

The Crystal Structures of Disordered Crystals of Adducts of Diphenylmercury with Bidentate Ligands

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(Received 29 December 1971)

The crystal structures of two adducts of diphenylmercury with neutral bidentate nitrogen donor atom ligands have been determined. The crystals are disordered in a manner giving rise to diffuse reflexions for which k is odd. The crystal of diphenylbis-(2,4,7,9-tetramethyl-1,10-phenanthroline)mercury(II) studied has $a = 21.09$, $b = 14.54$, $c = 14.65$ Å, $\beta = 120^\circ 34'$, $Z = 4$, and space group Cm . Crystals of diphenylbis-(2,9-dimethyl-1,10-phenanthroline)mercury(II) have $a = 15.42$, $b = 14.28$, $c = 14.55$ Å, $\alpha \approx \beta \approx \gamma \approx 90^\circ$, $Z = 4$, and space group $P1$ with approximate Pm symmetry. Both adducts have their planar ligands in (040) with the C-Hg-C units of Ph_2Hg moieties aligned in the [010] direction. Each Ph_2Hg moiety has one ligand adjacent to it with Hg-N distances of 2.8–3.0 Å, and one-half of the ligands in the crystals do not have a mercury atom adjacent to their nitrogen atoms. Within the unit cell of each adduct every second (040) plane has 0.5 occupancy for two Ph_2Hg moieties with mercury atoms in those planes, and every other (040) plane has 0.8 and 0.2 occupancy for two Ph_2Hg moieties in those planes, giving one Ph_2Hg for every two ligands.

Introduction

The adducts Ph_2HgL_2 [$L = 1,10$ -phenanthroline, 2,9-dimethyl-1,10-phenanthroline (dmp), and 2,4,7,9-tetramethyl-1,10-phenanthroline (tmp)] have stoichiometry suggesting six-coordination for mercury (Deacon & Canty, 1969), whereas coordination numbers lower than six are found for isolable complexes of diarylmercury compounds forming more stable complexes in solution with the same ligands (see e.g. Canty & Deacon, 1971). Deacon & Canty (1969) commented that no definite evidence for coordination of ligands could be found and that the adducts may well be 'lattice compounds'. Uncertainty of the nature of the adducts has prompted this study.

Preliminary experimental results

An infrared spectrum and melting point confirmed that crystals of $\text{Ph}_2\text{Hg(tmp)}_2$, $(\text{C}_6\text{H}_5)_2\text{Hg}(\text{C}_{16}\text{H}_{16}\text{N}_2)_2$, grown from benzene/hexane were identical with the pure compound. Crystals of $\text{Ph}_2\text{Hg(dmp)}_2$, $(\text{C}_6\text{H}_5)_2\text{Hg}(\text{C}_{14}\text{H}_{12}\text{N}_2)_2$, were obtained from an analytically pure sample of the adduct dissolved in benzene, and had melting point and powder photographs identical with those of the sample. Crystals of both adducts have similar crystal habit, being colourless and elongated in the [001] direction, and all crystals chosen for X-ray studies extinguished polarized light when viewed normal to the two pairs of crystal faces parallel to the needle axis.

(a) Diphenylbis-(2,9-dimethyl-1,10-phenanthroline)-mercury(II)

Crystals of $\text{Ph}_2\text{Hg(dmp)}_2$ mounted about the needle axis gave oscillation photographs with streaking normal to layer lines but not passing through all reflexions. Equi-inclination Weissenberg photographs for reciprocal lattice levels $hk0$ – $h,k,12$ and $0kl$ – $2kl$ have diffuse lines through reflexions along festoons with odd k and extending in the \mathbf{a}^* and \mathbf{c}^* directions respectively (Fig. 1). Each reflexion with odd k is present as a maximum in a diffuse line whereas all even k reflexions are sharp and generally of higher intensity.

Weissenberg photographs indicate triclinic symmetry, although cell angles of ca. 90° and similar intensities of reflexions in each octant of reciprocal space suggest that the structure has symmetry close to orthorhombic. The crystals have unit-cell dimensions $a = 15.42$, $b = 14.28$, $c = 14.55$, $\alpha \approx \beta \approx \gamma \approx 90^\circ$, $U = 3203.9$ Å³, and $D_{\text{meas}} = 1.60$ g.cm⁻³ (by flotation), $D_{\text{calc}} = 1.59$ g.cm⁻³ for $Z = 4$. The cell angles are only $8'$, $8'$, and $5'$ ($\pm 4'$) removed from 90° for α , β , and γ respectively. Since the differences in intensity of equivalent reflexions for monoclinic symmetry are slight, it was not possible to decide whether $90^\circ < \alpha, \beta$, or $\gamma > 90^\circ$ from Weissenberg photographs obtained with the crystal oscillated about different axes. The conditions for reflexions to be observed:

$$hk0: h + k = 2n$$

$$0k0: k = 4n$$

$$00l: l = 2n$$

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are not all required together for space groups of orthorhombic or lower symmetry.

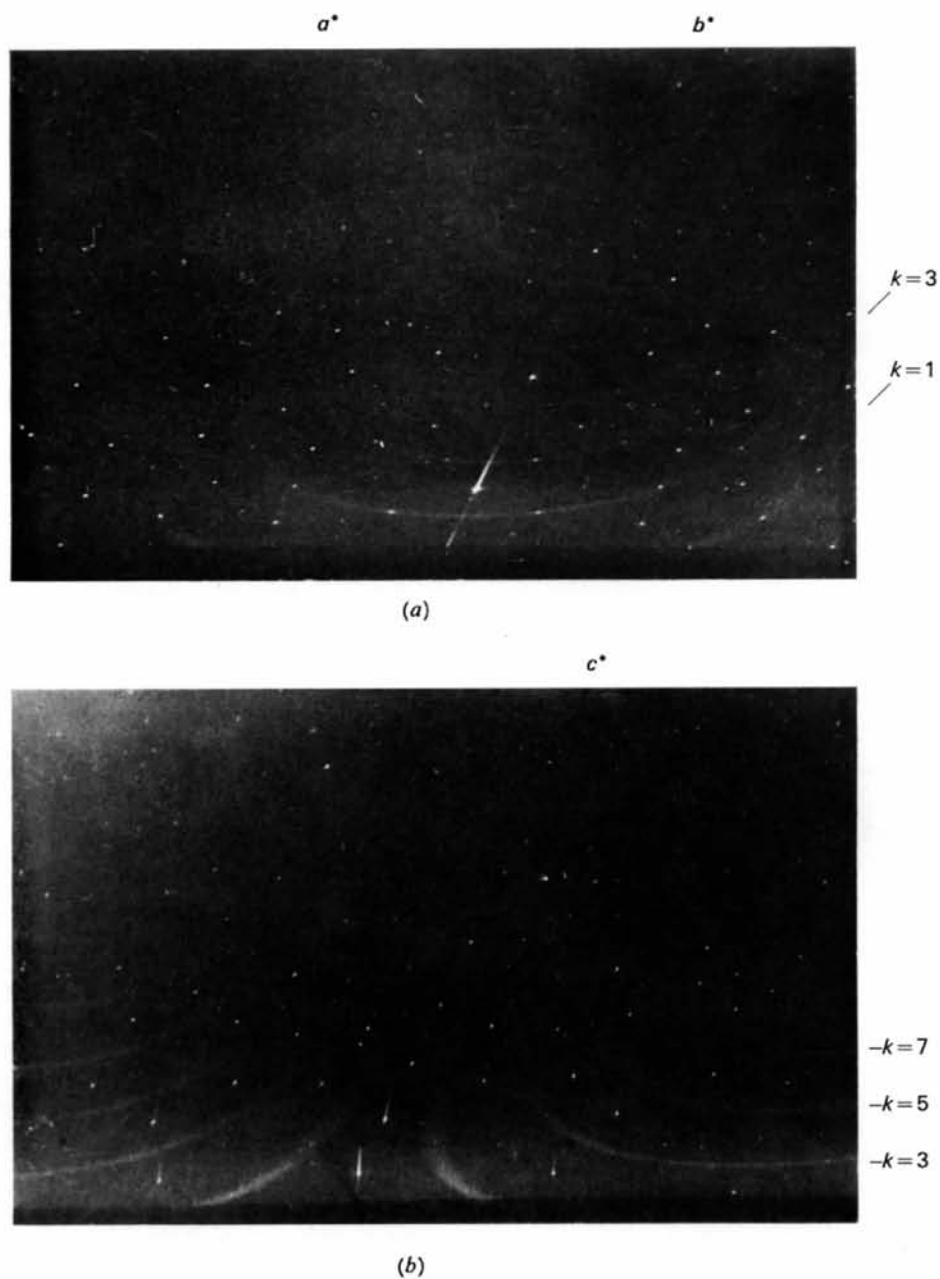


Fig. 1. Weissenberg photographs from a crystal of $\text{Ph}_2\text{Hg}(\text{dmp})_2$. (a) A region of the reciprocal lattice layer $hk0$. (b) A region of the reciprocal lattice layer $2kl$.

Table 1. Comparison of F_o and F_c for $\text{Ph}_2\text{Hg}(\text{tmp})_2$

Sharp reflexions are presented first. Unobserved reflexions are indicated with an asterisk.

