Inorganica Chimica Acta, 108 (1985) 221–226

Synthesis and X-ray Crystal Structure Determination of Two Pseudopolymorphic Forms of μ-[1,2-bis(diphenylphosphino)ethane]bis[chlorogold(I)]: a Digold(I) DNA Binder

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Received May 18, 1985

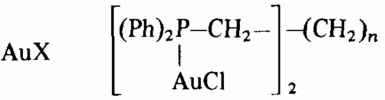
Abstract

An improved synthetic route to the linearly coordinated digold(I) complex, μ-[1,2-bis(diphenylphosphino)ethane]bis[chlorogold(I)], is reported. This complex crystallizes in two pseudopolymorphic forms from a chloroform/methylene chloride solution; the crystal and molecular structures of both are discussed and compared. In both crystal forms the potentially chelating diphenylphosphinoethane (dppe) ligand instead coordinates to two separate gold atoms. The coordination environment of each gold atom is linear in both pseudopolymorphs and the structures display normal gold–chloride and gold–phosphorus bond distances. On the molecular level, the pseudopolymorphs differ fundamentally by a twist about one of the gold phosphorous bonds with the phosphorous atoms of the dppe ligand adopting a transoid orientation relative to one another in both polymorphs. These conformations thus place the intramolecular gold atoms 6 Å apart and preclude intramolecular Au–Au bonding interactions. As has been observed for related gold(I) complexes there are short intermolecular Au–Au contacts of the order of 3.2 Å present in both structures. The conformational flexibility of the gold complex relative to its observed biological activity as a DNA binder is discussed.

Introduction

The phosphine coordinated gold(I) complexes **1** and **3** have been of interest as chrysotherapeutic agents useful in the treatment of rheumatoid arthritis upon oral administration [1].

Et_3PAuX



$$\left[\begin{array}{c}(Ph)_2P{-}CH_2{-}\\ |\\ AuCl\end{array}\right]_2{-}(CH_2)_n$$

1, X = Cl; **4**, $n = 0$;
2, X = Br; **5**, $n = 1$.
3, X = 1-thio-β-D-tetraacetyl glucopyranose.

Compound **3** (auranofin) ('Ridaura', Smith Kline and French Laboratories) has undergone extensive clinical trials [2]. The mechanism whereby these agents exert their therapeutic effect has been an area of intense pharmacological and biochemical investigation [3]. Dabrowiak recently observed, using absorption and circular dichroism spectroscopy, that **1** and the bromide **2**, but not auranofin **3**, interact in a non-denaturing fashion with calf thymus DNA apparently via the guanine and cytosine bases [4]. The only gold compounds previously observed to bind DNA were four-coordinate gold(III) halides [5] which are isoelectronic and, to a degree, isostructural with the known Pt(II) DNA binders [6].

Using an agarose gel electrophoretic assay we have observed that μ-[1,2-bis(diphenylphosphino)ethane]-bis[chlorogold(I)] (**4**) alters the mobility of pBR322 circular DNA after incubation [7]. Circular dichroism spectroscopy and an ethidium bromide fluorescence assay also revealed an interaction of **4** with DNA [8]. Although the reactive site is still to be identified, the interaction of **4** with DNA, like that of **1** and **2**, is likely to be aided by the presence of two easily displaced halides. Further, **1** shows a significant increase in life span in a p388 leukemia mouse model as does the *cis*-ethylene complex analogous to **4**. In contrast, the *trans*-ethylene complex shows little or no activity in this same model [9]. These findings suggest that conformational considerations are also important in understanding the activity of this molecular series. As part of an effort to obtain structural information on gold compounds of biological

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0020-1693/85/$3.30

TABLE I. Crystal and Intensity Measurement Data for μ-[1,2-bis(diphenylphosphino)ethane]bis[chlorogold(I)].

		Polymorph I		Polymorph II	
Molecular formula		$C_{26}H_{24}Au_2Cl_2P_2$		$C_{26}H_{24}Au_2Cl_2P_2(0.4CH_2Cl_2)$	
Molecular weight		863.27		897.24	
Crystal shape		Prism		plate	
Crystal dimensions (mm)		0.10 × 0.15 × 0.10		0.10 × 0.30 × 0.30	
Space group		$P2_{1/n}$		$C_{2/c}$	
Temperature (K)		293		293	
Cell constants	a (Å)	12.793(2)		19.305(11)	
	b (Å)	11.144(2)		10.921(6)	
	c (Å)	19.018(5)		27.202(11)	
	β (°)	109.29(2)		95.43(4)	
Volume (Å^3)		2559.0		5709.7	
Z		4		8	
μ (cm^{-1})		117.81		105.61	
$F(000)$		1608		3216	
Transmission coefficients		max: 99.67%	min: 50.83%	max: 99.90%	min: 25.00%
Agreement factors from data averaging		3.0% on I	4.5% on F	3.8% on I	4.4% on F
Range of data		$2° \leqslant 2\theta \leqslant 55°$	$+h, +k, \pm l$	$2° \leqslant 2\theta \leqslant 55°$	$\pm h, +k, +l$
Total number reflections		6442		7029	
Number of observed data		3060	$I \geqslant 4\sigma(I)$	3545	$I \geqslant 4\sigma(I)$
Number of variables		290		301	
P		0.04		0.05	
R (%)		3.5		4.7	
R_w (%)		4.1		6.4	
Goodness of fit		1.09		1.62	

interest [10] we undertook the X-ray crystal structure determination of **4**. Our interest in **4** was heightened by the structure determination of the propyl bridged compound **5** [11] as well as structural reports of other 1,2-bis(diphenylphosphino)ethane (dppe) complexes of gold(I) [12] and the *cis*-ethylene analog of **4** [13].

Experimental

Synthesis

μ-[1,2-bis(diphenylphosphino)ethane]bis[chlorogold(I)] was prepared as follows: thiodiglycol (11.0 g, 0.09 mol) in methanol (50 ml) was added dropwise over 15 min to a solution of chloroauric acid tetrahydrate (12.4 g, 0.03 mol) in water (100 ml)/methanol (150 ml) kept at 0 °C. After stirring an additional 15 min, 1,2-bis(diphenylphosphino)ethane (6.12 g, 0.015 mol) in chloroform (100 ml)/methanol (100 ml) was added to the colorless solution (immediate ppt upon addition). After warming to room temperature (2 h), methanol (0.5 l) was added and the product collected, slurried with methylene chloride/ethanol, filtered and dried to give 11.0 g (85%) of white product; m.p. 290–292 °C; lit. m.p. 291 °C [14]; 262–267 °C [12]. Crystals suitable for diffraction work were grown by slow evaporation from a chloroform–methylene chloride solution. These crystals displayed two distinct habits which were shown to be pseudopolymorphic forms of the complex with one form containing occluded solvent.

Collection of the X-ray Data

Crystal data for both polymorphs are presented in Table I. Cell constants were obtained from a least-squares analysis of the angular settings for 25 reflections with $32° \leqslant 2\theta(\text{Mo}) \leqslant 36°$. Diffraction data were collected on an Enraf-Nonius CAD4 diffractometer using Mo Kα radiation and a graphite monochromator. Peaks were scanned in an ω-θ mode and the data, along with their estimated standard deviations, were corrected for Lorentz-polarization effects and absorption. The absorption corrections were based on PSI scans of reflections with $80° \leqslant X \leqslant 90°$. Friedel pairs were averaged; the agreement factors are listed along with other intensity data information in Table I. Three intensity standards were monitored every 3 h during data collection. For polymorph **I** these standards showed no significant decay. Standards for polymorph **II** did display a decay; thus, data for this compound were corrected using the program CHORT*.

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*All programs used were from the Enraf-Nonius SDP Program Library with local modifications.

Solution and Refinement

Solution of each structure was effected by the use of a three-dimensional, origin removed Patterson function from which the positions of the gold atoms were obtained. After isotropic least-squares refinement of these positions difference Fourier maps were used to locate the remaining non-hydrogen atoms. Refinement of all non-hydrogen atoms with isotropic temperature factors yielded values of the usual agreement factors $R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$ and $R_2 = [\Sigma w(|F_o| - |F_c|)^2/\Sigma w(F_o)^2]^{1/2}$ of 0.059 and 0.066 for polymorph **I** and 0.084 and 0.096 for polymorph **II**, respectively. All least-squares analyses in this work were carried out on F; the function minimized was $\Sigma w(|F_o| - |F_c|)^2$. The weights, w, were initially given the value of unity, but were eventually assigned as $w = 4F_o^2/\sigma^2(F_o^2)$ where $\sigma(F_o^2)$ is given by $\sigma(F_o^2) = [\sigma^2(I) + p^2I^2]^{1/2}$ and p was assigned the value listed in Table I.

After conversion to anisotropic librational parameters and additional refinement cycles the R values decreased to 0.042 and 0.045 for polymorph **I** and 0.048 and 0.068 for polymorph **II**. For polymorph **I**, hydrogen atom positions were calculated from geometric considerations assuming a C–H bond length of 1.0 Å and were added to the model in fixed positions with fixed isotropic temperature factors of 4.0 Å^2 for the methylene hydrogens and 5.0 Å^2 for the phenyl hydrogens. Further refinement converged after 4 cycles; the largest parameter shift was 0.17 times its estimated standard deviation. An extinction coefficient included in the later stages refined to an insignificant value and thus was not included in the final cycles. One reflection, the $(10\bar{1})$, which showed an asymmetric background when collected, was omitted from the refinement. A final difference Fourier map showed five peaks of heights 1.01 to 1.519 e Å^{-3} all of which were within 1 Å of either of the two gold atoms and therefore presumably reflect an inefficiency in the modeling of the thermal motion of these atoms.

For polymorph **II** some significant electron density remained after the anisotropic refinement in locations distant from that of the parent molecule. This was modeled as a partial occupancy methylene chloride molecule with an occupancy factor of 40%. Hydrogen atom positions were assigned from geometric considerations assuming a C–H bond length of 1.0 Å. The positions and their assigned isotropic thermal parameter of 6.0 Å^2 were held fixed in the final refinement cycles. The largest parameter shift in the final cycle was 0.18 times its estimated standard deviation. One reflection, the (002), which showed an asymmetric background when collected, was omitted. A final difference Fourier map displayed significant residual density (four peaks of heights 1.9 to 1.2e Å^{-3}) in the vicinity of the randomly distributed solvent molecule. Since the solvent molecule sits in a cavity within the structure and is not involved in hydrogen bonding interactions, and in view of the observed decay in data intensity which may indicate loss of solvent by the crystal, the observation of this residual density is to be expected.

Neutral atom scattering factors as well as values of $\Delta f'$ and $\Delta f''$ for anomalous dispersion for all non-hydrogen atoms were taken from the International Tables for X-ray Crystallography [15].

Results and Discussion

The title compound **4** was first synthesized from chloroauric acid using dppe as both reducing agent and ligand [14]. Recently, Sadler prepared **4** from sodium tetrachloroaurate using thiodiglycol as the reducing agent followed by complexation with dppe [12]. Reduction of chloroauric acid with thiodiglycol followed by phosphine complexation had been reported previously to be a good method for the preparation of monophosphine gold(I) complexes [1]. Application of this method, from chloroauric acid directly, also was found suitable for preparing the bidentate digold(I) complex **4** in good yield.

The compound was found to crystallize in two colorless pseudopolymorphic forms. Positional parameters, along with their standard deviations as estimated from the inverse least-squares matrix, are listed in Tables II and III. Additional crystallographic information including tables of anisotropic librational parameters, tables of observed and calculated structure factors and additional bond distances and angles are available upon request.*

The structure of pseudopolymorph **I** is displayed as Fig. 1 and that of pseudopolymorph **II** is displayed as Fig. 2. A comparison of metrical details is afforded by Tables IV and V which list principal bond distances and angles, respectively. In both structures the potentially chelating ligand coordinates instead two AuCl moieties in a fashion quite similar to that observed in the structure of *μ-cis*-1,2-bis(diphenylphosphino)ethylene]bis[chlorogold(I)] [13] and in the propyl derivative [prophos]bis[chlorogold(I)] [11]. The two gold atoms are linearly coordinated in both structures although there are slight deviations from 180° in the P–Au–Cl bond angles. Within experimental error, chemically equivalent bonds are equivalent in length both intrastructurally and interstructurally. The Au–P and Au–Cl bonds are similar in length to those reported in numerous phosphino–chlorogold(I) structures with

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*From the Director of the Cambridge Crystallographic Data Centre, University Chemical Laboratory, Lensfield Road, Cambridge CB2 1EW, U.K. Any request should be accompanied by the full literature citation for this paper.

TABLE II. Positional Parameters for μ-[1,2-bis(diphenylphosphino)ethane]bis[chlorogold(I)] Polymorph **I**.

Atom	*x*	*y*	*z*
Au1	0.44871(3)	0.13283(4)	0.10435(2)
Au2	0.77337(3)	0.01943(5)	−0.07018(2)
Cl1	0.3431(3)	0.2993(3)	0.0577(2)
Cl2	0.7252(3)	−0.0252(3)	−0.1948(2)
P1	0.5655(2)	−0.0206(3)	0.1435(1)
P2	0.8346(2)	0.0623(3)	0.0511(2)
C1	0.6604(8)	−0.032(1)	0.0896(5)
C2	0.7273(8)	0.083(1)	0.0942(5)
C3	0.5018(8)	−0.166(1)	0.1360(6)
C4	0.490(1)	−0.241(1)	0.0766(6)
C5	0.436(1)	−0.351(1)	0.0712(7)
C6	0.389(1)	−0.381(1)	0.1256(8)
C7	0.398(1)	−0.307(1)	0.1837(7)
C8	0.4544(9)	−0.200(1)	0.1900(6)
C9	0.6566(8)	−0.005(1)	0.2395(5)
C10	0.739(1)	−0.092(1)	0.2701(6)
C11	0.812(1)	−0.077(1)	0.3400(7)
C12	0.802(1)	0.024(1)	0.3805(7)
C13	0.722(1)	0.108(1)	0.3513(7)
C14	0.648(1)	0.094(1)	0.2807(6)
C15	0.9243(8)	−0.053(1)	0.1076(6)
C16	0.9608(9)	−0.048(1)	0.1861(6)
C17	1.031(1)	−0.134(1)	0.2254(8)
C18	1.067(1)	−0.222(1)	0.1909(8)
C19	1.030(1)	−0.231(1)	0.1133(8)
C20	0.958(1)	−0.144(1)	0.0726(7)
C21	0.9130(8)	0.202(1)	0.0726(5)
C22	1.0251(9)	0.201(1)	0.1125(6)
C23	1.084(1)	0.309(1)	0.1252(7)
C24	1.029(1)	0.416(1)	0.0996(8)
C25	0.918(1)	0.418(1)	0.0592(8)
C26	0.860(1)	0.309(1)	0.0447(8)

TABLE III. Positional Parameters for μ-[1,2-bis(diphenylphosphino)ethane]bis[chlorogold(I)] Polymorph **II**.

Atom	*x*	*y*	*z*
Au1	0.02310(3)	0.06636(6)	−0.42630(2)
Au2	0.6480(3)	0.18571(5)	−0.20810(2)
Cl1	−0.0902(2)	0.0389(5)	−0.4563(2)
Cl2	0.0096(2)	0.3443(4)	−0.1735(2)
Cl3	0.008(1)	−0.551(2)	−0.0497(8)
Cl4	0.033(1)	0.382(2)	−0.0112(9)
P1	0.1323(2)	0.0998(3)	−0.3955(2)
P2	0.1224(2)	0.0267(3)	−0.2362(2)
C1	0.1389(7)	0.122(1)	−0.3279(6)
C2	0.1118(8)	0.010(1)	−0.3030(6)
C3	0.1937(7)	−0.024(1)	−0.4052(6)
C4	0.2649(9)	−0.013(2)	−0.3884(7)
C5	0.3116(9)	−0.106(2)	−0.3962(8)
C6	0.288(1)	−0.210(2)	−0.4219(8)
C7	0.222(1)	−0.219(2)	−0.4391(8)
C8	0.172(1)	0.129(1)	−0.4304(6)
C9	0.1699(7)	0.234(1)	−0.4228(6)
C10	0.2177(9)	0.310(1)	−0.3975(6)
C11	0.247(1)	0.408(2)	−0.4213(7)
C12	0.2259(9)	0.426(1)	−0.4711(8)
C13	0.180(1)	0.351(2)	−0.4972(7)
C14	0.1525(8)	0.253(1)	−0.4731(6)
C15	0.2157(7)	0.032(1)	−0.2200(6)
C16	0.2431(8)	0.119(1)	−0.1863(6)
C17	0.3125(9)	0.118(2)	−0.1722(7)
C18	0.3565(8)	0.032(2)	−0.1910(8)
C19	0.3303(8)	−0.050(2)	−0.2229(7)
C20	0.2593(9)	−0.054(1)	−0.2390(7)
C21	0.0968(8)	−0.112(1)	−0.2092(5)
C22	0.083(1)	−0.110(1)	−0.1609(6)
C23	0.067(1)	−0.217(2)	−0.1348(7)
C24	0.065(1)	−0.327(2)	−0.1577(9)
C25	0.083(2)	−0.329(2)	−0.2040(8)
C26	0.099(1)	−0.210(2)	−0.2295(9)
C27	−0.025(2)	−0.405(4)	−0.046(2)

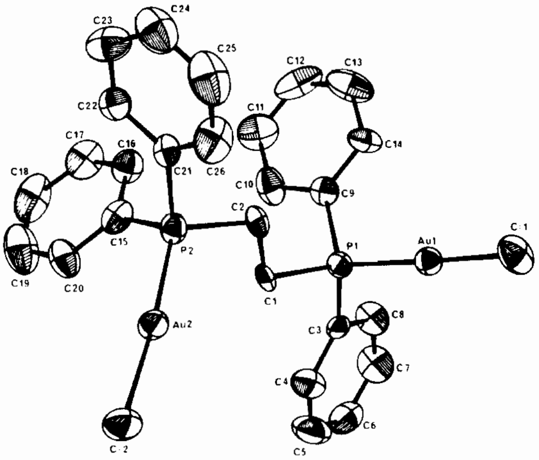

Fig. 1. View of the μ-[1,2-bis(diphenylphosphino)ethane]bis[chlorogold(I)] molecule: polymorph I. Thermal ellipsoids are drawn at the 50% probability level.

the Au–P bonds distinctly shorter than the Au–Cl bonds, as has been noted by others [13, 16].

There are no close intramolecular contacts between gold atoms in either of the dppe(AuCl)$_2$ structures since, as may be seen from the figures, the two halves of the molecule point away from one another. This conformation contrasts sharply to the solid state conformation adopted by the *cis*-ethylenic analogue [13], but is quite similar to the conformation reported for the prophos(AuCl)$_2$ molecule [11]. The two dppe pseudopolymorphs adopt slightly different conformations in the solid state as reflected in the torsion angle comparison presented in Table VI. The largest difference occurs in the torsion angle about the Au1–P1 bond with

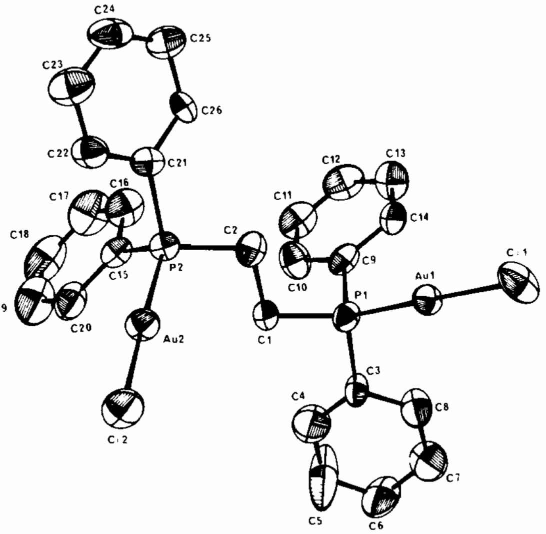

Fig. 2. View of the μ-[1,2-bis(diphenylphosphino)ethane]-bis[chlorogold(I)] molecule: polymorph **II**. Thermal ellipsoids are shown at the 50% probability level.

TABLE IV. Principal Internuclear Separations (Å) in μ-[1,2-bis(diphenylphosphino)ethane]bis[chlorogold(I)].

Atoms	Distances	
	Polymorph **I**	Polymorph **II**
Au1–Cl1	2.294(3)	2.281(3)
Au2–Cl2	2.298(3)	2.283(3)
Au1–P1	2.231(3)	2.224(3)
Au2–P2	2.229(3)	2.235(3)
P1–C1	1.84(1)	1.85(1)
P1–C3	1.80(1)	1.82(1)
P1–C9	1.82(1)	1.77(1)
P2–C2	1.83(1)	1.82(1)
P2–C15	1.82(1)	1.83(1)
P2–C21	1.82(1)	1.83(1)
C1–C2	1.53(2)	1.52(2)

TABLE V. Principal Internuclear Angles (°) in μ-[1,2-bis-(diphenylphosphino)ethane]bis[chlorogold(I)].

Atoms	Angles	
	Polymorph **I**	Polymorph **II**
Cl1–Au1–P1	173.2(1)	175.6(2)
Cl2–Au2–P2	175.3(1)	177.7(1)
Au1–P1–C1	111.2(4)	114.0(4)
Au1–P1–C3	115.4(3)	113.9(4)
Au1–P1–C9	113.6(4)	110.8(4)
Au2–P2–C2	115.6(4)	111.8(4)
Au2–P2–C15	113.5(4)	115.3(4)
Au2–P2–C21	113.3(3)	112.2(4)
C1–P1–C3	105.0(5)	105.1(6)
C1–P1–C9	104.2(5)	108.7(6)
C3–P1–C9	106.7(5)	103.8(6)
C2–P2–C15	104.1(5)	103.8(6)
C2–P2–C21	103.3(5)	104.9(6)
C15–P2–C21	106.0(5)	107.8(6)
P1–C1–C2	111.7(7)	110.9(8)
P2–C2–C1	110.9(7)	110.1(9)

TABLE VI. Principal Torsion Angles (°) for μ-[1,2-bis-diphenylphosphino)ethane]bis[chlorogold(I)].

Atoms	Polymorph **I**	Polymorph **II**
Cl1–Au1–P1–C1	−18.6	50.7
Au1–P1–C1–C2	58.1	59.2
P1–C1–C2–P2	172.1	176.8
C1–C2–P2–Au2	62.3	60.9
C2–P2–Au2–Cl2	179.6	177.2

atoms Cl1 and C1 nearly eclipsed in pseudopolymorph **I** (torsion angle = −18.6°) but in a gauche relationship in pseudopolymorph **II** (torsion angle = 50.7°). Thus, the two molecules have a slightly different twist which places the P–Au–Cl moieties in different orientations relative to one another in each structure. The dihedral angle between the planes defined by atoms Cl1, Au1, P1 and C1 and atoms Cl2, Au2, P2 and C2 is 116.3° in pseudopolymorph **I** but only 60.0° in pseudopolymorph **II**. The resulting intramolecular Au–Au separations are then 6.243(1) Å in pseudopolymorph **I** and 6.056(1) Å in pseudopolymorph **II**. In both pseudopolymorphs the large Au–Au internuclear separations preclude any intramolecular bonding interactions between the metal centers. This situation contrasts to the short intramolecular Au–Au contact observed in the *cis*-ethylenic structure where restricted rotation of the ethylenic bridge imposes conformational rigidity on the complex. The isolation of two conformationally distinct pseudopolymorphs of this gold dppe complex suggests, however, that freedom of rotation about the ethane bridge will allow adoption of a wide manifold of conformations by this complex, including a conformation comparable to that observed for the *cis*-ethylenic complex. Further support for this conclusion is derived from the observations [9] that **4** and the *cis*-ethylene complex show comparable significant *in vivo* activity against mouse p388 leukemia cells (in contrast to the insignificant activity of the *trans*-ethylene complex).

Short Au–Au intermolecular and intramolecular contacts (2.7 to 3.4 Å) are commonly observed in Au(I) compounds [13, 16]. While the structures described here show no short Au–Au intramolecular contacts there are notable short intermolecular Au–Au contacts. Thus, in pseudopolymorph **I**, Au1 and Au2 of inversion related molecules are within 3.187(1) Å of each other and in pseudopolymorph **II** the closest approach is 3.221(1) Å between two

Au2 atoms related by the c-glide. These intermolecular contacts are quite similar in distance to values reported for the propyl bridged analog **5** [11]. Thus, as noted by those authors, a polymeric network of gold molecules is presented by these crystal structures. Considering: (1) the absence of intramolecular Au–Au effects and (2) the lack of significant rigidifying influence by the dppe ligand on the conformation(s) of these pseudopolymorphs, the short intermolecular contacts may represent the dominating influence in dictating both the crystal packing and the observed molecular conformations.

References

1 B. M. