

Mercury(II) complexes of bis(diphenylphosphinomethyl)phenylphosphine

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Abstract

Treatment of mercury(II) chloride with dpmp (dpmp is bis(diphenylphosphinomethyl)phenylphosphine) yields a mixture of $\text{HgCl}_2(\text{dpmp})$ and $[\text{Hg}_3(\mu\text{-Cl})_2(\mu\text{-dpmp})_2]\text{Cl}_2$. With mercury(II) bromide and iodide, only $\text{HgX}_2(\text{dpmp})$ forms. $[\text{Hg}_3(\mu\text{-Cl})_2\text{Cl}_2(\mu\text{-dpmp})_2]\text{Cl}(\text{HCO}_3) \cdot 5\text{CHCl}_3$ crystallizes in the monoclinic space group $P2_1/n$ with $a = 16.424(5)$, $b = 26.915(9)$, $c = 19.896(5)$ Å, $\beta = 96.99(2)^\circ$, at 130 K with $Z = 4$. Refinement of 434 parameters with 5277 reflections yields $R = 0.080$, $R_w = 0.067$. The complex consists of three roughly tetrahedrally coordinated mercury ions that are connected to the two bridging phosphine ligands. Each mercury ion is coordinated to two phosphorus atoms and two chloride ligands. The $\text{Hg} \cdots \text{Hg}$ separations are 3.339(2) and 3.331(2) Å which suggest that there is little or no bonding between these d^{10} centers.

Introduction

Recently there has been considerable interest in the weak metal–metal interactions between closed-shell ions such as Au(I) [1, 2] and Tl(I) [3]. For the d^{10} Au(I) complexes, $\text{Au} \cdots \text{Au}$ separations of less than 3.5 Å have been considered unusually short and taken to indicate that an attractive interaction was involved [2]. In the context of these studies, which included the recent demonstration that weak $\text{Au} \cdots \text{Au}$ interactions could be observed in solution and these were implicated in guiding a reaction [4], we have undertaken studies to examine Hg(II) complexes for evidence of weak $\text{Hg} \cdots \text{Hg}$ bonding. Interesting examples of Hg(II)/Au(I) bonding are known [5]. To do so, we have used the triphosphine ligand dpmp (bis(diphenylphosphinomethyl)phenylphosphine), which has been shown to form a number of nearly linear, trimetallic complexes with varying degrees of metal–metal bonding [6, 7].

A number of contemporary studies have also shown that novel, mixed valence, or non-integral oxidation state mercury complexes that involve Hg–metal bonds can be formed [8–10]. Frequently the synthesis of these involves the incorporation of mercury atoms from elemental mercury. In our examination of mercury complexes of dpmp, we have also looked for

evidence of the formation of species with unusual oxidation states of mercury.

Results

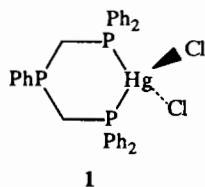
Treatment of mercury(II) chloride with dpmp under a variety of conditions including different solvents and reaction stoichiometries yielded mixtures of two complexes that can readily be identified by their $^{31}\text{P}\{\text{H}\}$ NMR spectra. The relevant spectral parameters are set out in Table 1. One, formulated as $\text{HgCl}_2(\text{dpmp})$ by analogy with the bromo and iodo complexes *vide infra*, exhibits a spectrum that consists of a doublet at 20.3 ppm and a triplet at –38.2 ppm. The doublet is surrounded by low intensity satellites that are consistent with bonding of the phosphorus atoms to mercury. No satellites are present around the triplet. The pattern of resonances is consistent with observations made previously on dpmp complexes in which dpmp forms a six-membered chelate ring that leaves the internal phosphorus atom uncoordinated [11, 12]. Consequently we formulate $\text{HgCl}_2(\text{dpmp})$ as a simple four-coordinated, tetrahedral complex of mercury(II), 1. The other species shows multiplets at 24.5 and 21.0 ppm due to the internal and terminal phosphorus atoms of dpmp. Both resonances have satellites that indicate direct bonding to mercury [13]. On the basis of the

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TABLE 1. $^{31}\text{P}\{^1\text{H}\}$ NMR spectral parameters^a

