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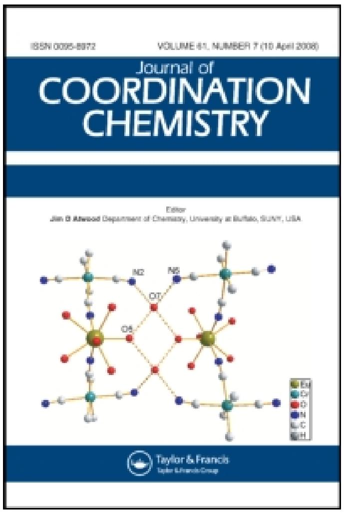

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Synthesis and chemistry of *tris*(2-pyridyl)phosphine and *bis*(2-pyridyl)phenylphosphine complexes of mercury(II) X (X = Br, Cl) and X-ray crystal structural determination of [$HgBr_2(PPh(2\text{-}py)_2)_2$]

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Synthesis and chemistry of *tris*(2-pyridyl)phosphine and *bis*(2-pyridyl)phenylphosphine complexes of mercury(II) X (X = Br, Cl) and X-ray crystal structural determination of [$HgBr_2(PPh(2\text{-}py)_2)_2$]

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Mercury(II) halide complexes [$HgX_2(P(2\text{-}py)_3)_2$] (X = Br (**1**), Cl (**2**)) and [$HgX_2(PPh(2\text{-}py)_2)_2$] (X = Br (**3**), Cl (**4**)) containing $P(2\text{-}py)_3$ and $PPh(2\text{-}py)_2$ ligands ($P(2\text{-}py)_3$ is *tris*(2-pyridyl) phosphine and $PPh(2\text{-}py)_2$ is *bis*(2-pyridyl)phenylphosphine) were synthesized in nearly quantitative yield by reaction of corresponding mercury(II) halide and appropriate ligands. The synthesized complexes are fully characterized by elemental analysis, melting point determination, IR, ^1H, and ^{31}P-NMR spectroscopies. Furthermore, the crystal structure of [$HgBr_2(PPh(2\text{-}py)_2)_2$] determined by X-ray diffraction is also reported.

Keywords: Mercury(II) halide complex; $P(2\text{-}py)_3$; $PPh(2\text{-}py)_2$; ^{31}P-NMR; X-ray diffraction

1. Introduction

Pyridylphosphines are convenient building blocks for the construction of transition metal complexes [1–4]. Many transition metals preferentially bind through nitrogen of pyridyl rings to form sandwich or half-sandwich complexes [5–8]. Especially, for the ligands, such as $P(2\text{-}py)_3$ and $PPh(2\text{-}py)_2$, P-monodentate coordination modes are rare [9–11]. Coordination and structural chemistry of mercury(II) halide complexes with ligands containing a variety of donors has been a matter of interest over several decades [12–15]. Following the seminal work of Mann and co-worker in 1940 [16], mercury(II) halides are known to form a wide range of stoichiometrics with tertiary phosphine ligands. By far, the most common are 1 : 1 and 2 : 1 complexes, $(R_3P)_nHgX_2$ ($n = 1, 2$; $X = Cl, Br, I$). The 2 : 1 complexes have a monomeric pseudo-tetrahedral structure with degree of distortion depending on the σ-donor ability and steric requirement of the phosphines [17–20]. In this article, four complexes of Hg(II) with the previously described [21, 22] pyridylphosphine, $P(2\text{-}py)_3$ and $PPh(2\text{-}py)_2$, are reported.

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2. Experimental

2.1. *Materials and instruments*

All manipulations for preparation of ligands were carried out under air and moisture-free conditions under nitrogen, using standard Schlenk or glove box techniques. Diethyl ether was dried over lithium aluminum hydride ($LiAlH_4$) and freshly distilled prior to use. Methanol, methylene chloride, *n*-butyllithium (15% solution in *n*-hexane), 2-bromopyridine, and phosphorus trichloride were purchased from Merck and except for *n*-butyllithium, purified before use according to standard methods. DMF, $HgCl_2$, and $HgBr_2$ were purchased from Fluka and were used as received. The ligands were prepared as described previously [21, 22]. Melting points are uncorrected and were obtained by an Electrothermal 9200 melting point apparatus. Infrared spectra from 250 to 4000 cm^{-1} of solid samples were taken as 1% dispersion in CsI pellets using a Shimadzu-470 spectrometer. ^1H and ^{31}P-NMR spectra were recorded at room temperature in DMSO-d_6 on a Bruker AVANCE 300 and 121.49 MHz, respectively. The NMR spectra are referenced to Me_4Si (^1H) or H_3PO_4 85% (^{31}P) as external standards. Elemental analysis was performed using a Heraeus CHN–O Rapid analyzer.

