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Mercuric Halide Complexes of Tertiary Phosphines. Part I. The Crystal Structures of 1:1 Complexes of Triphenylphosphine, Tributylphosphine and 1,2,5-triphenylphosphole

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Complete crystal structure analyses have been carried out for 1:1 adducts of mercuric chloride with triphenylphosphine and 1,2,5-triphenylphosphole. In each case the structure consists of discrete centrosymmetric chlorine-bridged dimers, with mercury atoms in distorted tetrahedral environments, but with different Hg–Cl bridge distances. Preliminary single-crystal X-ray photographs indicate that $HgX_2(PPh_3)$ [X = Br or I] and $HgBr_2$(1,2,5-triphenylphosphole) are isostructural with their chloro analogues.

A full X-ray study has shown that the α-form of $HgCl_2(PBu_3)$ comprises a discrete tetrameric unit, in which two unsymmetric dimers, related by a centre of symmetry, are loosely linked by further Hg–Cl bridges.

Introduction

Although the literature contains numerous reports of 1:1 complexes of mercuric halides with neutral unidentate ligands [1], alleged to have discrete centrosymmetric halogen-bridged dimeric structures, there is a marked lack of unambiguous evidence for their structure. The most common structural probe employed has been infrared spectroscopy. For a C_{2h} model, one predicts that there will be one $\nu(HgX)_{terminal}$ and two $\nu(HgX)_{bridge}$ modes active in the infrared spectrum. There are several instances in the literature [2–7] where the assignment of just the infrared-active $\nu(HgX)_{terminal}$ mode has been used in evidence for asserting the dimeric structure. Such assertions must be viewed with suspicion since the presence of halogen-bridges has been inferred without actually observing $\nu(HgX)_{bridge}$; moreover, alternative interpretations are equally valid. Even when $\nu(HgX)_{bridge}$ modes have been assigned there appears to be poor agreement as to their wavenumber positions [2, 3, 8–13].

There are now three compounds of the type HgX_2(L) for which the halogen-bridged dimeric structure has been confirmed by crystallographic studies (L = Ph_3PSe [14], methyl pyrolidine-1-carbodithioate (MPC) [15], or 1-methylcytosine [16]), but there are even more examples of rather different arrangements [17]. The factors determining the type of structure adopted are not clear, largely because the range of ligands used is very wide and systematic studies have not yet been made, nor is it clear to what extent vibrational spectroscopy may be used to characterise the structures.

We have therefore undertaken a systematic study of complexes $HgCl_2$(TPP) (I; TPP = 1,2,5-triphenylphosphole), $HgCl_2(PPh_3)$ (II), and α-$HgCl_2(PBu_3)$ (III). A preliminary report has been published [17].

Experimental

Preparation of Complexes

α-$HgCl_2(PBu_3)$, as distinct from the β-form [11], was prepared by dissolving PBu_3 (0.005 mol) and $HgCl_2$ (0.005 mol) separately in minimal quantities of ethanol. The BPu_3 solution was added dropwise to the solution of $HgCl_2$, whereupon a fine white precipitate formed immediately. When approximately half of the PBu_3 solution had been added, the precipitate was collected, washed in ethanol, then ether, and dried *in vacuo*; it was then recrystallised from benzene/heptane (*Anal.* Found: C, 30.4; H, 5.7%. Calcd. for $C_{12}H_{27}PHgCl_2$: C, 30.4; H, 5.7%). Separate experiments showed that if all the PBu_3 solution was added to the $HgCl_2$ solution, the white precipitate initially formed redissolved and subsequent attempts to isolate a complex of 1:1 stoicheiometry led to the formation of an impure sample of a 3:2 complex. *Anal.* Found: C, 35.8; H, 6.8%. Calcd. for $C_{36}H_{81}P_3Hg_2Cl_4$: C, 37.6; H, 7.1%). All other complexes studied have been previously described [1].

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TABLE I. Crystal Data and Details of Data Collection.