Compound	$\delta(\text{P}_i)$ (ppm)	$^1J(\text{Hg}, \text{P}_i)$ (Hz)	$\delta(\text{P}_t)$ (ppm)	$^1J(\text{Hg}, \text{P}_t)$ (Hz)	$J(\text{P}_i, \text{P}_t)$ (Hz)
$[\text{Hg}_3(\mu\text{-Cl})_2\text{Cl}_2(\mu\text{-dpmp})_2]^{2+}$	24.5	6041	21.0	5267	21
$\text{HgCl}_2(\text{dpmp})$	-38.2		20.3	3505	85
$\text{HgBr}_2(\text{dpmp})$	-37.1		11.4	2931	79
$\text{HgI}_2(\text{dpmp})^b$	-39.7		4.1	2251	86

^aIn chloroform solution P_i and P_t refer to the internal and terminal phosphorus atoms of dpmp. ^bRecorded at -81°C . The resonance due to P_t broadens on warming, presumably due to exchange processes.



crystallographic data (*vide infra*) and this ^{31}P data, the complex is formulated as the salt $[\text{Hg}_3(\mu\text{-Cl})_2\text{Cl}_2(\mu\text{-dpmp})_2]\text{Cl}_2$. Small quantities of the mixed salt $[\text{Hg}_3(\mu\text{-Cl})_2(\mu\text{-dpmp})_2](\text{HCO}_3)\text{Cl}$ were obtained by slow evaporation for the X-ray study. The bicarbonate ion arises from contact of the sample with the atmosphere during the crystallization process. Another mixed salt, $[\text{Hg}_3(\mu\text{-Cl})_2(\mu\text{-dpmp})_2](\text{PF}_6)\text{Cl}$, can be obtained nearly free of $\text{HgCl}_2(\text{dpmp})$ by the procedure outlined in 'Experimental'. It has not been possible to obtain samples of $\text{HgCl}_2(\text{dpmp})$ that were free of contamination of the tri-mercury cation. Treatment of dpmp with mercury(I) chloride yielded a black precipitate presumed to contain elemental mercury and a mixture of $\text{HgCl}_2(\text{dpmp})$ and $[\text{Hg}_3(\mu\text{-Cl})_2\text{Cl}_2(\mu\text{-dpmp})_2]^{2+}$. No evidence for the formation of complexes of mercury in lower oxidation states was found.

The reaction of mercury(II) bromide with dpmp yields only $\text{HgBr}_2(\text{dpmp})$ which was isolated in pure form. In this case there was no evidence for the formation of any other species. Mercury(II) iodide reacted similarly to form $\text{HgI}_2(\text{dpmp})$. These mercury phosphine complexes did not show any reaction toward elemental mercury.

The $^{31}\text{P}\{^1\text{H}\}$ NMR data on the chelated complexes, $\text{HgX}_2(\text{dpmp})$, show spectra characteristics that are similar to those noted by Pregosin and co-workers for other tetrahedral mercury complexes with P_2X_2 coordination [13]. Thus as the halide changes from Cl to Br and to I, the chemical shift of the coordinated phosphorus moves to higher field, while the one-bond Hg-P coupling constant decreases.

Structure of $[\text{Hg}_3(\mu\text{-Cl})_2\text{Cl}_2(\mu\text{-dpmp})_2](\text{HCO}_3)\text{Cl}$

Atomic coordinates are given in Table 2. A selection of important bond distances and angles is given in

Table 3. Figure 1 shows a perspective drawing of the cation. Figure 2 shows another view. The cation is well separated from the two anions. It does not display any crystallographically imposed symmetry. Nevertheless its core, without the phenyl rings, has approximate C_{2v} symmetry with the two-fold axis passing through Hg(3).

The complex involves a slightly bent chain of three mercury ions that are each coordinated by two phosphorus atoms and two chloride ions. Two of the chloride ligands, those surrounding Hg(3), are bridging, while two are terminal. The terminal Hg-Cl bond distances are shorter, as may be expected, than the bridging Hg-Cl distances. However, both are within normal ranges for tetrahedral Hg(II) complexes [13-15]. All of the Hg-P distances are similar.

As seen in Fig. 2, the two dpmp ligands are not *trans* to one another as is generally seen in other complexes where two dpmp ligands span three transition metal ions [6, 7]. Rather, these two ligands are bent toward one another to give P-Hg-P angles that range from 128.3 to 143.9°. This bending allows each mercury to obtain a quasi-tetrahedral geometry. Nevertheless, the range of bond angles at each mercury is quite large. For Hg(3), for example, it extends from 90.2 to 143.9°. The angular distortion extends to the bridging chloride ligands where the Hg-Cl-Hg angles are compressed to 77.4 and 77.6°. The problem of accommodating these bridging halides is probably responsible for the inability to form the corresponding complex cation with bromide or iodide ligands. The larger size of bromide or iodide ions simply does not allow them to fit into such a crowded bridging environment.

The Hg(1)···Hg(3) distance (3.339(2) Å) is similar to the Hg(2)···Hg(3) distance (3.331(2) Å). These distances are much greater than the range (2.4-2.7 Å) of Hg-Hg single bond distances found for compounds containing Hg_2^{2+} [16]. It seems that these distances, along with the quasi-tetrahedral geometry at each Hg ion in the complex cation, indicate that there is little, if any, bonding between these mercury ions along this bent chain (Hg(1)···Hg(3)···Hg(2))

TABLE 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement coefficients ($\text{\AA}^2 \times 10^3$)

	x	y	z	U_{eq}^a
Hg(1)	1835(1)	2052(1)	4495(1)	18(1)
Hg(2)	4394(1)	769(1)	2827(1)	20(1)
Hg(3)	3066(1)	1302(1)	3741(1)	17(1)
P(1)	1060(5)	2141(3)	3367(5)	20(3)
P(2)	2114(5)	1381(3)	2692(4)	17(3)
P(3)	3386(6)	939(3)	1842(5)	22(3)
P(4)	5417(5)	1335(3)	3409(4)	17(3)
P(5)	4243(5)	1687(3)	4419(5)	19(3)
P(6)	3090(5)	2363(3)	5145(5)	19(3)
Cl(1)	694(5)	2108(4)	5207(5)	37(3)
Cl(2)	5177(5)	57(3)	2435(5)	31(3)
Cl(3)	2075(5)	1076(3)	4663(4)	22(3)
Cl(4)	3568(5)	351(3)	3726(5)	29(3)
Cl(5)	9167(6)	1900(4)	6153(6)	55(5)
Cl(6)	7470(6)	2121(4)	6249(6)	58(4)
Cl(7)	8153(6)	1180(4)	6739(6)	53(4)
Cl(8)	3263(8)	211(5)	9116(6)	78(6)
Cl(9)	2981(9)	665(4)	7825(8)	99(7)
Cl(10)	2165(7)	-241(4)	8054(6)	59(5)
Cl(11)	1556(11)	259(5)	6022(9)	132(9)
Cl(12)	-42(9)	615(5)	5498(7)	102(7)
Cl(13)	733(7)	977(4)	6765(6)	68(5)
Cl(14)	1005(8)	849(5)	8816(8)	99(7)
Cl(15)	33(9)	1091(6)	9861(7)	109(7)
Cl(16)	-156(9)	1630(5)	8623(8)	118(8)
Cl(17)	6997(8)	2684(4)	3615(6)	78(6)
Cl(18)	7974(7)	3444(4)	4323(6)	70(5)
Cl(19)	8470(7)	2404(4)	4464(6)	59(5)
Cl(20)	3459(5)	2425(3)	2995(5)	34(3)
C(1)	866(12)	2806(5)	3297(10)	12(8)
C(2)	1368	3107	2951	25(9)
C(3)	1248	3621	2931	18(8)
C(4)	627	3833	3256	20(8)
C(5)	126	3532	3602	13(8)
C(6)	245	3019	3623	17(8)
C(7)	107(11)	1810(8)	3282(12)	25(9)
C(8)	-12	1410	3707	43(11)
C(9)	-751	1150	3624	42(11)
C(10)	-1372	1288	3117	46(11)
C(11)	-1254	1687	2693	45(11)
C(12)	-514	1948	2776	43(11)
C(13)	1551(17)	1977(10)	2639(14)	11(8)
C(14)	1398(10)	889(6)	2545(10)	7(7)
C(15)	1343	531	3045	23(9)
C(16)	755	156	2943	32(10)
C(17)	223	138	2341	34(10)
C(18)	278	496	1841	19(8)
C(19)	865	871	1943	35(10)
C(20)	2672(17)	1426(10)	1977(14)	10(8)
C(21)	2756(12)	422(7)	1518(11)	27(9)
C(22)	2660	2	1911	16(8)
C(23)	2103	-367	1670	18(8)
C(24)	1642	-316	1037	52(12)
C(25)	1739	105	644	45(11)
C(26)	2296	474	885	38(10)
C(27)	3987(12)	1141(8)	1198(10)	19(9)
C(28)	4386	768	879	38(10)

(continued)

TABLE 2. (continued)

	x	y	z	U_{eq}^a
C(29)	4945	892	432	56(13)
C(30)	5104	1390	303	33(10)
C(31)	4704	1764	622	39(11)
C(32)	4145	1639	1070	29(10)
C(33)	5890(12)	1628(8)	2730(8)	11(8)
C(34)	6386	1323	2385	21(8)
C(35)	6704	1501	1812	36(10)
C(36)	6526	1983	1584	38(10)
C(37)	6030	2288	1929	52(12)
C(38)	5712	2111	2502	26(9)
C(39)	6212(12)	1031(8)	3979(10)	26(9)
C(40)	6951	1274	4184	40(10)
C(41)	7521	1062	4678	30(10)
C(42)	7354	606	4965	32(10)
C(43)	6615	363	4759	35(10)
C(44)	6044	576	4266	17(8)
C(45)	5027(19)	1851(11)	3878(17)	27(9)
C(46)	4695(11)	1326(7)	5138(8)	6(7)
C(47)	5489	1417	5449	11(8)
C(48)	5791	1157	6033	11(8)
C(49)	5298	807	6307	28(9)
C(50)	4504	717	5996	26(9)
C(51)	4202	976	5412	20(9)
C(52)	3977(21)	2290(12)	4709(18)	39(11)
C(53)	3303(12)	2090(8)	5963(8)	31(10)
C(54)	4094	2060	6307	19(8)
C(55)	4215	1875	6966	7(7)
C(56)	3545	1720	7282	39(11)
C(57)	2755	1750	6938	26(9)
C(58)	2633	1934	6278	24(9)
C(59)	3021(12)	3028(5)	5247(10)	7(7)
C(60)	3510	3262	5776	16(8)
C(61)	3447	3773	5869	35(10)
C(62)	2894	4051	5432	28(9)
C(63)	2405	3817	4903	37(10)
C(64)	2468	3305	4810	25(9)
C(65)	8367(21)	1811(12)	6664(17)	28(9)
C(66)	3082(26)	93(15)	8245(22)	60(13)
C(67)	872(23)	767(14)	5908(19)	44(11)
C(68)	546(22)	1329(15)	9207(19)	45(11)
C(69)	7583(26)	2817(15)	4393(21)	58(13)
C(70)	6004(20)	2922(13)	5630(17)	21(8)
O(1)	6587(17)	3231(10)	5585(14)	64(9)
O(2)	5661(15)	2895(9)	6077(13)	50(8)
O(3)	5902(18)	2661(10)	5103(15)	71(9)

^aEquivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

angle, 168.1(1)°. While these Hg···Hg separations are in the range where contacts between Au(I) centers are believed to be significant [1, 2], the orbital contraction expected for Hg(II) requires that the mercury centers be much closer to experience any direct interaction.

TABLE 3. Selected interatomic distances and angles in $[\text{Hg}_3(\mu\text{-Cl})_2\text{Cl}_2(\mu\text{-dpmp})_2]\text{Cl}(\text{HCO}_3)$

Distances (Å)			
AtHg(1)		AtHg(2)	
Hg(1)–P(1)	2.454(9)	Hg(2)–P(3)	2.450(9)
Hg(1)–P(6)	2.442(9)	Hg(2)–P(4)	2.453(8)
Hg(1)–Cl(1)	2.488(9)	Hg(2)–Cl(2)	2.484(9)
Hg(1)–Cl(3)	2.674(8)	Hg(2)–Cl(4)	2.626(9)
AtHg(3)			
Hg(3)–P(2)	2.461(8)		
Hg(3)–P(5)	2.449(9)		
Hg(3)–Cl(3)	2.667(9)		
Hg(3)–Cl(4)	2.691(9)		
Hg(3)...Hg(1)	3.339(2)		
Hg(3)...Hg(2)	3.331(2)		
Angles (°)			
AtHg(1)		AtHg(2)	
P(1)–Hg(1)–P(6)	139.6(3)	P(3)–Hg(2)–P(4)	128.3(3)
P(1)–Hg(1)–Cl(1)	99.9(3)	P(3)–Hg(2)–Cl(2)	102.6(3)
P(1)–Hg(1)–Cl(3)	105.2(3)	P(3)–Hg(2)–Cl(4)	105.7(3)
P(1)–Hg(1)–Hg(3)	85.6(2)	P(3)–Hg(2)–Hg(3)	86.2(2)
P(6)–Hg(1)–Cl(1)	108.9(3)	P(4)–Hg(2)–Cl(2)	106.0(3)
P(6)–Hg(1)–Cl(3)	99.7(3)	P(4)–Hg(2)–Cl(4)	109.0(3)
P(6)–Hg(1)–Hg(3)	85.8(2)	P(4)–Hg(2)–Hg(3)	86.0(2)
Cl(1)–Hg(1)–Cl(3)	95.6(3)	Cl(2)–Hg(2)–Cl(4)	102.3(3)
AtHg(3)		AtCl(3)	
P(2)–Hg(3)–P(5)	143.9(3)	Hg(1)–Cl(3)–Hg(3)	77.4(2)
P(2)–Hg(3)–Cl(3)	103.0(3)		
P(2)–Hg(3)–Cl(4)	103.7(3)	AtCl(4)	
P(2)–Hg(3)–Hg(2)	87.9(2)	Hg(2)–Cl(4)–Hg(3)	77.6(2)
P(5)–Hg(3)–Cl(3)	103.0(3)		
P(5)–Hg(3)–Cl(4)	100.7(3)		
P(5)–Hg(3)–Hg(1)	88.7(2)		
P(5)–Hg(3)–Hg(2)	87.5(2)		
Cl(3)–Hg(3)–Cl(4)	90.2(3)		
Hg(1)–Hg(3)–Hg(2)	168.1(1)		

