Synthesis, spectroscopic characterization and catalytic significance of Palladium(II) complexes derived from 1,1 bis(diphenylphosphinomethyl)ethane

Ismail Warad [a],*, Mohammad Azam [a], Usama Karama [a], Saud Al-Resayes [a], Ahmed Aouissi [a], Belkheir Hammouti [b]

[a] Department of Chemistry, King Saud University, P.O. Box 2455, Riyadh 11451, Saudi Arabia
[b] LCAE-URAC18, Faculté des Sciences, Université Mohammed I er B.P. 717, 60000 Oujda, Morocco

ARTICLE INFO

ABSTRACT

Two novel and neutral bidentate Palladium(II) complexes of the type, [PdX$_2$(dpme)] (X = Cl **1**, OAc **2**], have been synthesized from 1,1-bis(diphenylphosphinomethyl)ethene (dpme) ligand and characterized on the basis of elemental analyses, infrared, FAB-MS and ^{1}H-, ^{13}C-, and ^{31}P-NMR spectroscopy. The structure of **1** has been ascertained by single crystal X-ray crystallography and shows *cis* configuration in a square planar structure while the structural behavior of the dpme ligand through the formation of these complexes has been monitored by ^{31}P{^{1}H} NMR and IR spectra. These Pd(II) complexes have shown a very significant catalytic role in Heck reaction giving high yield under mild condition.

© 2011 Elsevier B.V. All rights reserved.

1. Introduction

The coordination chemistry of phosphine ligands have played an important role in the development and the structural design of novel metal complexes and their applications in some of major chemical reaction of homogeneous catalysis since 1960 [1]. Among them, bidentate phosphines such as bis(diphenylphosphino)-methane (dppm), 1,2 bis(diphenylphosphino)ethane, 1,3-bis(diphenylphosphino)propane (dppp) or ether–phosphine (P∼O) have received considerable attention because of their excellent 100% *cis* ligating properties to all most all transition metals [2,3]. Recently, the designs of conformationally rigid polyphosphine ligands for the coordination of late transition metals have been a subject of much interest in organometallic chemistry and homogeneous catalysis. Indeed, decreasing the flexibility of the supporting ligand decrease the number of the possible conformations of the metal ligand assembly, thus leading to improved selectivity. Unlike selectivity, the effect of the structural rigidity on the activity is unpredictable as it mainly depends on the reaction under investigation rather than on the structural and conformational properties of the metal diphosphine precursor [4–7]. Metal complexes containing phosphorus ligands have always been important due to their possible catalytic activity, and variety of them has been reported [8]. Over the last 30 years, Palladium catalyzed coupling reactions have gained immense importance for the carbon-

heteroatom bond formation [9] and are now one of the most valuable catalyzed reaction in organic synthesis [10–13]. In continuation of our ongoing interest in chemistry of 1,1-bis(diphenylphosphinomethyl)ethene (dpme) [14], Herein, we describe the synthesis, structural characterization of Palladium(II) complexes derived from 1,1-bis(diphenylphosphinomethyl)ethene and their catalytic significance in Heck reaction.

2. Experimental

2.1. Materials, and instrumentations

All reactions were carried out in an inert atmosphere (argon) by using standard high vacuum and Schlenk-line techniques unless otherwise noted.

1,1-Bis(diphenylphosphinomethyl)ethene (dpme) was prepared according to literature methods [14]. Ph$_3$P, *n*-BuLi, H$_2$C=(CH$_2$Cl)$_2$ and PdCl$_2$(PhCN)$_2$ and Pd(OAc)$_2$ were available from Merck and ChemPur, respectively, and used as received. Elemental analyses were carried out on an Elementar Varrio EL analyzer. High-resolution ^{1}H, ^{13}C{^{1}H}, DEPT 135, and ^{31}P{^{1}H} NMR spectra were recorded on a Bruker DRX 250 spectrometer at 298 K. FT-IR and FAB-MS data were obtained on a Bruker IFS 48 FT-IR spectrometer and Finnigan 711A (8 kV), modified by AMD and reported as mass/charge (*m/z*), respectively. The analyses of the C—C coupling experiments were performed on a GC 6000 Vega Gas 2 (Carlo Erba Instrument) with a FID and capillary column PS 255 [20 m, carriergas, He(40 kPa), integrator3390A (HewlettPackard)].

* Corresponding author. Tel./fax: +96 61 4675992.
E-mail address: warad@ksu.edu.sa (I. Warad).