2.2. *Synthesis of [$HgBr_2(P(2\text{-}py)_3)_2$] (1)*

$HgBr_2$ (0.077 mmol, 0.028 g) was dissolved in 10 mL MeOH and a solution of P(2-py)$_3$ (0.039 mmol, 0.01 g) in 5 mL CH_2Cl_2 was added with vigorous stirring. A white precipitate was immediately formed. The slurry was stirred for 30 min. The solvent was completely removed, and the resulting white residue was washed with cold MeOH (10 mL) and Et_2O (10 mL) and finally dried *in vacuo*. White powder was recrystallized from concentrated DMF solution. Yield 94.4%; m.p. = 308°C (dec). IR (cm^{-1}): 3047(w), 1651(b), 1570(m), 1508(w), 1447(m), 1422(s), 1283(w), 1151(w), 1082(w), 1042(w), 986(m), 764(s), 736(m), 617(w), 523(s), 510(s), 496(s), 420(w), 395(w), 279(w). ^1H-NMR (DMSO-d_6, 300 MHz, 25°C): $\delta = 7.57$–7.59(d) ($J = 4.5$), 7.82–7.86(m), 7.97(b), 8.69–8.70(d) ($J = 4.2$). ^{31}P-NMR (DMSO-d_6, 121.4 MHz, 25°C): $\delta = 16.73$ ppm. Anal. found: N 9.30%, C 40.58%, H 2.75%; Calcd for $C_{30}H_{24}Br_2HgN_6P_2$ (890): N 9.43%, C 40.44%, H 2.72%.

2.3. *Synthesis of [$HgCl_2(P(2\text{-}py)_3)_2$] (2)*

This complex was prepared in a manner analogous to **1** where $HgCl_2$ (0.077 mmol, 0.021 g) dissolved in 10 mL MeOH was used. Resulting white powder was recrystallized from hot concentrated DMF solution. Yield 93.8%, m.p. = 320°C < (dec). IR (cm^{-1}): 3058(w), 2977(w), 1966(w), 1859(w), 1569(s), 1448(s), 1423(s), 1283(m), 1152(m), 1083(m), 1043(m), 987(s), 769(s), 738(s), 525(s), 508(s), 447(w), 422(w), 396(w), 292(w), 247(m). ^1H-NMR (DMSO-d_6, 300 MHz, 25°C): $\delta = 7.59$, 7.98, 8.68. ^{31}P-NMR (DMSO-d_6, 121.4 MHz, 25°C): $\delta = 16.05$ ppm. Anal. found: N 10.36%, C 45.06%, H 3.05%; Calcd for $C_{30}H_{24}Cl_2HgN_6P_2$ (802): N 10.48%, C 44.93%, H 3.02%.

