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Synthesis, spectra and crystal structures of zwitterionic mercury(II) complexes formed by the ligand, [Ph$_2$PCH$_2$PPh$_2$CH$_2$C(O)Ph]$^+$

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ABSTRACT

The reactions of phosphonium salt, [PPh$_2$CH$_2$PPh$_2$CH$_2$C(O)Ph]Br with Hg(II) halides in methanol formed the zwitterionic products with the composition [HgCl$_2$(Br)(PPh$_2$CH$_2$PPh$_2$CH$_2$C(O)Ph)] (**2**), [HgBr$_3$(PPh$_2$CH$_2$PPh$_2$CH$_2$C(O)Ph)] (**3**), [HgBr$_2$(I)(PPh$_2$CH$_2$PPh$_2$CH$_2$C(O)Ph)] (**4**). A product of composition, [HgCl$_3$(PPh$_2$CH$_2$PPh$_2$CH$_2$C(O)Ph)] (**5**) was obtained when the ylide, Ph$_2$PCH$_2$PPh$_2$=CHC(O)Ph was treated with HgCl$_2$ in CH$_2$Cl$_2$ and crystallized under aerobic conditions. The complexes were characterized by elemental analysis, IR, ^{1}H, ^{31}P NMR spectra and also by X-ray crystallography. The absence of significant coordination chemical shifts in the ^{31}P NMR spectra indicates the presence of formal negative charge on mercury. The single crystal X-ray structures confirm the presence of Hg–P bond and also reveal a distorted tetrahedral geometry around the mercury atom in all the structures.

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1. Introduction

Phosphonium salts are useful intermediates in organic synthesis, exhibiting variety of reactivity when compared with the ammonium or pyridinium salts [1]. In the field of cationic polymerization, phosphonium salts showed promise as latent thermal- or photoinitiators due to their ability to form the corresponding stable ylides by releasing a proton [2–4]. Different types of homo- and heteropolynuclear ylide complexes of Hg(II), Pd(II) and Au(I) were prepared using the corresponding phosphonium salts, by deprotonation [5]. In the course of our ongoing research on the coordination chemistry of keto-stabilized phosphorus ylides, we have been interested to investigate the different bonding modes adopted by ylides upon ligation to Hg(II) [6–8] and U(VI) [9]. The α-keto-stabilized ylides derived from bisphosphines, viz., Ph$_2$PCH$_2$PPh$_2$=C(H)C(O)R and Ph$_2$PCH$_2$CH$_2$PPh$_2$=C(H)C(O)R (R = Me, Ph or OMe) [10] form an important class of hybrid ligands containing both phosphine and ylide functionalities, and can exist in ylidic and enolate forms. We recently observed that the mono-keto ylide with an ethylenic spacer, Ph$_2$PCH$_2$CH$_2$PPh$_2$=C(H)C(O)Ph, forms polymeric Hg(II) complexes with HgCl$_2$ via P, C-bridging mode while HgBr$_2$ and HgI$_2$ react with the same ylide giving poly-

meric halogen bridged phosphine complexes with dangling ylide [11]. On the other hand, Ph$_2$PCH$_2$PPh$_2$=C(H)C(O)Ph affords P, C-chelate complexes with mercury(II) halides [12]. Trihalomercu-rates such as [(CH$_3$)$_4$N]HgX$_3$ and [(CH$_3$)$_4$P]HgX$_3$ (where X = Cl, Br, I) are also useful as novel ferroelectric materials [13]. It is therefore of interest to investigate the reactivity of the hybrid phosphine–phosphonium salt, [PPh$_2$CH$_2$PPh$_2$CH$_2$COPh]Br with mercury(II) halides which can in principle form (i) mercury-ylide complexes by deprotonation, (ii) phosphonium metalates where the metal remains uncoordinated to the ligand or (iii) zwitterionic complexes containing Hg–P bond. In this paper, we report the formation of zwitterionic complexes of mercury(II) and their solution and solid state structures.

2. Experimental

All reactions were carried out in an atmosphere of nitrogen. Reactants and reagents were obtained from Aldrich Chemical Company and used without further purification. The solvents were dried and distilled using standard methods [14]. The ^{1}H and ^{31}P-{^{1}H}NMR spectra were recorded on a Bruker DPX400 spectrometer at 400.13 and 161.98 MHz, referenced relative to residual solvent and external 85% H$_3$PO$_4$, respectively. The chemical shifts (δ) and the coupling constants (J) were expressed in ppm and Hz, respectively. The IR spectra in the interval of 4000–400 cm^{-1} were recorded on a Perkin–Elmer 1720X FT-IR spectrophotometer using

KBr pellets. Elemental analyses were performed at the Ecole d'ingénieurs de Fribourg, Switzerland.

