Synthesis and molecular structure of a dinuclear phosphorus ylide complex: μ-dichlorobis[chloro(benzoylmethylenetri-n-butylphosphorane)palladium(II)]

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Abstract

The synthesis and molecular structure of μ-dichlorobis[chloro(benzo-ylmethylenetri-n-butylphosphorane)palladium(II)] is reported. The characterization of the dinuclear ylide species, wherein the ylide is not *ortho*-metalated, but solely coordinated through the ylide-methine carbon, is discussed. The role of the title compound in the overall formation of an *ortho*-metalated dinuclear species is also discussed. X-ray crystal data for $C_{40}H_{66}O_2P_2Pd_2Cl_4$: $P2_1/n$, monoclinic, a 12.928(4), b 13.318(4), c 15.948(5) Å; β 110.16(2)°, V 2504(1) Å^3, $Z = 2$, R_f 5.98%, wR_f 6.24%, for 3776 observed reflections with $F_0 > 5\sigma(F_0)$.

Introduction

The ability of keto-stabilized phosphorus ylides of the type $R_3PCR'C(O)R''$ to act as ambidentate ligands has been recognized for quite some time [1,2]. However, recent reports by our group [3–5], as well as others [6,7], have revealed the true versatility of these ligands in adopting numerous bonding modes. Single crystal X-ray diffraction studies have confirmed four different bonding modes: *O*-bonding via the carbonyl oxygen in an axial position in the trigonal bipyramidal complex $[Sn(CH_3)_3(Ph_3PCHC(O)CH_3)Cl]$ [2], *C*-bonding via the methine carbon atom, coupled with *ortho*-metalation of a phenyl ring in the dinuclear complex $[\overline{Pt(\mu\text{-}Cl)(CH_3C(O)CHP(C_6H_4)Ph_2)}]_2$ [3], metal–metal bridging via the methine carbon atom in the dinuclear complex $[\{Au(PPh_3)\}_2\{\mu\text{-}C(PPh_3)CO_2Et\}]ClO_4$ [6], and simple *C*-bonding in the complexes $[PdCl(\eta^3\text{-}2\text{-}MeC_3H_4)(Ph_3PCHC(O)CH_3)]$ [7] and *trans*-$[PdCl_2(PhC(O)CHP(n\text{-}C_4H_9)_3)_2]$ [5].

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In order to rationalize the ligating properties of these ligands [1,3–5,8,9], we have studied [4] the possibility of combining C-bonding via the methine carbon atom with *ortho*-metalation of a phenyl ring bound to the carbonyl group, rather than to the phosphorus atom, as was previously observed [3]. The synthesis of the complex [Pd(μ-Cl)(C$_6$H$_4$C(O)CHP(n-C$_4$H$_9$)$_3$)]$_2$ was reported [4], but not confirmed by a single crystal X-ray diffraction study. In an attempt to prepare the *ortho*-metalated complex, so that single crystals could be obtained, the title compound was unexpectedly formed. We now report characterization of the dinuclear phosphorus ylide complex, including the determination of its molecular structure via a single crystal X-ray diffraction study.

Experimental

All preparative procedures were performed without any precautions to exclude oxygen. Glassware was dried at 110 °C for a minimum of 2 h prior to use. The ylide ligand was prepared according to the method of Weleski et al. [8]. PdCl$_2$ was used as received.

Proton, carbon-13, and phosphorus-31 NMR spectra were obtained on a Bruker AM-250 spectrometer. The ^{1}H NMR spectra were recorded at 250.13 MHz, the ^{13}C{^{1}H} spectra were obtained at 62.9 MHz, and the ^{31}P{^{1}H} NMR spectra were obtained at 101.25 MHz. Proton and carbon NMR spectra were referenced to residual protonated solvent in acetone-d_6 (δ 2.04 ppm downfield from tetramethylsilane). Phosphorus chemical shifts were referenced relative to 85% phosphoric acid (external standard). These data are summarized in Table 1. Infrared spectra were obtained on a Nicolet 5 DXB FTIR spectrometer as KBr pellets. Elemental analyses were performed by Guelph Chemical Laboratories, Ltd., Guelph, Ontario, Canada, N1H1E7. Melting points were obtained on a Thomas Hoover capillary melting point apparatus, and are reported uncorrected.

