

Synthesis and Characterization of Ag(I) and Pd(II) Complexes of Some Phosphorus Ylides

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Reaction of three previously reported phosphorus ylides $\text{Ph}_3\text{PCHCOC}_6\text{H}_4\text{CH}_3$ (Y^1), $\text{Ph}_3\text{PCHCOC}_6\text{H}_4\text{NO}_2$ (Y^2) and $\text{Ph}_3\text{PCHCOC}_6\text{H}_4\text{Cl}$ (Y^3) with AgNO_3 in 1:2 molar ratios using dichloromethane as solvent yielded mononuclear complexes of the type $[(\text{Y})_2\text{Ag}]\text{NO}_3$ ($\text{Y} = \text{Y}^1$ (**1**), Y^2 (**2**) and Y^3 (**3**)). The reaction of PdCl_2 with (Y^2) in equimolar ratio in acetonitrile at the reflux temperature gave the complex $[\text{PdCl}(\mu\text{-Cl})\text{Ph}_3\text{PCHCOC}_6\text{H}_4\text{NO}_2]_2$ (**4**) along with the phosphonium salt $[\text{Ph}_3\text{PCH}_2\text{COC}_6\text{H}_4\text{NO}_2]\text{Br}$. The same reaction in a 1:2 molar ratio with (Y^1) at the reflux temperature gave a binuclear complex of the type $[\text{Pd}\{\text{CH}\{\text{P}(\text{C}_6\text{H}_4)(\text{Ph}_3)_2\}\text{COC}_6\text{H}_4\text{CH}_3\}(\mu\text{-Cl})_2]$ (**5**) along with the phosphonium salt $[\text{Ph}_3\text{PCH}_2\text{COC}_6\text{H}_4\text{CH}_3]\text{Br}$. This complex reacts with PPh_3 in a 1:2 molar ratio in dichloromethane to give the product of bridge-splitting $[\text{Pd}\{\text{CH}\{\text{P}(\text{C}_6\text{H}_4)(\text{Ph}_3)_2\}\text{COC}_6\text{H}_4\text{CH}_3\}\text{Cl}(\text{PPh}_3)]$ (**6**). The characterization of these compounds was carried out by IR, ^1H , ^{31}P and ^{13}C NMR spectroscopy and elemental analysis.

Key Words: Orthopalladation, Silver(I) nitrate, Phosphorus ylide, Triphenylphosphine.

INTRODUCTION

Phosphorus ylides are important reagents in organic chemistry, especially in the synthesis of naturally-occurring products with biological and pharmacological activities¹. The utility of metalated phosphorus ylides in synthetic chemistry has been well documented²⁻⁵. Juxtaposition of the keto group and carbanion in phosphorus ylides allows for the resonance delocalization of the ylidic electron density while providing additional stabilization to the ylide species. The α -keto-stabilized phosphorus ylides $\text{R}_3\text{P}=\text{CR}'\text{COR}''$ (R , R' and R'' = alkyl or aryl groups) show interesting properties such as their high stability and their ambidentate character as ligands (C- versus O-coordination).

This ambidentate character can be rationalized in terms of the resonance forms A-C, together with the isomeric form D (Fig. 1). Form B can be considered as leading to coordination by the carbon atom to give a complex of form E, whereas isomers C and D would both lead to coordination by the oxygen atom, affording structures F (*transoid*) and G (*cisoid*), respectively. Although many coordination

modes are possible for keto ylides⁶, coordination through carbon is more predominant and observed with soft metal ions, *e.g.* Pd(II), Pt(II), Ag(I), Hg(II), Au(I) and Au(III)⁷⁻¹¹, whereas O-coordination dominates when the metals involved are hard, *e.g.*, Ti(IV), Zr(IV) and Hf(IV)¹². Only W(0) complexes of the type W(CO)₅L (L = ylide)¹³ and Pd(II) complexes of stoichiometry [Pd(C₆F₅)L₂](APPY)(ClO₄)⁸ [APPY = Ph₃PCHCOMe; L = PPh₃ and PBU₃; L₂ = bipy] contain stable ylides O-linked to a soft metal centre. In this work, we describe the synthesis and spectroscopic (IR and NMR) characterization of Ag(I) and Pd(II) complexes of the above ylides.