2.1. Synthesis

2.1.1. [Ph₂PCH₂PPh₂CH₂C(O)Ph]Br (1)

The phosphonium salt was prepared following the literature procedure [10] using $BrCH_2COPh$ (1.55 g, 7.8 mmol) and $PPh_2CH_2PPh_2$ (3.00 g, 7.8 mmol). Yield: 4.10 g (90%). M.p. 194–196 °C (Reported: 195–197 °C). IR (cm^{-1}): 1667 (vC=O). ^{1}H NMR (CDCl₃): δ 4.32 (d, 2H, PCH_2P, $^2J_{P-H}$ = 14.6), 5.93 (d, 2H, PCH_2COPh, $^2J_{P-H}$ = 12.5), 7.20–8.13 (m, 25H, Ph). ^{31}P NMR (CDCl₃): δ −26.18 (d, PPh₂, $^2J_{P-P}$ = 63.2), 24.07 (d, PCH₂COPh, $^2J_{P-P}$ = 63.2).

2.1.2. [HgCl₂(Br)(PPh₂CH₂PPh₂CH₂C(O)Ph)] (2)

A mixture of HgCl₂ (0.09 g, 0.34 mmol) and [PPh₂CH₂PPh₂CH₂C(O)Ph]Br (0.18 g, 0.34 mmol) in methanol (15 ml) was stirred for 3 h. The white precipitate obtained was isolated, washed twice with 15 ml methanol and recrystallised in dichloromethane. Yield: 0.25 g (86%). M.p. 158–160 °C. *Anal.* Calc. for $C_{33}H_{29}BrCl_2HgOP_2$: C, 46.36; H, 3.42. Found. C, 46.34; H, 3.35%. IR (cm^{-1}): 1672 (vC=O). ^{1}H NMR (DMSO–d_6): δ 4.43 (d, 2H, PCH_2P, $^2J_{P-H}$ = 15.5), 5.68 (d, 2H, PCH_2COPh, $^2J_{P-H}$ = 12.2), 7.26–7.99 (m, 25H, Ph). ^{31}P NMR (DMSO–d_6): δ −19.79 (d, PPh₂, $^2J_{P-P}$ = 63.2), 23.43 (d, PCH₂COPh, $^2J_{P-P}$ = 56.6).

2.1.3. [HgBr₃(PPh₂CH₂PPh₂CH₂C(O)Ph)] (3)

This complex was obtained using the same procedure as adopted for the preparation of 2 using HgBr₂ (0.12 g, 0.34 mmol). The product was recrystallized using dichloromethane containing a few drops of hexane. Yield: 0.29 g (90%). M.p. 135–137 °C. *Anal.* Calc. for $C_{33}H_{29}Br_3HgOP_2$: C, 41.99; H, 3.10. Found. C, 41.49; H, 3.00%. IR (cm^{-1}): 1671 (vC=O). ^{1}H NMR (DMSO–d_6): δ 4.27 (d, 2H, PCH_2P, $^2J_{P-H}$ = 15.4), 5.58 (d, 2H, PCH_2COPh, $^2J_{P-H}$ = 12.2), 7.26–7.97 (m, 25H, Ph). ^{31}P NMR (DMSO–d_6): δ −26.72 (d, PPh₂, $^2J_{P-P}$ = 65.4), 23.74 (d, PCH₂COPh, $^2J_{P-P}$ = 65.4).