	$HgCl_2(TPP)$	$HgCl_2(PPh_3)$	α-$HgCl_2(PBu_3)$	
Crystal Data				
M_r	583.84	533.78	473.75	
Crystal System	triclinic	monoclinic	monoclinic	
a/Å	11.854(10)	12.304(8)	13.698(12)	13.472(9)[a]
b/Å	10.041(10)	11.356(7)	25.475(17)	24.960(9)[a]
c/Å	9.443(9)	13.444(10)	10.621(9)	10.391(14)[a]
α/°	84.16(5)			
β/°	103.38(5)	92.50(5)	100.75(5)	102.44(4)[a]
γ/°	114.29(5)			
U/Å^3	996.6	1876.6	3641.2	3412.1
D_m/g cm^{-3}	1.97	1.92	1.70	
Z	2	4	8[b]	
F(000)	556	1008	1824	
μ(Mo-K$_\alpha$)/cm^{-1}	77.56	82.33	84.79	
Space Group	$P\bar{1}$	$P2_1/n$	$P2_1/n$	
Collection of Intensity Data				
Reflections collected	2932	3340	4380	
Number of observed reflections[c]	1272	1616	1464	
$I/\sigma(I)$	4.0	3.0	4.0	
Final R value	0.070	0.083	0.083	
Final R' value	0.074			

[a] Unit cell parameters at *ca.* 100 K. [b] Two independent molecules in the asymmetric unit. [c] Those reflections having $I/\sigma(I)$ greater than the indicated value were considered to be observed. The net intensity $I = T - B$, where T = scan count, B = mean background count over the scan width; $\sigma(I) = (T + Bc/2t)^{1/2}$, where c = scan time, t = time for background measurements at each end of the scan.

Far-infrared Spectra

Spectra were obtained in the 50–450 cm^{-1} region using a Beckmann-RIIC FS-720 Fourier spectrometer, with the samples as pressed discs in polyethene cooled to *ca.* 30 K.

Crystal Data

These are summarised in Table I.

X-Ray Intensity Measurements

The crystals for all three compounds were mounted with their *b*-axes coincident with the rotation (ω) axis of a Stöe Stadi 2 two-circle diffractometer. Data were collected using the background-ω scan-background technique. Lorentz and polarisation corrections were applied and corrections for absorption effects were made for (I) [18].

Structure Determination and Refinement

The position of the mercury atoms were determined from the three-dimensional Patterson function for all three compounds. The remaining non-hydrogen atoms were located from successive difference electron-density maps. All phenyl rings were refined as regular C_6 hexagons (C–C, 1.395 Å; C–C–C bond angles 120°). Scattering factors were calculated [19] using an analytical approximation. Full matrix refinement was used with anisotropic temperature factors applied to mercury, phosphorus and chlorine and with isotropic temperature factors assigned to all carbons. Unit weights were applied to data for (II) and (III), while the weighting scheme $w = 1.4848/[\sigma^2(F_o) + 0.0027(F_o)^2]$ was adopted for (I). Final atomic parameters are listed in Table II; bond distances and angles are in Table III. Observed and calculated structure factors and thermal parameters have been deposited and are available from the Editor.

Structure Calculations

All calculations, apart from preliminary processing, were carried out on an IBM 370/165 computer using the SHELX computing package [20].

TABLE II. Final Fractional Coordinates ($\times 10^4$) for Non-hydrogen Atoms with Estimated Standard Deviations in Parentheses.