Preparation of μ-dichlorobis[chloro(benzoylmethylenetri-n-butylphosphorane)palladium-(II)] (1)

In 70 ml of acetonitrile was placed PdCl$_2$ (0.514 g, 2.90 mmol) and 17.2 ml (5.00 mmol, 0.29 mmol/ml) of a benzene solution of benzoyltri-n-butylphosphorane (BBuPY). The solution was magnetically stirred and heated to reflux for 40 min. After cooling to room temperature, the solvent was removed using a rotary evaporator, resulting in the formation of a red oil. The oil was dissolved in CHCl$_3$ (100 ml) and diethyl ether (40 ml) and allowed to stand for two days, resulting in the formation of red-orange crystals. The red supernatant liquid was decanted and the crystals were collected and dried in vacuo. Analysis Found: C, 46.66; H, 6.68; P, 6.11. Pd$_2$Cl$_4$C$_{40}$H$_{66}$P$_2$O$_2$ calcd.: C, 48.26; H, 6.68; P, 6.22%. M.p. 168–170 °C (dec.).

Conversion of 1 to trans-[PdCl$_2$(BBuPY)$_2$]

To 0.102 g of **1** (0.103 mmol) in 35 ml of acetonitrile was added 0.107 g (0.257 mmol) BBuPY in 25 ml of acetonitrile. The solution was stirred at room temperature for 3 h and then heated to reflux for 4 h. The solvent was removed under reduced pressure to give a yellow oil. A portion of the oil was dissolved in acetone-d_6 and subjected to proton and phosphorus-31 NMR analysis. The NMR

Table 1

^{1}H, ^{13}C and ^{31}P NMR data ^{a}

	Compound	δ (ppm) ^{b,c}	Assignment	Coupling ^{d}
^{1}H	BBuPY	1.017(t)	PCH$_2$CH$_2$CH$_2$CH_3	$^1J(^1$H–^{1}H) 7.1
		1.3-1.7(m)	PCH$_2$CH_2CH_2CH$_3$	
		2.1-2.2(m)	PCH_2CH$_2$CH$_2$CH$_3$	
		3.69(d)	CHP	$^2J(^{31}$P–^{1}H) 14.7
		7.30(m)	*ortho-* and *para-*(C$_6$H$_5$)	
		7.85(m)	*meta-*(C$_6$H$_5$)	
	[C$_{20}$H$_{33}$OPCl$_2$Pd]$_2$	0.93(m)	PCH$_2$CH$_2$CH$_2$CH_3	
		1.43(m)	PCH$_2$CH$_2$CH_2CH$_3$	
		1.72(m)	PCH$_2$CH_2CH$_2$CH$_3$	
		2.40(m)	PCH_2CH$_2$CH$_2$CH$_3$	
		4.63(d)	CHP	$^2J(^{31}$P–^{1}H) 2.6
		7.52(m)	*ortho-* and *para-*(C$_6$H$_5$)	
		8.40(m)	*meta-*(C$_6$H$_5$)	
^{13}C{^{1}H}	BBuPY	13.95(s)	PCH$_2$CH$_2$CH$_2$CH$_3$	
		22.10(d)	PCH$_2$CH$_2$CH$_2$CH$_3$	$^1J(^{31}$P–^{13}C) 55.2
		24.64(d)	PCH$_2$$CH_2CH_2CH_3$	$^2J(^{31}$P–^{13}C) 14.9
		24.87(d)	PCH$_2$CH$_2$$CH_2CH_3$	$^3J(^{31}$P–^{13}C) 2.9
		46.97(d)	CH	$^1J(^{31}$P–^{13}C) 103.9
		phenyl region obscured by benzene		
		184.31(s)	C=O	
	[C$_{20}$H$_{33}$OPCl$_2$Pd]$_2$	13.86(s)	PCH$_2$CH$_2$CH$_2$CH$_3$	
		22.11(d)	PCH$_2$CH$_2$CH$_2$CH$_3$	$^1J(^{31}$P–^{13}C) 48.3
		24.72(d)	PCH$_2$$CH_2CH_2CH_3$	$^2J(^{31}$P–^{13}C) 15.9
		25.24(d)	PCH$_2$CH$_2$$CH_2CH_3$	$^3J(^{31}$P–^{13}C) 2.3
		not found	CH	
		129.17(s),129.85(s)	(C$_6$H$_5$)	
		133.30(s),139.25(s)		
		not found	C=O	
^{31}P{^{1}H}	BBuPY	20.5(s)	PCH$_2$CH$_2$CH$_2$CH$_3$	
	[C$_{20}$H$_{33}$OPCl$_2$Pd]$_2$	34.2(s)	PCH$_2$CH$_2$CH$_2$CH$_3$	

^{a} All spectra were obtained in acetone-d_6, unless otherwise specified. ^{b} ^{1}H and ^{13}C{^{1}H} peaks referenced to solvent signals and indirectly referenced to Si(CH$_3$)$_4$; ^{31}P{^{1}H} peaks referenced to external phosphoric acid (85%). ^{c} s = singlet, d = doublet, m = multiplet. ^{d} Coupling constants are reported in Hertz.

spectra indicated that *trans*-[PdCl$_2$(BBuPY)$_2$] [5] was formed. Also, single crystals were obtained of the product and were confirmed to be *trans*-[PdCl$_2$(BBuPY)$_2$] [5] based on unit cell parameters.

Crystal data, structure solution, and refinement

A red-orange crystal of [C$_{20}$H$_{33}$OPCl$_2$Pd]$_2$ (0.40 × 0.40 × 0.45 mm), obtained from the slow evaporation of an acetone solution, was mounted on a glass fiber with epoxy cement. Unit cell parameters were determined through least squares refinement of 25 reflections (20 ≤ 2θ ≤ 25°). Data were collected on a Nicolet R3m/μ diffractometer with a graphite monochromator, (λ(Mo-K_α) 0.71073 Å). An empirical absorption correction (6 reflections, ψ scans, 216 data, T_{max}/T_{min} 1.11) was applied to the data collected. Crystal data and collection parameters are sum-

Table 2

Crystal data for $[C_{20}H_{33}OPCl_2Pd]_2$

(a) Crystal data			
Formula	$[C_{20}H_{33}OPCl_2Pd]_2$	V, Å^3	2504(1)
Formula weight	995.53 g	Z	2
Crystal system	monoclinic	D_{calc}, g cm^{-3}	1.29
Space group	$P2_1/n$	μ, cm^{-1}	10.06
a, Å	12.928(4)	T, K	296
b, Å	13.318(4)	Crystal size, mm	$0.40 \times 0.40 \times 0.45$
c, Å	15.948(5)	Color	red-orange
β, (deg)	110.16(2)	$\lambda(\text{Mo-}K_\alpha)$ Å	0.71073

(b) Data collection	
Scan type	Wykoff
2θ limits, (deg)	4–55
h,k,l collected	$\pm 17, +18, +21$
Standard reflections, decay < 1%	3 std/197 reflns
Independent reflections ($R_{int} = 2.76\%$)	5745
Observed reflections $F_0 > 5\sigma(F_0)$,	3776
Diffractometer	Nicolet R3m/μ
Monochromator	highly oriented graphite

(c) Structure solution	
Least square parameters	214
Data/parameter	17.6
R_f	5.98%,
wR_f	6.24%
GOF	1.529
g	0.002
Δ/σ_{max}	0.021
$(\Delta/\rho)_{max}$, e Å^{-3}	1.79
$(\Delta/\rho)_{min}$, e Å^{-3}	−0.71
$w^{-1} = \sigma^2(F_0) + g(F_0)^2$,	

marized in Table 2. Table 3 contains the atomic coordinates; selected bond lengths and bond angles are given in Table 4. The molecular structure and numbering scheme of the compound are shown in Fig. 1. Supplementary materials (25 pp.) are available from the senior authors.

The structure was solved via direct methods (SOLV), which located the Pd and Cl atoms. The remaining atoms were determined through subsequent Fourier analyses and least-squares refinement. Systematic absences within the data uniquely determined the space group as $P2_1/n$. The n-butyl groups exhibit expected high thermal activity.

All non-hydrogen atoms were refined anisotropically; hydrogen atoms were calculated and fixed at idealized positions (d(C–H) 0.96 Å, $U = 1.2U$ of attached C). The phenyl ring was constrained to a rigid planar hexagon (d(C–H) 1.395 Å). The final difference map contained 3 peaks (1.79, 1.44, and 1.17 eÅ^{-3}, ave. c 2.5 e Å^{-3}), situated on a $\bar{1}$ site forming a square planar structure with an average bond distance of 1.625 Å. No chemical relevance could be ascribed to this formation, although it was well separated from the Pd complex. No attempt was made to refine these positions. All programs used in data collection and structure solution and

Table 3

Atomic coordinates ($\times 10^4$) and isotropic thermal parameters ($\text{Å}^2 \times 10^3$)

	x	y	z	U^a
Pd	259.8(4)	156.1(4)	1170.4(4)	44.5(2)
Cl(1)	234(2)	−1194(1)	122(1)	56.6(7)
Cl(2)	582(2)	−945(1)	2384(1)	61.0(7)
P	1791(2)	1846(2)	2378(1)	50.0(7)
O(1)	−286(5)	2892(4)	1358(4)	72(3)
C(1)	−456(6)	2109(6)	1718(5)	55(3)
C(2)	425(5)	1325(5)	2078(5)	49(3)
C(3)	−1885(5)	966(4)	1988(5)	81(4)
C(4)	−2898	843	2109	102(6)
C(5)	−3567	1674	2076	125(7)
C(6)	−3223	2629	1922	111(6)
C(7)	−2211	2753	1801	90(5)
C(8)	−1542	1922	1834	62(3)
C(11)	2151(6)	2273(6)	1416(5)	62(3)
C(12)	2645(8)	1457(9)	965(7)	90(5)
C(13)	2657(14)	1854(15)	−39(13)	171(11)
C(14)	3459(18)	2666(22)	100(19)	304(21)
C(15)	2745(6)	896(6)	3018(5)	59(3)
C(16)	3952(6)	1262(7)	3464(6)	68(3)
C(17)	4738(7)	421(9)	3684(7)	89(5)
C(18)	5905(8)	741(11)	4194(8)	108(6)
C(19)	1919(7)	2930(6)	3106(5)	60(3)
C(20)	1654(10)	2693(7)	3985(7)	86(5)
C(21)	1443(10)	3655(10)	4482(8)	105(6)
C(22)	398(11)	4204(12)	3935(10)	129(8)

^{a} Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

refinement are from the P3 (G. Sheldrick, Nicolet RXD, Madison WI) and SHELXTL program libraries.

Discussion

μ-Dichlorobis[chloro(benzoylmethylenetri-n-butylphosphorane)palladium(II)] (**1**) was prepared by the reaction of PdCl$_2$ and the α-keto ylide BBuPy in acetonitrile. Red-orange crystals were obtained by slow evaporation of an acetone solution and submitted for X-ray structure determination. The geometry about each Pd is square-planar, with the ylide ligand bound via the methine carbon atom. The non-bonding distance of 2.914 Å observed for Pd–H attached to C(3) would seem to indicate that this is a potential intermediate in the formation of an *ortho*-metalated dinuclear species. The Pd–Pd separation is 3.480 Å. The P–C(methine), C(methine)–C(carbonyl), and C=O bond lengths are in agreement with those previously reported for C-bound ylide complexes [3,5–7].