2.1.4. [HgBr₂(I)(PPh₂CH₂PPh₂CH₂C(O)Ph)] (4)

A mixture of HgI₂ (0.08 g, 0.17 mmol) and [PPh₂CH₂PPh₂CH₂C(O)Ph]Br (0.10 g, 0.17 mmol) in methanol (15 ml) was stirred for 3 h. The clear colourless solution was evaporated to dryness giving an oily residue. Addition of diethyl ether (30 ml) resulted in the formation of a pale yellow solid. It was recrystallized in a mixture of dichloromethane–pentane and dried. Yield: 0.075 g (44%). M.p. 103–105 °C. *Anal.* Calc. for $C_{33}H_{29}Br_2HgIOP_2$: C, 40.00; H, 2.95. Found. C, 39.53; H, 2.90%. IR (cm^{-1}): 1675 (vC=O). ^{1}H NMR (DMSO–d_6): δ 4.24 (d, 2H, PCH_2P, $^2J_{P-H}$ = 15.4), 5.57 (d, 2H, PCH_2COPh, $^2J_{P-H}$ = 12.3), 7.24–7.97 (m, 25H, Ph). ^{31}P NMR (DMSO–d_6): δ −28.29 (d, PPh₂, $^2J_{P-P}$ = 67.0), 23.39 (d, PCH₂COPh, $^2J_{P-P}$ = 67.0).

2.1.5. [HgCl₃(PPh₂CH₂PPh₂CH₂C(O)Ph)] (5)

To a suspension of HgCl₂ (0.10 g, 0.36 mmol) in dichloromethane (10 ml) a solution of Ph₂PCH₂PPh₂=C(H)C(O)Ph [10] (0.21 g, 0.36 mmol) in dichloromethane (10 ml) was added dropwise. The clear suspension immediately turned turbid. After completion of the addition the solution became clear and the stirring was continued for two hours. The solution was reduced to about 5 ml and the addition of excess n-pentane resulted in a white precipitate. The solid was dissolved in CH₂Cl₂ and layered with n-pentane. After two days colourless diffraction quality crystals were obtained. Yield: 0.06 g (21% based on ylide). M.p. 184–186 °C. *Anal.* Calc. for $C_{33}H_{29}Cl_3HgOP_2$: C, 48.90; H, 3.61. Found. C, 48.84; H, 3.58%. IR (cm^{-1}): 1674 (vC=O). ^{1}H NMR (400 MHz, DMSO–d_6): δ 4.34 (d, 2H, PCH_2P, $^2J_{P-H}$ = 15.7), 5.64 (d, 2H, PCH_2COPh, $^2J_{P-H}$ = 12.2), 7.27–8.05 (m, 25H, Ph). ^{31}P NMR (162 MHz, DMSO–d_6): δ −22.78 (br, PPh₂), 23.55 (d, PCH₂COPh, $^2J_{P-P}$ = 60.2).

2.2. X-ray crystallography

All the crystallizations were carried out under aerobic conditions. Single crystals of 2, 3 and 4 were obtained by slow evaporation of dichloromethane, dichloromethane/hexane and dichloromethane/pentane solutions, respectively. Well formed crystals of 5 were grown by layering pentane over a dichloromethane solution. The intensity data were collected at 173 K (−100 °C) on a Stoe Mark II-Image Plate Diffraction System [15] equipped with a two-circle goniometer and using Mo Kα graphite monochromated radiation. The structures were solved by direct methods using the programme SHELXS-97 [16]. The refinement and all further calculations were carried out using SHELXL-97 [16]. The H-atoms were included in calculated positions and treated as riding atoms using SHELXL default parameters. The non-H atoms were refined anisotropically, using weighted full-matrix least-squares on F^2. Further crystallographic data are given in Table 1. The molecular structure and crystallographic numbering schemes are illustrated in ORTEP [17] drawings, Figs. 1–3.

3. Results and discussion

3.1. Synthesis

The reactions of mercury(II) halides with the phosphine–phosphonium salt, [Ph₂PCH₂PPh₂CH₂C(O)Ph]Br in 1:1 molar ratio in methanol afford the zwitterionic complexes 2–4 as shown in Scheme 1.

While 2 and 3 are simple complexation products, the formation of 4 involves a halogen exchange followed by complexation as shown below,

