

Synthesis and Multinuclear NMR Study of Novel Complexes of Zn(II) and Hg(II) Containing Phosphorus Ylides

SEYYED JAVAD SABOUNCHEI*, MOHSEN AHMADI GHARACHEH and MARIAN HOSSEINZADEH

Faculty of Chemistry, University of Bu-Ali Sina, Hamedan 65174, Iran

Fax: (98)(811)8257407; E-mail: jsabouchei@yahoo.co.uk

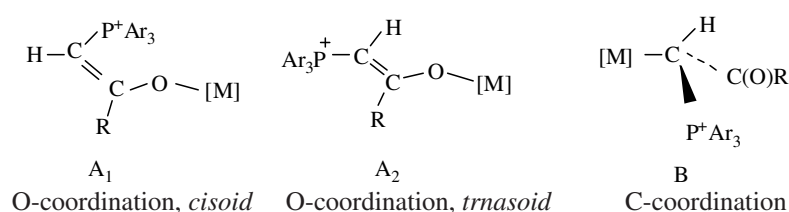
Reaction of phosphorus ylides of the type R-COCH = PPh₃ (R = C₆H₅C₆H₄ (**1a**) and C₁₀H₇ (**1b**)), with M(NO₃)₂.nH₂O (M = Hg(II), n = 1 and Zn(II), n = 6) in equimolar ratios using methanol as solvent are reported. The analytical data, IR, ¹H, ¹³C and ³¹P NMR indicate a 1:1 stoichiometry between the ylides and M(II) nitrate [M = Zn(II) or Hg(II)] in each of new polymeric complexes of the type {Hg[(R-COCH = PPh₃)(μ-NO₃)(NO₃)]_n.(DMSO)_n (R = C₆H₅C₆H₄ (**2a**), C₁₀H₇ (**2b**)) and {Zn[(R-COCH = PPh₃)(μ-NO₃)(NO₃)]_n.(DMSO)_n (R = C₆H₅C₆H₄ (**3a**), C₁₀H₇ (**3b**)). Also, reaction of ylides with ZnCl₂ in equimolar ratios, gave novel binuclear complexes of the type [(R-COCH = PPh₃).ZnCl₂]₂ (R = C₆H₅C₆H₄ (**4a**), C₁₀H₇ (**4b**)). Variation of temperature in a ³¹P NMR study for complex **2a** indicates that the disappearance of satellites, due to coupling to ¹⁹⁹Hg, occurs with increasing temperature.

Key Words: Phosphorus ylide, Dimeric complexes, Polymeric complexes, Bridging nitrate, Triphenylphosphine.

INTRODUCTION

The utility of metalated phosphorus ylides in synthetic chemistry has been well documented^{1,2}. Phosphorus ylides are interesting ligands which have attracted much attention in synthetic, catalytic and theoretical fields of transition metal chemistry³. Ylides containing a second functional group have become increasingly interesting since they can provide new synthetic and catalytic routes in organometallic chemistry. Synthesis of complexes derived from phosphorus ylides and Hg(II) salts was limited to Hg(II) halides and was started in 1965 by Nesmeyanov⁴ and Weleski⁵ in 1975 proposed a symmetric halide-bridged dimeric structure for Hg(II) halides complexes while Kaliasundari in 1995 reported an asymmetric halide-bridged structure⁶. Recently, we reported the first examples of polymeric Hg(II) phosphorus ylide complexes wherein the nitrate anions are bridging, confirming the general belief that the seven-coordinated complexes would be formed⁷. As far as we are aware, no Zn(II) nitrate polymeric complexes with these ylides have been reported to date. We expect that Zn give seven-coordinated complexes wherein the nitrate anions are bridging; this is similar to polymeric Hg(II) complexes. The aims of

present work are to describe the preparation, spectroscopic characterization of Hg(II) and Zn(II) polymeric complexes. We also describe the preparation and characterization of novel complexes prepared from ZnCl₂ with phosphorus ylides. Juxtaposition of the keto group and carbanion in the phosphorus ylides allows for the resonance delocalization of the ylidic electron density providing additional stabilization to the ylide species (**Scheme-I**). This so-called α -stabilization provides ylides with the potential to act as an ambidentate ligand and thus bond to an Hg(II) center through the either carbon (B) or oxygen (A1 and A2) (**Scheme-I**).



Scheme-I

Although many bonding modes are possible for keto ylides⁸, 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)⁹ whereas, O-coordination dominates when the metals involved are hard, *e.g.*, Ti(IV), Zr(IV) and Hf(IV)¹⁰.

EXPERIMENTAL

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. The phosphorus ligand **1b** was synthesized according to the published procedures¹¹.

Preparation of [PPh₃CHCOC₆H₄C₆H₅] (1a): 2-Bromo-4-phenylacetophenone (0.31 g, 1.1 mmol) was dissolved in 20 mL of acetone and then a solution of triphenylphosphine (0.3 g, 1.1 mmol) in the same solvent (5 mL) was added dropwise to the above solution. The resulting brown solution was stirred for 10 h. The pale brown solid product (phosphonium salt), was filtered off, washed with diethyl ether and dried under reduced pressure, yield: 0.47 g, 77 %. Addition of the phosphonium salt (0.92 mmol, 0.47 g) to an aqueous solution of NaOH (0.5 M) led to the elimination of HBr and gave the free ligand. Yield: 0.35 g, 74.5 %. ¹³C NMR (CDCl₃) δ_{C} : 49.52 (d, *J* = 113.72, CH), 141.11 (d, *J* = 23.89, CPh(i)), 133.03 (s, CPh(o)), 132.50

(s, C₆H₅(m)), 134.70 (s, C₆H₅(p)), 126.13 (s, Ph(i')), 128.82 (s, Ph(o')), 126.72 (s, Ph(m')), 127.17 (s, Ph(p')), 126.54 (d, $J = 84.71$, PPh₃(i)), 127.35 (d, $J = 11.44$, PPh₃(o)), 130.89 (d, $J = 8.89$, PPh₃(m)), 131.64 (s, PPh₃(p)), 183.90 (d, $J = 11.82$, CO).

Preparation of [PPh₃CHCOC₆H₄C₆H₅.Hg(NO₃)₂] (2a): General procedure; to a magnetically stirred solution of Hg(NO₃)₂.H₂O (0.075 g, 1 mmol) in methanol (10 mL), was added a methanolic solution (5 mL) of ylide **1a** (0.2 g, 1 mmol). After 0.5 h the solvent was removed under reduced pressure to 3 mL. After addition of diethylether (30 mL) the pale brown solid product was separated by filtration. Yield: 0.20 g, 74.18 %. ¹³C NMR (DMSO-*d*₆) δ_C: 129.49 (s, C₆H₅(i)), 135.82 (s, C₆H₅(o)), 130.02 (s, C₆H₅(m)), 139.56 (s, C₆H₅(p)), 127.57 (s, Ph(i')), 129.45 (s, Ph(o')), 127.97 (s, Ph(m')), 135.61 (s, Ph(p')), 126.54 (d, $J = 84.71$, PPh₃(i)), 134.66 (d, $J = 10.60$, PPh₃(o)), 130.67 (d, $J = 12.50$, PPh₃(m)), 130.02 (s, PPh₃(p)), 192.61 (s, CO).

Data for [PPh₃CHCOC₁₀H₇.Hg(NO₃)₂] (2b): Pale brown solid, Yield: 0.22 g, 80.0 %. ¹³C NMR (DMSO-*d*₆) δ_C: 135.80 (s, CO.NaPh(i)), 122.08 (d, $J = 88.53$, PPh₃(i)), 134.28 (d, $J = 10.04$, PPh₃(o)), 130.27 (d, $J = 12.75$, PPh₃(m)), 134.76 (s, PPh₃(p)), 193.78 (s, CO).

Data of [PPh₃CHCOC₆H₄C₆H₅.Zn(NO₃)₂] (3a): Pale yellow solid, Yield: 0.55 g, 72.3 %. ¹³C NMR (DMSO-*d*₆) δ_C: 140.61 (s, C₆H₅(i)), 133.14 (s, C₆H₅(o)), 127.93 (s, C₆H₅(m)), 141.79 (s, C₆H₅(p)), 128.06 (s, Ph(i')), 127.64 (s, Ph(o')), 127.21 (s, Ph(m')), 132.29 (s, Ph(p')), 126.54 (d, $J = 84.71$, PPh₃(i)), 133.82 (br, PPh₃(o)), 129.87 (br, PPh₃(m)), 128.46 (s, PPh₃(p)).