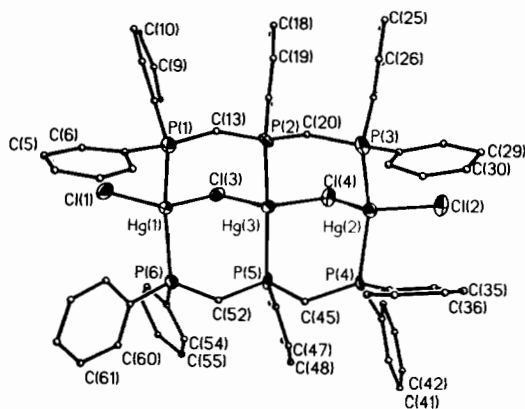


Fig. 1. A perspective view of $[\text{Hg}_3(\mu\text{-Cl})_2\text{Cl}_2(\mu\text{-dpmp})_2]^{2+}$ showing 50% thermal contours for Hg, P and Cl and uniform arbitrarily sized circles for carbon atoms.

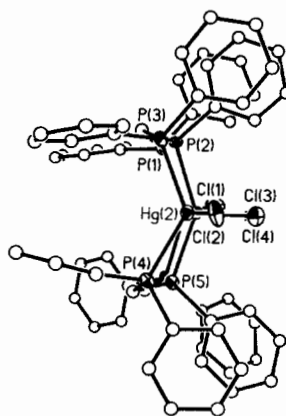


Fig. 2. A view of $[\text{Hg}_3(\mu\text{-Cl})_2\text{Cl}_2(\mu\text{-dpmp})_2]^{2+}$ that emphasizes the orientation of the two dpmp ligands.

Experimental

Preparation of compounds

The ligand dpmp was prepared by the reported route [17].

$[\text{Hg}_3(\mu\text{-Cl})_2\text{Cl}_2(\mu\text{-dpmp})_2]\text{Cl}(\text{PF}_6)$

A solution of 103.0 mg (0.204 mmol) of dpmp in a mixture of 20 ml of dichloromethane and 30 ml of methanol was added dropwise to a solution of 82.5 mg (0.304 mmol) of mercury(II) chloride in 200 ml of methanol. The formation of a transient white precipitate was noted. A solution of 75.5 mg of ammonium hexafluorophosphate in 5 ml of methanol was added. The solution was stirred for 30 min and filtered. The volume of the solution was reduced to 10 ml by rotary evaporation. Chloroform was added to dissolve all of the solid present. After filtration of this solution, its volume was reduced to 20 ml. The solution was allowed to stand at room temperature for 18 h. The white crystalline product was collected by filtration and washed with methanol; yield 153.4 mg (78%). *Anal. Calc.* for $\text{C}_{64}\text{H}_{58}\text{Cl}_5\text{F}_6\text{-Hg}_3\text{P}_7$: C, 39.67; H, 3.02. *Found*: C, 39.79; H, 2.63%. Integration of the $^{31}\text{P}\{\text{H}\}$ NMR spectrum confirmed that only one hexafluorophosphate ion was present in the salt.