2.4. *Synthesis of [HgBr$_2$(PPh(2-py)$_2$)$_2$] (3)*

HgBr$_2$ (0.077 mmol, 0.028 g) was dissolved in 10 mL MeOH and a solution of PPh(2-py)$_2$ (0.039 mmol, 0.01 g) in 5 mL CH$_2$Cl$_2$ was added with vigorous stirring. A white precipitate was immediately formed. The slurry was stirred for 30 min. The mixture of solvents was removed by rotary evaporator and the white residue was washed with cold MeOH (10 mL) and Et$_2$O (10 mL) and dried *in vacuo*. Resulting white powder was recrystallized from concentrated DMF solution. Yield 94.7%; m.p. = 313°C (dec). IR (cm^{-1}): 3043(m), 2981(w), 1568(s), 1481(m), 1450(s), 1422(s), 1320(w), 1283(m), 1155(w), 1099(m), 1045(w), 987(m), 768(s), 742(s), 691(s), 617(w), 511(s), 492(sh), 428(w), 395(w), 277(w). ^{1}H-NMR (DMSO-d$_6$, 300 MHz, 25°C): $\delta = 7.48$–7.52(m), 7.56–7.61(m), 7.66–7.72(m), 8.58. ^{31}P-NMR (DMSO-d$_6$, 121.4 MHz, 25°C): $\delta = 17.3$ ppm ($J = 284$). Anal. found: N 6.19%, C 45.21%, H 2.99%; Calcd for C$_{32}$H$_{26}$Br$_2$HgN$_4$P$_2$ (888): N 6.30%, C 43.24%, H 2.95%. Crystals suitable for crystallographic structure determination were crystallized from hot concentrated DMF solution.

2.5. *Synthesis of [HgCl$_2$(PPh(2-py)$_2$)$_2$] (4)*

This complex was prepared in a manner analogous to **3** where HgCl$_2$ (0.077 mmol, 0.021 g) dissolved in 10 mL MeOH was used. White powder was recrystallized from hot concentrated DMF solution. Yield 96.1%; m.p. = 318°C (dec). IR (cm^{-1}): 3044(m), 2985(w), 1969(w), 1568(s), 1482(w), 1450(s), 1425(m), 1322(w), 1283(w), 1187(w), 1100(m), 1045(w), 988(s), 770(s), 744(s), 693(s), 618(w), 512(s), 429(w), 395(w), 248(w). ^{1}H-NMR (DMSO-d$_6$, 300 MHz, 25°C): $\delta = 7.49$–7.54(m), 7.58–7.61, 7.72–7.78(m), 7.89(b), 8.56(b). ^{31}P-NMR (DMSO-d$_6$, 121.4 MHz, 25°C): $\delta = 17.31$ ppm. Anal. found: N 6.89%, C 48.19%, H 3.31%; Calcd for C$_{32}$H$_{26}$Cl$_2$HgN$_4$P$_2$ (800): N 7.00%, C 48.04%, H 3.28%.

2.6. *Crystal structure determination and refinement*

X-ray diffraction data were collected at room temperature on small crystals glued on top of glass capillaries by means of a Stoe imaging plane. The X-Area program was used for indexing and integrating the single crystal reflections. The structure of the title compound was solved by direct methods using SHELXS-97 and refined by full matrix least squares on F^2, SHELXL-97 [23]. Minimum and maximum final electron densities were −0.969 to 0.614 eÅ^{-3}, respectively. Absorption corrections were performed with the programs X-RED [24] and X-SHAPE [25]. Symmetry equivalent reflections were used to optimize crystal shape and size. All non-hydrogen atoms were refined anisotropically. Aromatic hydrogens were placed in calculated positions (C–H = 0.93 Å) and treated as riding on the respective carrier atom, with $U_{\text{iso}}(\text{H}) = 1.2$ (C). Plots were produced with Diamond, and Mercury programs, and PLATON [26] software was used to prepare materials for publication. A summary of the crystal data, experimental details, and refinement results is given in table 1.

Table 1. Crystallographic and structure refinements data for **3**.

Formula	$C_{32}H_{26}Br_2HgN_4P_2$
Formula weight	888.90
Temperature (K)	295(2)
Wavelength, λ (Å)	0.71073
Crystal system	Monoclinic
Space group	$P2_1/c$
Unit cell dimensions (Å, °)	
a	9.766(2)
b	19.316(4)
c	17.598(3)
β	100.72(3)
Volume (Å^3), *Z*	3261.8(11), 4
Absorption coefficient (mm^{-1})	7.30
F(000)	1704
Crystal size (mm^3)	0.24 × 0.20 × 0.11
θ range for data collection (°)	2.1–26.0
Index ranges	$-9 \le h \le 12$; $-23 \le k \le 23$; $-21 \le l \le 21$
Data collected	6402
R_{int}	0.058
Restraints/parameters	0/370
Goodness of fit on F^2 (S)	1.01
Final *R* indices (obs. data)[a]	$R_1 = 0.0410$, $wR_2 = 0.0586$
R indices (all data)[a]	$R_1 = 0.0684$, $wR_2 = 0.0634$
Largest difference peak and hole (e Å^{-3})	0.61 and −0.97

[a] $R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$, $wR_2 = [\Sigma(w(F_o^2 - F_c^2)^2)/\Sigma w(F_o^2)^2]^{1/2}$.