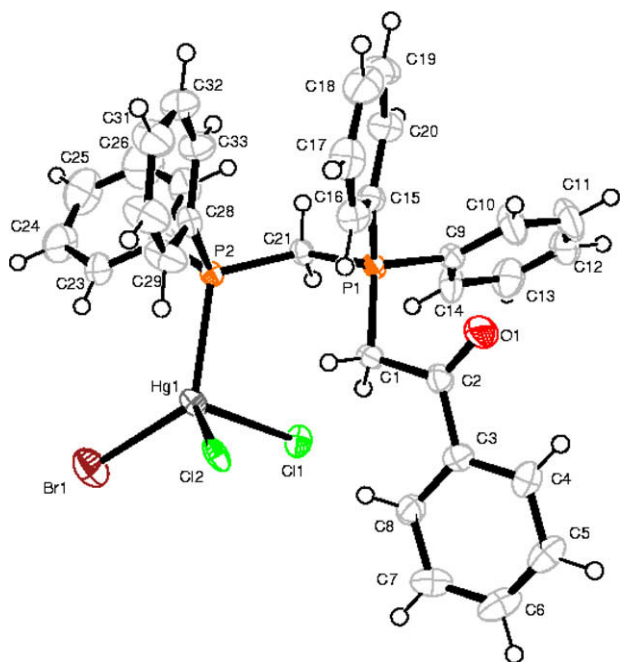2[Ph₂PCH₂PPh₂CH₂C(O)Ph]Br + HgI₂
→ [Br₂(I)Hg(PPh₂CH₂PPh₂CH₂C(O)Ph)] + [Ph₂PCH₂PPh₂CH₂C(O)Ph]I
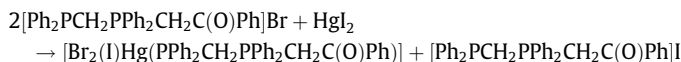
The stability of 4 consisting of the HgIBr₂ moiety can be traced to the softness of Hg(II) and I$^-$. The non-formation of HgI₂Br$^-$ or HgI₃$^-$ may be ascribed to steric effects caused by I$^-$ ion. Complex 5 has been obtained by the reaction of the ylide, Ph₂PCH₂PPh₂=C(H)C(O)Ph with HgCl₂ in dichloromethane and subsequent crystallization. The preferential formation of zwitterionic complexes as opposed to the formation of phosphonium metalates can be ascribed to the strength of Hg–P bond.

3.2. Spectroscopy

In the IR spectra of complexes 2–5, a strong absorption around 1675 cm^{-1}, which is close to the same frequency in free phosphonium salt (1667 cm^{-1}) indicates the non involvement of the –PCH₂C(O)Ph group in the reactions. The ^{31}P NMR spectra of complexes 2–5 exhibit two mutually coupled doublets corresponding to 'phosphonium' and 'phosphine' groups. The former peak remains sharp and unaltered, while the latter peak is relatively broad. In contrast to the ^{31}P NMR of Hg(II)–phosphine complexes [18], the coordination of phosphine to mercury in the present cases did not cause significant downfield shifts. Complexes 2 and 5 show downfield shifts (−19.79 and −22.78 ppm, respectively) compared to that of phosphine of the phosphonium salt (−26.18 ppm). The chemical shift for 3 (−26.72 ppm) appears close to that of the free ligand whereas 4 (−28.29 ppm) shows a slight upfield shift. These data indicate that the presence of a formal negative charge on the metal may effectively reduce the deshielding experienced by the phosphorus due to complexation. In the ^{1}H NMR spectra, the dou-

Table 1
Crystal data and refinement details for compounds $2 \cdot CH_2Cl_2$, $3 \cdot CH_2Cl_2$, $4 \cdot CH_2Cl_2$, $5 \cdot 2CH_2Cl_2$.

	$2 \cdot CH_2Cl_2$	$3 \cdot CH_2Cl_2$	$4 \cdot CH_2Cl_2$	$5 \cdot 2CH_2Cl_2$
Empirical formula	$C_{33}H_{29}BrCl_2HgOP_2$, CH_2Cl_2	$C_{33}H_{29}Br_3HgOP_2$, CH_2Cl_2	$C_{33}H_{29}Br_2HgIOP_2$, CH_2Cl_2	$C_{33}H_{29}Cl_3HgOP_2$, $2(CH_2Cl_2)$
Formula weight	939.83	1028.75	1075.74	980.29
Temperature (K)	173(2)	173(2)	173(2)	173(2)
Wavelength (Å)	0.71073	0.71073	0.71073	0.71073
Crystal size (mm)	$0.50 \times 0.15 \times 0.10$	$0.50 \times 0.40 \times 0.30$	$0.40 \times 0.30 \times 0.25$	$0.50 \times 0.50 \times 0.29$
Crystal system	monoclinic	monoclinic	monoclinic	monoclinic
Space group	$P2_1/c$	$P2_1/c$	$P2_1/c$	$P2_1/n$
a (Å)	10.1464(4)	10.2043(4)	10.451(2)	10.5390(4)
b (Å)	21.7503(10)	21.7893(10)	21.771(4)	22.6369(8)
c (Å)	15.8516(7)	16.1032(6)	16.265(3)	16.4398(6)
β (°)	96.702(3)	96.698(3)	96.299(16)	106.031(3)
V (Å^3)	3474.3(3)	3556.0(3)	3678.4(12)	3769.5(2)
Z	4	4	4	4
$F(000)$	1824	1968	2040	1920
μ (mm^{-1})	6.007	7.966	7.454	4.693
Reflections measured	42556	47650	19814	53038
Independent reflections	6175	9578	6572	10137
R_{int}	0.0555	0.0642	0.0695	0.0287
Observed reflections [$I > 2\sigma(I)$]	5272	7728	3974	9669
R_1 ($I > 2\sigma(I)$)	0.0399	0.0339	0.0492	0.0310
wR_2 (all data)	0.1107	0.0723	0.1278	0.0674

