Mercury(II) Halide Complexes of Tertiary Phosphines. Part IV.[†] The Crystal Structure of $HgCl_2(PCy_3)$ and a Re-interpretation of its Vibrational Spectra

NORMAN A. BELL, TERENCE D. DEE, MICHAEL GOLDSTEIN* and IAN W. NOWELL

Department of Chemistry, Sheffield City Polytechnic, Pond Street, Sheffield S1 1WB, U.K.

Received August 3, 1982

The previously proposed dimeric structure of $HgCl_2(PCy_3)$ has been confirmed by full single-crystal X-ray diffraction analysis. However, rather than consisting of asymmetrically halogen-bridged dimeric molecules, the unit cell contains two independent centrosymmetric dimers. Differences between the molecular parameters of the two types of dimer are discussed and their origins considered. The structure determination has shown that the previous assignments of the far-infrared and Raman spectra are erroneous, and a correct re-interpretation is given.

Introduction

On the basis of multiplicity of $\nu(HgCl)_t$ bands in the far-infrared and Raman spectra of the 1:1 adduct $HgCl_2(PCy_3)$, Moers and Langhout [2] proposed a non-centrosymmetrically halogen-bridged dimeric structure in the solid state. Subsequently, Allman, Goel and Pilon [3] argued that since only one infrared and one Raman band were observed in the $\nu(HgP)$ region, and because only a single strong polarized Raman band was shown in the $\nu(HgCl)_t$ region for solutions in benzene and dichloromethane, the structure of the complex must be centrosymmetric. However, $\nu(HgP)$ bands are notoriously difficult to locate, and no explanation of the splitting of $\nu(HgCl)_t$ in the solid state was given.

During the course of our investigations of the variations in structure of complexes of the type $HgX_2(PR_3)$ [1, 4, 5], we had cause to determine the crystal and molecular structure of $HgCl_2(PCy_3)$. The results, which are presented herein, have allowed correct assignments to be made of the vibrational spectra, and provide a clarification of the previous [2, 3] assertions; they also afford a striking illustration of the dangers in making detailed proposals of molecular structure from vibrational spectroscopic data alone.

Experimental

Dichloro(tricyclohexylphosphine)mercury(II) was prepared essentially as in [3] by addition of solid $HgCl_2$ to a solution of the phosphine in dichloromethane in 1:1 molar proportions. A clear solution was obtained from which the white complex was recovered on slow evaporation at room temperature followed by recrystallisation from ethanol.

Far-infrared Spectrum

The spectrum in the region 450–50 cm^{-1} was obtained using a Beckman-RIIC FS-720 interferometer, with the sample as a pressed disc in polyethene cooled to *ca.* 50 K using a CTi Model 20 closed-cycle helium cryostat.

Crystal Data

$C_{18}H_{33}Cl_2HgP$, $M_r = 551.93$. Triclinic, $P\bar{1}$, $a = 10.843(2)$, $b = 14.118(2)$, $c = 14.792(3)$ Å, $\alpha = 94.76(1)$, $\beta = 80.38(1)$, $\gamma = 110.62(2)°$, $U = 2088.8$ $Å^3$, D_m (by flotation) $= 1.73$, $D_c = 1.76$ g cm^{-3}. $Z = 4$, Mo-K_α, $\lambda = 0.7107$ Å, μ(Mo-K_α) $= 73.97$ cm^{-1}, $F(000) = 1080$.

Intensity Measurements

A colourless crystal $0.30 \times 0.25 \times 0.30$ mm was selected. Cell dimensions were calculated from the centred settings of 25 reflections with $9 < \theta < 11°$, on an Enraf-Nonius CAD-4 diffractometer with monochromated Mo-K_α radiation. Intensities for reflections having θ in the range 1.3–20° were measured on the same instrument with a variable scan speed and an ω–$4/3\theta$ (goniometer-counter) scanning ratio, as optimised by peak analysis routines. Of the 5116 reflections measured, 3563 had $I \geqslant 3\sigma(I)$ and were considered to be observed. Corrections were made for Lorentz, polarisation and absorption effects. The intensities of two central reflections were monitored and showed negligible deterioration.