H K FO FC				H K FO FC				H K FO FC				H K FO FC				H K FO FC				H K FO FC									
0	0	149	143	0	4	79	17*	0	10	84	181	0	8	95	131	0	2	93	99*	0	1	5	39	79*	0	1	5	42	62*
0	0	62	72	0	8	141	195	0	8	248	332	0	0	380	371	0	1	7	37	92	71*	0	1	7	39	87*			
0	0	281	273	0	10	215	219	0	8	205	224	0	2	97	83	0	10	2	131	114	0	1	9	39	90*				
0	0	100	139	0	4	155	149	0	4	224	171	0	4	267	249	0	12	0	132	123	0	3	3	88	82				
0	0	110	99	0	2	372	391	0	6	159	176	0	4	0	122	118	0	3	3	88	82								
0	0	38	133*	0	2	40	36*	0	4	141	175	0	4	8	128	127	0	14	2	96	71*								
0	0	159	183	0	2	118	138	0	10	0	67	30*	0	0	0	67	29*	0	10	2	740	735							
0	0	217	199*	0	2	35	36*	0	10	2	99	99*	0	4	6	100	18*	0	3	5	41	81*							
0	0	92	44	0	2	241	197	0	2	99	99*	0	2	8	102	105	0	2	8	102	105								
0	0	35	62*	0	2	10	39	34*	0	4	69	18*	0	4	6	100	81*	0	3	5	37	87*							
0	0	34	44*	0	4	81	93	0	12	0	76	27*	0	8	110	157	0	7	4	98	63*								
0	0	225	185	0	4	193	252	0	14	0	172	154	0	8	110	157	0	7	4	98	63*								
0	0	45	32*	0	4	102	143	0	14	2	190	105	0	8	110	157	0	7	4	98	63*								
0	0	141	186	0	5	0	267	234	0	10	4	140	136	0	8	110	157	0	7	4	98	63*							
0	0	70	29	0	5	110	103	0	10	4	140	136	0	8	110	157	0	7	4	98	63*								
0	0	259	185	0	5	110	103	0	10	4	140	136	0	8	110	157	0	7	4	98	63*								
0	0	99	266	0	5	110	103	0	10	4	140	136	0	8	110	157	0	7	4	98	63*								
0	0	276	256	0	5	110	103	0	10	4	140	136	0	8	110	157	0	7	4	98	63*								
0	0	101	104	0	5	110	103	0	10	4	140	136	0	8	110	157	0	7	4	98	63*								
0	0	141	179	0	5	110	103	0	10	4	140	136	0	8	110	157	0	7	4	98	63*								
0	0	103	108	0	5	110	103	0	10	4	140	136	0	8	110	157	0	7	4	98	63*								
0	0	81	104*	0	5	110	103	0	10	4	140	136	0	8	110	157	0	7	4	98	63*								
0	0	80	39	0	5	110	103	0	10	4	140	136	0	8	110	157	0	7	4	98	63*								
0	0	90	89	0	5	110	103	0	10	4	140	136	0	8	110	157	0	7	4	98	63*								
0	0	109	187	0	5	110	103	0	10	4	140	136	0	8	110	157	0	7	4	98	63*								
0	0	134	184	0	5	110	103	0	10	4	140	136	0	8	110	157	0	7	4	98	63*								
0	0	51	24	0	0	511	502	0	0	282	244	0	0	86	83*	0	0	86	83*										
0	0	403	388	0	0	246	232	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	136	84	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	282	407	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	10	114	236	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*									
0	0	414	418	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	34	344	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	228	213	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	41	204	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	118	172	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	45	13*	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	100	99	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	331	279	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	4	122	42	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*									
0	0	100	214	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	70	86	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	144	79	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	349	308	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	80	874	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	183	216	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	67	93*	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	10	97	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	203	198	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	2	88	53	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*									
0	0	110	177	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	186	126	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	85	120*	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	165	189	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	132	93	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	132	87	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	141	89	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	27	12*	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	326	285	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	132	2	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	273	338	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	31	43*	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	113	159	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	409	399	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	40	40*	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	203	217	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	43	33*	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	123	188	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	102	49	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	377	325	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	79	6*	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	205	236	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	57	37*	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	124	162	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	282	249	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	90	77	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	192	178	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86	83*										
0	0	97	113	0	0	153	124	0	0	125	103	0	0	86	83*	0	0	86											

A powder photograph can be indexed with the above unit-cell dimensions, and upon estimation of intensities of reflexions it was revealed that the most intense reflexions are accounted for by the powder lines.

(b) *Diphenylbis-(2,4,7,9-tetramethyl-1,10-phenanthroline)mercury(II)*

Oscillation photographs obtained from crystals of $\text{Ph}_2\text{Hg}(\text{tmp})_2$ have diffuse lines in the same orientation as in photographs of $\text{Ph}_2\text{Hg}(\text{dmp})_2$. The $hk0$ reciprocal lattice level is similar to that of $\text{Ph}_2\text{Hg}(\text{dmp})_2$, having identical requirements for reflexions to be observed $-hk0:h+k=2n$ and $0k0:k=4n$, but $hk1$ and $h0l$ Weissenberg photographs revealed the crystals to be twinned, with components of monoclinic symmetry having diffuse lines for odd k extending in the \mathbf{a}^* and \mathbf{c}^* directions of reciprocal space. Four crystals were examined and found to be twinned in this manner.

A very small crystal of habit different from that of the other crystals was found to have cell dimensions and intensities of reflexions similar ($\pm 0.1 \text{ \AA}$, $\pm 20'$) to those of the twin components. The crystal was mounted about the b axis and since it was a very small crystal it proved too difficult to mount about another direct axis, so the b -axis length was determined from an oscillation photograph. The crystal has $a=21.09$, $b=14.54$, $c=14.65 \text{ \AA}$, $\beta=126^\circ 34'$, $U=3607.8 \text{ \AA}^3$, D_{meas} (twinned crystals, by flotation) $=1.52 \text{ g.cm}^{-3}$, $D_{\text{calc}}=1.52 \text{ g.cm}^{-3}$ for $Z=4$, requirements for reflexions to be observed $hkl:h+k=2n$, and gave an oscillation photograph symmetrical about the $h0l$ layer line, indicating one of the C -centred monoclinic space groups $C2$, Cm , or $C2/m$.

All except one line (of medium intensity) of a powder photograph can be indexed with the unit cell of the single crystal. The line not indexed does not result from either free ligand or diphenylmercury.

(c) *The diffuse lines*

The presence of diffuse lines usually indicates micro-synthetic twinning, often resulting from mistakes in crystal growth of layer structures. When twinning in this way leads to ordered regions displaced by $\frac{1}{2}b$ from each other, there is a lowering in intensity of odd k reflexions, and if the regions are small enough, diffuse streaks are observed through odd k reflexions (Dornberger-Schiff & Dunitz, 1965). Diffuse lines may be caused by the mode of disorder found in the urea-hydrocarbon (Smith, 1952) and urea-di-n-alkylmercury inclusion compounds (Bähr & Meier, 1958). In crystals of these compounds, the urea molecules form an ordered structure with channels of appropriate diameter for the inclusion of n-alkanes or di-n-alkylmercurials. One-dimensional disorder giving rise to diffuse lines occurs when the n-alkanes are randomly distributed along the channels. If discrete spots occur in the diffuse lines, then the hydrocarbon chains are partially ordered in their positions in the channels.

Diffuse lines may also arise from thermal diffuse scattering, particularly for structures involving large planar molecules. Thermal diffuse scattering resulting from vibrations of molecules is expected to be temperature dependent (see *e.g.* Wooster, 1962). With a crystal of $\text{Ph}_2\text{Hg}(\text{dmp})_2$ mounted about the needle axis an oscillation photograph obtained at a temperature below -115°C had diffuse lines of the same intensity relative to sharp reflexions, as diffuse lines in a photograph obtained at *ca.* 20°C . In addition, the Patterson syntheses for each adduct indicate that the planar aromatic ligands are in ac planes and the diffuse lines are observed to lie parallel to these planes in reciprocal space ($k=1,3,5,7,\dots$) instead of normal to them as expected for thermal diffuse scattering.

Since the low temperature study of $\text{Ph}_2\text{Hg}(\text{dmp})_2$ shows that the diffuse lines probably result from dis-

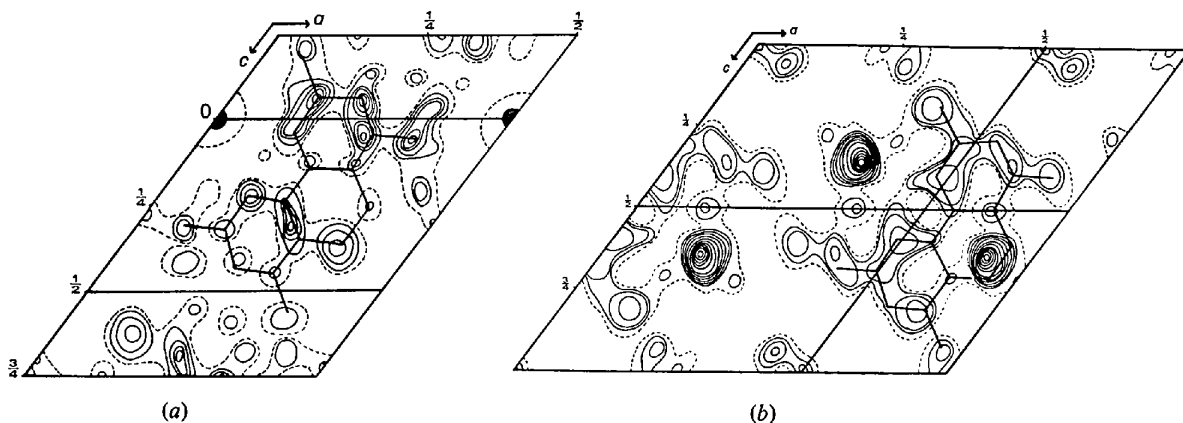


Fig. 2. Patterson syntheses for $\text{Ph}_2\text{Hg}(\text{tmp})_2$. The contours are evenly spaced on an arbitrary scale. Negative regions are bounded by broken lines. (a) The plane $y=0$. A skeleton of the ligand is placed to coincide with vectors near the origin. The asymmetric section is bounded by $x=0 \rightarrow \frac{1}{2}$, $z=0 \rightarrow \frac{1}{2}$. (b) The plane $y=\frac{1}{2}$. Mercury-ligand atom vectors are shown for mercury and ligand atoms separated by $x, \frac{1}{2}, z$, together with the large mercury-mercury vector at $0.33, \frac{1}{2}, 0.36$.

order, structure solution for both adducts was initially sought with all atoms fully occupied until packing requirements indicated the presence of partial occupancy. The observed data represents the sum of diffraction from all ordered regions of the crystal, and the structure solution obtained by this procedure will be referred to as the superposition structure. The possible structures of ordered regions have been estimated from the superposition structure.

Structure solutions

(1) Solution of the superposition structure for $\text{Ph}_2\text{Hg}(\text{tmp})_2$

From the very small monoclinic crystal of dimensions $0.05 \times 0.05 \times 0.025$ mm (absorption coefficient $\mu(\text{Cu } K\alpha) = 8.47 \text{ mm}^{-1}$, $t_{\text{opt}} = 0.236$ mm) mentioned earlier, 414 independent non-zero reflexions were recorded photographically for reciprocal lattice levels $h0l-h, 10, l$ with an Enraf-Nonius equi-inclination Weissenberg camera. Reflexions of odd k index are so weak that only 36 reflexions of this type were observed, and no odd k reflexions were observed for $k > 3$. Reflexion intensities were measured visually by comparison with a standard strip of twenty spots.

After Lorentz and polarization corrections had been applied, a Patterson synthesis was calculated. Vector peaks were found to be predominantly in the (040)

planes with very few vectors between these planes. The (010) plane has mercury–ligand vectors grouped near the origin [Fig. 2(a)]. The positions of these vectors reveal that the ligands are oriented with N(1)–N(10) parallel to the [001] direction and lie in ac planes (the atom numbering scheme for 2,4,7,9-tetramethyl-1,10-phenanthroline is given in Fig. 3). The only other vector in (010) is a peak at $\frac{1}{2}, 0, 0$ almost as large as the origin, suggesting that it is not only a mercury–mercury vector or vectors, but also a sum of smaller vectors between light atoms separated by $\frac{1}{2}a$. The $y = \frac{1}{4}$ plane has one large peak at $0.33, \frac{1}{4}, 0.36$ [Fig. 2(b)]. Both $C2$ and $C2/m$ space groups require a Harker plane ($2x, 0, 2z$) in the Patterson synthesis. Since the peak $0.33, \frac{1}{4}, 0.36$ is almost certainly a mercury–mercury vector, a large vector in (010) is expected in addition to the peak at $\frac{1}{2}, 0, 0$ for $C2$ or $C2/m$ space groups.

