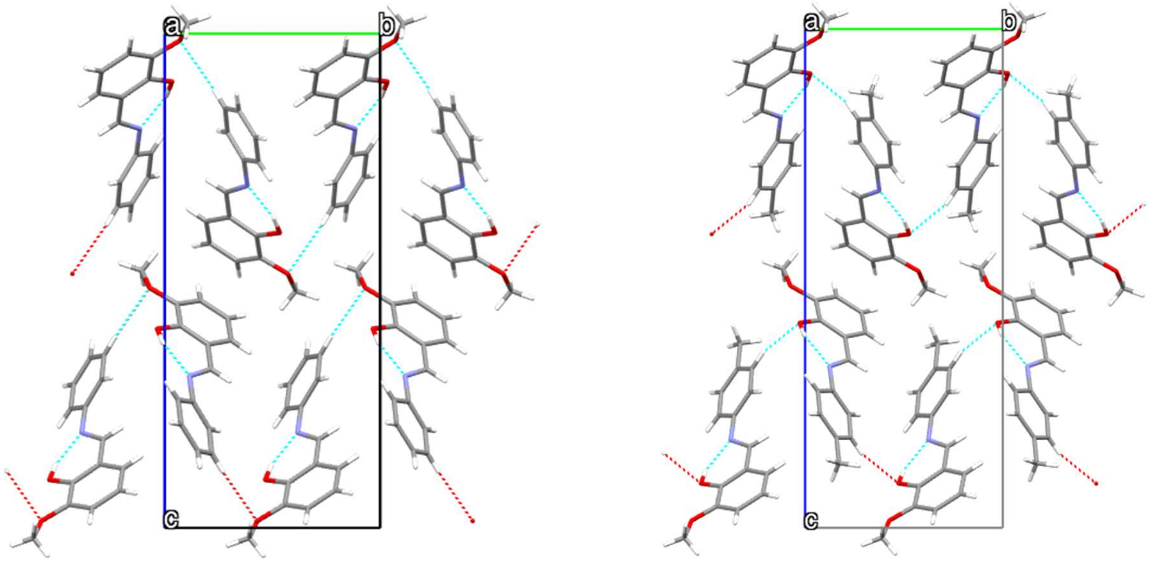
The packing diagram for B1H view down the crystallographic a-axis is shown in Figure 6. There is an intramolecular hydrogen bond O1—H1···N1 and the intermolecular hydrogen bond is drawn as dashed lines at C10—H10···O2 in the compound, as shown in Table 5. The packing diagram for the B1Me view along the a-axis is shown in Figure 6. There is an intramolecular hydrogen bond O1—H1···N1, and the intermolecular hydrogen bond at C10—H10···O1 is drawn as dashed lines in the compound (Table 5).

Table 5. Intramolecular and intermolecular hydrogen bonds of B1H and B1Me (Å, °)

D—H···A	**D—H**	**H···A**	**D ···A**	**D—H···A**
B1H				
O1—H1···N1	0.90(3)	1.83(3)	2.619(2)	146(2)
C10—H10···O2[(a)]	0.93	2.57	3.488(2)	167

D—H···A	**D—H**	**H···A**	**D ···A**	**D—H···A**
B1Me				
O1—H1···N1	0.82	1.90	2.621(2)	146
C10—H10···O1$^{(b)}$	0.93	2.47	3.365(3)	162