Structure Determination and Refinement

The positions of the two independent mercury atoms were located from the three-dimensional

---

[†]Part III: ref. [1].

*Author to whom correspondence should be addressed.

Patterson function and the remaining atoms were located from successive difference electron-density maps. Hydrogen atoms were included in positions calculated from the geometry of the molecule (C–H, 1.08 Å). A common isotropic temperature factor was applied to the cyclohexyl hydrogen atoms and refined to a final value of $U = 0.113(22)$ Å^2. Scattering factors were calculated [6] using an analytical approximation, and the weighting scheme adopted was $w = 1.4963/[\sigma^2(F_o) + 0.0003\ (F_o)^2]$. Full matrix refinement with anisotropic temperature factors for all non-hydrogen atoms gave the final $R = 0.039$ and $R' = 0.040$. The final difference-Fourier map showed no peaks greater than 1.21 e Å^{-3}. Final atomic parameters are listed in Table I; bond distances and angles are in Table II. A list of observed and calculated structure factors is available from the Editor.

Calculations

All calculations, apart from preliminary data processing, were carried out on an IBM 370/165 computer at the SERC Computing Centre, Daresbury, using the SHELX computing package [7].

Results and Discussion

HgCl$_2$(PCy$_3$) is found to be dimeric, but while the present study confirms the centrosymmetric nature of these units, it also reveals that the two dimers contained within the unit cell are crystallographically independent (Figs. 1 and 2). Examination of the bond distances and angles within the two dimers (Table II), shows that although the Hg–P distances are almost identical, being 2.416(3) and 2.412(3) Å for dimers 1 and 2 respectively, in almost all other respects the two dimers show quite striking differences:

(i) The Hg–Cl$_{terminal}$ distances [2.391(5) Å, dimer 1; 2.413(3) Å, dimer 2] differ significantly.

(ii) The Hg–Cl$_{bridge}$ distances in dimer 1 are far less asymmetric [2.641, 2.665(4) Å] compared with those in dimer 2 [2.602, 2.779(4) Å].

(iii) Analogous bond angles about mercury are all significantly different, the differences ranging from a minimum of 3.4° [Cl(2)–Hg–Cl(2')] to maximum values of 8.5° [Cl(2)–Hg–P] and 8.9° [Cl(2')–Hg–P].

In an attempt to isolate the origins of these marked differences between the two independent dimers, the short intra- and inter-dimer contacts have been examined and are summarised in Table III. It seems likely that differing H···Cl interactions are a significant influence upon the geometry adopted by the two dimeric units, with such intra-dimer contacts being of prime importance for the bridging chlorine atoms, and inter-dimer interactions being of more significance for the terminal chlorines. Projection down the Hg–P bond in the two dimers shows subtle differences in the arrangements of the tricyclohexylphosphine ligands within the dimeric units (Figs. 3 and 4). Thus the orientation of the Cl(1)HgCl(2)Cl(2') unit with respect to the three P–C bonds is significantly different, with dimer 1 adopting the more eclipsed arrangement. In addition, while the orientation of the cyclohexyl rings C21–C26 and C31–C36 with respect to the Hg–P bond is very similar in the two dimers, that of the C11–C16 rings is quite different. On going from dimer 1 to dimer 2, the C11–C16 ring is effectively inverted such that H111 is directed towards H322 in dimer 1 and towards H221 in dimer 2. The resulting arrangements lead to a greater number of short intra-dimer H···H contacts in dimer 1 (Table III) and it may be relevant that the C11–C16 ring in dimer 1 is found to be distorted (Table II).