Table 1
Crystal data collection and refinement of 1.

Empirical formula	$C_{28} H_{26} Cl_2 P_2 Pd$
Molecular weight	601. 73
Crystal color	Yellow
Crystal description	Block
Crystal system	Monoclinic
Temperature (K)	293(2)
Wavelength (Å)	0.71073
Space group	*P21/c*
Unit cell dimension	
a (Å)	11.194(2)
b (Å)	17.345(4)
c (Å)	13.8043(12)
α (Å)	90.00
β (Å)	92.114(15)
γ (Å)	90.00
Volume (Å³)	2678.6(8)
Z	4
Theta range for data collection (°)	7.901–14.043
F (0 0 0)	1216
D (g cm⁻³)	1.492
Date completeness	0.87369
Absorption coefficient μ (mm⁻¹)	0.98447

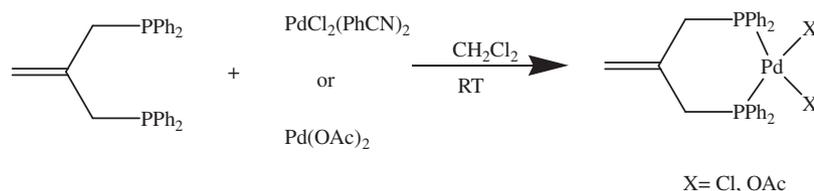## 2.2. Synthesis of complexes, PdCl₂[dpme] 1 and Pd(OAc)₂[dpme] 2

A solution of Pd(II) salt (0.26 mmol) dissolved in 15 ml dichloromethane was added in the 10 ml dichloromethane solution of dpme ligand (0.26 mmol). The resultant reaction mixture was stirred for 10 min resulting a clear yellow colored solution. The resulting solution was kept for evaporation at room temperature leading to the formation of yellow colored microcrystalline solid product which on recrystallisation in dichloromethane yielded the yellow crystals for complex **1** suitable for single crystal diffraction. No crystal for complex **2** was found suitable for single crystal diffraction.

1: Yield: 88%, $^{31}P\{^1H\}$ NMR (CDCl₃): δ (ppm) 16.14 (s, 2P, dpme), ¹H NMR (CDCl₃): δ (ppm) 2.93 (m, 4H, CH₂P), 4.65 (s, 2H, H₂C=C), 7.00–7.82 (m, 20H, Ph—H), ¹³C NMR (CDCl₃): δ (ppm) 37.86, 38.11 (2d, 2C, P—CH₂), 115.55 (s, 1C, H₂C=C), 128.66–133.55 (3 m, 20C, Ph—H), 135.53 (s, 1C, H₂C=C̲), Anal. Found: C, 55.87; H, 4.32; Cl, 11.80%, Calculated, C, 55.79; H, 4.28; Cl, 11.73%.

2: Yield: 90%, $^{31}P\{^1H\}$ NMR (CDCl₃): δ (ppm) 17.22 (s, 2P, dpme), ¹H NMR (CDCl₃): δ (ppm) 2.88 (m, 4H, CH₂P), 3.72 (br, 3H, CH₃O), 4.77 (s, 2H, H₂C=C), 7.00–7.88 (m, 20H, Ph—H), ¹³C NMR (CDCl₃): δ (ppm) 39.21 (br, 2C, PCH₂), 55.54 (s, 2C, CH₃O), 117.21 (s, 1C, H₂C=C), 128.00–134.00 (3 m, 20C, Ph—H), 136.34 (s, 1C, H₂C=C). Anal. Found: C, 59.25; H, 4.93%, Calculated, C, 59.17; H, 4.75%.

2.3. Heck reaction

Pd(II) salt (1.0 μmol), phenyl halide (1.0 mmol), metyl acrylate (1.16 mol), Base (4 mmol), DMF (10 mL) were added to 100 ml Schlenk tube and the reaction mixture was subjected to a freeze–thaw cycle before it heated at 80 °C for 14 h. During the coupling process samples were taken from the reaction mixture using syringe to control the conversion. At the conclusion of the reaction, the mixture was cooled, extracted with ethylacetate–hexane (1:5), filtered through a pad of silica gel with copious washing, concentrated and purified by flash chromatography on silica gel. All reactions were monitored by GC. The purity of the compounds was checked by NMR.