The space group was assumed to be Cm and a mercury atom was placed at $\frac{1}{4}, \frac{1}{4}, 0$ satisfying the Patterson peak at $\frac{1}{2}, 0, 0$ and consistent with space groups Cm , $C2$, and $C2/m$. The residual index $R = \sum ||F_o| - |F_c|| / \sum |F_o|$ was 0.33 and a difference Fourier synthesis had four large peaks at $0.08, 0, 0.36$; $\frac{1}{2} + 0.08, 0, 0.36$; $\frac{1}{2} - 0.08, 0, 1 - 0.36$ and $1 - 0.08, 0, 1 - 0.36$. Vectors from $\frac{1}{4}, \frac{1}{4}, 0$ to each of these positions satisfy the vector at $0.33, \frac{1}{4}, 0.36$ but vectors between all of these positions are not present. The coordinates $0.08, 0, 0.36$ were chosen for a mercury atom position, R was reduced to 0.24, and a subsequent difference Fourier synthesis revealed positions of ligand (Fig. 4) and phenyl group atoms. The phenyl groups lie immediately above and below each mercury atom, and the C–Hg–C skeleton is linear and in the [010] direction.

No improvement in phasing of light atom positions occurred on adding ligand atoms singly to the structure factor calculation. Consequently, coordinates of ligand and phenyl group atoms were chosen to coincide with peaks in difference Fourier syntheses as closely as possible, maintaining C–C and C–N bond lengths within the range 1.29–1.52 Å, Hg–C bond lengths of 2.0–2.15 Å, and planar phenyl rings.

The difference syntheses of Fig. 4 indicate eight ligands per unit cell as required from the density of the crystals. Ligands were added singly to the structure factor calculation. When the ligand in (010) with x coordinates $< \frac{1}{2}$ is added peaks corresponding to the ligand with $x > \frac{1}{2}$ disappear from the synthesis as well as those of the ligand added. However, when both of these ligands are added the subsequent difference synthesis is devoid of negative regions, confirming the presence of both ligands.

At this stage six mercury atoms had been placed in the unit cell (fourfold position $\frac{1}{4}, \frac{1}{4}, 0$; twofold $0.08, 0, 0.36$) whereas the measured density of the crystals suggests four. Phenyl group positions indicated in difference Fourier syntheses were confirmed by the Patterson synthesis. Mercury–phenyl-group atom vectors are present immediately below the origin of the synthesis. Vectors between mercury atoms and phenyl

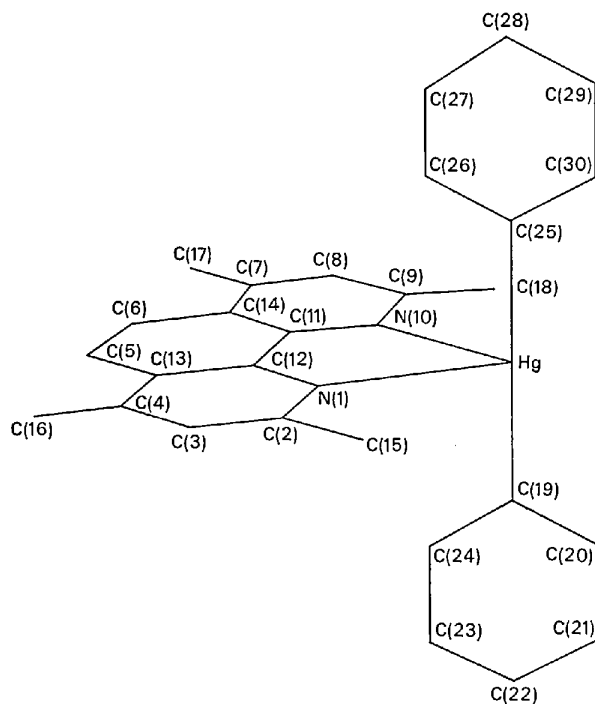


Fig. 3. Atom numbering scheme for $\text{Ph}_2\text{Hg}(\text{tmp})_2$. Adjacent ligand and Ph_2Hg moieties in the plane $y = \frac{1}{4}$ are shown. Adjacent ligand and Ph_2Hg moieties in (020) have an atom numbering scheme related by a twofold axis about the C–Hg–C unit.

group atoms attached to mercury atoms in adjacent (040) planes are also present, being situated immediately above and below the mercury-mercury vector $0.33, \frac{1}{4}, 0.36$. All peaks in the Patterson synthesis are now accounted for, and all calculated Hg-light-atom vectors are present. However, phenyl groups of mercury atoms at $x, \frac{1}{4}, z$ overlap with phenyl groups of mercury atoms at $x, \frac{3}{4}, z$.

The coincidence of phenyl groups is so close that atoms of separate groups cannot be distinguished in difference syntheses. A mirror plane in (020) is required for C_m symmetry, but whenever a Ph_2Hg moiety is placed at $x, \frac{1}{4}, z$ (e.g. $\frac{1}{4}, \frac{1}{4}, 0$) a Ph_2Hg moiety cannot be present at $x, \frac{3}{4}, z$ (e.g. $\frac{1}{4}, \frac{3}{4}, 0$) as required for space group C_m . With the fourfold Ph_2Hg moieties half-occupancy to maintain a mirror plane in the superposition structure in (020) and prevent overlap of phenyl groups, and with the Ph_2Hg moiety in (010) fully occupied, there are four formula units of $\text{Ph}_2\text{Hg}(\text{tmp})_2$ in the unit cell as required.

The residual index R was 0.19 for the sharp reflexions and 0.57 for the diffuse reflexions. A small peak was present in a subsequent difference synthesis at $\frac{1}{2} + 0.08, 0, 0.36$ suggesting the presence of a mercury atom in position for coordination of the ligand in (010) with x coordinates $> \frac{1}{2}$ [Fig. 4(a)]. Placing a Ph_2Hg moiety at this position requires partial occupancy, since overlap of phenyl groups occurs with Ph_2Hg moieties having mercury atoms in the $y = \frac{1}{2}$ plane. A mercury atom was placed at $\frac{1}{2} + 0.08, 0, 0.36$ and the occupancies of Ph_2Hg moieties in the (020) planes varied such that with an occupancy of m for one Ph_2Hg moiety in (010) the occupancy of the other is $1 - m$, since it is translated to a position $\frac{1}{2}b$ removed from the Ph_2Hg moiety of occupancy m ($\frac{1}{2}, \frac{1}{2}, 0$ operation). Several models were tried, best agreement being obtained for $m = 0.8$. The calculated structure factors for diffuse reflexions are more sensitive than the sharp reflexions to occupancy variations since only the Ph_2Hg moieties with mercury atoms in (020) contribute to these re-

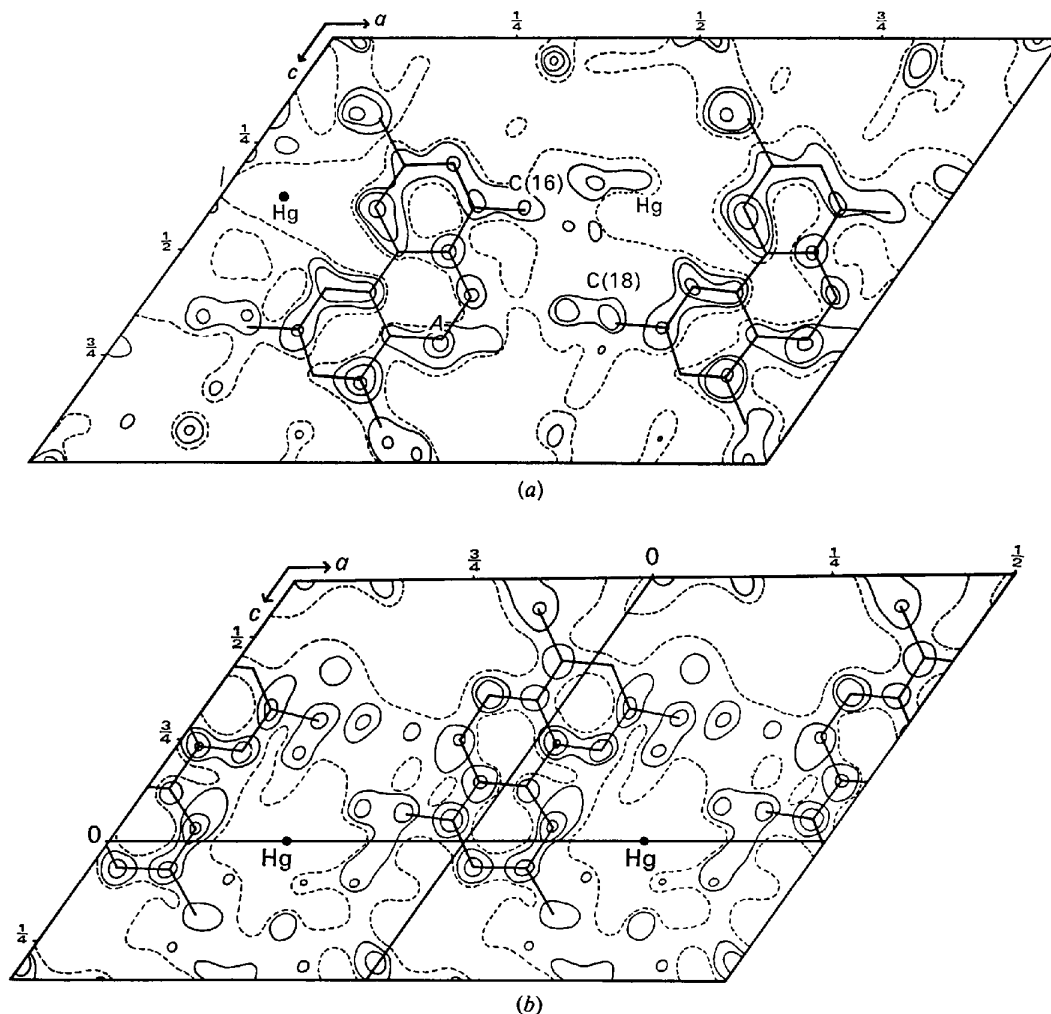


Fig. 4. Difference Fourier syntheses for $\text{Ph}_2\text{Hg}(\text{tmp})_2$ with mercury atoms at $\frac{1}{4}, \frac{1}{4}, 0$ and $0.08, 0, 0.36$ as the phasing atoms. (a) The plane $y = 0$. (b) The plane $y = \frac{1}{4}$.

flexions, the remainder of the cell contents conforming to a subcell of dimensions $a, \frac{1}{2}b, c$.