In dimer 2 only one intra-dimer H···Cl contact is less than the sum of the van der Waals' radii (3.0 Å). The presence of this short contact (Cl(2)···H321, 2.801 Å) is consistent with the large tetrahedral distortion observed for the P–Hg–Cl(2) angle (120.3-(1)°). Accommodation of such a large P–Hg–Cl(2) angle, bearing in mind the crystallographically imposed planarity of the [HgCl$_2$Hg] bridging unit and the small variation in Cl(2)–Hg–Cl(2') angles found in such chlorine-bridged dimers [1, 4, 5], can be achieved by an increase in the Hg–Cl(2') bond distance and a decrease in the P–Hg–Cl(2') angle. The observed values of 2.779(4) Å for Hg–Cl(2') and 99.8(1)° for P–Hg–Cl(2') are in accord with this view. In dimer 1 however it is found that both bridging chlorine atoms are involved in a short intra-dimer contact (Table III) and this is consistent with the similarity of the P–Hg–Cl$_{bridge}$ angles (111.8, 108.7(1)°) and of the Hg–Cl$_{bridge}$ distances (2.641, 2.665(4) Å).

The closest H···Cl contacts for the terminal chlorine atom in each dimer involve neighbouring dimeric units and it may well be that the differences in the P–Hg–Cl(1) angles [139.6(2)°, dimer 1; 132.0(1)°, dimer 2] can be attributed to significant differences in these inter-dimer H···Cl(1) interactions. We have previously found [1, 4, 5] that the magnitude of the P–Hg–Cl$_{terminal}$ angle strongly reflects the strength of the interaction between phosphorus and the metal. In the light of the similarity of the enthalpies of ligation reported [8] for tricyclohexylphosphine and tributylphosphine in their reaction with mercury(II) halides, it is surprising that the P–Hg–Cl$_{terminal}$ angles in the present complex are considerably less than the values found [1, 4] in the α- and β-forms of HgCl$_2$(PBu$_3$) [α-form, 147.8(7), 150.6(7)°; β-form, 150.9(3)°]. The value of 132.0(1)° is comparable with that found [4] in HgCl$_2$(PPh$_3$) and this is particularly unexpected

TABLE I. Final Atomic Parameters.[a] Estimated Standard Deviations for Non-Hydrogen Atoms are Given in Parentheses.

Fractional Co-ordinates (Hg × 10^5; remaining atoms × 10^4)

	Dimer 1			Dimer 2		
	x	*y*	*z*	*x*	*y*	*z*
Hg	37978(5)	−43541(4)	−1577(3)	33714(5)	190(3)	45919(3)
Cl(1)	3336(7)	−4930(4)	−1672(3)	1539(4)	−1482(3)	5112(3)
Cl(2)	6400(3)	−3922(2)	−566(2)	5224(3)	−731(2)	3938(2)
P	3236(3)	−3259(2)	1059(2)	3440(3)	1602(2)	4045(2)
C11	1869(13)	−2934(10)	764(9)	2026(11)	1443(7)	3426(6)
C12	867(17)	−3667(12)	344(14)	1354(12)	341(8)	3107(9)
C13	−108(15)	−3382(12)	−44(11)	66(13)	266(9)	2733(8)
C14	−504(18)	−2564(12)	505(13)	374(13)	958(10)	1939(8)
C15	541(16)	−1795(12)	871(12)	1111(13)	2024(10)	2206(9)
C16	1498(14)	−2102(9)	1275(9)	2368(12)	2144(8)	2602(8)
C21	2804(12)	−3843(8)	2176(7)	3247(10)	2431(7)	5052(7)
C22	1637(13)	−4819(9)	2225(9)	2023(12)	1911(8)	5703(8)
C23	1488(17)	−5347(11)	3146(10)	1852(16)	2584(9)	6554(8)
C24	1365(17)	−4695(12)	3945(10)	1873(16)	3592(10)	6282(9)
C25	2500(20)	−3693(12)	3880(9)	3121(15)	4119(9)	5624(9)
C26	2633(17)	−3147(9)	2992(8)	3279(12)	3465(8)	4770(7)
C31	4651(11)	−2074(7)	1123(7)	5034(11)	2231(8)	3326(7)
C32	5164(12)	−1598(8)	161(8)	5336(13)	1530(9)	2577(8)
C33	6328(14)	−637(9)	179(9)	6671(12)	2042(11)	1984(8)
C34	7445(13)	−757(10)	561(9)	7814(13)	2411(12)	2554(9)
C35	6975(13)	−1220(10)	1513(9)	7498(12)	3115(10)	3324(9)
C36	5809(12)	−2222(9)	1501(8)	6164(11)	2623(9)	3934(7)
H111	2483	−2533	166	1336	1666	3936
H121	309	−4228	851	2019	127	2568
H122	1366	−4007	−212	1113	−155	3677
H131	315	−3122	−731	−597	473	3278
H132	−992	−4043	−68	−413	−504	2503
H141	−1269	−2911	1065	974	712	1377
H142	−903	−2213	60	−547	923	1726
H151	1104	−1282	327	462	2295	2710
H152	85	−1399	1403	1393	2476	1601
H161	1060	−2345	1965	3069	1949	2079
H162	2395	−1451	1295	2807	2919	2832
H211	3680	−4018	2235	4088	2583	5411
H221	742	−4659	2196	1166	1723	5353
H222	1794	−5313	1654	2098	1226	5923
H231	2356	−5557	3143	2647	2696	6949
H232	609	−6020	3191	906	2211	6967
H241	437	−4557	3997	1007	3485	5960
H242	1367	−5073	4550	1850	4065	6892
H251	3419	−3827	3891	3982	4289	5967
H252	2332	−3215	4461	3058	4814	5417
H261	1739	−2975	2987	2475	3355	4384
H262	3482	−2454	2951	4220	3836	4350
H311	4293	−1571	1596	4975	2870	3004
H321	5456	−2133	−312	5365	867	2877
H322	4367	−1436	−70	4559	1307	2150
H331	6001	−80	595	6624	2683	1658
H332	6686	−374	−513	6861	1505	1465
H341	7857	−1245	106	7918	1770	2844
H342	8205	−22	615	8731	2814	2125
H351	6660	−694	1984	7462	3773	3021
H352	7793	−1353	1742	8280	3346	3747
H361	6140	−2769	1071	6210	1998	4286
H362	5469	−2496	2191	5963	3174	4431