2.4. Crystallography data collection and refinement of 1

The X-ray structure determination of **1** was performed on a Bruker SMART diffractometer, using graphite monochromated Mo Kα radiation (λ = 0.71069). The structure in *P21/c* space group was determined by direct method and refined by full matrix least-square using SHELXL-97 program package. Data reduction and cell refinement were performed by SAINT Program. All calculation was performed using the programs contained in the SHEL-XL-97 packages [15]. Parameters associated with unit cell dimensions, intensity data collection and refinement for the structure of **1** are given in Table 1.

3. Results and discussion

The complexes, **1** and **2** have been synthesized by interacting ligand, 1,1-bis(diphenylphosphinomethyl)ethane with Pd(II) ions in 1:1 M ratio (Scheme 1). Both the isolated complexes are yellow solids, stable at room temperature, soluble in chlorinated solvent such as CH₂Cl₂, CHCl₃ and insoluble in polar and non-polar solvent.

Formation of isolated complexes has been confirmed on the basis of characteristic bands in IR spectra, Uv–vis, elemental analysis, resonance signals in ¹H, ¹³C and ³¹P NMR, molecular ion peak (m/z) in FAB-MS and single crystal X-ray crystallography.
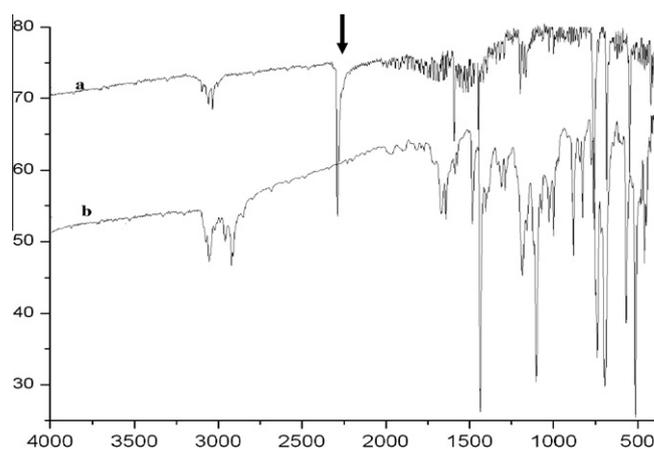

Fig. 1. IR spectra of: (a) PdCl₂(PhCN)₂ before addition of dpme ligand and (b) after the addition to prepare 1.
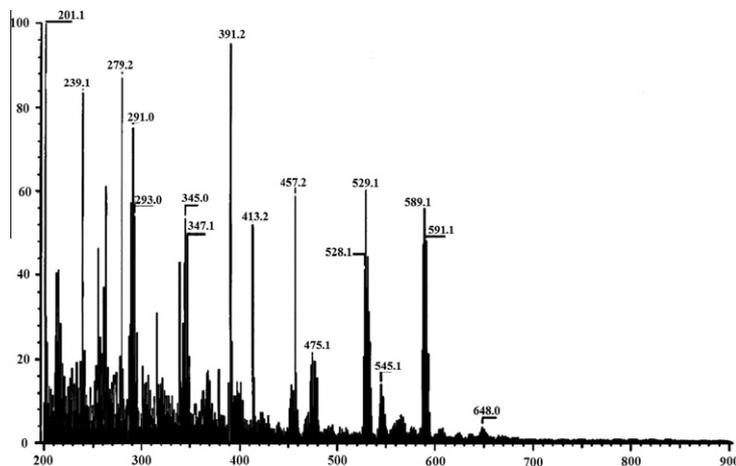

Scheme 1. The synthetic route to prepare 1 and 2 complexes.
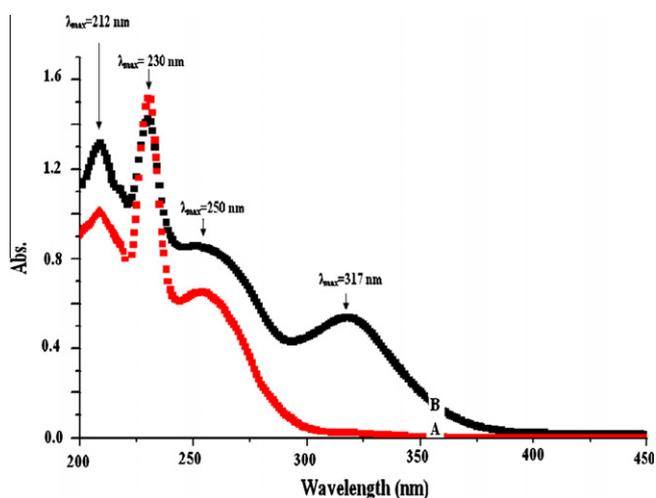
Fig. 2. FAB-MS Spectrum of 2.