	$HgCl_2(TPP)$				$HgCl_2(PPh_3)$				α-$HgCl_2(PBu_3)$		
	x	*y*	*z*		*x*	*y*	*z*		*x*	*y*	*z*
Hg	722(2)	1573(3)	1380(2)	Hg	1325(1)	899(1)	−143(1)	Hg1	4787(2)	1815(1)	2140(3)
Cl(1)	2907(10)	2799(17)	1209(14)	Cl(1)	2219(9)	239(13)	−1566(7)	Cl(1)	3283(16)	1735(7)	789(24)
Cl(2)	202(11)	−1152(14)	1563(9)	Cl(2)	759(7)	−1055(7)	737(6)	Cl(3)	5777(16)	1006(6)	1499(19)
P	−517(9)	2502(13)	2410(11)	P	1908(6)	2365(7)	1058(6)	P1	6079(16)	2373(9)	3140(24)
C1	−1458(31)	3336(46)	1022(37)	C11	3398(12)	2251(18)	1329(15)	C11	6902(117)	2767(62)	2446(181)
C2	−845(35)	4791(51)	1243(41)	C12	4012(12)	3243(18)	1598(15)	C12	6499(75)	2703(40)	864(116)
C3	419(34)	5375(50)	2399(41)	C13	5121(12)	3131(18)	1839(15)	C13	6944(81)	3049(42)	−144(123)
C4	575(31)	4262(48)	3192(37)	C14	5617(12)	2027(18)	1810(15)	C14	6875(64)	2967(33)	−1466(97)
C11	−1477(23)	1345(28)	3652(26)	C15	5003(12)	1035(18)	1541(15)	C21	5712(119)	2929(55)	4331(150)
C12	−1618(23)	−102(28)	3907(26)	C16	3893(12)	1147(18)	1300(15)	C22	4671(81)	2995(39)	4111(103)
C13	−2380(23)	−976(28)	4853(26)	C21	1241(15)	2272(19)	2180(12)	C23	4406(128)	3338(66)	5440(181)
C14	−3001(23)	−403(28)	5544(26)	C22	173(15)	1859(19)	2173(12)	C24	3510(91)	3455(42)	5530(115)
C15	−2860(23)	1045(28)	5289(26)	C23	−392(15)	1854(19)	3050(12)	C31	7184(109)	1986(60)	3346(164)
C16	−2098(23)	1919(28)	4343(26)	C24	111(15)	2262(19)	3934(12)	C32	7213(88)	1807(46)	4340(138)
C21	1667(19)	4387(33)	4455(21)	C25	1179(15)	2675(19)	3941(12)	C33	7990(151)	1531(69)	5391(176)
C22	2873(19)	5367(33)	4297(21)	C26	1744(15)	2680(19)	3064(12)	C34	8745(128)	1336(68)	5409(170)
C23	3895(19)	5666(33)	5479(21)	C31	1680(19)	3808(15)	532(16)	Hg2	4870(3)	403(1)	3283(3)
C24	3711(19)	4985(33)	6819(21)	C32	1259(19)	4720(15)	1096(16)	Cl(2)	4326(15)	1309(6)	4195(18)
C25	2504(19)	4005(33)	6977(21)	C33	1168(19)	5851(15)	696(16)	Cl(4)	6327(17)	271(7)	4727(23)
C26	1483(19)	3706(33)	5795(21)	C34	1496(19)	6071(15)	−266(16)	P2	3504(14)	74(7)	1846(23)
C31	−2647(19)	2424(29)	65(26)	C35	1916(19)	5160(15)	−829(16)	C41	3555(51)	252(24)	157(67)
C32	−3536(19)	3021(29)	−491(26)	C36	2008(19)	4028(15)	−430(16)	C42	2610(54)	147(26)	−661(76)
C33	−4664(19)	2208(29)	−1434(26)					C43	2753(57)	429(30)	−2108(76)
C34	−4903(19)	798(29)	−1820(26)					C44	1804(60)	387(31)	−3229(79)
C35	−4014(19)	200(29)	−1264(26)					C51	2657(77)	453(38)	2545(100)
C36	−2886(19)	1013(29)	−321(26)					C52	1798(97)	186(46)	1976(126)
								C53	863(90)	456(45)	2889(120)
								C54	−121(66)	309(33)	1807(87)
								C61	3373(61)	−666(29)	1855(80)
								C62	4476(71)	−876(37)	2051(97)
								C63	4305(66)	−1589(34)	2058(86)
								C64	5474(59)	−1753(30)	2341(75)

TABLE III. Bond lengths (Å) and Angles (°) with Estimated Standard Deviations in Parentheses.