[a]Hydrogen atoms are labelled H*abc*, where *ab* corresponds to the carbon atom they are attached to and *c* = 1 or 2 for axial or equatorial hydrogens.

N. A. Bell, T. D. Dee, M. Goldstein and I. W. Nowell

TABLE II. Bond Lengths (Å) and Angles (°) with Estimated Standard Deviations in Parentheses.

Symmetry Code
none x, y, z ; (') $1 - x, - 1 - y, - z$ dimer 1
         (') $1 - x, - y, 1 - z$ dimer 2

Bond Lengths (Å)

	Dimer 1	Dimer 2
Hg–Cl(1)	2.391(5)	2.413(3)
Hg–Cl(2)	2.641(4)	2.602(4)
Hg–Cl(2')	2.665(4)	2.779(4)
Hg–P	2.416(3)	2.412(3)
P–C11	1.824(17)	1.853(12)
P–C21	1.823(11)	1.847(10)
P–C31	1.837(9)	1.835(10)
C11–C12	1.402(21)	1.540(14)
C11–C16	1.482(20)	1.546(16)
C12–C13	1.467(29)	1.554(21)
C13–C14	1.501(25)	1.508(19)
C14–C15	1.413(23)	1.489(17)
C15–C16	1.480(27)	1.523(20)
C21–C22	1.505(15)	1.493(14)
C21–C26	1.521(16)	1.538(16)
C22–C23	1.565(21)	1.540(17)
C23–C24	1.455(21)	1.503(22)
C24–C25	1.511(21)	1.517(18)
C25–C26	1.538(20)	1.524(17)
C31–C32	1.555(15)	1.497(17)
C31–C36	1.540(19)	1.557(16)
C32–C33	1.495(15)	1.527(15)
C33–C34	1.484(23)	1.529(19)
C34–C35	1.533(18)	1.526(20)
C35–C36	1.531(15)	1.532(15)
Hg···Hg	3.773(1)	3.937(1)

Bond Angles (°)