Fig. 3. Electronic spectra of free dpme ligand (A), and 1 (B), dissolved in CH_2Cl_2 at.

3.1. IR spectra

The IR spectra of the desired complexes showed characteristic bands at 3265, 3190 cm^{-1}, 2880, 2895 cm^{-1}, 1108, 1115 cm^{-1} and 510, 512 cm^{-1} assigned to υ(C-HPh), υ(C-H alph), υ(C-P) and υ(Pd-P) for **1** and **2** respectively [16]. The carboxalyate bands in complex **2** appeared at around 1550 cm^{-1} while the other bands were found at their expected positions [17] (Fig. 1b). The fully exchange of PhCN in PdCl$_2$(PhCN)$_2$ (Scheme 1) was confirmed by the disappearance of PhCN appearing at 2260 cm^{-1} (Fig. 1a).

3.2. Elemental analysis and FAB-MS

The elemental analyses of these complexes are in a good agreement with the proposed composition which is further confirmed by molecular ion peaks of **1** and **2** observed at 601.2 and 648.1, respectively.

The FAB-MS of complex **2** shows molecular ion peak at 648.1 [M]$^+$ corresponding to its molecular formula $C_{32}H_{32}O_4P_2Pd$. It also shows a series of peaks at (m/z) 589.1 [M-OAc]$^+$, 545.1 [M-OAc-Ac]$^+$ 529.1 [M-2OAc]$^+$, 457.1, corresponding to various fragments (Fig. 2).

3.3. Electronic spectra

The electronic absorption spectra of the complexes **1** and **2** show four intense bands between 200 and 400 nm. The UV re-

gion shows the bands at 212, 230, 250 nm assigned to $\pi -\pi^*$ charge transfer transitions of the dpme ligand similar to the free dpme ligand. A new band observed at 317 nm assignable d–d electron transfer confirms the coordination of the dpme in **1**. (Fig. 3).

3.4. NMR spectra

3.4.1. $^{31}P\{^1H\}$ NMR

$^{31}P\{^1H\}$ NMR spectra of complexes **1** and **2** exhibit singlets at 16.1 ppm and 17.3 ppm respectively, indicating that phosphorus atom of phosphine groups of dpme ligand is coordinated to Pd(II) to give chemically equivalent chelated complexes in solution.

On addition of the PdCl$_2$(PhCN)$_2$ into the dpme ligand solution in NMR tube, intensity of the free dpme $^{31}P\{^1H\}$ NMR signal is gradually decreased while the intensity of complexed dpme $^{31}P\{^1H\}$ NMR signal is gradually increased simultaneously confirming metal–ligand molar ratio 1:1 (Fig. 4). Thus, on the basis of $^{31}P\{^1H\}$ NMR and UV–vis analyses, a square planar geometry has been assigned around [PdCl$_2$(dpme)] complex [16,18,19].

3.4.2. ^1H and $^{13}C\{^1H\}$ NMR spectra

The ^1H and $^{13}C\{^1H\}$ NMR spectra of the complexes show characteristic sets of signals attributed to the aliphatic and aromatic protons and carbons of dpme diphosphine ligand, [CH$_2$P, H$_2$C=C, Ph—H]. An addition signal has been recorded in **2** attributed to CH$_3$ of acetate group. The integral values of the ^1H resonances are in agreement with the compositions.