Symmetry Code

none	*x*, *y*, *z*
(i)	−*x*, −*y*, −*z*
(ii)	1.0 − *x*, −*y*, 1.0 − *z*

Metal Coordination

Distances (Å)

	$HgCl_2$(TPP)	$HgCl_2(PPh_3)$
Hg–P	2.438(10)	2.406(7)
Hg–Cl(1)	2.404(11)	2.370(10)
Hg–Cl(2)	2.542(13)	2.623(8)
Hg–Cl(2$^{\mathrm{i}}$)	2.747(14)	2.658(8)
Hg–Hg$^{\mathrm{i}}$	3.855(4)	3.881(3)

α-$HgCl_2(PBu_3)$

Hg1–P1	2.363(21)	Hg2–Cl(3)	2.895(21)
Hg1–Cl(1)	2.289(21)	Hg2–Cl(4)	2.304(21)
Hg1–Cl(2)	2.709(20)	Hg2–Cl(4$^{\mathrm{ii}}$)	3.375(25)
Hg1–Cl(3)	2.626(19)	Hg1–Hg2	3.792(4)
Hg2–P2	2.337(19)	Hg2–Hg2$^{\mathrm{ii}}$	4.142(16)
Hg2–Cl(2)	2.664(18)		

Angles (°)

	$HgCl_2$(TPP)	$HgCl_2(PPh_3)$
P–Hg–Cl(1)	127.8(5)	128.7(4)
P–Hg–Cl(2)	118.4(4)	111.1(3)
P–Hg–Cl(2$^{\mathrm{i}}$)	110.3(4)	114.3(3)
Cl(1)–Hg–Cl(2)	107.4(5)	103.8(3)
Cl(1)–Hg–Cl(2$^{\mathrm{i}}$)	95.4(4)	104.7(3)
Cl(2)–Hg–Cl(2$^{\mathrm{i}}$)	86.5(4)	85.4(3)
Hg–Cl(2)–Hg$^{\mathrm{i}}$	93.5(4)	94.6(3)

α-$HgCl_2(PBu_3)$

P1–Hg1–Cl(1)	147.8(7)	P2–Hg2–Cl(4)	150.6(7)
P1–Hg1–Cl(2)	101.1(7)	P2–Hg2–Cl(4$^{\mathrm{ii}}$)	78.7(6)
P1–Hg1–Cl(3)	102.1(7)	Cl(2)–Hg2–Cl(3)	87.8(6)
Cl(1)–Hg1–Cl(2)	98.4(7)	Cl(2)–Hg2–Cl(4)	98.6(7)
Cl(1)–Hg1–Cl(3)	102.4(7)	Cl(2)–Hg2–Cl(4$^{\mathrm{ii}}$)	90.7(7)
Cl(2)–Hg1–Cl(3)	92.6(6)	Cl(3)–Hg2–Cl(4)	95.1(7)
Hg1–Cl(2)–Hg2	89.8(6)	Cl(3)–Hg2–Cl(4$^{\mathrm{ii}}$)	176.4(9)
Hg1–Cl(3)–Hg2	86.6(7)	Cl(4)–Hg2–Cl(4$^{\mathrm{ii}}$)	88.3(7)
P2–Hg2–Cl(2)	107.7(6)	Hg2–Cl(4)–Hg2$^{\mathrm{ii}}$	91.7(6)
P2–Hg2–Cl(3)	98.7(7)		

Ligand Geometries

$HgCl_2$(TPP)

Distances

P–C1	1.88(4)	P–C4	1.81(4)	P–C11	1.81(2)
C1–C2	1.35(6)	C1–C31	1.47(4)	C2–C3	1.56(2)
C3–C4	1.33(6)	C4–C21	1.52(4)		

Angles

Hg–P–Cl	113.7(11)	Hg–P–C4	106.3(12)	Hg–P–C11	118.7(10)
C1–P–C4	92.6(18)	C1–P–C11	110.1(14)	C4–P–C11	112.4(15)

(continued on facing page)