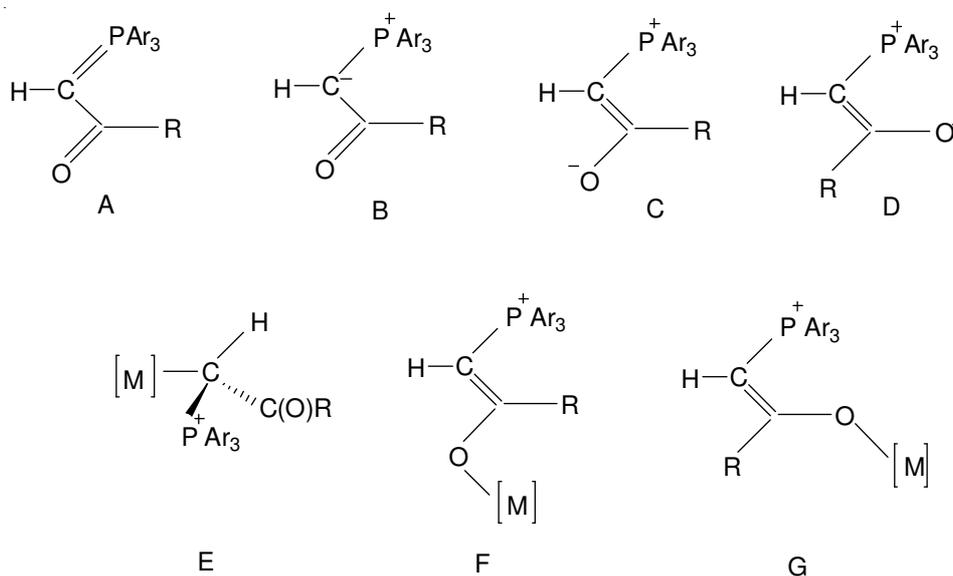


Fig. 1. Structures of phosphorus ylides and their metal complexes

EXPERIMENTAL

Dichloromethane was distilled over magnesium powder and diethyl ether was distilled over a mixture of sodium and benzophenone just before use. All other solvents were reagent grade and used without further purification. Elemental analysis for C, H and N were performed using a Perkin-Elmer 2400 series analyzer. IR spectra were recorded on a Shimadzu 435-U-04 spectrophotometer in the region 4000-550 cm⁻¹ and the measurements were made by the KBr disk method. Melting points were measured on a SMP3 apparatus. Solution-state ¹H, ³¹P and ¹³C NMR spectra at ambient temperature were obtained in DMSO-*d*₆ or CDCl₃ using a FT-NMR Bruker 500 (500 MHz) and FT-NMR JEOL FX 90 Q spectrometers. Chemical shifts (ppm) are reported according to internal TMS and external 85 % phosphoric acid. Coupling constants *J* are given in Hz. All of the ligands were prepared based on the published methods^{14,15}.

Synthesis of [(Y¹)₂Ag]NO₃ (1) general procedure: To a solution of AgNO₃ (0.06 g, 0.38 mmol) in dichloromethane (15 mL) was added Y¹ (0.3 g, 0.76 mmol) and stirred for 12 h. The solution was protected from light with aluminium foil. A white product formed by slow evaporation of the solvent and concentrated to 2 mL. The product was washed several times with petroleum ether. Yield: 0.266 g (74 %). m.p. 185-187 °C. Anal. calcd. (found) (%) for C₅₄H₄₆AgNO₅P₂: C, 67.64 (67.32); H, 4.83 (5.13); N, 1.46 (1.28). IR (KBr, ν_{\max} , cm⁻¹): 1598 (CO) and 863 (P-C). ¹H NMR (CDCl₃) δ_{H} ppm: 2.27 (s, 3H, CH₃), 5.19 (br, 1H, CH) and 6.98-7.91 (m, 19H, arom.). ³¹P NMR (CDCl₃) δ_{P} ppm: 21.72 ppm. ¹³C NMR (CDCl₃) δ_{C} ppm: 20.74 (s, CH₃), 36.65 (d, CH, ¹J_{PC} = 71.39), 124.23 (d, ¹J_{PC} = 89.86, PPh₃ (i)); 132.25 (d, ²J_{PC} = 8.83, PPh₃ (o)); 128.61 (d, ³J_{PC} = 12.57, PPh₃ (m)); 134.38 (d, ⁴J_{PC} = 10.31, PPh₃ (p)); 141.23 (s, CPh(i)); 132.05 (s, CPh(o)); 126.89 (s, CPh(m)); 128.33 (s, CPh(p)); 190.30 (s, CO).