3.5. Crystallography of PdCl$_2$(dpme) 1

The single crystal of **1** suitable for single crystal diffraction was obtained as yellow block crystals by recrystallization in CH_2Cl_2 solution and crystallized as a monomeric square planar structure (Fig. 5). This is the first time that molecular structure of PdCl$_2$(diphosphine) complex has been established by X-ray crystallography using dpme ligand. Earlier, the structures of same complexes using dpm, dpe, dpp have been reported [19]. The coordination geometry of two phosphorus atoms of dpme ligand and two chorine atoms coordinated to Pd(II) ion is a *cis* configuration as required by the geometry of the bidentate chelating diphosphine ligand. Bond distances and angles are given in Table 2.
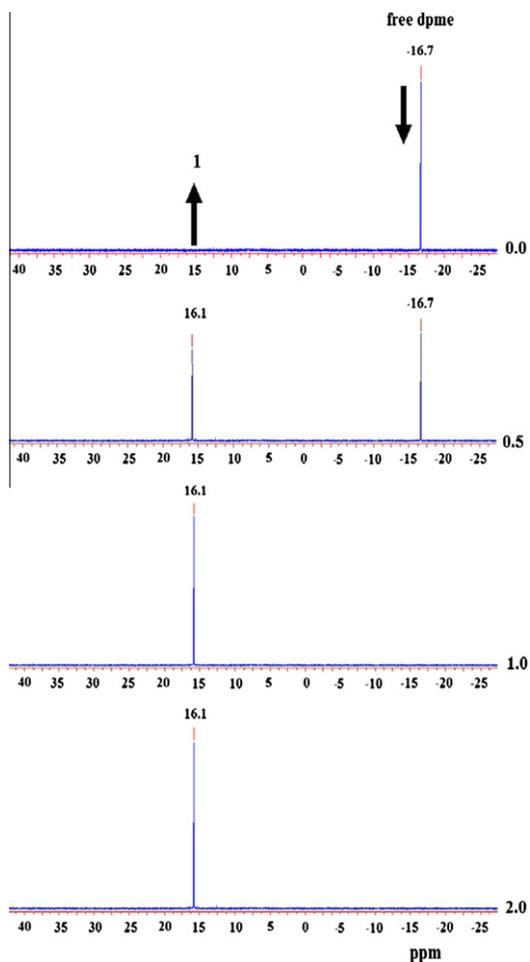
Fig. 4. ³¹P{¹H} NMR spectra at various [PdCl₂(PhCN)₂]/[dpme] mole ratios in CDCl₃ at RT.

3.6. Heck reaction

The Pd-catalyzed Heck reaction is an efficient way to prepare organic derivatives, which are important chemicals for many applications viz., the Heck couplings of metyl acrylate with iodobenzene, bromobenzene, and chlorobenzene to cinnamate. The reactions were performed with NEt_3, K_2CO_3, KOH as a base in DMF at 80 °C for 16 h (Scheme 2) (Table 3).

The choice of bases and solvents are usually important in achieving an efficient cross coupling reaction. The widely used solvents such as DMF, DMSO, toluene, dioxane, THF and CH_3CN had remarkable effects on the coupling reaction [20–23].

In general, all the catalysts studied exhibited a high degree of activity, the expected product being obtained in high yield as in Table 3. No product except cinnamate was observed. The Heck reaction of iodobenzene with methylate in the presence of theses complexes gave better yields under the studied conditions that those of bromobenzene (Table 3, runs 1, 2, 4 and 5), as expected no more than 5% coupling conversion was detected with chlorobenzene (Table 3, runs 3 and 6). Less significant changes in the activity of **1** and **2** was observed under identical Heck reaction condition (Table 3, runs 1–6). Unfortunately, the catalysts are oxygen sensitive and had to be kept under an inert atmosphere in order to avoid deactivation and maintain its high activity, no conversion was detected if non-degassed DMF served as solvent (Table 3, run 7), therefore DMF was dried and deoxygenated prior to use, and the reactions were kept under argon. By allowing oxygen to enter the reaction Schlenk tube during reaction process, no farther more conversion was observed even when reaction lifted for 24 h.

Enhancements in activities were recorded in order KOH > K_2CO_3 ~ NEt_3, respectively with the base strength (Table 3, run 1, 8 and 9). Control experiment indicate that the coupling reactions did not occur in the absence of base (Table 3, run 10). These result are in agreement with the previous studies [24,25].
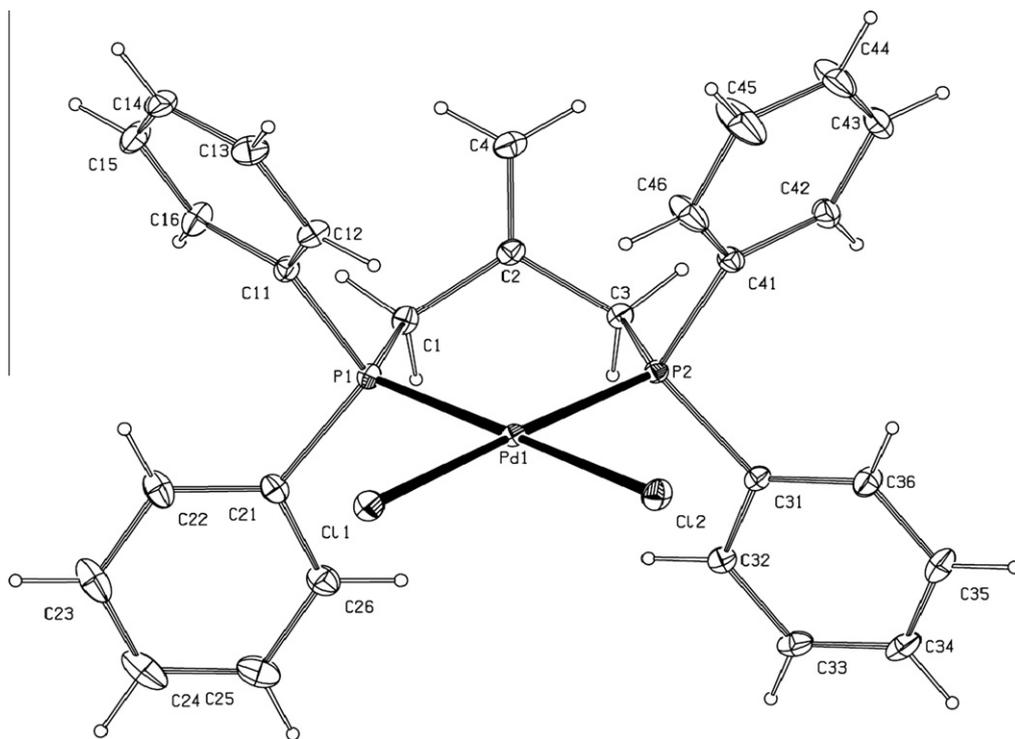

Fig. 5. ORTEP plots of 1 with thermal ellipsoids are drawn at the 50% probability.
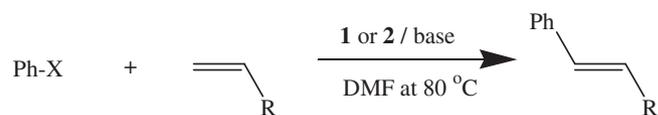
Table 2
Selected bond lengths (Å) and bond angles (°) of 1.

Bond lengths	
Pd(1)—P(2)	2.2458(7)
Pd(1)—P(1)	2.2465(7)
Pd(1)—Cl(1)	2.3490(8)
Pd(1)—Cl(1)	2.3534(7)
Pd(1)—C(l1)	2.3534(7)
P(1)—C(21)	1.811(3)
P(1)—C(11)	1.818(2)
P(1)—C(1)	1.834(2)
P(2)—C(41)	1.807(3)
P(2)—C(31)	1.816(2)
P(2)—C(3)	1.836(2)
C(11)—C(12)	1.377(4)
C(11)—C(16)	1.387(4)
C(12)—C(13)	1.394(4)
C(13)—C(14)	1.364(5)
C(14)—C(15)	1.373(5)
C(15)—C(16)	1.377(4)
C(21)—C(22)	1.382(4)
C(21)—C(26)	1.387(4)
C(22)—C(23)	1.390(5)
C(23)—C(24)	1.364(6)
C(24)—C(25)	1.358(6)
C(25)—C(26)	1.378(5)
C(31)—C(32)	1.380(4)
C(31)—C(36)	1.390(4)
C(44)—C(45)	1.356(5)
C(45)—C(46)	1.383(5)
C(1)—C(2)	1.496(4)
C(2)—C(4)	1.316(4)
Bond angles	
P(2)—Pd(1)—P(1)	96.82(2)
P(2)—Pd(1)—Cl(2)	86.09(3)
P(1)—Pd(1)—Cl(2)	176.45(2)
P(2)—Pd(1)—C(l1)	177.89(2)
P(1)—Pd(1)—C(l1)	85.24(3)
C(l2)—Pd(1)—C(l1)	91.87(3)
C(21)—P(1)—C(11)	108.58(12)
C(21)—P(1)—C(1)	103.22(12)
C(11)—P(1)—C(1)	103.66(12)
C(21)—P(1)—Pd(1)	110.19(9)
C(11)—P(1)—Pd(1)	112.57(9)
C(1)—P(1)—Pd(1)	117.85(8)
C(41)—P(2)—C(31)	109.48(12)
C(41)—P(2)—C(3)	102.23(12)
C(31)—P(2)—C(3)	101.81(11)
C(41)—P(2)—Pd(1)	112.37(8)
C(31)—P(2)—Pd(1)	111.90(8)
C(3)—P(2)—Pd(1)	118.12(8)
C(12)—C(11)—C(16)	118.9(2)
C(12)—C(11)—P(1)	120.4(2)
C(16)—C(11)—P(1)	120.7(2)
C(11)—C(12)—C(13)	120.2(3)
C(14)—C(13)—C(12)	120.2(3)
C(13)—C(14)—C(15)	119.9(3)
C(14)—C(15)—C(16)	120.3(3)
C(15)—C(16)—C(11)	120.5(3)
C(22)—C(21)—C(26)	118.7(3)
C(22)—C(21)—P(1)	124.5(2)
C(26)—C(21)—P(1)	116.8(2)
C(21)—C(22)—C(23)	119.9(4)
C(24)—C(23)—C(22)	120.0(4)
C(25)—C(24)—C(23)	121.0(4)
C(24)—C(25)—C(26)	119.5(4)
C(25)—C(26)—C(21)	120.9(3)
C(32)—C(31)—C(36)	119.2(2)
C(32)—C(31)—P(2)	118.04(19)
C(36)—C(31)—P(2)	122.7(2)
C(31)—C(32)—C(33)	120.5(3)
C(34)—C(33)—C(32)	120.0(3)
C(33)—C(34)—C(35)	120.4(3)
C(34)—C(35)—C(36)	120.3(3)
C(35)—C(36)—C(31)	119.6(3)
C(46)—C(41)—C(42)	118.3(2)
C(46)—C(41)—P(2)	119.9(2)