	Dimer 1	Dimer 2
Cl(1)–Hg–Cl(2)	95.2(2)	101.5(1)
Cl(1)–Hg–Cl(2')	100.8(2)	106.1(1)
Cl(1)–Hg–P	139.6(2)	132.0(1)
Cl(2)–Hg–Cl(2')	89.4(1)	86.0(1)
Cl(2)–Hg–P	111.8(1)	120.3(1)
Cl(2')–Hg–P	108.7(1)	99.8(1)
Hg–P–C11	107.4(4)	112.2(3)
Hg–P–C21	112.2(4)	108.0(4)
Hg–P–C31	110.2(4)	111.2(4)
C11–P–C21	111.7(6)	105.6(5)
C11–P–C31	107.4(6)	110.7(5)
C21–P–C31	107.9(5)	108.8(4)
P–C11–C12	119.6(12)	112.8(9)
P–C11–C16	119.7(10)	114.7(7)
C12–C11–C16	116.2(14)	109.0(9)
C11–C12–C13	120.5(15)	108.3(11)
C12–C13–C14	113.3(15)	111.3(10)
C13–C14–C15	115.1(16)	110.9(11)
C14–C15–C16	117.9(15)	113.2(13)
C15–C16–C11	114.4(12)	110.1(9)
P–C21–C22	113.5(9)	110.8(7)
P–C21–C26	115.2(9)	111.9(7)
C22–C21–C26	110.7(10)	112.3(10)
C21–C22–C23	109.6(11)	111.7(9)

Bond Angles (°)

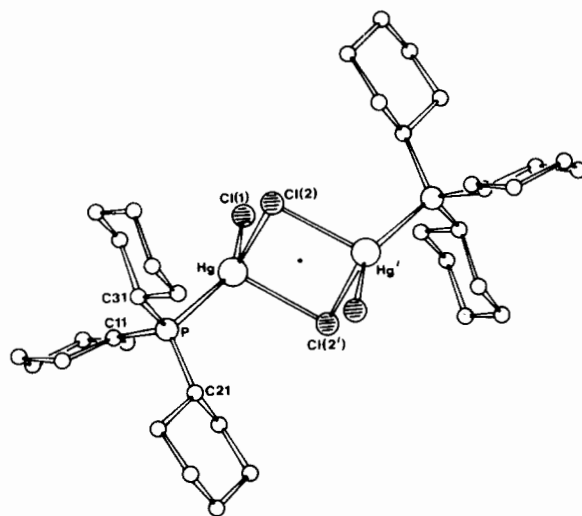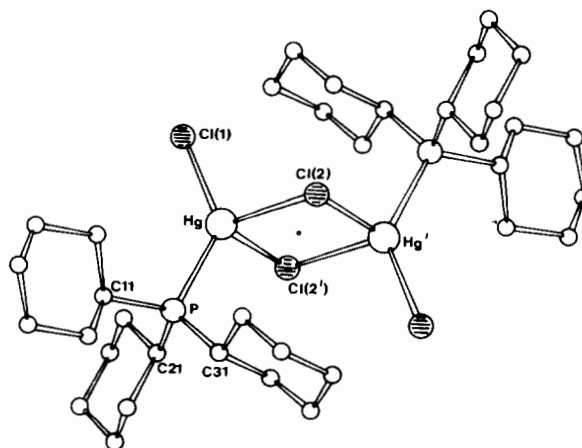|	Dimer 1	Dimer 2
C22–C23–C24	112.4(13)	110.9(10)
C23–C24–C25	111.5(12)	111.7(14)
C24–C25–C26	112.2(13)	111.8(10)
C25–C26–C21	108.8(12)	109.7(9)
P–C31–C32	110.2(7)	110.9(7)
P–C31–C36	112.6(8)	109.6(7)
C32–C31–C36	109.4(9)	110.9(11)
C31–C32–C33	112.0(9)	111.3(9)
C32–C33–C34	113.2(11)	111.6(10)
C33–C34–C35	111.2(11)	108.8(13)
C34–C35–C36	111.7(10)	113.1(10)
C35–C36–C31	110.5(11)	109.3(9)

Fig. 1. $HgCl_2(PCy_3)$. General view of dimer 1. Hydrogen atoms have been omitted for clarity.

Fig. 2. $HgCl_2(PCy_3)$. General view of dimer 2. Hydrogen atoms have been omitted for clarity.