3. Results and discussion

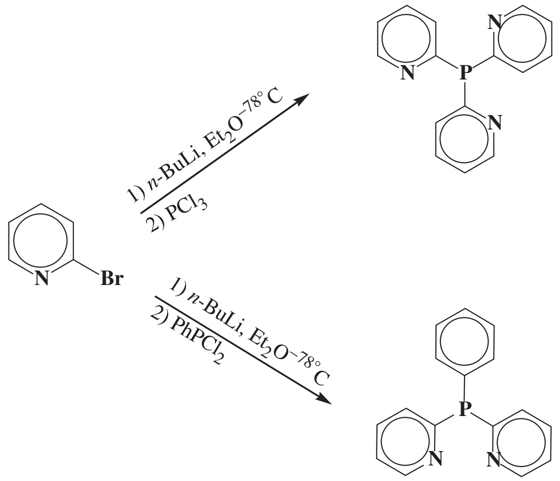
3.1. *Synthesis of ligands and complexes*

P(2-py)$_3$ and PPh(2-py)$_2$ were synthesized according to previously reported methods from 2-lithiopyridine and appropriate chlorophosphine [21, 22], as shown in scheme 1.

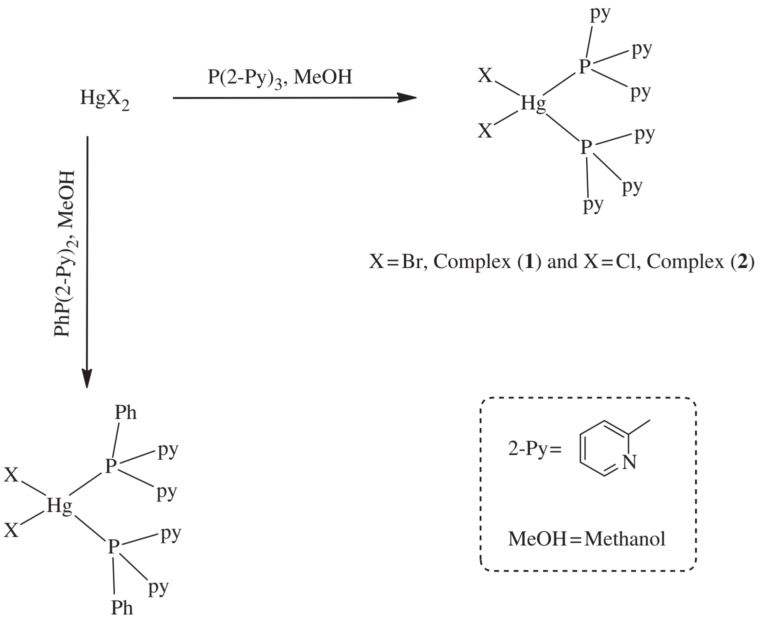
Reaction of mercury(II) halides in methanol with P(2-py)$_3$ and PPh(2-py)$_2$, dissolved in methylene chloride, in a 2 : 1 mole ratio resulted in white precipitates. Colorless crystals of all complexes, suitable for X-ray analysis, were grown by slow evaporation of concentrated DMF solution (scheme 2). The complexes were characterized by CHN elemental analysis, melting point determination, IR, ^{1}H and ^{31}P-NMR spectroscopies. The crystal structure of [HgBr$_2$(PPh(2-py)$_2$)$_2$] was determined by X-ray single crystal analysis. The *ν* Hg–Cl vibrations were found at 270 and 271 cm^{-1} [27]. The *ν* Hg–Br vibrations were not observed. The stretching vibrations of C=C and C=N bonds of pyridine rings were found between 1422–1450 cm^{-1} and 1568–1570 cm^{-1} as strong absorptions.