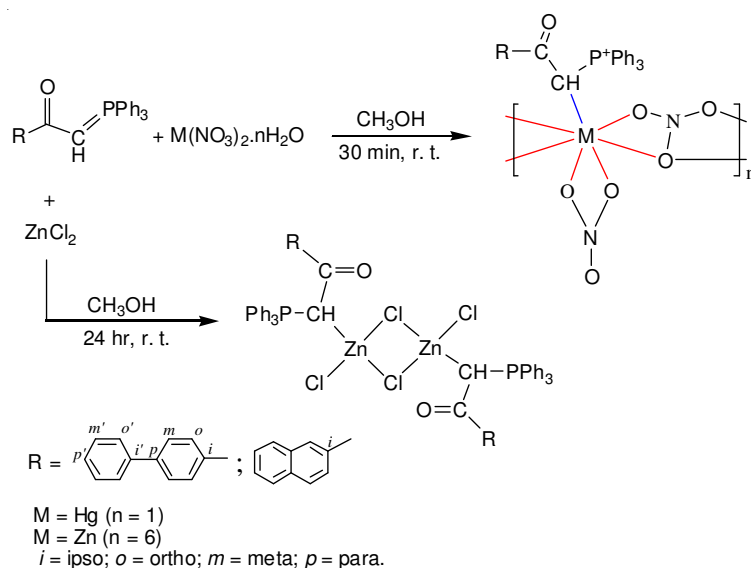
Data for [PPh₃CHCOC₁₀H₇.Zn(NO₃)₂] (3b): Pale yellow solid, Yield: 0.52 g, 69.8 %. ¹³C NMR (DMSO-*d*₆) δ_C: 50.51 (d, CH, $J = 112.98$), 138.51 (d, $J = 12.82$, CO.NaPh(i)), 125.81 (d, $J = 89.68$, PPh₃(i)), 133.18 (d, $J = 9.30$, PPh₃(o)), 129.49 (d, $J = 12.24$, PPh₃(m)), 132.73 (s, PPh₃(p)).

Preparation of [PPh₃CHCOC₆H₄C₆H₅.ZnCl₂]₂ (4a): General procedure: A solution of (0.036 g, 0.26 mmol) of ZnCl₂ in methanol (15 mL) was added to a solution of (0.120 g, 0.26 mmol) of ylide **1a** in methanol (15 mL) and stirred for 24 h. The pale yellow solution was concentrated to 2 mL and diethyl ether (15 mL) added to precipitate the white complex. Yield: 0.1 g, 64.1 %. ¹³CNMR (DMSO-*d*₆) δ_C: 139.69 (s, C₆H₅(i)), 132.91 (s, C₆H₅(o)), 132.45 (s, C₆H₅(m)), 134.72 (s, C₆H₅(p)), 127.29 (s, Ph(i')), 131.67 (s, Ph(o')), 139.15 (s, Ph(m')), 126.53 (s, Ph(p')), 128.04 (d, $J = 95.93$, PPh₃(i)), 131.36 (d, $J = 9.77$, PPh₃(o)), 128.59 (d, $J = 11.59$, PPh₃(m)), 131.88 (s, PPh₃(p)), 197.05 (s, CO).

Data for [PPh₃CHCOC₁₀H₇.ZnCl₂]₂ (4b): White solid, Yield: 0.11 g, 72.5 %. ¹³CNMR (DMSO-*d*₆) δ_C: 51.28 (d, CH, $J = 108.91$), 138.54 (d, $J = 14.79$, CO.NaPh(i)), 125.72 (d, $J = 79.1$, PPh₃(i)), 133.29 (d, $J = 9.8$, PPh₃(o)), 129.54 (d, $J = 12.0$, PPh₃(m)), 132.85 (d, PPh₃(p)), 195.45 (s, CO).

RESULTS AND DISCUSSION

The polymeric structures may be obtained from the reaction of $M(\text{NO}_3)_2 \cdot n\text{H}_2\text{O}$ ($M = \text{Hg}(\text{II})$, $n = 1$ and $\text{Zn}(\text{II})$, $n = 6$) with ylides in equimolar ratios using methanol as solvent (**Scheme-II**). The reaction of parent ylides with ZnCl_2 in a 1:1 molar ratio in methanol as solvent gave binuclear C-coordinated complexes (**Scheme-II**).