TABLE III. *(continued)*

P–C1–C2	105(3)	P–C1–C31	121(3)	C2–C1–C31	133(4)
C1–C2–C3	119(4)	C2–C3–C4	110(3)	P–C4–C3	113(3)
P–C4–C21	121(3)	C3–C4–C21	126(3)	P–C11–C12	121(1)
P–C11–C16	119(1)	C4–C21–C22	117(1)	C4–C21–C26	122(1)
C1–C31–C31	118(2)	C1–C31–C36	122(2)		
$HgCl_2(PPh_3)$					
Distances					
P–C11	1.86(2)	P–C21	1.75(2)	P–C31	1.80(2)
Angles					
Hg–P–C11	110.3(7)	Hg–P–C21	113.5(8)	Hg–P–C31	109.2(8)
C11–P–C21	108.8(10)	C11–P–C31	106.0(10)	C21–P–C31	108.7(11)
P–C11–C12	120.9(7)	P–C11–C16	119.1(7)	P–C21–C22	119.4(7)
P–C21–C26	120.4(7)	P–C31–C32	121.2(8)	P–C31–C36	118.7(8)
α-$HgCl_2(PBu_3)$					
*Distances**					
P–C	1.852	C–C	1.539		
*Angles**					
Hg–P–C	111.8		C–P–C	106.6	
P–C–C	104.7		C–C–C	113.8	

*Average values.

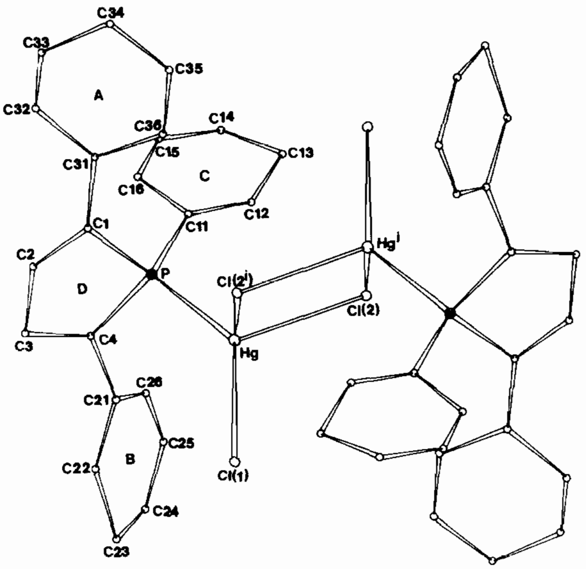

Fig. 1. The molecular structure of $HgCl_2$(1,2,5-triphenylphosphole). Molecular parameters are given in Table III.

Results and Discussion

Both $HgCl_2$(TPP) and $HgCl_2(PPh_3)$ consist of centrosymmetric dimers in which the ligands are mutually *trans* (Figs. 1 and 2). However, the halogen-bridged dimers differ in one important aspect, *viz.* the relative lengths of the $Hg–Cl_{bridge}$ distances. In (II) these bridging distances are almost equal [2.623 and 2.658(8) Å], while in the TPP adduct they are quite different [2.54 and 2.75(1) Å] and resemble the arrangements found in $HgCl_2(SePPh_3)$ (2.60, 2.78 Å; [14]) and $HgCl_2$(MPC) (2.57, 2.78 Å; [15]). Although the means of the $Hg–Cl_{bridge}$ distances in (I) and (II) are almost identical (2.641 and 2.645 Å, respectively), the asymmetry of the bridges leads to significant differences in the associated $\nu(HgCl)_{bridge}$ frequencies. Thus while both (I) and (II) contain one prominent band in the 280–300 cm^{-1} region of the IR spectrum which can be ascribed to $\nu(HgCl)_{terminal}$ [(I), 283; (II), 297 cm^{-1}], the two $\nu(HgCl)_{bridge}$ modes which are almost coincident for the triphenylphosphine adduct [183, 188 cm^{-1}] are found to be well separated in $HgCl_2$(TPP) [156, 219 cm^{-1}] [13, 21].