With occupancies of 0.8 and 0.2 for the Ph_2Hg

moieties in (020) R was 0.17 for both the 378 sharp and 36 diffuse reflexions and 0.25 for all reflexions, including 424 unobserved reflexions. A structure factor

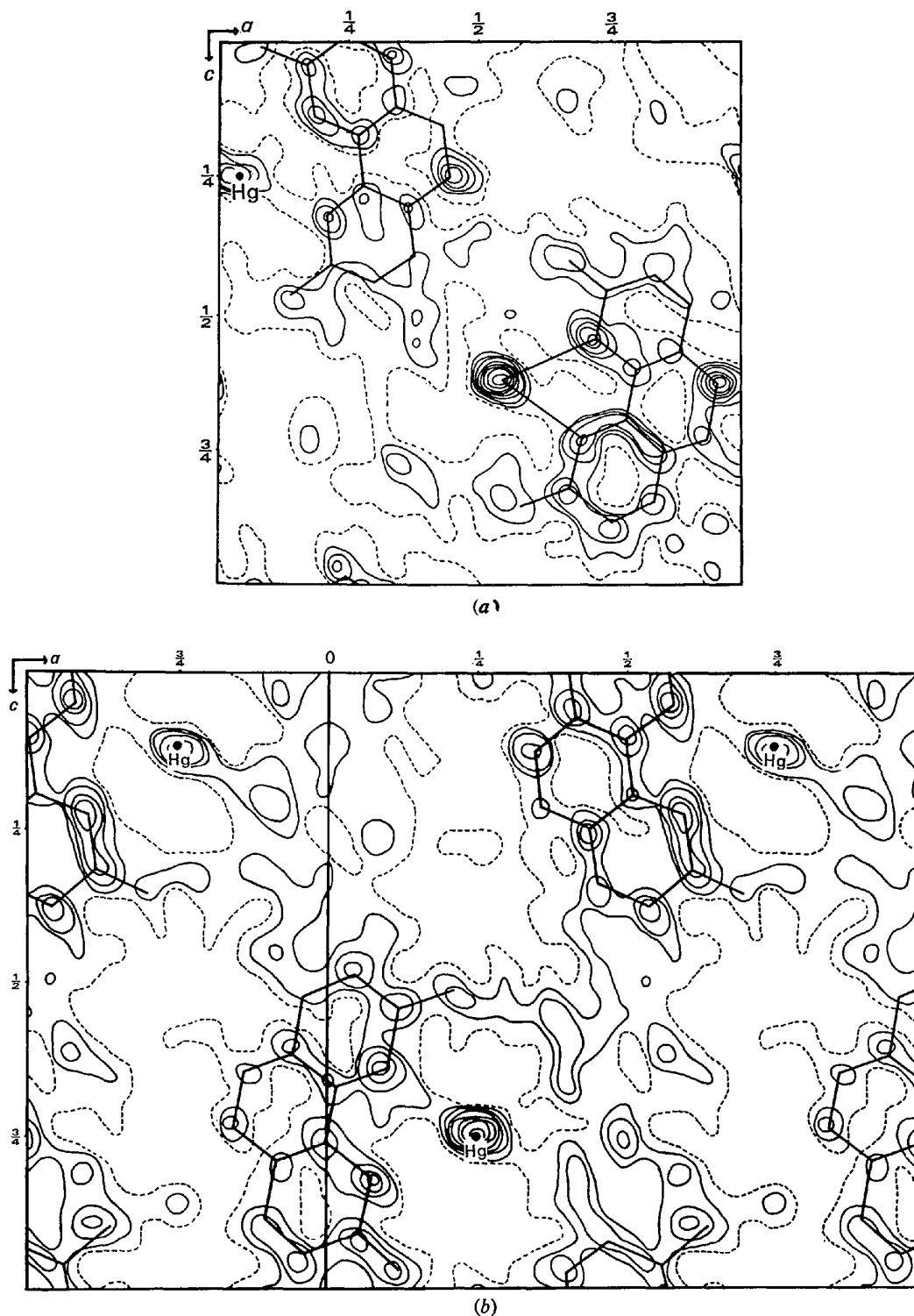


Fig. 5. Difference Fourier syntheses for $\text{Ph}_2\text{Hg}(\text{dmp})_2$ with mercury atoms at $\frac{1}{4}, \frac{1}{2}, \frac{3}{4}; \frac{1}{4}, \frac{3}{4}; \frac{1}{2}, \frac{1}{2}; \frac{1}{4}, \frac{3}{4}, \frac{1}{2}; 0.04, 0, \frac{1}{2}$ and $0.54, \frac{1}{2}, \frac{3}{4}$ as the phasing atoms calculated in space group $P1$. The contours are evenly spaced on an arbitrary scale. Negative regions are bounded by broken lines. (a) The plane $y=0$. (b) The plane $y=\frac{1}{4}$.

list is given in Table 1. A final difference Fourier synthesis calculated using observed reflexions has no peaks $> \frac{1}{2}$ or $< -\frac{1}{2}$ of a carbon atom, and difference syntheses calculated with mercury atoms only given the occupancy values of the final model are virtually identical with those shown in Fig. 4, which were calculated with an incomplete mercury atom contribution to the structure factor expression. Tables 2, 3 and 4 show the atomic coordinates, bond lengths and bond angles in $\text{Ph}_2\text{Hg}(\text{tmp})_2$.

Refinement was not attempted with 81 atoms in the asymmetric unit and only 414 observed reflexions.

Table 2. Coordinates of atoms of $\text{Ph}_2\text{Hg}(\text{tmp})_2$ within the asymmetric unit of space group Cm

	<i>x/a</i>	<i>y/b</i>	<i>z/c</i>	Occu- pancy
Hg(1)	0.2500	0.2500	0	0.5
Hg(2)	0.0839	0	0.3584	0.8
Hg(3)	0.5839	0	0.3584	0.2

Ligand coordinated to Hg(1)

	<i>x/a</i>	<i>y/b</i>	<i>z/c</i>	Occu- pancy
N(1)	0.1062	0.2500	-0.0250	1.0
N(10)	0.1000	0.2500	-0.2240	1.0
C(2)	0.1125	0.2500	0.0692	1.0
C(3)	0.0387	0.2500	0.0615	1.0
C(4)	-0.0362	0.2500	-0.0481	1.0
C(5)	-0.1062	0.2500	-0.2531	1.0
C(6)	-0.1094	0.2500	-0.3565	1.0
C(7)	-0.0425	0.2500	-0.4481	1.0
C(8)	0.0250	0.2500	-0.4327	1.0
C(9)	0.0975	0.2500	-0.3250	1.0
C(11)	0.0312	0.2500	-0.2346	1.0
C(12)	0.0325	0.2500	-0.1346	1.0
C(13)	-0.0375	0.2500	-0.1461	1.0
C(14)	-0.0400	0.2500	-0.3442	1.0
C(15)	0.1937	0.2500	0.1808	1.0
C(16)	-0.1125	0.2500	-0.0673	1.0
C(17)	-0.1244	0.2500	-0.5596	1.0
C(18)	0.1775	0.2500	-0.2961	1.0

Ligand coordinated to Hg(2)

	<i>x/a</i>	<i>y/b</i>	<i>z/c</i>	Occu- pancy
N(1)	0.2325	0	0.3904	1.0
N(10)	0.2375	0	0.5865	1.0
C(2)	0.2200	0	0.2885	1.0
C(3)	0.2850	0	0.2885	1.0
C(4)	0.3550	0	0.3904	1.0
C(5)	0.4412	0	0.6106	1.0
C(6)	0.4387	0	0.7048	1.0
C(7)	0.3812	0	0.8000	1.0
C(8)	0.3112	0	0.7961	1.0
C(9)	0.2437	0	0.6827	1.0
C(11)	0.3025	0	0.5961	1.0
C(12)	0.3000	0	0.4961	1.0
C(13)	0.3662	0	0.4923	1.0
C(14)	0.3750	0	0.7038	1.0
C(15)	0.1425	0	0.1846	1.0
C(16)	0.4312	0	0.4038	1.0
C(17)	0.4525	0	0.9231	1.0
C(18)	0.1700	0	0.6769	1.0

Atoms of ligand coordinated to Hg(3) have coordinates of atoms of ligand coordinated to Hg(2) + $\frac{1}{2}, 0, 0$ and occupancy 1.0.

Phenyl groups bonded to Hg(1)

	<i>x/a</i>	<i>y/b</i>	<i>z/c</i>	Occu- pancy
C(19)	0.2500	0.3946	0.0000	0.5
C(20)	0.3228	0.4482	0.0625	0.5
C(21)	0.3228	0.5445	0.0625	0.5
C(22)	0.2500	0.5870	0.0000	0.5

Table 2 (cont.)

	<i>x/a</i>	<i>y/b</i>	<i>z/b</i>	Occu- pancy
C(23)	0.1815	0.5445	-0.0481	0.5
C(24)	0.1815	0.4482	-0.0481	0.5
C(25)	0.2500	0.1054	0.0000	0.5
C(26)	0.1815	0.0518	-0.0481	0.5
C(27)	0.1815	-0.0445	-0.0481	0.5
C(28)	0.2500	-0.0870	0.0000	0.5
C(29)	0.3228	-0.0445	0.0625	0.5
C(30)	0.3228	0.0518	0.0625	0.5
Phenyl groups bonded to Hg(2)				
C(19)	0.0856	0.1462	0.3650	0.8
C(20)	0.0125	0.1979	0.3125	0.8
C(21)	0.0125	0.2911	0.3125	0.8
C(22)	0.0856	0.3446	0.3650	0.8
C(23)	0.1519	0.2911	0.4154	0.8
C(24)	0.1519	0.1979	0.4154	0.8

Atoms of phenyl groups bonded to Hg(3) have coordinates of atoms of phenyl groups bonded to Hg(1) + $\frac{1}{2}, 0, 0$ and occupancy 0.2.