Scheme-II

The elemental analyses and spectral data of the ligand **1a**, **1b** and their metal complexes are listed in Table-1. These data confirm the assigned composition of the ligands and complexes. The $\nu(\text{CO})$ which is sensitive to complexation occurs at higher frequencies comparing to parent ylides, indicating coordination of the ylide through carbon. C-coordination causes an increase in $\nu(\text{CO})$ and decrease in $\nu(\text{P}^+-\text{C}^-)$ whiles for O-coordination a lowering for both frequencies is expected. These assignments were confirmed by comparing the IR spectra of the corresponding ^{13}C substituted ylides¹². The $\nu(\text{P}^+-\text{C}^-)$ which is also diagnostic for the coordination occurs at lower frequencies for complexes, suggesting some removal of electron density in the P-C bond^{13,14}.

The ^1H NMR signals due to methinic protons are doublet or broad doublet (forming satellites due to coupling to ^{199}Hg) for $\text{Hg}(\text{II})$ complex. Similar behaviour was observed earlier in the case of ylide complexes of platinum(II) chloride^{15,16}. The signals due to methinic protons for other complexes are doublet. The expected downfield shifts of ^{31}P and ^1H signals for PCH group upon complexation in the case of C-coordination⁶ were observed in their corresponding spectra. It must be noted that O-coordination of the ylide generally leads to the formation of *cisoid* and *transoid*-isomers giving rise to two different signals in ^{31}P and ^1H NMR¹⁷.

TABLE-1
 ELEMENTAL ANALYSIS AND SPECTRAL DATA OF
 PHOSPHORANES AND THEIR METAL COMPLEXES

Ligand/Complex	Elemental analysis			IR (cm ⁻¹ , KBr)	³¹ P NMR (δ, ppm) (DMSO- <i>d</i> ₆)	¹ H NMR (δ, ppm) (DMSO- <i>d</i> ₆)
	C(%)	H(%)	N(%)			
1a Ph ₃ PCHCOC ₆ H ₄ C ₆ H ₅ C ₃₂ H ₂₅ OP FW= 456.16 MP= 82-84 °C	83.62* (84.19)**	5.50 (5.52)		v C=O v P-C	1568 (s) 881 (s)	4.45 (br, 1H, CH) 7.35-8.03 (m, 24H, arom.)
1b (Ref. ¹¹) Ph ₃ PCHCOC ₁₀ H ₇ C ₃₀ H ₂₃ OP FW= 430.48 MP= 193-194 °C (Ref. ⁷)	83.46 (83.70)	5.31 (5.39)		v C=O v P-C	1577 (s) 873 (s)	4.58 (d, ² J _{PH} = 25.26, 1H, CH) 7.52-8.49 (m, 22H, arom.)
C ₃₆ F ₂₀ ClH ₈ N ₂ O ₇ P FW= 740.04 MP= 154-155 °C	42.28 (42.23)	2.89 (2.73)	4.16 (3.79)	v C=O v P-C	1662 (s) 824 (s)	28.38 s with satellites, ² J _{HgP} = 295.2 Hz
2a C ₃₂ H ₂₅ HgN ₂ O ₇ P FW= 782.11 MP= 103-105 °C	48.92 (49.20)	3.30 (3.23)	3.91 (3.59)	v C=O v P-C v Hg-O v N-O	1659 (s) 816 (s) 259 (d) 358 (d)	28.41 s with satellites, ² J _{HgP} = 288.5 Hz
2b C ₃₀ H ₂₃ HgN ₂ O ₇ P FW= 755.08 MP= 162-164 °C	47.62 (47.72)	3.12 (3.07)	3.62 (3.71)	v C=O v P-C v Hg-O v N-O	1659 (s) 815 (s) 259 (d) 359 (d)	28.45 s with satellites, ² J _{HgP} = 290.5 Hz
3a C ₃₂ H ₂₅ N ₂ O ₇ PZn FW= 645.9 MP= 112-115 °C	59.53 (59.50)	3.82 (3.90)	4.58 (4.34)	v C=O v P-C v Zn-O v N-O	1672 (s) 816 (s) 291 (d) 360 (d)	16.40 (s)
3b C ₃₀ H ₂₃ N ₂ O ₇ PZn FW= 619.8 MP= 153-156 °C	58.25 (58.13)	3.65 (3.74)	4.91 (4.52)	v C=O v P-C v Zn-O v N-O	1668 (s) 825 (s) 295 (d) 359 (d)	17.65 (s)
4a C ₆₄ H ₅₀ Cl ₄ O ₂ P ₂ Zn ₂ FW= 1184.06 MP= 138-141 °C	65.03 (64.83)	4.46 (4.25)		v C=O v P-C v Zn-Cl (term.) v Zn-Cl (brid.)	1672 (s) 828 (s) 293 (s) 295 (s)	16.34 (s)
4b C ₆₀ H ₄₆ Cl ₄ O ₂ P ₂ Zn ₂ FW= 1113.55 MP= 127-130 °C	63.35 (63.57)	3.91 (4.09)		v C=O v P-C v Zn-Cl (term.) v Zn-Cl (brid.)	1668 (s) 822 (s) 292 (s) 297 (s)	16.34 (s)