3.2. *Investigation of 1–4 in the solution state*

The ^{31}P-NMR spectra of all complexes were recorded in DMSO-d$_6$ and expected satellites due to $^1J(^{199}\text{Hg}^{31}\text{P})$ coupling (^{199}Hg 16.84% natural abundance, nuclear spin



Scheme 1. Synthesis of ligands.



X = Br, Complex (**1**) and X = Cl, Complex (**2**)

X = Br, Complex (**3**) and X = Cl, Complex (**4**)

Scheme 2. Synthesis of **1**–**4**.

$I = 1/2$) were observed. The $\delta(^{31}P)$ and $^{1}J(^{199}Hg^{31}P)$ coupling constants are listed in table 2. The $^{31}P\{^{1}H\}$-NMR parameters of **1**–**4** are summarized in table 2. NMR data for **1**–**4** are consistent with structures in scheme 2, showing similar phosphorus.

The trend of the $\delta(P)$ values in **1**–**4** (table 2) is the same. The same is true for the ^{1}J(Hg–P) values for **1**–**4** in agreement with the same coordination mode for all complexes.

Table 2. $^{31}P\{^{1}H\}$-NMR data for **1**–**4**.[a]

Compound	$\delta(P)$	^{1}J(Hg–P)
1	16.73	349
2	16.05	2270
3	17.30	284
4	17.31	1048

[a] J values in Hz. Spectra were run at 298 K. DMSO-d_6 was used as solvent.

According to table 2, the ^{1}J(Hg–P) coupling constants for bromide complexes are much smaller than chloride complexes. Generally, the J value is related to the bond distance, the longer bond distance resulted in a low value of coupling constant [28–30].

The ^{1}H-NMR resonances (DMSO-d_6) for the aromatic protons of pyridine rings for **1**–**4** occur between 7.48 and 8.70. For all of them, the integrated intensities are in agreement with the proposed structures illustrated in scheme 2.

3.3. *IR spectra*

IR spectra of **1** and **2** show distinct vibrational bands at 1570, 1508, and 1569 cm^{-1} assigned as C=N bond vibrations (pyridine rings) and at 1447, 1423, 1448, and 1422 cm^{-1} which were attributed to the C=C bond vibrations. Bands at 617, 523, 510, and 496 for **1**, and 619, 525, and 508 cm^{-1} for **2** are attributed to the vibrations of the P–C bond. The IR spectra of **3** and **4** show distinct vibrational bands at 1568, 1481, and 1482 cm^{-1} assigned as C=N bond vibrations, and at 1450, 1422, and 1425 cm^{-1} which were attributed to the C=C bond vibrations. Bands at 617, 510, and 492 for **3**, and 618 and 512 cm^{-1} for **4** have been attributed to P–C vibrations. Further information about complex formation was obtained from Far-IR spectra. New bands at 247 and 248 cm^{-1} have been assigned to Hg–Cl vibrations in **2** and **4**, respectively [27].

3.4. *Description of the molecular structure of [$HgBr_2(PPh(2\text{-}py)_2)_2$] (3)*

Colorless prismatic crystals of **3** were obtained by recrystallization of **3** in hot DMF. Also, **3** crystallizes in the monoclinic space group $P2_1/c$ (no. 14) with four molecules in the unit cell. The structure consists of a mercury with two bromides and two *tris*(2-pyridyl)phosphines that are monodentate P-donors, giving a distorted tetrahedral geometry, figure 1. The Hg–P ($PhP(2\text{-}py)_2$) average bond distance is 2.5030(15) Å and the Hg–Br average bond distance is 2.6309(8) Å. These bond distances are comparable to similar mercuric bromide–tertiary phosphine complexes [27–30].

All pyridine rings have nitrogens pointing in the same direction as the phosphorus (figure 1). The phenyl rings A (C11–C16) and A′ (C27–C32) and pyridine rings B (N1/C1/C5–C2), C (N2/C6/C10–C7), B′ (N3/C17/C21–C18), and C′ (N4/C23–C26/C22) are, of course, planar. The dihedral angles between related rings in two different (2-pyridyl)phenylphosphine moieties A to A′, B to B′, and C to C′ are 34.6°, 10.6°, and 27.0°, respectively.