Coordination of the ylide ligand via the methine carbon was also indicated by NMR and IR spectroscopic results. In the proton NMR spectrum, the methine proton resonance was markedly shifted downfield relative to the free ylide, in accord with similar data previously reported for C(methine)-bound [3,4,6–8,10] (but not for O-bound) [3,11,12] ylides. Also, the lower $^2J(^{31}\text{P}-^1\text{H})$ coupling of **1** relative

Table 4

Selected bond lengths (Å) and bond angles ($^\circ$) for $[C_{20}H_{33}OPCl_2Pd]_2$

Bond lengths (Å)			
Pd–Cl(1)	2.416(2)	Pd–Cl(2)	2.307(2)
Pd–C(2)	2.060(7)	Pd–Cl(1A)	2.334(2)
Cl(1)–Pd(A)	2.334(2)	P–C(2)	1.802(7)
P–C(11)	1.802(9)	P–C(15)	1.806(7)
P–C(19)	1.805(8)	O(1)–C(1)	1.238(10)
C(1)–C(2)	1.503(10)	C(1)–C(8)	1.497(11)
C(11)–C(12)	1.544(15)	C(12)–C(13)	1.649(24)
C(13)–C(14)	1.461(33)	C(15)–C(16)	1.552(10)
C(16)–C(17)	1.471(14)	C(17)–C(18)	1.503(13)
C(19)–C(20)	1.548(15)	C(20)–C(21)	1.568(17)
C(21)–C(22)	1.512(17)		
Bond angles ($^\circ$)			
Cl(1)–Pd–Cl(2)	91.8(1)	Cl(1)–Pd–C(2)	175.0(2)
Cl(2)–Pd–C(2)	88.7(2)	Cl(1)–Pd–Cl(1A)	85.8(1)
Cl(2)–Pd–Cl(1A)	174.4(1)	C(2)–Pd–Cl(1A)	94.1(2)
Pd–Cl(1)–Pd(A)	94.2(1)	C(2)–P–C(11)	114.7(3)
C(2)–P–C(15)	107.2(3)	C(11)–P–C(15)	110.7(4)
C(2)–P–C(19)	109.9(4)	C(11)–P–C(19)	105.7(4)
C(15)–P–C(19)	108.6(3)	O(1)–C(1)–C(2)	121.5(7)
O(1)–C(1)–C(8)	121.0(6)	C(2)–C(1)–C(8)	117.5(6)
Pd–C(2)–P	109.2(4)	Pd–C(2)–C(1)	112.6(4)
P–C(2)–C(1)	112.2(5)	C(1)–C(8)–C(3)	122.7(4)
C(1)–C(8)–C(7)	117.3(4)	P–C(11)–C(12)	114.6(7)
C(11)–C(12)–C(13)	109.9(10)	C(12)–C(13)–C(14)	109.5(16)
P–C(15)–C(16)	114.6(6)	C(15)–C(16)–C(17)	111.9(7)
C(16)–C(17)–C(18)	113.2(10)	P–C(19)–C(20)	112.7(6)
C(19)–C(20)–C(21)	113.2(8)	C(20)–C(21)–C(22)	113.1(9)

to the free ylide is indicative of ylide coordination via the methine carbon. The coupling constant is directly related to the s character in the orbitals of the carbon atom to which the phosphorus and methine hydrogen atoms are bound [11,13]. Coordination of the methine carbon to the metal center changes its hybridization from sp^2 to sp^3, thereby reducing the s character in its bonding orbitals. The IR spectrum exhibited an absorption band at 1640 cm^{-1}, which was assigned to $\nu(CO)$. This is a change of $+120$ cm^{-1} in comparison to that of the free ylide ligand. This movement of the carbonyl stretching frequency to higher energy has been shown to be a definitive indication of the ylide's bonding mode [8].