α-$HgCl_2(PBu_3)$ also contains dimeric units, but in contrast to (I) and (II), pairs of dimers are weakly linked together in a centrosymmetric manner to give a tetrameric arrangement (Fig. 3). Although the Hg2–Cl(4^{ii}) distance of 3.38(3) Å is long, it does lie within the sum of the Van der Waals' radii (3.45 Å, [22]); moreover, examination of the packing arrangements shows that the tetramers are well separated. Since Hgl and Hg2 are not related by a centre of symmetry, the tetrameric unit contains

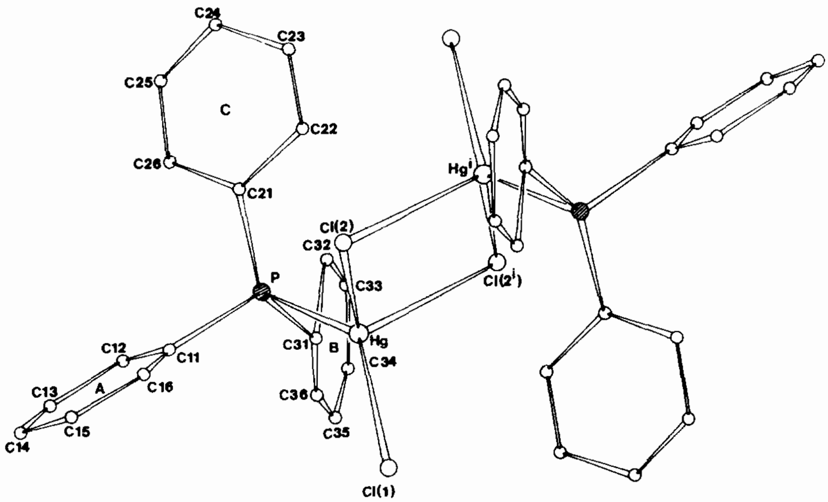

Fig. 2. The molecular structure of $HgCl_2(PPh_3)$. Molecular parameters are given in Table III.

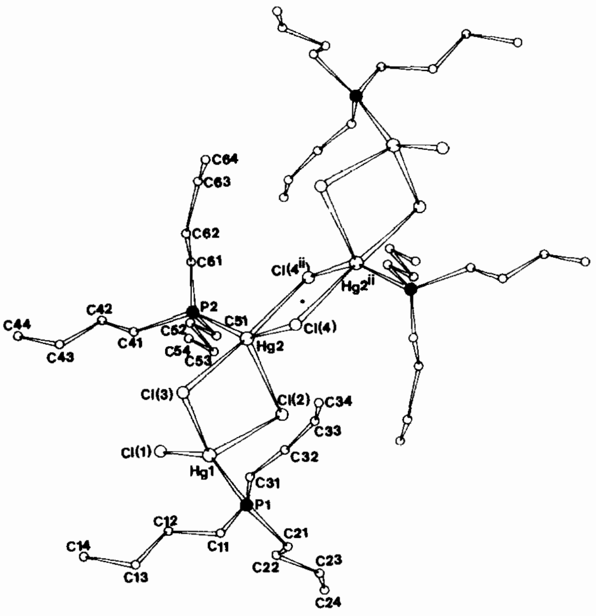

Fig. 3. The molecular structure of α-$HgCl_2(PBu_3)$. Molecular parameters are given in Table III.

a total of six unique Hg–Cl_{bridge} distances, ranging from 2.29 to 3.38 Å in length. The complex nature of the ir spectrum in the region characteristic of $\nu(HgCl)_{bridge}$ modes [21] is a reflection of this large number of differing Hg–Cl bridging distances.