Fig. 1. Molecular structure of [HgCl$_2$(Br)(PPh$_2$CH$_2$PPh$_2$CH$_2$COPh)] (**2**) with 30% probability ellipsoids. The dichloromethane solvent has been omitted for clarity.


Fig. 2. Molecular structure of [HgBr$_3$(PPh$_2$CH$_2$PPh$_2$CH$_2$COPh)] (**3**) with 30% probability ellipsoids. The dichloromethane solvent has been omitted for clarity.

blet in the region of 4.30 ppm attributed to PCH_2P, and the doublet in the region of 5.62 ppm attributed to PCH_2COPh, remains unaffected due to complexation.

3.3. Molecular structures of complexes 2–5

The crystal structures of $2 \cdot CH_2Cl_2$, $3 \cdot CH_2Cl_2$, $4 \cdot CH_2Cl_2$ and $5 \cdot 2CH_2Cl_2$ have been determined and the relevant bond parameters are given in Table 2. The molecular structures of **2**, **3**, **4** are represented in Figs. 1–3, respectively. The structure of **5** is given as Supplementary data (Fig. S1). The Hg atom in all four structures is in a distorted tetrahedral environment with one P, and three halide ligands. The Hg–P distances in all of these zwitterionic complexes fall within the range of 2.39(1)–2.606(3) Å observed in

Fig. 3. Molecular structure of [HgBr$_2$(I)(PPh$_2$CH$_2$PPh$_2$CH$_2$COPh)] (**4**) with 30% probability ellipsoids. The dichloromethane solvent has been omitted for clarity.
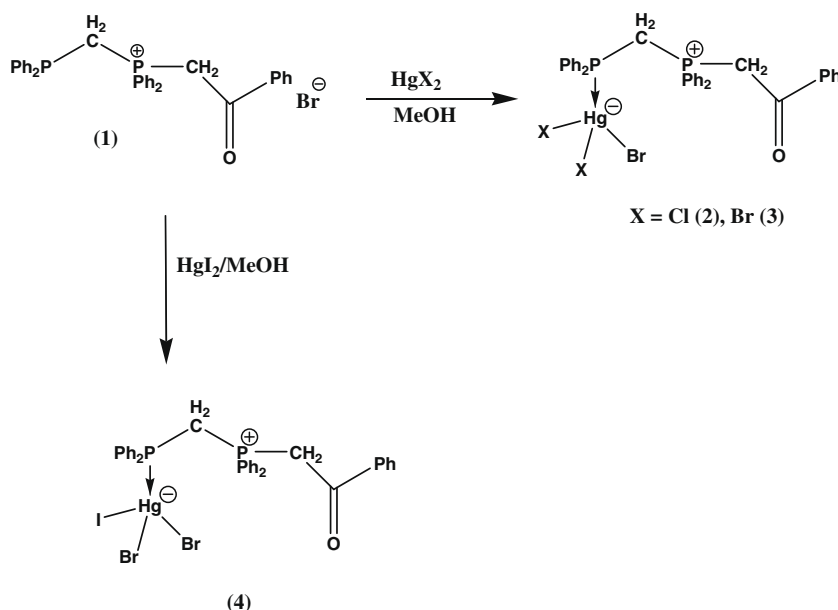
Scheme 1. Reactions of mercuric halides with phosphonium salt 1.