Symmetry code= (a): -x,1/2+y,3/2-z, (b): 1-x,-1/2+y,1/2-z



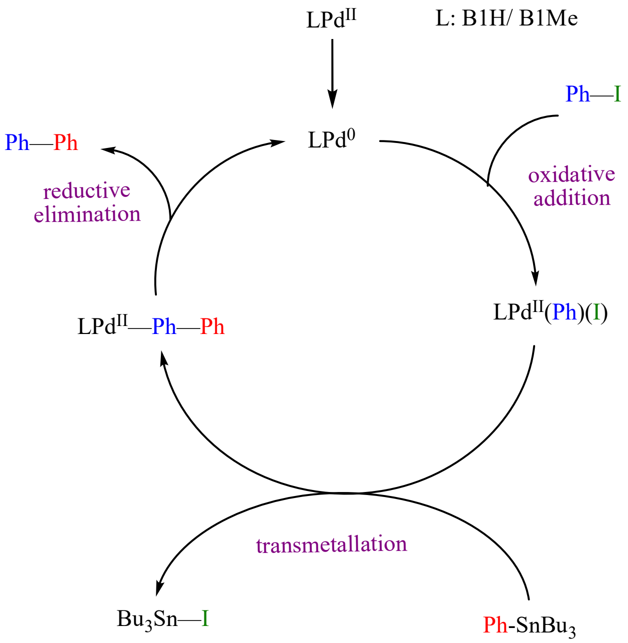
B1H B1Me

Figure 6. Packing B1H and B1Me view down crystallographic a-axis

Catalytic studies

The palladium(II) complexes in this study were screened for their catalytic activity in Stille reaction. The reaction was conducted by reacting iodobenzene with tributylphenyltin. The reaction conditions used were 1.0 mmol% catalyst loading in the presence of triethylamine as a base in dimethyl sulfoxide at 80 °C within 6 hours of the reaction time. Scheme 3 proposes the mechanism of the palladium-catalysed cross-coupling Stille reaction. The three key basic steps involved in the catalytic cycle are oxidative addition, transmetallation and reductive elimination. The mechanism begins with the oxidative addition of the organohalide to the Pd(0) in order to form a Pd(II) complex. Transmetallation with the organotin then proceeds where the R group of the organotin reagent replaces the halide anion on the palladium complex. Reductive elimination then gives the final coupled product, regenerates the palladium catalyst, and the catalytic cycle can recommence.

The reaction was monitored by measuring the % conversion of the aryl iodide starting material using gas chromatography [30]. The results are summarized in Figure 7. It was observed that PdB1Me displayed a better catalytic performance compared to PdB1H for the Stille reaction, as indicated by the 80% conversion of iodobenzene (PdB1Me) while for PdB1H, the % conversion is only 73%, this could be due to the presence of the electron donating group (-CH_3) [31, 32]. The presence of an electron-donating group in ligands will activate the benzene ring, thus as the π system in the benzene ring is activated, the electron density will increase [33]. By increasing the electron density, the formed metal-N bond will be shorter and stronger, thus this will improve the catalytic performance.



Scheme 3. Mechanism of palladium-catalyzed Stille reaction

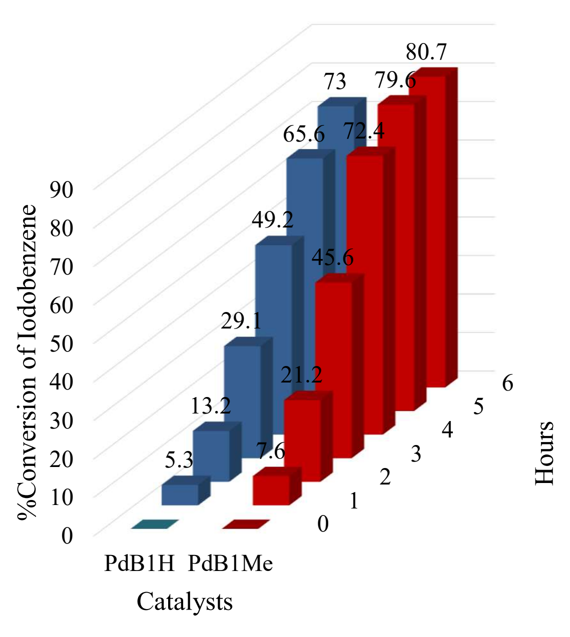


Figure 7. Activity of palladium catalysts on the Stille reaction between iodobenzene and tributylphenyltin

Conclusion

Two ligands and their palladium(II) complexes have been successfully synthesized and confirmed by characterization via various physico-spectral techniques. It can be concluded that the ligands act as bidentate chelators coordinated to the Pd(II) ion upon complexation based on the spectroscopic results and exhibited a square planar geometry. The good agreement between experimental and predicted spectral data of the Schiff bases strongly supported the elucidation of Pd(II) complexes. It was observed that both Pd(II) complexes displayed the properties of good catalysts for the reaction. PdB1Me was the most efficient catalyst for the

Stille reaction, which could be due to the presence of an electron donating group (-CH_3).

Acknowledgement

The authors would like to acknowledge the Ministry of Higher Education (MoHE) for the MyBrainSc scholarship, and Universiti Teknologi MARA for research funding via the grant 600-RMC/YTR/5/3 (007/2020).

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