TABLE III. Inter- and Intra-Dimer Contacts Less than 3.000 Å (H···Cl) and 2.200 Å (H···H).[a]

Intra-dimer contacts (Å)			
Dimer 1		*Dimer 2*	
Cl(2)–H361	2.834	Cl(2)–H321	2.801
Cl(2')–H222	2.782		
H111–H322	2.078	H111–H221	2.071
H121–H221	2.293		
H161–H261	2.163		

Inter-dimer contacts	
Dimer 1	
Inter-dimer distance (Å)	Symmetry of second dimer
Cl(1)–H242 2.855	dimer 2; $x, -1+y, -1+z$
Cl(2)–H152 2.845	dimer 2; $1-x, -y, -z$
H142–H341 2.220	dimer 1; $-1+x, y, z$

Dimer 2	
Inter-dimer distance (Å)	Symmetry of second dimer
Cl(1)–H131 2.898	dimer 2; $-x, -y, 1-z$
Cl(1)–H252 2.909	dimer 1; x, y, z
Cl(2)–H262 2.951	dimer 1; x, y, z
H132–H152 2.093	dimer 1; x, y, z

[a]Intra-dimer H···H contacts do not include close contacts between hydrogen atoms in the same cyclohexyl ring.

as triphenylphosphine is generally regarded as being a much weaker donor towards mercury than is tricyclohexylphosphine. Tricyclohexylphosphine is a more sterically demanding and less flexible ligand than are either triphenylphosphine or tributylphosphine, and it may be that in the present complex the inter-dimer H···Cl$_{terminal}$ interactions are dominated by the inflexibility of the phosphine. Thus any close contacts would be principally relieved by movement of the terminal chlorine atom giving rise to high sensitivity of the P–Hg–Cl$_{terminal}$ angle to inter-dimer H···Cl$_{terminal}$ interactions. The differences in such contacts for the two dimers (Table III), could well be responsible both for the unexpectedly small P–Hg–Cl(1) angle found in dimer 2 and also for the magnitude of the difference in the P–Hg–Cl(1) angle (7.6°) observed in the two dimers.

Vibrational Spectra

The far-infrared spectrum observed in the present work (Fig. 5 and Table IV) is in general agreement with those reported previously [2, 3], although cooling the sample to *ca.* 50 K has given greater clarity and detail to the far-infrared spectrum, while causing the usual small shifts in band positions to higher wavenumbers.
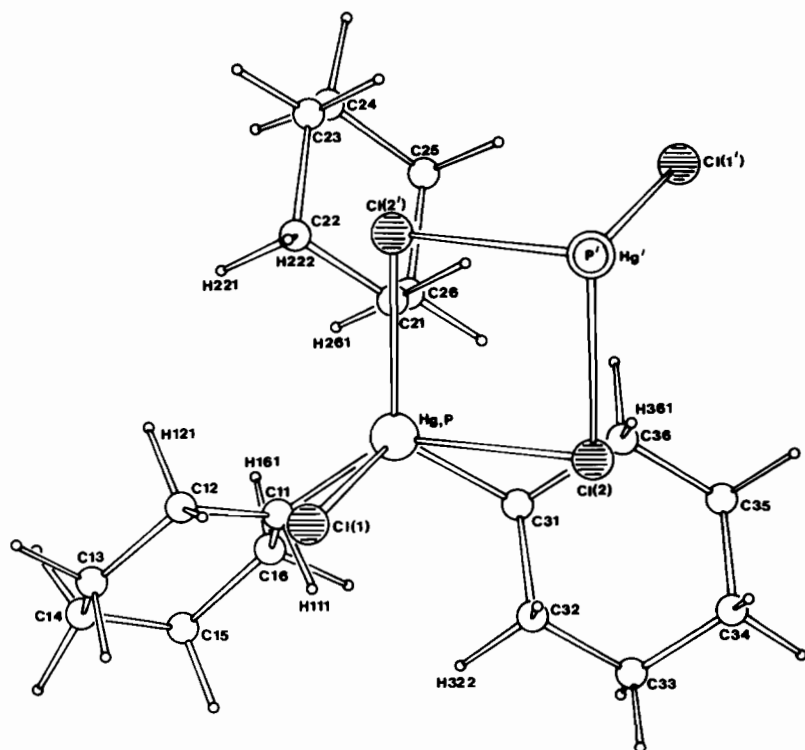