$\text{Hg}(\text{dpmp})\text{Br}_2$

A solution of 107.9 mg (0.299 mmol) of mercury bromide in 12 ml of ethanol was added dropwise to a solution of 101.1 mg (0.1996 mmol) of dpmp in 15 ml of dichloromethane. The turbid mixture was allowed to stand for 30 min. The volume of the sample was reduced to 5 ml by evaporation under vacuum. The white solid product was collected by filtration and washed with ethanol and ethyl ether. The product was purified by dissolving the solid in dichloromethane, filtering the solution and adding

TABLE 4. Crystal data and data collection parameters

Empirical formula	C ₇₀ H ₆₃ Cl ₂₀ Hg ₃ O ₃ P ₆
Color; habit	colorless parallelepipeds
Crystal size (mm)	0.08 × 0.13 × 0.33
Crystal system	monoclinic
Space group	<i>P</i> 2 ₁ / <i>n</i>
Unit cell dimensions	
<i>a</i> (Å)	16.424(5)
<i>b</i> (Å)	26.915(9)
<i>c</i> (Å)	19.896(5)
β (°)	96.99(2)
Volume (Å ³)	8730(5)
Z	4
Formula weight	2448.8
Density (calc.) (mg/m ³)	1.86
Absorption coefficient (mm ⁻¹)	6.030
Radiation	Mo Kα (λ = 0.71069 Å)
Temperature (K)	130
Monochromator	highly oriented graphite crystal
2θ Range (°)	0.0–45.0
Scan type	ω
Scan speed	constant, 15.00°/min in ω
Scan range (ω)	1.00°
Standard reflections	2 measured every 198 reflections
Index ranges	–17 ≤ <i>h</i> ≤ 17, 0 ≤ <i>k</i> ≤ 28, 0 ≤ <i>l</i> ≤ 21
Reflections collected	11417
Independent reflections	10060
Observed reflections	5277 (<i>F</i> > 4.0σ(<i>F</i>))
No. parameters refined	434
Final R indices (obs. data)	<i>R</i> = 8.0%, <i>R</i> _w = 6.7%
Goodness-of-fit	1.19

methanol slowly to the filtrate to precipitate the white crystalline sample; yield 73.8 mg (43%). *Anal.* Calc. for C₃₂H₂₉Br₂HgP₃: C, 44.34; H, 3.37. Found: C, 44.90; H, 3.22%.

Hg(dpmp)I₂

This was prepared from HgI₂ by the method described for Hg(dpmp)Br₂. *Anal.* Calc. for C₃₂H₂₉HgI₂P₃: C, 40.00; H, 3.04; P, 9.67. Found: C, 39.84; H, 2.88; P, 9.69%.

X-ray data collection

A solution of 99.3 mg (0.20 mmol) of dpmp in 30 ml of water-saturated chloroform was added to a suspension of 89.0 mg (0.19 mmol) of mercury(I) chloride in 100 ml of wet chloroform. The mixture which contained a gray solid was stirred in air for 24 h. The colorless solution was filtered to separate it from the metallic mercury that had precipitated. After reducing the volume of the filtrate to 60 ml, the white solid, largely HgCl₂(dpmp) by ³¹P NMR analysis, was removed by filtration. The volume of the filtrate was reduced to 15 ml by evaporation under vacuum. The white solid which formed was

collected by filtration and redissolved in chloroform. Suitable crystals of [Hg₃(μ-Cl)₂Cl₂(μ-dpmp)₂]Cl·(HCO₃)·5CHCl₃ were obtained by slow evaporation of this solution in air. The crystals were coated with a light hydrocarbon oil and mounted on a glass fiber in the cold stream of a Syntex P2₁ diffractometer that was equipped with a modified LT-1 apparatus. The space group *P*2₁/*n* was uniquely determined by the observed conditions: *h*0*l*, *h* + *l* = 2*n* and 0*k*0, *k* = 2*n*. No decay in the intensities of two check reflections was observed. Data collection parameters are given in Table 4. The data were corrected for Lorentz and polarization effects.

Solution and refinement of structure

All calculations were performed using the SHELXTL PLUS (VMS) software. Scattering factors and corrections for anomalous dispersion were taken from a standard source [18]. The structure was solved by Patterson and difference Fourier methods. The phenyl rings were treated as rigid groups. One reflection (1,0,1) was omitted due to extinction. An absorption correction was applied [19]. A value of 1.58 Å⁻³ was found for the largest feature in the final difference map.

Supplementary material

Listings of thermal parameters, hydrogen atom positions, bond distances and angles and observed and calculated structure factors are available from the authors.

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