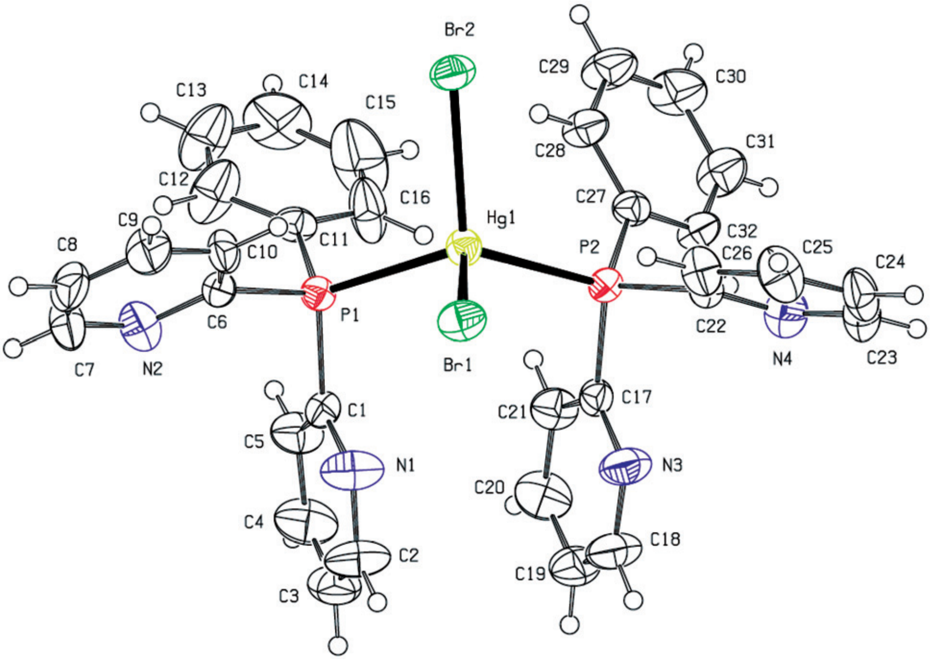


Figure 1. ORTEP diagram of [$HgBr_2(PPh(2\text{-}py)_2)_2$] (**3**) with atom numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 40% probability level.

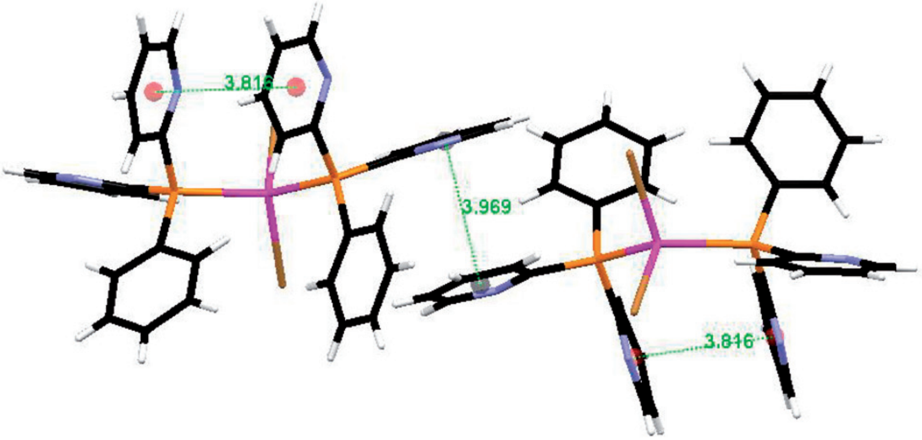


Figure 2. Inter- and intramolecular $\pi\cdots\pi$ interactions in [$HgBr_2(PPh(2\text{-}py)_2)_2$] (**3**).