Table 2
Selected bond distances (Å) and angles (°) in 2–5.[a]

	2	3	4	5
Hg(1)–P(2)	2.4868(15)	2.5077(9)	2.550(3)	2.4465(7)
Hg(1)–X(*l*)	2.5221(8)	2.6545(4)	2.7030(9)	2.5197(8)
Hg(1)–X(*m*)	2.5743(15)	2.6474(4)	2.7038(13)	2.6210(8)
Hg(1)–X(*n*)	2.5792(14)	2.5504(4)	2.7084(13)	2.4263(8)
P(1)–C(1)	1.810(6)	1.806(4)	1.813(10)	1.803(3)
C(1)–C(2)	1.511(9)	1.522(5)	1.510(15)	1.518(4)
O(1)–C(2)	1.230(8)	1.220(5)	1.230(12)	1.213(4)
P(1)–C(21)	1.816(6)	1.816(3)	1.811(10)	1.815(3)
P(2)–C(21)	1.839(6)	1.841(3)	1.836(10)	1.843(3)
X(*n*)–Hg(1)–P(2)	109.81(5)	124.51(2)	112.10(7)	133.53(3)
X(*n*)–Hg(1)–X(*l*)	106.30(4)	107.656(15)	108.67(3)	99.16(3)
P(2)–Hg(1)–X(*l*)	126.85(4)	99.77(2)	121.83(7)	113.45(3)
X(*n*)–Hg(1)–X(*m*)	104.90(5)	106.554(14)	108.55(4)	107.90(3)
P(2)–Hg(1)–X(*m*)	100.50(5)	109.85(2)	95.30(7)	92.71(2)
X(*l*)–Hg(1)–X(*m*)	106.39(4)	107.300(15)	109.05(3)	108.36(3)
P(1)–C(1)–C(2)–O(1)	21.7(8)	24.7(5)	−14.6(15)	9.3(4)

[a] For 2 read *l* = Br(1), *m* = Cl(1), *n* = Cl(2). For 3 read *l* = Br(1), *m* = Br(2), *n* = Br(3). For 4 read *l* = I(1), *m* = Br(1), *n* = Br(2). For 5 read *l* = Cl(1), *m* = Cl(2), *n* = Cl(3).

Hg(II)–phosphine complexes [19]. The Hg–Cl distances in 5 and 2 are comparable to those reported in the literature [20,21]. In complex 3, one of the Hg–Br distances is shorter [2.550(1) Å] than the other two. It is worth mentioning that in 4, both the Hg–Br and Hg–I [2.703(1) Å] distances are very similar. One of the P–Hg–halogen bonds in the complexes exhibits a major deviation from an ideal tetrahedral angle. The most striking feature is the large distortion shown by P–Hg–Cl bond angle in complex 5 (which contains three chlorides). It has been observed previously [22] that the weaker bonding of chlorine to mercury compared to that of bromine and iodine, allows the Hg–P bonding to be strengthened leading to a linear P–Hg–Cl arrangement. In accordance with this fact the Hg–P bond length is much shorter in complex 5 (containing three chlorides) and 2 (containing two chlorides) compared to that in 3 and 4 (containing no chloride). The structural features in the phosphonium moiety are very similar in all four complexes. The significant shortening of the C2–O1 bond, as well as the elongation of the P1–C1 and C1–C2 bonds in all four complexes, compared with ylide, PPh₂CH₂PPh₂CHCOPh [12] indicates the absence of any significant additional resonance delocalization in the ylide moiety.

4. Conclusions

The reactions of the phosphine–phosphonium salt, [PPh₂CH₂PPh₂CH₂COPh]Br with mercury(II) halides offers an easy method for the preparation of zwitterionic mixed halogen mercurates. Formation of the above complexes can be ascribed to the strength of Hg–P bond. In the ^{31}P NMR spectra the absence of significant downfield shifts due to complexation could be ascribed to the presence of formal negative charge on the metal which effectively reduces the desheilding experienced by the phosphine phosphorus atom. The crystal structures of the above complexes reveal a distorted tetrahedral geometry around the mercury atom.

Appendix A. Supplementary data

CCDC 662097, 662098, 662099 and 662096 contain the supplementary crystallographic data for 2 · CH₂Cl₂, 3 · CH₂Cl₂, 4 · CH₂Cl₂ and 5 · 2CH₂Cl₂, respectively. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.poly.2009.01.001.

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