Data for [(Y²)₂Ag]NO₃ (2): Yield: 0.213 g (71 %). m.p. 196-199 °C. Anal. calcd. (found) (%) for C₅₂H₄₀AgN₃O₉P₂: C, 61.18 (61.49); H, 3.95 (4.35); N, 4.11 (4.31). IR (KBr, ν_{\max} , cm⁻¹): 1618 (CO) and 872 (P-C). ¹H NMR (CDCl₃) δ_{H} ppm: 5.15 (d, 1H, CH, ¹J = 8.24) and 7.24-8.09 (m, 19H, arom.). ³¹P NMR (CDCl₃) δ_{P} ppm: 20.75. ¹³C NMR (CDCl₃) δ_{C} ppm: 40.33 (d, CH, ¹J_{PC} = 80.65), 124.48 (d, ¹J_{PC} = 90.08, PPh₃ (i)); 132.82 (d, ²J_{PC} = 9.88, PPh₃ (o)); 129.22 (d, ³J_{PC} = 12.66, PPh₃ (m)); 128.33 (s, PPh₃ (p)); 143.23 (d, ³J_{PC} = 11.60 CPh(i)); 133.04 (s, CPh(o)); 123.12 (s, CPh(m)); 149.15 (s, CPh(p)); 187.73 (s, CO).

Data for [(Y³)₂Ag]NO₃ (3): Yield: 0.231 g (77 %). m.p. 196-199 °C. Anal. calcd. (found) (%) for C₅₂H₄₀AgCl₂NO₅P₂: C, 62.47 (62.12); H, 4.03 (4.41); N, 1.40 (1.62). IR (KBr, ν_{\max} , cm⁻¹): 1609 (CO) and 861 (P-C). ¹H NMR (CDCl₃) δ_{H} ppm: 5.19 (br, 1H, CH) and 7.14-8.00 (m, 19H, arom.). ³¹P NMR (CDCl₃) δ_{P} ppm: d 21.15. ¹³C NMR (CDCl₃) δ_{C} ppm: 37.68 (d, CH, ¹J_{PC} = 74.07), 124.49 (d, ¹J_{PC} = 89.90, PPh₃ (i)); 132.52 (d, ²J_{PC} = 9.16, PPh₃ (o)); 128.88 (d, ³J_{PC} = 12.32, PPh₃ (m)); 135.96 (d, ⁴J_{PC} = 10.92, PPh₃ (p)); 137.00 (s, CPh(i)); 132.31 (s, CPh(o)); 127.91 (s, CPh(m)); 128.60 (s, CPh(p)); 189.05 (s, CO).

Synthesis of [PdCl(μ-Cl)Ph₃PCHCOC₆H₄NO₂]₂ (4): To a saturated solution of PdCl₂ (0.08 g, 0.46 mmol) in acetonitrile (15 mL) at the reflux temperature, was added solid Y² (0.2 g, 0.46 mmol) and the suspension was refluxed for 15 min. The resulting orange solution was concentrated (2 mL) and diethyl ether (20 mL) was added to precipitate the orange solid (4). Yield 0.210 g (75 %). m.p. 165-168 °C. Anal. calcd. (found) (%) for C₅₂H₄₀Cl₄N₂O₆P₂Pd: C, 56.82 (57.29); H, 3.66 (4.02); N, 2.54 (2.92). IR (KBr, ν_{\max} , cm⁻¹): 1650 (CO), 857(P-C). ¹H NMR (CDCl₃) δ_{H} (ppm): 5.60 (br, 1H, CH), 7.21-8.55 (19H, m, arom.). ³¹P NMR (CDCl₃) δ_{P} (ppm): 23.51. ¹³C NMR (DMSO-*d*₆) δ_{C} (ppm): 41.28 (d, ¹J_{PC} = 79.56 Hz, CH), 122.26-149.85 (m, arom.), 195.16 (CO).

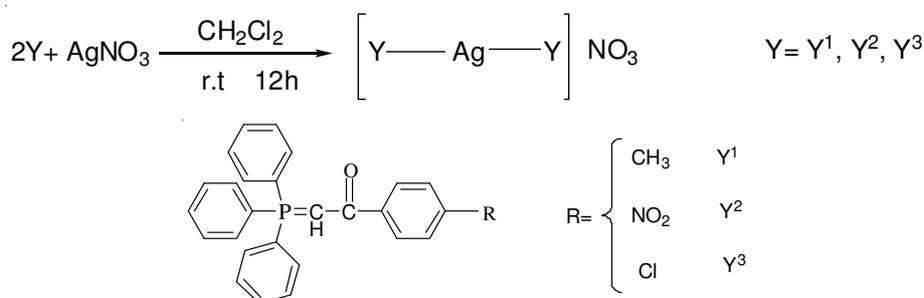
Synthesis of [Pd{CH{P(C₆H₄)(Ph₃)₂}COC₆H₄CH₃}(μ-Cl)]₂ (5): To a suspension of PdCl₂ (0.04 g, 0.25 mmol) in acetonitrile (20 mL) was added solid Y¹ (0.2 g, 0.5 mmol). The mixture was refluxed for 3 h and then allowed to cool to room tempe-

perature. The suspension was filtered and the solid was washed with diethyl ether (20 mL) to give a yellowish-orange solid as product **5**. The phosphonium salt $[\text{Ph}_3\text{PCH}_2\text{COC}_6\text{H}_4\text{CH}_3]\text{Br}$ separated, as a byproduct, *via* the mother liquor by washing solid **5**. Yield 0.190 g (78 %). m.p. 273-275 °C, Anal. calcd. (found) (%) for $\text{C}_{54}\text{H}_{44}\text{Cl}_2\text{O}_2\text{P}_2\text{Pd}_2$: C, 60.57 (60.99); H, 4.14 (4.60). IR (KBr, ν_{max} , cm^{-1}): 1622 (CO), 877 (P-C).

Synthesis of $[\text{Pd}\{\text{CH}\{\text{P}(\text{C}_6\text{H}_4)(\text{PH}_3)_2\}\text{COC}_6\text{H}_4\text{CH}_3\}\text{Cl}(\text{PPh}_3)]$ (6**):** To a suspension of complex **5** (0.06 g, 0.06 mmol) in dichloromethane (20 mL) was added solid PPh_3 (0.03 g, 0.12 mmol). The mixture was stirred for 0.5 h. The resulting colourless solution was concentrated (2 mL) and diethyl ether (30 mL) was added to precipitate the white solid **6**. Yield 0.082 g (81 %). m.p. 217-220 °C Anal. calcd. (found) (%) for $\text{C}_{45}\text{H}_{37}\text{ClOP}_2\text{Pd}$: C, 67.76 (67.98); H, 4.67 (5.17). IR (KBr, ν_{max} , cm^{-1}): 1654 (CO), 864 (P-C). ^1H NMR (CDCl_3) δ_{H} (ppm): 2.32 (s, 3H, CH_3), 5.45 (dd, $^2J_{\text{PH}} = 8.78$ Hz, $^3J_{\text{PH}} = 8.69$ Hz, 1H, CH), 6.88-8.34 (33H, m, arom.). ^{31}P NMR (CDCl_3) δ_{P} (ppm): 12.26 (PPh_3) and 28.67 (PPh_2) (2d, $^3J_{\text{PP}} = 19.70$ Hz). ^{13}C NMR ($\text{DMSO}-d_6$) δ_{C} (ppm): 21.92 (s, CH_3), 36.04 (d, $^1J_{\text{PC}} = 62.88$ Hz, CH), 119.71-146.65 (m, arom.), 192.70 (CO).

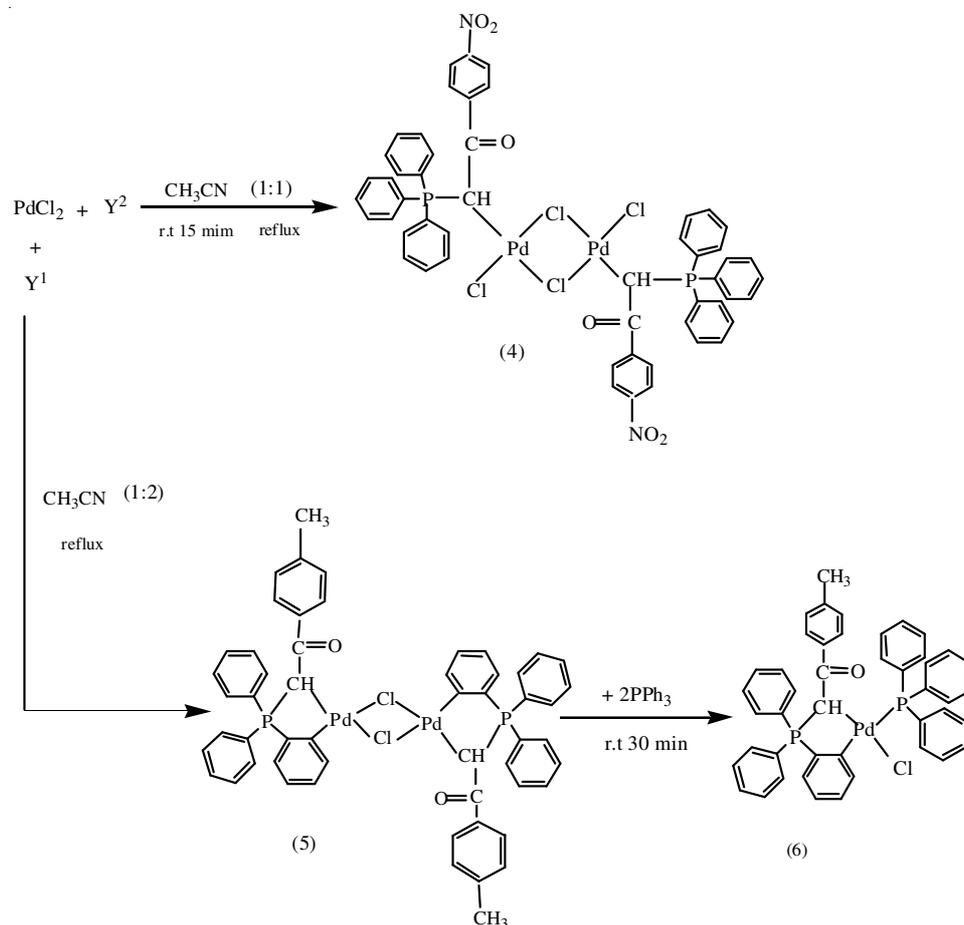
RESULTS AND DISCUSSION

The reaction of ylides with AgNO_3 in 1:2 molar ratios in dichloromethane as solvent yielded mononuclear complexes **16** (Scheme-I).



Scheme-I

On the other hand, the presence of only one chiral C centre in **4** is shown in the ^1H , ^{31}P and ^{13}C NMR spectra by one set of signals for CH, CO and PCH groups. Thus there are not two geometries of *cis* and *trans* and perhaps a binuclear complex of the type $[\text{PdCl}(\mu\text{-Cl})\text{Y}^2]_2$ is formed¹⁷ (Scheme-II). An NMR study could not be performed on complex **5** because it is highly insoluble in the most common solvents. However its elemental analysis, IR spectra and the synthesis of **6** from complex **5** adequately establish its identity, thus we propose for **5** the same structure as reported for the related complexes¹⁸⁻²⁴ (Scheme-II). The bridge-splitting reaction of **5** with PPh_3 is highly selective, affording exclusively the corresponding mononuclear compound **6** (Scheme-II).



Scheme-II

The IR data of the ligands as well as those of the corresponding metal complexes are listed in Table-1.

The $\nu(\text{CO})$ band, which is sensitive to complexation, occurs at 1519, 1529 and 1579 cm^{-1} in the parent ylides Y^1 , Y^2 and Y^3 , respectively^{14,15}. Coordination of ylide through the carbon atom causes an increase in the $\nu(\text{CO})$ band, whereas for O-coordination a lowering of the $\nu(\text{CO})$ band is expected¹¹. Thus the IR absorption bands for the complexes at higher frequencies indicate that C-coordination has occurred. The $\nu(\text{P-C})$ band frequencies, which are also diagnostic of the coordination mode, occur at 885, 884 and 882 cm^{-1} in the parent ylides Y^1 , Y^2 and Y^3 , respectively and are shifted to lower frequencies for the complexes, suggesting some removal of the electron density of the P-C bonds³⁰.

In the ^1H NMR spectra for **1**, **3** and **4**, the signals due to the methinic proton are broad. This broadening is probably due to very low solubility of the complexes in CDCl_3 as solvent. The expected downfield shifts of ^{31}P and ^1H signals for PCH

TABLE-1
 $\nu(\text{CO})$ OF PHOSPHORANES AND THEIR Ag(I) AND Pd(II) COMPLEXES

Compound	$\nu(\text{CO})$ (cm^{-1})	Ref.
$\text{Ph}_3\text{PCHCON}(\text{CH}_3)_2$	1530	[25]
$\text{Ph}_3\text{PCHCOCH}_3$ (APPY)	1530	[26]
$\text{Ph}_3\text{PCHCOPh}$ (BPPY)	1525	[27]
$\text{Ph}_3\text{PCHCOC}_6\text{H}_4\text{CH}_3$ (Y^1)	1519	[14]
$\text{Ph}_3\text{PCHCOC}_6\text{H}_4\text{NO}_2$ (Y^2)	1529	[15]
$\text{Ph}_3\text{PCHCOC}_6\text{H}_4\text{Cl}$ (Y^3)	1579	[15]
C-coordination		
$[(\text{Y}^1)_2\text{Ag}]\text{NO}_3$	1598	This work
$[(\text{Y}^2)_2\text{Ag}]\text{NO}_3$	1618	This work
$[(\text{Y}^3)_2\text{Ag}]\text{NO}_3$	1609	This work
$[\text{PdCl}(\mu\text{-Cl})\text{Ph}_3\text{PCHCOC}_6\text{H}_4\text{NO}_2]$	1650	This work
$[\text{Pd}\{\text{CH}\{\text{P}(\text{C}_6\text{H}_4)(\text{Ph}_3)_2\}\text{COC}_6\text{H}_4\text{CH}_3\}(\mu\text{-Cl})_2]$	1622	This work
$[\text{Pd}\{\text{CH}\{\text{P}(\text{C}_6\text{H}_4)(\text{Ph}_3)_2\}\text{COC}_6\text{H}_4\text{CH}_3\}\text{Cl}(\text{PPh}_3)]$	1654	This work
$[\text{Pd}\{\text{CH}\{\text{P}(\text{C}_7\text{H}_6)(p\text{-tolyl})_2\}\text{COCH}_3\}(\mu\text{-Cl})_2]$	1645	[28]
$[\text{Pd}\{\text{CHP}(\text{C}_6\text{H}_4)\text{Ph}_2\text{CO}_2\text{CH}_2\text{Ph}\}(\mu\text{-Cl})_2]$	1695	[28]
$[\text{Pd}\{\text{CH}\{\text{P}(\text{C}_7\text{H}_6)(p\text{-tolyl})_2\}\text{COCH}_3\}\text{Cl}\{\text{P}(p\text{-tolyl})_3\}]$	1641	[28]
$[(\text{Ph}_3\text{PCHCOC}_{10}\text{H}_7)_2\text{Ag}]\text{NO}_3$	1603	[16]
O-coordination		
$[(\text{Sn}(\text{CH}_3)_3\text{BPPY})\text{Cl}]$	1480	[29]
$[\text{Pd}(\text{C}_6\text{F}_5)(\text{PPh}_3)(\text{APPY})]\text{ClO}_4$	1513	[12]

group upon complexation were observed in their corresponding spectra. The appearance of a sharp doublet signal and expected downfield shift for CH proton show the coordination of carbon through methine group. The proton decoupled $^{31}\text{P}\{^1\text{H}\}$ -NMR spectra show only one sharp singlet between $\delta \approx 20.75\text{-}23.51$ ppm in the complexes **1**, **2**, **3** and **4**. Chemical shift values for these complexes, appear to be downfield by about $\delta \approx 6\text{-}9$ ppm with respect to the parent ylides^{14,15}, indicating the coordination of the ylides have occurred. The ^1H NMR spectra of complex **6** show two doublet for the CH group that must arise from the simultaneously coupling with two phosphorus centres

In ^{13}C NMR spectra, a higher shielding of the ylidic carbon atoms compared to the parent ylides is expected and we observed this in all of mentioned complexes. Such a higher shielding was observed in $\text{PdCl}(\eta^3\text{-}2\text{-XC}_3\text{H}_4)(\text{C}_6\text{H}_5)_3\text{PCHCOR}$ ($\text{X} = \text{H}, \text{CH}_3$; $\text{R} = \text{CH}_3, \text{C}_6\text{H}_5$) and is due to the change in hybridization from sp^2 to sp^3 of the ylidic carbon atom on coordination³¹. Similar upfield shifts of 2-3 ppm with reference to the parent ylide were also observed in the case of $[(\text{C}_6\text{H}_5)_3\text{PC}_5\text{H}_4\text{HgI}_2]_2$ ³² and in our synthesized complexes^{33,34}. The downfield shift of the carbonyl C atom in all of mentioned complexes compared to the same carbon atom in the parent ylides, indicate a much lower shielding of the CO group in these complexes^{7,33-35}.

Conclusion

The present study describes the synthesis and characterization of some mono-nuclear Ag(I), binuclear and orthometallated Pd(II) complexes of phosphorus ylides. On the basis of the spectroscopic data we propose that the ligands herein exhibit C-coordination to the metal centres.

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