Table 2 (continued)

Bond lengths	
C(42)—C(41)—P(2)	121.7(2)
C(43)—C(42)—C(41)	121.3(3)
C(44)—C(43)—C(42)	119.5(3)
C(43)—C(44)—C(45)	120.4(3)
C(44)—C(45)—C(46)	120.9(3)
C(41)—C(46)—C(45)	119.5(3)
C(2)—C(1)—P(1)	111.91(17)
C(4)—C(2)—C(1)	123.5(3)
C(4)—C(2)—C(3)	122.0(3)
C(1)—C(2)—C(3)	114.4(2)
C(2)—C(3)—P(2)	111.71(16)

Ph-X   +   =⟨R   →[**1** or **2** / base][DMF at 80 °C]   Ph⟩=⟨R

X = Cl, Br, I, R = CO₂ Me

Scheme 2. The Heck coupling reaction of benzene halide with metyl acrylate to prepare cinnamate.

Table 3
The Heck coupling reaction of benzene halaide with metyl acrylate.

Run	PhX	Complex	Base	Yield[a]
1	PhI	**1**	KOH	>99
2	PhBr	**1**	KOH	86
3	PhCl	**1**	KOH	5
4	PhI	**2**	KOH	98
5	PhBr	**2**	KOH	84
6	PhCl	**2**	KOH	4
7	PhI	**1**	KOH	0[b]
8	PhI	**1**	K₂CO₃	75
9	PhI	**1**	NEt₃	73
10	PhI	**1**	–	0[c]

[a] *Reaction conditions*: Pd(II) 1.0 μmol, phenyl halide 1 mmol, metyl acrylate 1.16 mmol, Base 4 mmol, in 10 mL DMF at 80 °C for 14 h, isolated yields are based on phenyl halide, TON = 1000, all reactions were monitored by GC.
[b] Non-degassed solvent was used,
[c] No-base was added.

4. Conclusions

Two novel and neutral Palladium(II) complexes have been prepared using 1,1-bis(diphenylphosphinomethyl)ethene (dpme) ligand. The ligand exchange through these complexes have been monitored by $^{31}P\{^1H\}$-NMR and IR and characterized by elemental analyses, infrared, FAB-MS and ^1H-, ^{13}C-, H, and ^{31}P-NMR spectroscopy data. Complex **1** was additionally characterized by X-ray crystallography.

These complexes showed efficient catalytic activity under mild basic conditions when subjected as catalysts to C—C Heck cross coupling of metyl acrylate with phenyl halide to produce cinnamate.

Supplementary material

Crystallographic data has been deposited with the Cambridge Crystallographic Data Centre as supplementary publication number CCDC 816387. Copies of this information may be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html (or

from the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk).

References

[1] W. Hewerston, H.R. Watson, J. Chem. Soc. 1490 (1962).
[2] R.J. Puddephatt, Chem. Soc. Rev. 12 (1983) 99.
[3] (a) B. Chavdret, B. Delavaux, R. Poilblanc, Coord. Chem. Rev. 86 (1988) 191;
    (b) I. Warad, Z. Kristallogr. NCS 225 (2010) 753.;
    (c) M.A. Khanfar, I. Warad, M.A. Al-Damen, Acta Cryst. E66 (2010) 731;
    (d) I. Warad, Molecules 15 (2010) 4652.
[4] A.J. Hierso, R. Amardeil, E. Bentabet, R. Broussier, E. Bentabet, R. Broussier, B. Gautheron, P. Meunier, P. Kalck, Coord. Chem. Rev. 236 (2003) 143.
[5] R.H. Crabtree, The Organometallic Chemistry of Transition Metals, 3rd ed., John-Wiley and Sons, New York, 2001.
[6] Z. Freixa, PW.N.M. Van Leeuwen, Dalton Trans (2003) 1890.
[7] K.L. Arthur, Q.L. Wang, D.M. Bregel, N.A. Symthe, B.A. O'Neil, K.I. Goldberg, K.G. Moloy, Organometallics 24 (2005) 4624.
[8] (a) E. Linder, H.A. Mayer, I. Warad, K. Eichele, J. Organomet. Chem. 665 (2003) 176;
    (b) I. Warad, E. Lindner, K. Eichele, A.H. Mayer, Inorg. Chim. Acta 357 (2004) 1847;
    (c) J.K. Whitesell, Chem. Rev. 89 (1989) 1581;
    (d) J.M. Brown, Chem. Soc. Rev. 22 (1993) 25.
[9] A.S. Guram, R.A. Rennels, S.L. Rennels, S.L. Buchwald, Angew. Chem. Int. Ed. Engl. 34 (1995) 2379.
[10] R.F. Heck, Palladium Reagents in Organic Synthesis, Academic Press, London, 1985.
[11] A. demeijere, F.E. Meyer, Angew. Chem. Int. Ed. Engl. 33 (1994) 2379.
[12] J. Jeffery, Adv. Met-Org. Chem. 5 (1996) 153.
[13] W.A. Herrmann, V.P.W. Boehm, C.-P. Reisinger, J. Organomet. Chem. 576 (1999) 23.
[14] I. Kh. Warad, M.R.H. Siddiqui, S. Al-Resayes, A. Al-Warthan, R. Mahfouz, Trans. Met. Chem. 34 (2009) 347.
[15] G.M. Sheldrick, SHELXS-97, University of Göttingen, Göttingen, Germany, 1997.
[16] D.C. Gilheany, C.M. Mitchell, in: F.R. Hartley (Ed.), The Chemistry of Organophosphorus Compounds, Wiley, New York, 1990, p. 151.
[17] . Tsutomu Ishioka, Youko Shibata, Mizuki Takahashi, Isao Kanesaka, Yasuyuki Kitagawa, Kazuo T. Nakamura, Spectrochim. Acta Part A 54 (1998) 1827–1836.
[18] L. Chahen, B. Therrien, G. Suess-Fink, J. Organomet. Chem. 691 (2006) 4257.
[19] W.L. Steffen, G.J. Palenik, Inorg. Chem. 15 (1976) 2432.
[20] X-J. Yu, R. Zhou, Y. Zhang, H.-Y. Fu, R.-X. Li, H. Chen, X.-J. Li, Catal. Commun. 12 (2010) 222.
[21] A.M. Trzeciak, J.J. Ziolkowski, Coord. Chem. Rev. 249 (2005) 2308.
[22] M. Aydemir, F. Durap, A. Baysal, O. Akba, B. Guemgüma, S. Oezkar, L.T. Yıldırım, Polyhedron 28 (2009) 2313.
[23] M. Shiotsuka, T. Tanamachi, T. Urakawa, Y. Matsuda, J. Mol. Struct. 794 (2006) 215.
[24] F. Alono, I.P. Beletskaya, M. Yus, Tetrahedron 61 (2005) 11771. Fig. 5.
[25] G.P. Chiusoli, M. Catellani, M. Costa, E. Motti, N.D. Ca, G. Maestri, Coord. Chem. Rev. 254 (2010) 456.