S= singlet, d= doublet, br= broad, m= multiplet.

* Found; ** Analytical calculate

The proton decoupled ³¹P {¹H} NMR spectra show only one sharp singlet between δ ≈ 16-30 ppm in these complexes. The ³¹P chemical shift values for complexes appear to be downfield by about δ ≈ 2-15 ppm with respect to the parent ylide; this also indicating that coordination of the ylide has occurred^{6,18}. The most interesting aspect of the ³¹P NMR spectra at room temperature for **2a** and **2b** is that clearly exhibit mentioned satellites (8 % intensity each, ²J_{PHg} ~ 288.5 Hz and ²J_{PHg} ~ 290.5 Hz, respectively), while these satellites for ylidic complexes of mercury(II) halides are only observed in low temperature^{14,18} or by solid-state ³¹P NMR¹⁸. Variable temperature ³¹P NMR study on **2a**, using DMSO-*d*₆ as solvent showed that disappearing of satellites occurs when the temperature is increased up to 55 °C (Fig. 1). Greater solvent strength caused by increasing temperature¹⁸ or decreasing concentration, led to a faster exchange of the ylide with the metal¹⁹, which caused exchange decoupling. Thus it seems that DMSO can affect directly on the nature of the polymeric structure in the solution state and probably it is cleaved to smaller species.

The coordination number in Hg(II) and Zn(II) polymeric complexes are seven (one of ylide, two of one bidentate nitrate anion and four of two bridged nitrates). There are only a few inorganic examples of bridging nitrates with different coordination modes: bidentate²⁰, tridentate²¹ and tetradentate²² but in polymeric complexes there are two different nitrate anions: one of them acts as bidentate and the other one acts as both bidentate and bridging group (totally tetradentate).