Table 3. Bond lengths in $\text{Ph}_2\text{Hg}(\text{tmp})_2$

(a) Mercury stereochemistry

	Hg(1)	Hg(2)
Hg-N(1)	2.83 Å	2.88 Å
Hg-N(10)	2.90	2.96
Hg-C(19)	2.10	2.13
Hg-C(25)	2.10	

(b) Ligands

	Ligand coordinated to:	
	Hg(1)	Hg(2)
N(1)—C(2)	1.31 Å	1.35 Å
N(1)—C(12)	1.42	1.34
C(2)—C(3)	1.49	1.37
C(3)—C(4)	1.43	1.34
C(4)—C(13)	1.42	1.37
C(5)—C(6)	1.48	1.41
C(5)—C(13)	1.36	1.50
C(6)—C(14)	1.36	1.34
C(7)—C(8)	1.30	1.44
C(7)—C(14)	1.49	1.34
C(8)—C(9)	1.40	1.40
C(9)—N(10)	1.45	1.34
N(10)—C(11)	1.36	1.29
C(11)—C(12)	1.45	1.43
C(11)—C(14)	1.40	1.40
C(12)—C(13)	1.38	1.43
C(2)—C(15)	1.51	1.42
C(4)—C(16)	1.46	1.50
C(7)—C(17)	1.51	1.51
C(9)—C(18)	1.48	1.51

(c) Phenyl groups

	For Hg(1)	For Hg(2)
C(19)—C(20)	1.46	1.46
C(19)—C(24)	1.41	1.36
C(20)—C(21)	1.40	1.36
C(21)—C(22)	1.38	1.47
C(22)—C(23)	1.32	1.37
C(23)—C(24)	1.40	1.36
C(25)—C(26)	1.41	
C(25)—C(30)	1.46	
C(26)—C(27)	1.40	
C(27)—C(28)	1.32	
C(28)—C(29)	1.38	
C(29)—C(30)	1.40	

Table 4. Bond angles in $\text{Ph}_2\text{Hg}(\text{tmp})_2$

(a) Mercury stereochemistry		
	Hg(1)	Hg(2)
N(1)—Hg—N(10)	59°	57°
C(19)—Hg—C(25)	180	
N(1), N(10)—Hg—C(19), C(25)	90	90

(b) Ligands		
	Ligand coordinated to:	
	Hg(1)	Hg(2)
Hg—N(1)—C(2)	115°	110°
Hg—N(1)—C(12)	121	120
C(2)—N(1)—C(12)	123	130
N(1)—C(2)—C(3)	119	118
N(1)—C(2)—C(15)	119	122
C(3)—C(2)—C(15)	123	121
C(2)—C(3)—C(4)	119	116
C(3)—C(4)—C(13)	118	125
C(3)—C(4)—C(16)	125	122
C(13)—C(4)—C(16)	117	112
C(6)—C(5)—C(13)	123	120
C(5)—C(6)—C(14)	118	128
C(8)—C(7)—C(14)	117	120
C(8)—C(7)—C(17)	128	108
C(14)—C(7)—C(17)	115	131
C(7)—C(8)—C(9)	123	110
N(10)—C(9)—C(8)	120	130
N(10)—C(9)—C(18)	112	120
C(8)—C(9)—C(18)	128	111
Hg—N(10)—C(9)	120	123
Hg—N(10)—C(11)	120	120
C(9)—C(10)—C(11)	120	117
N(10)—C(11)—C(12)	120	120
N(10)—C(11)—C(14)	118	120
C(12)—C(11)—C(14)	121	120
N(1)—C(12)—C(11)	119	123
N(1)—C(12)—C(13)	120	110
C(11)—C(12)—C(13)	120	127
C(4)—C(13)—C(5)	122	130
C(4)—C(13)—C(12)	120	120
C(5)—C(13)—C(12)	118	110
C(6)—C(14)—C(7)	119	122
C(6)—C(14)—C(11)	119	115
C(7)—C(14)—C(11)	121	123

(c) Phenyl groups		
	Hg(1)	Hg(2)
Hg—C(19)—C(20)	122°	120°
Hg—C(19)—C(24)	124	124
C(20)—C(19)—C(24)	114	115
C(19)—C(20)—C(21)	122	121
C(20)—C(21)—C(22)	117	122
C(21)—C(22)—C(23)	125	113
C(22)—C(23)—C(24)	118	125
C(19)—C(24)—C(23)	124	124
Hg—C(25)—C(26)	124	
Hg—C(25)—C(30)	122	
C(26)—C(25)—C(30)	114	
C(25)—C(26)—C(27)	124	
C(26)—C(27)—C(28)	118	
C(27)—C(28)—C(29)	114	
C(28)—C(29)—C(30)	122	
C(25)—C(30)—C(29)	117	

(2) Solution of the superposition structure for $\text{Ph}_2\text{Hg}(\text{dmp})_2$

A crystal of dimensions $0.05 \times 0.08 \times 0.2$ mm, less than optimum size (absorption coefficient $\mu(\text{Cu } K\alpha) = 9.53 \text{ mm}^{-1}$, $t_{\text{opt}} = 0.21$ mm) was chosen and mounted about the needle axis; a total of 1868 independent

non-zero reflexions (including 537 diffuse reflexions) were collected for reciprocal lattice levels $hk0-hk12$. Since only very small variations from monoclinic symmetry could be seen in intensities of reflexions, structure solution was sought with a monoclinic data set in space group $P1$ until the appropriate monoclinic space group became apparent. All calculations were carried out in $P1$ except for the final occupancy determination and structure factor list.

A three-dimensional Patterson synthesis calculated in $P\bar{1}$ has $P2/m$ symmetry and only vectors within the asymmetric unit of $P2/m$ are discussed. The synthesis has a large number of peaks in (040) and a small number of peaks between these planes, as observed for $\text{Ph}_2\text{Hg}(\text{tmp})_2$. Very large peaks are present in the synthesis at $\frac{1}{2}, 0, \frac{3}{8}$; $\frac{1}{2}, 0, \frac{5}{8}$; $0.21, \frac{1}{4}, \frac{1}{2}$; $0.29, \frac{1}{4}, \frac{1}{8}$; and $0.29, \frac{1}{4}, \frac{7}{8}$. With b and c axis lengths almost identical with that of $\text{Ph}_2\text{Hg}(\text{tmp})_2$, and vectors almost entirely in (040), similarities between mercury-mercury vectors in Patterson syntheses were sought. Coincidence of b and c axes in Patterson syntheses brings one large vector at $\frac{1}{2}, 0, \frac{3}{8}$ almost into coincidence with the $\frac{1}{2}, 0, 0$ vector in the Patterson synthesis of $\text{Ph}_2\text{Hg}(\text{tmp})_2$, and there are many smaller vectors near the origin at about the correct distance from it for mercury-ligand vectors.

Using this orientation of the Patterson syntheses, and hence unit cells, an initial mercury atom position was chosen to satisfy the Patterson synthesis and coincide as closely as possible with the initial mercury position used for $\text{Ph}_2\text{Hg}(\text{tmp})_2$.

With Hg(1) at $\frac{1}{4}, \frac{1}{4}, \frac{3}{4}$ R was 0.53 and a difference synthesis had strong peaks at $0.04, 0, \frac{1}{4}$ (A); $\frac{1}{2} - 0.04, 0, \frac{1}{4}$ (B); $\frac{1}{2} + 0.04, 0, \frac{5}{8}$ (C); $\frac{1}{2} + 0.04, 0, \frac{7}{8}$ (D); $\frac{3}{4}, \frac{1}{4}, \frac{1}{8}$ (E) and $\frac{3}{4}, \frac{1}{4}, \frac{3}{8}$ (F). The $y = \frac{1}{2}$ and $\frac{3}{4}$ planes are identical to the $y = 0$ and $\frac{1}{4}$ planes respectively, except for a peak at $\frac{1}{4}, \frac{3}{4}, \frac{3}{4}$ corresponding to Hg(1) translated $\frac{1}{2}b$. Vectors from $\frac{1}{4}, \frac{1}{4}, \frac{3}{4}$ to E and F satisfy the Patterson synthesis peaks $\frac{1}{2}, 0, \frac{3}{8}$ and $\frac{1}{2}, 0, \frac{5}{8}$ respectively. Position E [Hg(2)] was added ($R = 0.50$) and peak F , together with the peak of the same x and z coordinates in the $y = \frac{3}{4}$ plane, was absent from a subsequent difference synthesis. Peaks at $\frac{1}{4}, \frac{3}{4}, \frac{3}{4}$ and $\frac{3}{4}, \frac{3}{4}, \frac{1}{8}$ became more intense, and mercury atoms were added with these coordinates together with $0.04, 0, \frac{1}{4}$ [Hg(3)]. A structure factor calculation had $R = 0.32$ and a difference synthesis had only one strong peak at $\frac{1}{2} + 0.04, \frac{1}{2}, \frac{5}{8}$. This position [Hg(4)] was added to the structure factor calculation ($R = 0.26$), and subsequent difference Fourier syntheses are shown in Fig. 5. The $y = \frac{1}{4}$ and $\frac{3}{4}$ planes are identical, as also are the $y = 0$ and $\frac{1}{2}$ planes but with a small peak in the $y = 0$ plane having the same x and z coordinates as the mercury position in the $y = \frac{1}{2}$ plane [Hg(4)], and a small peak in $y = \frac{1}{2}$ corresponding to the mercury position in $y = 0$ [Hg(3)].

As for $\text{Ph}_2\text{Hg}(\text{tmp})_2$ the planar ligands lie in (040). The ligands were added singly to the structure factor calculation and all ligands were found to be present, difference syntheses having only very small

Table 5 (cont.)

Table with 12 columns (M, K, FO, FC) and 12 rows of numerical data. The table is divided into several sections by horizontal lines and contains various numerical values, some with negative signs and some with asterisks. There are also some text markers like 'L = 7' and 'L = 8' interspersed within the data.

DISORDERED CRYSTALS OF ADDUCTS OF DIPHENYLMERCURY

Table 5 (cont.)