Relatively strong intra $\pi\cdots\pi$ interactions exist between two pyridine rings (B and B′) from two different (2-pyridyl)phenylphosphine moieties ($Cg1\cdots Cg3 = 3.82$ Å, $Cg1$ and $Cg3$ being the rings B and B′, respectively; figure 2). Crystal packing is dominated by C–H$\cdots\pi$ and $\pi\cdots\pi$ interactions (figures 2 and 3) between neighboring molecules

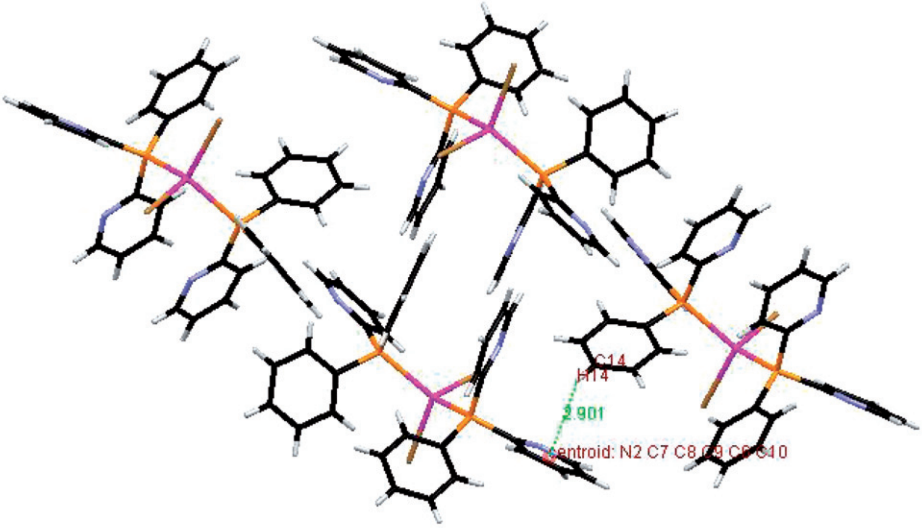


Figure 3. C–H $\cdots\pi$ interaction observed in packing of **3**.

Table 3. Selected bond distances (Å) and angles (°) for **3**.

Hg(1)–P(1)	2.5124(15)	P(2)–Hg(1)–(P1)	116.59(5)
Hg(1)–P(2)	2.4937(15)	P(2)–Hg(1)–Br(1)	111.43(4)
Hg(1)–Br(1)	2.6194(8)	P(1)–Hg(1)–Br(1)	105.97(4)
Hg(1)–Br(2)	2.6424(11)	P(2)–Hg(1)–Br(2)	111.31(4)
P(1)–C(1)	1.824(5)	P(1)–Hg(1)–Br(2)	107.07(4)
P(1)–C(6)	1.811(6)	Br(1)–Hg(1)–Br(2)	103.52(3)
P(1)–C(11)	1.816(6)		
P(2)–C(17)	1.827(5)		
P(2)–C(22)	1.815(6)		
P(2)–C(27)	1.826(6)		

(C14–H14 $\cdots Cg2^{i} = 2.90$ Å, $Cg4 \cdots Cg2^{ii} = 3.97$ Å, $Cg2$ being the ring N2/C6/C10/C9/C8/C7, and Cg4, N4/C22/C26/C25/C24/C23, with $^{i} = x$, $0.5 - y$, $-0.5 + z$, and $^{ii} = -z$, $0.5 + y$, $0.5 - z$). Crystallographic data, selected bond lengths (Å) and angles (°) are listed in table 3.

4. Conclusion

Four 2 : 1 mercuric halide (bromide and chloride) complexes with two multidentate pyridylphosphines, *tris*(2-pyridyl)phosphine, P(2-py)$_3$, and *bis*(2-pyridyl)phenylphosphine, PhP(2-py)$_2$, were synthesized. These ligands are monodentate P-donors. The synthesized complexes were characterized fully with IR and NMR. The ^{31}P-NMR spectra of these complexes are similar suggesting similar coordination modes. The solid structure of [HgBr$_2$(PPh(2-py)$_2$)$_2$] was determined by X-ray crystallography.

The packing of [$HgBr_2(PPh(2\text{-py})_2)_2$] is stabilized by weak C–H $\cdots\pi$ and $\pi\cdots\pi$ interactions.

Supplementary material

Full crystallographic details are deposited with the Cambridge Structural Database Centre (CCDC No. 766530 for **3**). Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB21EZ, UK (Fax: (44)01223336033; Email: deposit@ccdc.ac.uk).

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