Fig. 3. Projection of part of dimer 1 down the Hg–P bond.
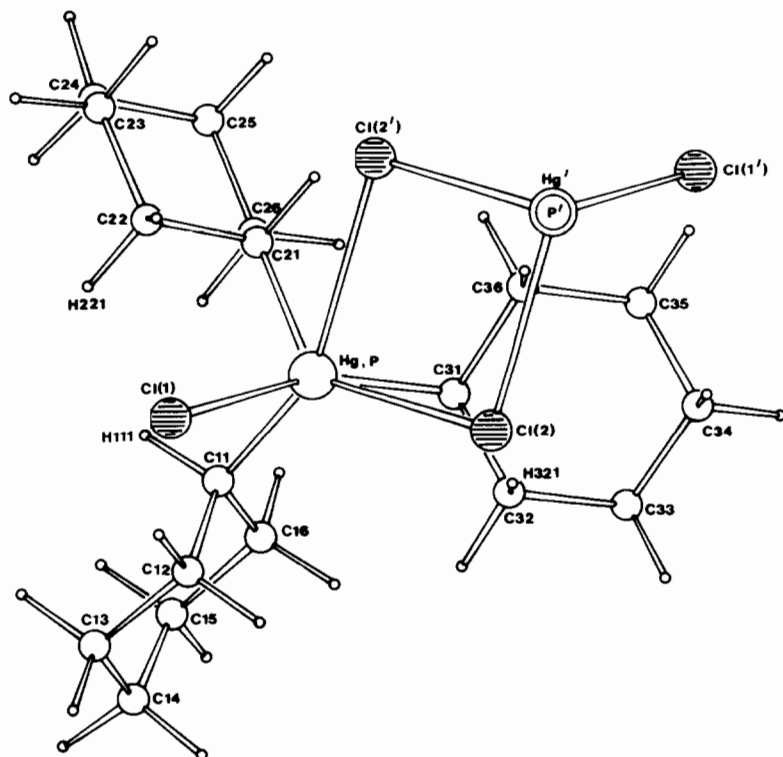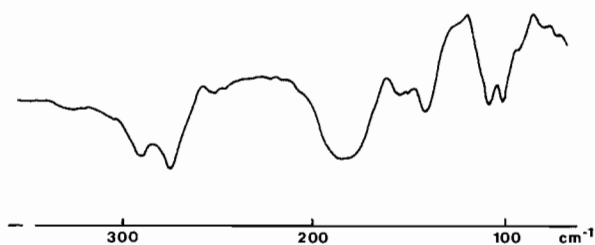
Fig. 4. Projection of part of dimer 2 down the Hg–P bond.

Fig. 5. Far-infrared spectrum of $HgCl_2(PCy_3)$ at ca. 50 K.

Assignments of the bands, made by comparison with spectra of the bromo-, iodo- and cyano-analogues [2, 3, 9], are also, in general terms, in agreement with earlier results [2, 3], but the interpretation is more subtle. For an isolated dimer of C_{2h} point group, the following modes are predicted (PCy_3 ligands taken as point masses):

$$\Gamma_{vib} = 6A_g(Ra) + 3B_g(Ra) + 3A_u(i.r.) + 6B_u(i.r.)$$

$$\Gamma\nu(HgCl)_t = A_g(Ra) + B_u(i.r.)$$

$$\Gamma\nu(HgCl)_b = A_g(Ra) + B_g(Ra) + A_u(i.r.) + B_u(i.r.)$$

The observation of two $\nu(HgCl)_t$ bands in each of the i.r. and Raman spectra is thus at first sight inconsistent with the structure deduced from the X-ray

TABLE IV. Vibrational Assignments (cm^{-1}) for $HgCl_2$-(PCy_3).[a]