H	K	FO	FC	H	K	FO	FC	H	K	FO	FC	H	K	FO	FC	H	K	FO	FC	H	K	FO	FC					
0	0	137	171	10	6	49	43*	11	4	31	28*	+1	0	115	136	****	L	M	B	*****	+9	1	94	129				
0	2	118	142	11	8	49	93*	11	6	39	15*	-1	10	95	102								+9	3	80	118		
0	4	48	63*	11	8	75		12	4	41	8*	-1	12	87	92									-9	8	84	121	
0	6	86	135	12	2	44	60*	14	4	101	141	-2	0	40	2	0	3	22	0*					+10	1	48	117	
10	2	47	39*	12	2	43	30*	12	6	30	43	-2	2	30	43	0	5	30	0*					+10	3	47	39*	
1	4	18	4	12	4	68	96	12	6	96	117*	-2	4	32	44*	0	7	37	8*					+10	3	91	8*	
10	4	47	94*	13	2	77	92	13	10	30	7*	-2	4	37	48*	0	9	44	1*					+10	7	93	3*	
11	2	109	79	13	4	40	68*	13	4	36	47*	-3	0	119	80	0	11	51	0*					+10	9	55	4*	
11	4	77	89	13	6	67	115*	13	6	92	13*	-3	2	142	171	0	13	58	0*					+10	11	93	3*	
11	4	78	106	-1	2	273	299	14	2	139	15*	-3	4	70	30	0	15	9*	0*					+11	1	92	16*	
12	2	47	10*	-1	2	165	112	14	4	31	33*	-3	4	170	140	1	3	143	187					+11	3	91	0*	
13	2	48	114	-1	4	197	183	14	6	82	123	-3	6	75	44	1	5	188	187					+11	5	52	84*	
13	4	45	104*	-1	4	192	192	-1	2	21	93*	-3	6	70	30	0	7	150	168					+12	3	94	0*	
13	4	102	126	-1	8	198	121	-1	4	31	12*	-4	0	108	140	1	9	143	104					+12	3	94	7*	
13	6	79	94	-1	10	109	109	-1	6	93	85	-4	2	197	127	1	11	72	81					+12	3	93	1*	
+1	0	259	297	-1	12	102	121	+2	0	348	378	-4	4	81	122	1	13	94	83					+12	3	94	0*	
-1	3	248	241	-1	14	62	73	-2	2	80	76	+4	6	87	86	2	1	18	17*					+12	7	93	1*	
-1	4	187	140	-2	0	160	87	-2	4	230	244	+5	0	313	348	2	3	24	8*					+12	9	93	3*	
-2	2	133	28	-2	2	132	147	-2	4	80	10	+5	2	34	39*	5	9	31	3*					+12	9	91	2*	
-1	0	313	89	-2	4	102	99	-2	0	164	179	+5	4	244	207	2	7	30	8*					+13	3	94	7*	
-1	10	173	197	-2	6	213	186	-2	10	43	50*	+5	6	39	28*	2	9	49	9*					+13	5	95	67*	
+1	12	78	41	-2	8	44	40*	-2	12	110	127	+5	8	231	289	2	11	52	3*					+14	1	99	1*	
-1	14	90	130	-2	10	101	116	-3	0	81	91	+5	10	37	124	2	13	93	1*					+14	3	99	0*	
-2	0	286	278	-2	12	42	29*	-3	2	28	60*	+5	12	126	201	3	1	162	177					+14	5	94	0*	
-2	2	133	28	-2	14	86	83	-3	4	34	28*	+6	0	36	29*	3	3	194	170					+14	7	92	9*	
-2	4	214	179	-3	0	127	92	-3	6	35	49	+6	2	36	24*	3	5	149	181					+14	9	94	0*	
-3	0	39	33*	-3	2	207	198	+4	0	42	95	+6	4	38	22*	3	7	143	139					+14	9	93	9*	
-3	0	114	107	-3	4	67	107	+4	2	334	320	+6	6	40	23*	3	9	119	118					+14	9	92	9*	
+3	0	420	408	-3	6	192	207	+4	4	31	11	+7	0	38	32*	4	11	71	84					+14	9	94	0*	
+3	2	84	46	-3	8	44	42*	+4	6	304	282	-7	2	140	179	4	1	29	16*					+16	3	90	1*	
+3	2	301	282	-3	10	62	115*	+4	8	30	29*	-7	4	39	26*	4	3	29	8*					+16	5	48	0*	
+3	4	56	69	-3	12	102	121	+4	10	124	105	-7	6	149	127	5	5	34	16*					+16	7	94	0*	
+3	6	226	233	-3	14	90	92	+4	12	36	37*	-8	0	39	9*	4	7	40	6*					+17	3	46	49*	
+3	8	143	134*	-4	0	142	136	+4	16	97	126	-8	2	40	31*	4	9	47	6*					+17	5	42	40*	
+4	0	29	66*	-4	2	160	98	+4	18	139	99	-8	4	40	8*	4	11	55	1*					+18	1	41	1*	
+4	0	120	77	-4	4	93	90	+5	2	110	76	-8	0	40	20*	5	13	145	178									
+4	0	230	277	-4	6	41	87*	+5	4	120	125	-9	0	164	172	5	15	144	143									
+4	0	318	92*	-4	8	160	98	+5	6	78	87	-9	2	40	90*	5	17	146	136									
+4	0	108	76	+5	2	270	250	+5	8	96	81	-9	4	126	131	5	19	107	117									
+4	0	83	64	+5	2	35	13*	+5	0	99	49	+9	0	30	97*	6	1	32	4*									
+5	0	31	59	+5	4	221	205	+6	2	193	239	+9	0	149	137	6	3	35	2*									
+5	2	294	276	+5	6	42	13*	+6	4	31	6	+10	0	40	48*	6	5	39	9*									
+5	4	38	7*	+5	8	202	229	+6	6	134	183	+10	2	40	23*	6	7	44	1*									
+5	4	233	222	+5	10	49	3*	+6	8	80	80	+10	4	39	31*	6	9	50	2*									
+5	6	46	6	+5	12	139	137	+6	10	39	44*	+10	6	37	17*	6	11	71	14									
+5	10	102	94	+6	0	141	96	+7	2	40	38*	+11	0	71	134	6	13	54	0*									
+5	12	44	5*	+6	2	217	176	+7	4	41	80*	+11	2	70	80	7	1	130	143									
+5	14	74	94	+6	4	184	74	+7	6	43	20*	+11	4	61	93	7	3	113	146									
+6	0	64	94	+6	6	149	135	+8	0	207	207	+11	6	90	110	7	5	101	118									
+6	2	52	68	+6	8	74	92	+8	2	120	131	+11	8	93	73	7	7	89	104									
+6	4	40	83*	+6	10	72	65	+8	4	174	149	+12	0	37	11*	7	9	115	98									
+6	6	44	43*	+6	12	125	102	+8	6	101	117	+12	2	36	94	8	1	30	9*									
+7	0	373	35*	+6	14	266	305	+8	8	161	183	+12	4	34	7*	8	3	41	1*									
+7	2	40	28*	+6	16	95	96	+8	10	37	48*	+12	6	31	22*	8	5	44	1*									
+7	4	292	301	+6	18	216	229	+8	12	110	127	+13	0	33	80*	8	7	42	12*									
+7	6	46	24*	+6	20	42	92*	+9	0	42	17*	+13	2	60	85	8	9	53	2*									
+7	8	287	312	+6	22	209	188	+9	2	43	31*	+13	4	30	41*	8	11	55	2*									
+7	10	48	10*	+6	24	44	75*	+9	4	45	24*	+13	6	57	110	9	13	55	4*									
+7	12	181	215	+6	26	157	152	+9	6	42	90*	+13	8	57	110	9	15	42	180*									
+8	0	278	297	+6	28	160	138	+10	0	61	81	+13	10	33	113	10	17	110	118									
+8	2	257	196	+6	30	162	123	+10	2	147	188	+13	12	59	121*	10	19	111*	111*									
+8	4	200	219	+6	32	101	99	+10	4	167	183	+13	14	7	93	11	21	93	117									
+8	6	121	113	+6	34	131	135	+10	6	141	186	+13	16	1	46	7*	4	3	110	108								
+8	8	137	198	+6	36	133	111	+10	8	38	97*	+13	18	10	3	3*	4	5	140	130								
+8	10	49	62*	+10	0	82	140	+10	10	89	80	+13	20	10	9	8*	4	7	111	111								
+8	12	89	137	+10	2	45	80*	+11	0	43	37*	+13	22	7	53	3*	4	9	94	0*								
+8	14	45	43*	+10	4	74	118	+11	2	42	27*	+13	24	10	9	5*	4	11	85	7*								
+9	0	44	24*	+10	6	63	82	+11	4	21	24*	+13	26	11	1	8*	4	13	86									

positive or negative regions at ligand atom positions as each ligand was added to the calculation.

Phenyl groups are located immediately above and below each mercury atom, and overlap for mercury atoms in the $y=\frac{1}{4}$ and $\frac{3}{4}$ planes. Difference Fourier syntheses indicate that the appropriate monoclinic space group is Pm , having mirror planes in (020). Ph_2Hg moieties in $y=\frac{1}{4}$ and $\frac{3}{4}$ were given half-occupancy and a subsequent difference synthesis had a larger peak in $y=0$ at $x > \frac{1}{2}$ [Fig. 5(a)], and in $y=\frac{1}{2}$ at $x < \frac{1}{2}$ than previously. Packing requirements again indicate the presence of partial occupancy for the Ph_2Hg moieties with mercury atoms in (020), and best agreement between calculated and observed structure factors was obtained with Ph_2Hg occupancies of 0.8 and 0.2, as found for $\text{Ph}_2\text{Hg}(\text{tmp})_2$.

This model has a unit cell with the required number of four formula units of $\text{Ph}_2\text{Hg}(\text{dmp})_2$, and for structure factors calculated for space group Pm , $R=0.16$ for both the 1331 sharp reflexions and the 537 diffuse reflexions, and 0.24 when 1981 unobserved reflexions are included. The structure factor list (Table 5) includes both $hk0$ and $\bar{h}k0$ reflexions, which must be identical for Pm symmetry, to indicate their similarity. Refinement was not attempted with 150 atoms in the asymmetric unit of space group Pm (232 in $P1$). Tables 6, 7 and 8 show the atomic coordinates, bond lengths and bond angles in $\text{Ph}_2\text{Hg}(\text{dmp})_2$.

Table 6. Coordinates of atoms of $\text{Ph}_2\text{Hg}(\text{dmp})_2$ within the asymmetric unit of space group Pm

The atom numbering scheme is similar to that of $\text{Ph}_2\text{Hg}(\text{tmp})_2$ (Fig. 2), with methyl groups having carbon atoms C(15) and C(16) and phenyl groups commencing with C(17) and C(23).

	x/a	y/b	z/c	Occu- pancy
Mercury positions				
Hg(1)	0.25	0.25	0.75	0.5
Hg(2)	0.75	0.25	0.1196*	0.5
Hg(3)	0.0433	0	0.25	0.8
Hg(4)	0.5433	0.50	0.6196†	0.8
Hg(5)	0.5433	0	0.6196†	0.2
Hg(6)	0.0433	0.50	0.25	0.2

* Given as *ca.* $\frac{1}{3}$ in discussing the structure solution.

† Given as *ca.* $\frac{2}{3}$ in discussing the structure solution.