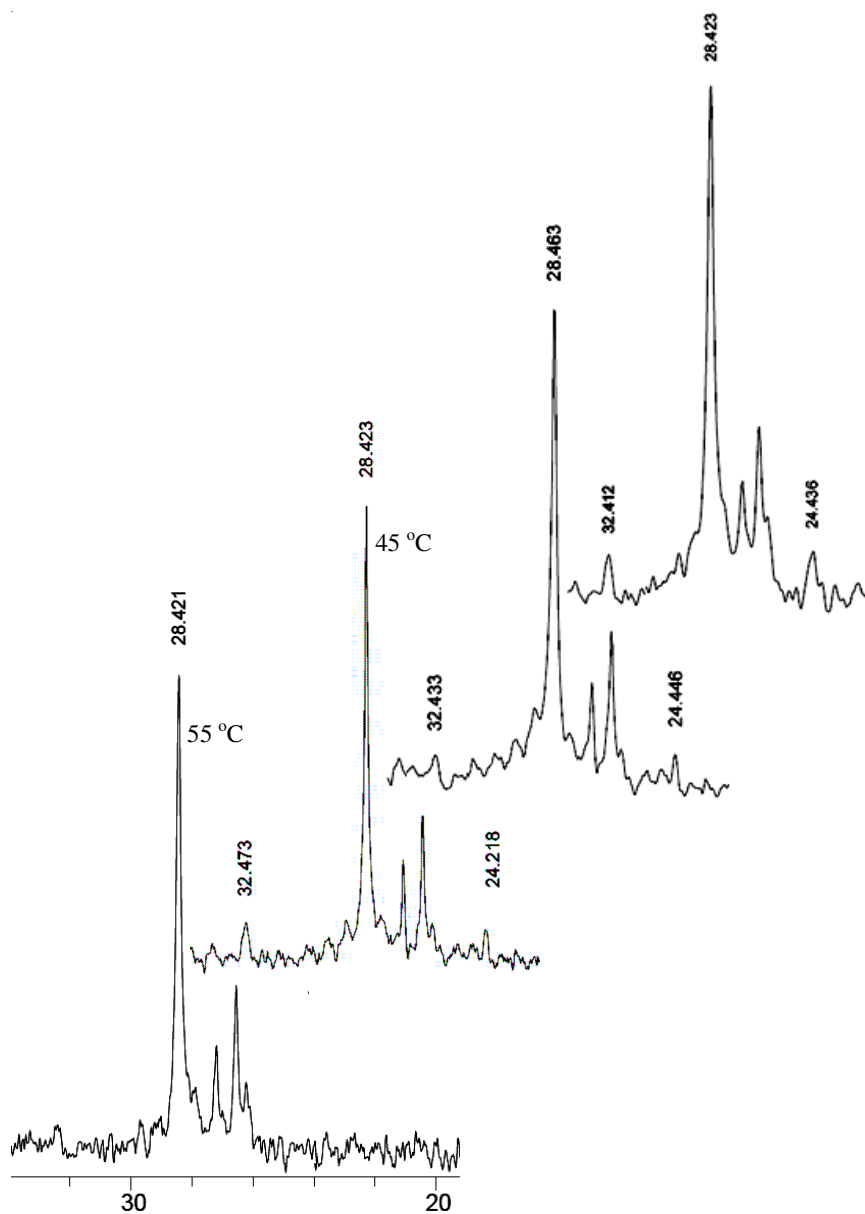


Fig. 1. Variable temperature ³¹P NMR study for **2a** (*impurity).

^{13}C NMR spectra of the complexes are the up field shift of the signals due to ylidic carbon. Such up field shifts observed in $[\text{PdCl}(\eta^3\text{-2-XC}_3\text{H}_4)(\text{C}_6\text{H}_5)_3\text{PCHCOR}]$ ($\text{X} = \text{H}, \text{CH}_3$; $\text{R} = \text{CH}_3, \text{C}_6\text{H}_5$) was attributed to a change in hybridization of the ylidic carbon²³. Similar up field shift of 2-3 ppm with reference to the parent ylide were also observed in the case of $[\text{Ph}_3\text{PC}_5\text{H}_4\text{HgI}_2]_2$ ²⁴ and in synthesized mercury complexes^{14,25}. High solubility of **1a** and **1b** in CDCl_3 allowed us to record the spectrum in this solvent but for the complexes we had to use $\text{DMSO-}d_6$, presence of a solvent septet peak at 39.5 ppm in the CH resonance region caused that these signals were not seen. The ^{13}C NMR shifts of CO group in the complexes are around 190 ppm, lower field than 183 ppm noted for the same carbon in the parent ylides, indicating much lower shielding of carbon of the CO group in these complexes.

In summary we describe the synthesis and characterization (spectral, variable temperature NMR and elemental analysis) of α -keto stabilized phosphorus ylides and novel polymeric complexes. In Hg(II) polymeric complexes, comparing with those of mercury halide complexes, satellites due to coupling to ^{199}Hg could be seen at room temperature but disappear at higher temperatures. Novel binuclear complexes of **4a** and **4b** have also been synthesized and characterized. The C-coordination of **1a** and **1b** is in stark contrast to the O-coordination of the phosphorus ylide $\text{Ph}_3\text{PC}(\text{COMe})(\text{COPh})$ (ABPPY) to the Hg(II) centre²⁶. The difference in coordination mode between ABPPY, **1a** and **1b** to Hg(II) and Zn(II) can be rationalized in terms of the electronic properties and steric requirements of the ylides. On the basis of the spectroscopic data and electronic properties we propose that these ligands exhibit C-coordination to the metal centre.

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