We had initially anticipated that, given the reaction conditions, *ortho*-metalation of the BBuPy ylide would occur, as had been previously observed [4]. In this context, the isolation of **1** was rather surprising. However, this finding supports the formulation of similar Pd^{II} ylide complexes prepared by Itoh [10]. The proposed mechanism of formation of **1** is given in Fig. 2. PdCl$_2$ is a polymer that is readily solubilized by acetonitrile, most likely resulting in the formation of an equilibrium mixture of **2** and **3**. **2** can react directly with 2 mol of ligand to give the *trans*-square planar complex **5**. However, the ylide ligand can react with **3** resulting in the formation of **1**. The dimer, **1**, can be solvated to give **4** which reacts with excess ligand to form **5**.

To test this hypothesis, a portion of **1** was refluxed in acetonitrile with excess ligand. The resulting product obtained was *trans*-[PdCl$_2$(BBuPY)$_2$], identified by its
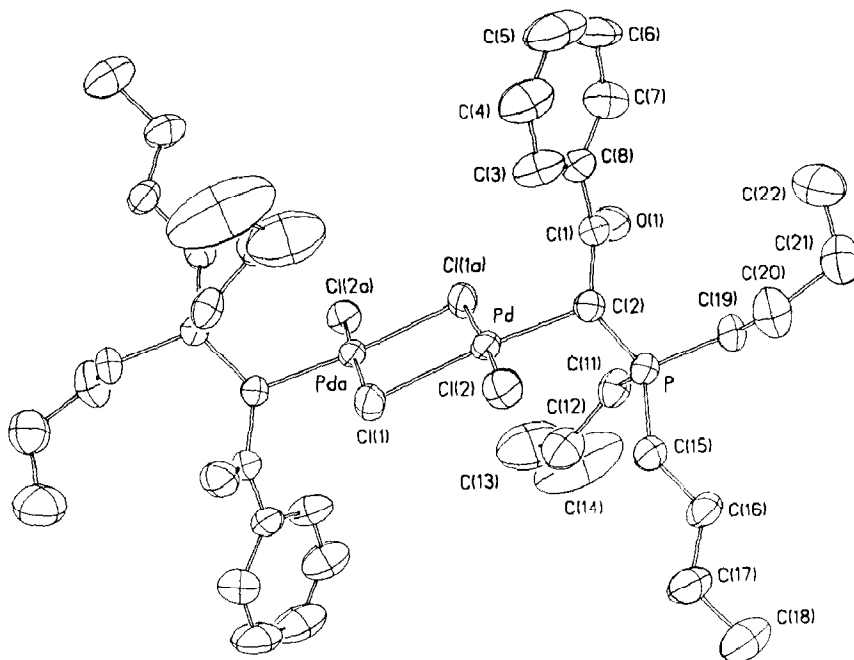
Fig. 1. Molecular structure and labeling scheme for $[C_{20}H_{33}OPCl_2Pd]_2$. Hydrogen atoms have been deleted. Thermal ellipsoids are shown at the 40% probability level.

characteristic proton and phosphorus-31 NMR resonances [4,5], and by unit cell parameters determined for a single crystal that was obtained. This result supports the postulated mechanism.
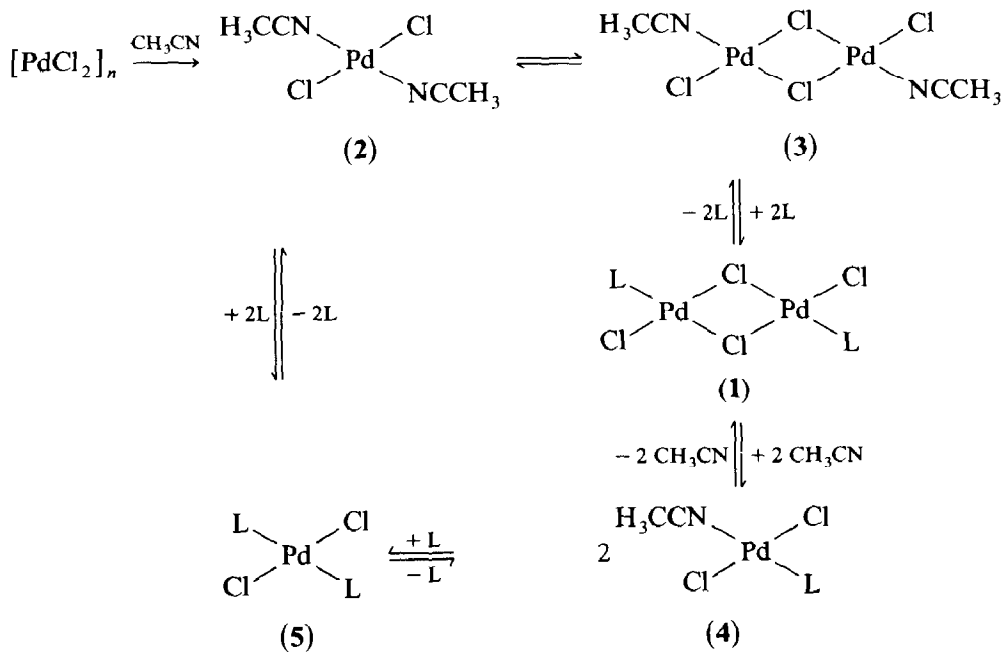

Fig. 2. Proposed mechanism of formation of 1.

Table 5. Reaction conditions for $PdCl_2$ and ylide ligands

Compound	$PdCl_2$(mmol)	Ligand(mmol)	Reference
trans-[$PdCl_2$(PhC(O)CHP(n-C_4H_9)$_3$)$_2$]	1.0	2.5	[5,8]
[Pd(μ-Cl)(C_6H_4C(O)CHP(n-C_4H_9)$_3$]$_2$	1.0	2.5	[4]
[PdCl(μ-Cl)(C_6H_5C(O)CHP(n-C_4H_9)$_3$]$_2$	2.9	5.0	this work
[PdCl(μ-Cl)(CH_3C(O)CHPPh$_3$)]$_2$	3.0	5.0	[10]

Our data suggest that the formation of **1** and **5** is dependent upon the ratio of ligand to $PdCl_2$. Table 5 shows the ligand to $PdCl_2$ ratio used in the preparation of the various Pd^{II} ylide compounds. It can be seen that **1** is the favored product if the ratio is less than 2 mol ligand to 1 mol $PdCl_2$, while **5** is the predominant product if the ratio is greater than 2/1. This is further supported by the observation that when **1** was refluxed in the absence of **BBuPY**, no reaction occurred (i.e., no **5** or *ortho*-metalated species was isolated.) This also suggests that **1** is very thermodynamically stable. Formation of an *ortho*-metalated species from **5** does not seem to be affected by the ligand concentration but rather was attributed to being a function of reaction time [5].

Finally, it should be noted that Vicente and his co-workers have recently reported [14] the synthesis of both the simple *C*-bound palladium(II) complex and the *ortho*-metalated palladium(II) dinuclear complex for the carbonyl-stabilized ylide Ph_3PCHC(O)OCH_3, i.e., [$PdCl_2${Ph_3PCHC(O)OCH_3}$_2$] and [Pd{CH{P(2-C_6H_4)Ph_2}(C(O)OCH_3)}(μ-Cl)]$_2$. Neither of the complexes was structurally characterized by single crystal X-ray diffraction studies, although the bridge-splitting reactions of the latter with a variety of mono- and bi-dentate ligands were investigated. Vicente et al. have also reported [15,16] the first crystal structures of silver complexes of carbonyl-stabilized ylides. Both mononuclear [15] and dinuclear complexes [16] involved simple C(methine)-bonding on the part of the ylides.

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