Ligand coordinated to Hg(1)

N(1)	0.0683	0.25	0.8179	1.0
N(10)	0.0958	0.25	0.6384	1.0
C(2)	0.0500	0.25	0.9107	1.0
C(3)	-0.0417	0.25	0.9375	1.0
C(4)	-0.1083	0.25	0.8804	1.0
C(5)	-0.1667	0.25	0.7357	1.0
C(6)	-0.1458	0.25	0.6429	1.0
C(7)	-0.0433	0.25	0.5179	1.0
C(8)	0.0417	0.25	0.4875	1.0
C(9)	0.1200	0.25	0.5429	1.0
C(11)	0.0125	0.25	0.6679	1.0
C(12)	-0.0067	0.25	0.7571	1.0
C(13)	-0.0917	0.25	0.7857	1.0
C(14)	-0.0583	0.25	0.6125	1.0
C(15)	0.1167	0.25	0.9777	1.0
C(16)	0.2125	0.25	0.5134	1.0

Table 6 (cont.)

	x/a	y/b	z/c	Occu- pancy
Ligand coordinated to Hg(2)				
N(1)	0.6000	0.25	0.2321	1.0
N(10)	0.5800	0.25	0.0491	1.0
C(2)	0.6117	0.25	0.3214	1.0
C(3)	0.5433	0.25	0.3804	1.0
C(4)	0.4583	0.25	0.3446	1.0
C(5)	0.3583	0.25	0.2161	1.0
C(6)	0.3467	0.25	0.1250	1.0
C(7)	0.4033	0.25	-0.0232	1.0
C(8)	0.4750	0.25	-0.0768	1.0
C(9)	0.5633	0.25	-0.0393	1.0
C(11)	0.5050	0.25	0.1071	1.0
C(12)	0.5167	0.25	0.1964	1.0
C(13)	0.4433	0.25	0.2536	1.0
C(14)	0.4167	0.25	0.0714	1.0
C(15)	0.7000	0.25	0.3571	1.0
C(16)	0.6333	0.25	-0.0982	1.0

Ligand coordinated to Hg(3)

N(1)	0.1917	0	0.1339	1.0
N(10)	0.2133	0	0.3250	1.0
C(2)	0.1733	0	0.0446	1.0
C(3)	0.2500	0	-0.0089	1.0
C(4)	0.3333	0	0.0268	1.0
C(5)	0.4250	0	0.1518	1.0
C(6)	0.4500	0	0.2500	1.0
C(7)	0.3792	0	0.3982	1.0
C(8)	0.3125	0	0.4553	1.0
C(9)	0.2292	0	0.4196	1.0
C(11)	0.2833	0	0.2678	1.0
C(12)	0.2667	0	0.1741	1.0
C(13)	0.3417	0	0.1205	1.0
C(14)	0.3700	0	0.3071	1.0
C(15)	0.0833	0	0.0089	1.0
C(16)	0.1458	0	0.4732	1.0

Ligand coordinated to Hg(4)

N(1)	0.7233	0.5	0.5464	1.0
N(10)	0.6967	0.5	0.7321	1.0
C(2)	0.7500	0.5	0.4554	1.0
C(3)	0.8367	0.5	0.4241	1.0
C(4)	0.9017	0.5	0.4839	1.0
C(5)	0.9517	0.5	0.6304	1.0
C(6)	0.9333	0.5	0.7277	1.0
C(7)	0.8333	0.5	0.8464	1.0
C(8)	0.7500	0.5	0.8839	1.0
C(9)	0.6717	0.5	0.8250	1.0
C(11)	0.7800	0.5	0.6982	1.0
C(12)	0.8000	0.5	0.6027	1.0
C(13)	0.8800	0.5	0.5714	1.0
C(14)	0.8500	0.5	0.7571	1.0
C(15)	0.6750	0.5	0.4018	1.0
C(16)	0.5767	0.5	0.8536	1.0

Atoms of ligand coordinated to Hg(5) have coordinates of atoms of ligand coordinated to Hg(3) $+0, \frac{1}{2}, 0$ and occupancy 1.0.

Atoms of ligand coordinated to Hg(6) have coordinates of atoms of ligand coordinated to Hg(4) $+0, \frac{1}{2}, 0$ and occupancy 1.0.

Phenyl groups bonded to Hg(1)

C(17)	0.25	0.3967	0.75	0.5
C(18)	0.3283	0.4467	0.7571	0.5
C(19)	0.3283	0.5433	0.7571	0.5
C(20)	0.25	0.5933	0.75	0.5
C(21)	0.1767	0.5433	0.7429	0.5
C(22)	0.1767	0.4467	0.7429	0.5
C(23)	0.25	0.1033	0.75	0.5

Table 6 (cont.)

	<i>x/a</i>	<i>y/b</i>	<i>z/c</i>	Occu- pancy
C(24)	0.1767	0.0533	0.7429	0.5
C(25)	0.1767	-0.0433	0.7429	0.5
C(26)	0.25	-0.0933	0.75	0.5
C(27)	0.3283	-0.0433	0.7571	0.5
C(28)	0.3283	0.0533	0.7571	0.5
Phenyl groups bonded to Hg(2)				
C(17)	0.75	0.3967	0.1196	0.5
C(18)	0.8283	0.4467	0.1196	0.5
C(19)	0.8283	0.5433	0.1196	0.5
C(20)	0.75	0.5933	0.1196	0.5
C(21)	0.6733	0.5433	0.1196	0.5
C(22)	0.6733	0.4467	0.1196	0.5
C(23)	0.75	0.1033	0.1196	0.5
C(24)	0.6733	0.0533	0.1196	0.5
C(25)	0.6733	-0.0433	0.1196	0.5
C(26)	0.75	-0.0933	0.1196	0.5
C(27)	0.8283	-0.0433	0.1196	0.5
C(28)	0.8283	0.0533	0.1196	0.5
Phenyl groups bonded to Hg(3)				
C(17)	0.0433	0.1467	0.2500	0.8
C(18)	-0.0417	0.1967	0.2500	0.8
C(19)	-0.0417	0.2933	0.2500	0.8
C(20)	0.0433	0.3433	0.2500	0.8
C(21)	0.1167	0.2933	0.2500	0.8
C(22)	0.1167	0.1967	0.2500	0.8
Phenyl groups bonded to Hg(4)				
C(17)	0.5433	0.6467	0.6196	0.8
C(18)	0.4633	0.6967	0.6161	0.8
C(19)	0.4633	0.7933	0.6161	0.8
C(20)	0.5433	0.8433	0.6196	0.8
C(21)	0.6233	0.7933	0.6232	0.8
C(22)	0.6233	0.6967	0.6232	0.8

Atoms of phenyl groups bonded to Hg(5) have coordinates of the phenyl groups of Hg(3) + 0, $\frac{1}{2}$, 0 and occupancy of 0.2.

Atoms of phenyl groups bonded to Hg(6) have coordinates of the phenyl groups of Hg(4) + 0, $\frac{1}{2}$, 0 and occupancy of 0.2.

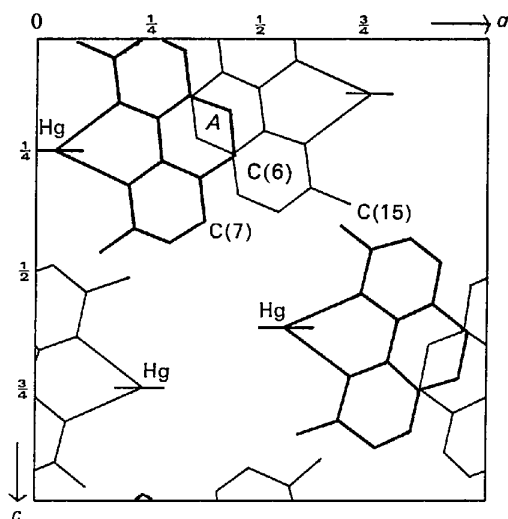


Fig. 6. Projection of the structure of $\text{Ph}_2\text{Hg}(\text{dmp})_2$ onto (010). The ligand and Ph_2Hg moieties in (020) are shown by bold lines.

Table 7. Bond lengths in $\text{Ph}_2\text{Hg}(\text{dmp})_2$

(a) Mercury stereochemistry				
	Hg(1)	Hg(2)	Hg(3)	Hg(4)
Hg-N(1)	2.97 Å	2.83 Å	2.84 Å	2.97 Å
Hg-N(10)	2.88	2.82	2.84	2.88
Hg-C(17)	2.10	2.10	2.10	2.10
Hg-C(23)	2.10	2.10		
(b) Ligands				
	Ligand coordinated to:			
	Hg(1)	Hg(2)	Hg(3)	Hg(4)
N(1)-C(2)	1.38 Å	1.31 Å	1.33 Å	1.39 Å
N(1)-C(12)	1.46	1.39	1.30	1.44
C(2)-C(3)	1.47	1.36	1.42	1.41
C(3)-C(4)	1.32	1.41	1.39	1.33
C(4)-C(13)	1.40	1.34	1.37	1.32
C(5)-C(6)	1.39	1.34	1.48	1.44
C(5)-C(13)	1.37	1.42	1.36	1.40
C(6)-C(14)	1.42	1.33	1.49	1.35
C(7)-C(8)	1.38	1.35	1.32	1.40
C(7)-C(14)	1.40	1.39	1.33	1.33
C(8)-C(9)	1.45	1.47	1.39	1.48
C(9)-N(10)	1.44	1.31	1.40	1.41
N(10)-C(11)	1.35	1.43	1.36	1.38
C(11)-C(12)	1.33	1.31	1.39	1.42
C(11)-C(14)	1.36	1.46	1.45	1.38
C(12)-C(13)	1.38	1.41	1.40	1.32
C(2)-C(15)	1.42	1.46	1.48	1.40
C(9)-C(16)	1.49	1.38	1.50	1.52
(c) Phenyl groups				
	For Hg(1)	For Hg(2)	For Hg(3)	For Hg(4)
C(17)-C(18)	1.41 Å	1.40 Å	1.34 Å	1.43 Å
C(17)-C(22)	1.34	1.38	1.49	1.43
C(18)-C(19)	1.38	1.38	1.38	1.38
C(19)-C(20)	1.41	1.40	1.34	1.43
C(20)-C(21)	1.34	1.38	1.49	1.43
C(21)-C(22)	1.38	1.38	1.38	1.38
C(23)-C(24)	1.34	1.38		
C(23)-C(28)	1.41	1.40		
C(24)-C(25)	1.38	1.38		
C(25)-C(26)	1.34	1.38		
C(26)-C(27)	1.41	1.40		
C(27)-C(28)	1.38	1.38		

In both structure determinations all atoms were given isotropic temperature factors of 3.0 \AA^2 and scattering factors given by Cromer & Waber (1965) were used. No corrections were made for anomalous dispersion. The full-matrix least-squares program used was that of Busing & Levy (1962) modified by White (1965) to operate on the Monash University CDC 3200 computer. The function minimized was $\sum_{hkl} w_{hkl} (|F_o| - |kF_c|)^2$ for which all reflexions were given unit weight and an overall scale used. Unobserved reflexions were assigned an arbitrary intensity value of one-half of the weakest observed reflexions. The Fourier program was written by White (1965). Powder photographs were obtained using a Philips Debye-Scherrer 114.6 mm powder camera with nickel-filtered $\text{Cu K}\alpha$ radiation.