The coordination geometry about mercury is considerably distorted in all three complexes. In (I) and (II) mercury is four-coordinate, with bond angles about the metal varying from 86.5 to 127.8° for (I), and from 85.4 to 128.7° for (II). In each case it is the P–Hg–$Cl_{terminal}$ bond angle which is the largest. In the tetrameric arrangement of (III), both four- and five-coordinate mercury atoms are found, and it is significant that the P–Hg–$Cl_{terminal}$ angle opens up to 147.8° and that the analogous angle about Hg2, P2–Hg2–Cl(4), has a similarly large value of 150.6°. The variation within the series of this P–Hg–Cl bond angle, which involves the chlorine nearest to mercury, may be rationalised in terms of the relative strength of interaction between phosphorus and the metal. Considering the structures of the present three complexes and of $HgCl_2(PR_3)$, (R = Me or Et [17]), there appears to be a tendency with more strongly interacting phosphines for the linear Cl–Hg–Cl arrangement, found in mercury(II) chloride itself, to be replaced by a R_3P–Hg–Cl arrangement in which the P–Hg–Cl angle approaches linearity for the very small trimethylphosphine ligand [162.1(1)°]. Certainly, among the three complexes presently under consideration the greatest angular distortion about mercury occurs with the adduct of tributylphosphine, which is known [23] to give rise to a larger enthalpy of ligation with mercury(II) halides than either TPP or triphenylphosphine. It is also significant that tributylphosphine apparently gives rise to the shortest Hg–P distance though the differences are not significant.

Intermolecular distances indicate that no further association occurs beyond the dimer stage for (I) and (II) or beyond the tetramer stage for (III). The very open manner in which the tetrameric units pack together in (III) (Fig. 4) is reflected in the low melting point of the compound (*ca.* 80 °C), the high thermal parameter of the atoms, and in the reduction in the volume of the unit cell upon cooling (6.3% reduction from ambient to *ca.* 100 K, Table I).

The molecular parameters of the three ligands are as expected, and in particular the geometries of TPP

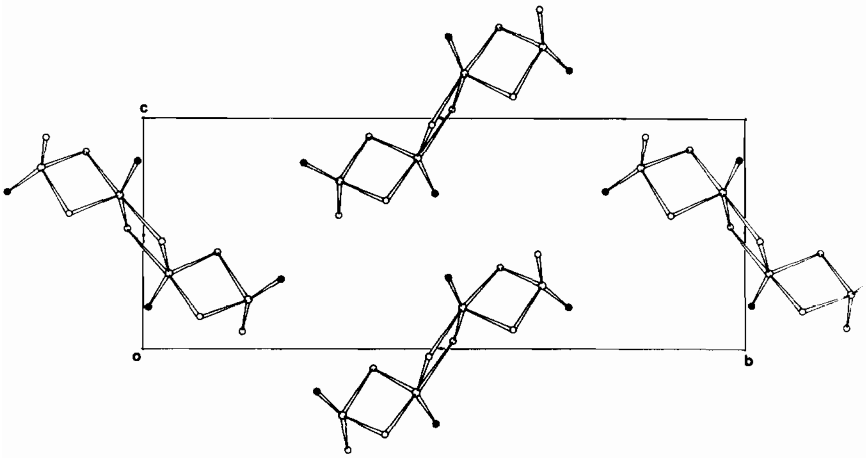

Fig. 4. View of the crystal structure of α-$HgCl_2(PBu_3)$ along the *a**-axis. Carbon atoms have been omitted for clarity.

TABLE IV. Angles Between Mean Planes (°).

$HgCl_2(TPP)$					
A/B	76.1	A/C	78.5	A/D	25.6
B/C	85.1	B/D	52.5	C/D	87.4
$HgCl_2(PPh_3)$					
A/B	84.4	A/C	75.4	B/C	80.1

and triphenylphosphine are similar to those found in the free ligands themselves [24, 25]. The heterocyclic ring in (I) is almost planar (maximum deviation from the mean plane being 0.071 Å for C3), and the attached phenyl rings are rotated with respect to the phosphole ring and to each other so as to minimise steric interactions (Table IV). The phenyl rings of the triphenylphosphine ligand take up the characteristic propeller arrangement, with angles between the rings (Table IV) being similar to those found in the free phosphine [25].

Preliminary photographic data indicate that $HgBr_2(TPP)$ and $HgX_2(PPh_3)$ [X = Br, I] have similar structures to those of the respective chloride complexes.

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