I.r. (50 K)	Raman [3]		Assignment	
293m		B_u	$\nu_a(HgCl)_t$	dimer 1[b]
	280sh	A_g	$\nu_s(HgCl)_t$	dimer 1[b]
276s		B_u	$\nu_a(HgCl)_t$	dimer 2[b]
	270s	A_g	$\nu_s(HgCl)_t$	dimer 2[b]
	184m[c]	A_g, B_g	$\nu(HgCl)_b$	
183sbr		A_u, B_u	$\nu(HgCl)_b$	
	145vs	A_g	$\nu_s(HgP)$	
139m		B_u	$\nu_a(HgP)$	

[a]Internal modes of the PCy_3 ligand have been eliminated by reference to spectra of other PCy_3 complexes [2, 3, 9]. [b]Dimer 1 is the one with the shorter Hg–Cl terminal bonds (Fig. 1), while dimer 2 is the other independent dimer in the unit cell (Fig. 2). [c]Assumed, in the present work, to be coincident with an internal mode of PCy_3.

study, but it is equally clear that the multiplicity of bands cannot be due to a non-centrosymmetric arrangement. The only plausible explanation is that a set of one i.r. and one Raman $\nu(HgCl)_t$ band arises from each of the two independent centrosymmetric dimers. Evidently, the higher wavenumber set, at

293 (i.r.) and 280 cm⁻¹ (Ra), are, respectively, the antisymmetric (B_u) and symmetric (A_g) $\nu(HgCl)_t$ modes of the dimer having the shorter Hg-Cl terminal bonds (2.391 Å, dimer 1, Fig. 1), while the other set of bands, 276 (i.r., B_u) and 270 cm⁻¹ (Ra, A_g), are $\nu(HgCl)_t$ of the other dimer (Hg—Cl terminal bonds of 2.413 Å, dimer 2, Fig. 2). It is in many respects remarkable that bands due to the separate dimers can be observed in the spectra, especially since the difference in bond lengths involved is only 0.022 Å, but there is no other possible explanation compatible with the facts.

The pattern of $\nu(HgCl)_b$ modes observed does not illustrate this same phenomenon, and indeed rather fewer bridging modes are observed than is expected for a single dimer. The failure to identify $\nu(HgCl)_b$ bands characteristic of the individual dimers is somewhat surprising, particularly because the bridging arrangements in the two dimers are distinctly different. However, even at low temperatures the $\nu(HgCl)_b$ bands are observed to be very broad, particularly in the i.r. spectrum, and we are forced to conclude that the expected multiplicity is undetectable because of this.

We do not regard the absence of multiplicity in the $\nu(HgP)$ modes as contradicting the above interpretations, since the Hg—P distances in the two dimers, 2.416(3) and 2.412(3) Å, are not significantly different.

Spectra of the bromo- and iodo-analogues do not show multiplicity of $\nu(HgX)_t$ bands, and we are thus able to concur with previous workers [3] that centrosymmetric structures obtain.

Acknowledgements

References

1 N. A. Bell, M. Goldstein, T. Jones, L. A. March and I. W. Nowell, *Inorg. Chim. Acta, 61*, 83 (1982).
2 F. G. Moers and J. P. Langhout, *Rec. Trav. Chim., 92*, 996 (1973).
3 T. Allman, R. G. Goel and P. Pilon, *Spectrochim. Acta, 35A*, 923 (1979).
4 N. A. Bell, M. Goldstein, T. Jones and I. W. Nowell, *Inorg. Chim. Acta, 43*, 87 (1980).
5 N. A. Bell, M. Goldstein, T. Jones and I. W. Nowell, *Inorg. Chim. Acta, 48*, 185 (1981).
6 'International Tables for X-Ray Crystallography', Volume IV, Kynoch Press, Birmingham (1974).
7 G. M. Sheldrick, 'SHELX. Programs for Crystal Structure Determination', University of Cambridge, U.K.
8 M. J. Gallagher, D. P. Graddon and A. R. Sheikh, *Austral. J. Chem., 29*, 759 (1976).
9 R. G. Goel, W. P. Henry and W. O. Ogini, *Canad. J. Chem., 57*, 762 (1979).