(a) The superposition structures

Within their respective unit cells, each adduct has two ligands in each (040) plane with Ph_2Hg moieties in

Table 8. Bond angles in $\text{Ph}_2\text{Hg}(\text{dmp})_2$

(a) Mercury stereochemistry				
	Hg(1)	Hg(2)	Hg(3)	Hg(4)
N(1)—Hg—N(10)	54°	57°	59°	56°
C(17)—Hg—C(23)	90	90		
N(1), N(10)—Hg—C(17), C(23)	90	90		
(b) Ligands				
	Ligands coordinated to:			
	Hg(1)	Hg(2)	Hg(3)	Hg(4)
Hg—N(1)—C(2)	121°	117°	114°	128°
Hg—N(1)—C(12)	123	123	117	124
C(2)—N(1)—C(12)	116	120	129	107
N(1)—C(2)—C(3)	117	121	111	126
N(1)—C(2)—C(15)	122	119	123	107
C(3)—C(2)—C(15)	121	120	126	127
C(2)—C(3)—C(4)	126	119	125	120
C(3)—C(4)—C(13)	118	122	117	116
C(6)—C(5)—C(13)	109	120	125	117
C(5)—C(6)—C(14)	122	118	109	120
C(8)—C(7)—C(14)	118	117	123	124
C(7)—C(8)—C(9)	128	123	119	122
N(10)—C(9)—C(8)	109	123	122	110
N(10)—C(9)—C(16)	122	117	111	122
C(8)—C(9)—C(16)	129	120	127	129
Hg—N(10)—C(9)	109	123	123	109
Hg—N(10)—C(11)	127	123	120	124
C(9)—N(10)—C(11)	123	115	117	127
N(10)—C(11)—C(12)	121	118	117	123
N(10)—C(11)—C(14)	125	123	119	121
C(12)—C(11)—C(14)	114	119	124	116
N(1)—C(12)—C(11)	115	120	127	112
N(1)—C(12)—C(13)	125	122	119	125
C(11)—C(12)—C(13)	120	118	113	123
C(4)—C(13)—C(5)	112	122	115	113
C(4)—C(13)—C(12)	118	116	119	125
C(5)—C(13)—C(12)	130	121	127	122
C(6)—C(14)—C(7)	118	117	118	120
C(6)—C(14)—C(11)	125	123	123	123
C(7)—C(14)—C(11)	117	119	119	117
(c) Phenyl groups				
	For Hg(1)	For Hg(2)	For Hg(3)	For Hg(4)
Hg—C(17)—C(18)	121°	121°	119°	120°
Hg—C(17)—C(22)	122	121	122	120
C(18)—C(17)—C(22)	117	118	119	120
C(17)—C(18)—C(19)	121	121	119	120
C(18)—C(19)—C(20)	121	121	119	120
C(19)—C(20)—C(21)	117	118	119	120
C(20)—C(21)—C(22)	122	121	122	120
C(17)—C(22)—C(21)	122	121	122	120
Hg—C(23)—C(24)	122	121		
Hg—C(23)—C(28)	121	121		
C(24)—C(23)—C(28)	117	118		
C(23)—C(24)—C(25)	122	121		
C(24)—C(25)—C(26)	122	121		
C(25)—C(26)—C(27)	117	118		
C(26)—C(27)—C(28)	121	121		
C(23)—C(28)—C(27)	121	121		

position for coordination of each ligand to form a 1:1 complex [Figs. 4 and 6]. Every second (040) plane has 0.5 occupancy for two Ph_2Hg moieties, and every other (040) plane has 0.8 and 0.2 occupancy for two Ph_2Hg moieties in that plane, giving one Ph_2Hg for every two ligands.

Molecular groups are packed at approximately van

der Waals distances from each other, with ligand planes separated by 3.63 Å in $\text{Ph}_2\text{Hg}(\text{tmp})_2$ and 3.57 Å in $\text{Ph}_2\text{Hg}(\text{dmp})_2$. Methyl groups in adjacent planes have slightly different x and z coordinates allowing closer approach in the b direction, but their x and z coordinates are closer in $\text{Ph}_2\text{Hg}(\text{tmp})_2$ than in $\text{Ph}_2\text{Hg}(\text{dmp})_2$, possibly accounting for the greater distance between planes in $\text{Ph}_2\text{Hg}(\text{tmp})_2$.

Excluding Hg—N distances closest approaches of adjacent mercury and/or ligand carbon atoms in the same (040) plane are 4.17 Å [C(16)—C(18)] and 3.66 Å [C(16)—Hg] in $\text{Ph}_2\text{Hg}(\text{tmp})_2$ [Fig. 4(a)], and 4.10 Å [C(6)—C(15) and C(7)—Hg] in $\text{Ph}_2\text{Hg}(\text{dmp})_2$ (Fig. 6).

(b) The structures of possible ordered regions

The derivation of possible ordered regions from the superposition structure must account for the diffuse lines and Ph_2Hg occupancies. With this requirement, the superposition structure can be most readily discussed in terms of disorder arising from a channel structure similar to the urea-di- n -alkylmercury inclusion compounds.

Inspection of a projection diagram of $\text{Ph}_2\text{Hg}(\text{dmp})_2$ (Fig. 6) and Fig. 4 indicate that for both adducts there are channels through the ligand structure perpendicular to the planes containing the ligands, and that the channels have appropriate dimensions to allow inclusion of Ph_2Hg moieties aligned with the C—Hg—C unit along the [010] direction. A random distribution of Ph_2Hg moieties, with the constraint that weak Hg—N interaction causes the mercury atoms to lie in (040), would lead to the presence of diffuse lines but without maxima in the lines since this structure would have cell dimensions $a, \frac{1}{2}b, c$. Some degree of ordering of Ph_2Hg occupancies in (020) causes the appearance of maxima in the lines, which have been used to determine occupancies in these planes.

Disordering of crystals is consistent with the solution stability of the adducts. The adducts are completely dissociated in benzene and this may indicate that crystal growth occurs by addition of ligand and Ph_2Hg moieties, rather than addition of complexes. Failure of a Ph_2Hg molecule to add to the growth plane would hardly affect the packing of ligand groups, since the presence of Ph_2Hg does not affect the distance between planes, and the ligands themselves are fairly closely packed in the (040) planes (Fig. 6), and thus mistakes may occur readily with a new ordered region extending from each mistake in crystal growth. A possible ordered region containing one Ph_2Hg moiety in each (040) plane is illustrated in Fig. 7. Ordered regions of this structure have space group $P1$ for both adducts.

There is no apparent reason why the crystals of each adduct selected for structure determination have the same set of occupancies for Ph_2Hg moieties in (040) when the disorder is explained by either a channel structure or ordered regions related by translations of $\frac{1}{2}b$. One possible explanation is that the composition of each channel is influenced, but not determined, by the

composition of neighbouring channels during crystal growth (channel model), or that mistakes during crystal growth have a fixed probability for each adduct. The close similarity of each model is apparent since mistakes in addition of Ph_2Hg moieties give essentially the same result – either a new ordered region displaced by $\frac{1}{2}b$ with ligand positions coincident, or a new channel with Ph_2Hg positions displaced by $\frac{1}{2}b$ but with ligand positions unaltered.

(c) *Twining of disordered crystals of $\text{Ph}_2\text{Hg}(\text{tmp})_2$*

Preliminary X-ray photographs indicated that disordered crystals of $\text{Ph}_2\text{Hg}(\text{tmp})_2$ readily form twins, with components related by a mirror plane in a^*b . Fig. 8 indicates that reflection in this plane followed by translation in ac leads to perfect coincidence of atom positions within the unit cell of one component.

(d) *Symmetry of the molecular packing*

The requirements for reflexions to be observed for $\text{Ph}_2\text{Hg}(\text{dmp})_2$, none of which are required for space group Pm , are explained from the molecular packing. The requirement $hk0:h+k=2n$ results from the presence of an n glide in projection on (001), and the requirement $00l:l=2n$ results from every ab plane having a neighbouring plane $\frac{1}{2}c$ removed containing exactly the same total contribution of C, N, and Hg electron density to the structure factor expression for Pm . The requirement $0k0:k=4n$ results from a similar situation in all ac planes separated by $\frac{1}{4}b$, even though a fourfold screw axis which normally causes this requirement is not present. Inspection of Weissenberg photographs of twinned crystals of $\text{Ph}_2\text{Hg}(\text{tmp})_2$ (the data crystal was only mounted about b) reveals the requirement $0k0:k=4n$ for the components, but since there is no periodicity of $\frac{1}{2}c$ in content of ab planes (Fig. 4) the requirement $00l:l=2n$ does not occur.

If the origin of the unit cell is moved to A and translated $\frac{1}{8}b$ for both superposition structures [Fig. 4(a) and Fig. 6] maintaining the directions of axes as before, then atom positions conform to $P\bar{1}$, although atoms of different occupancy are related by centres of inversion.

The molecular structure

In both adducts the C–Hg–C unit of the diphenylmercury moieties is linear, as in the parent mercurial. Mercury–nitrogen distances are 2.8–3.0 Å for both adducts. For ligands having mercury atoms close to their nitrogen atoms the N–Hg–N angle is *ca.* 54–59°. Since the accuracy of Hg–N distances determined from difference Fourier syntheses is uncertain, it is not possible to ascertain whether the nitrogen atoms are definitely within the sum of van der Waals radii from mercury (3.0 Å with mercury radius of 1.50 Å, or 3.23 Å with the upper limit of 1.73 Å suggested by Grdenic (1965) as a criterion to indicate some form of bonding) and thus whether a mercury–nitrogen interaction is present in the adducts.



Fig. 7. Stereoscopic illustration of a possible ordered cell of $\text{Ph}_2\text{Hg}(\text{dmp})_2$ containing a 1:1 'complex' and uncoordinated ligand in each (040) plane. The unit cell is viewed almost normal to (001). The figure was drawn by a Fortran thermal ellipsoid plot program for crystal structure illustrations (Johnson, 1965).

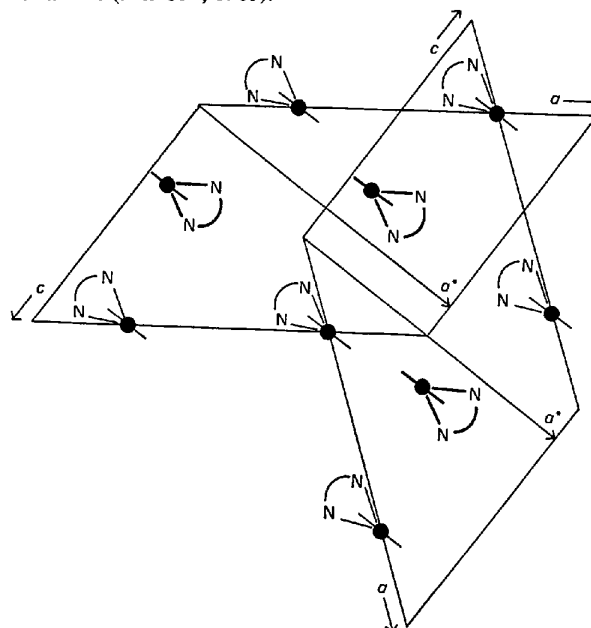


Fig. 8. Orientation of components of twinned crystals of $\text{Ph}_2\text{Hg}(\text{tmp})_2$. The (010) projection is shown with ligand and Ph_2Hg moieties in (020) drawn with bold lines.

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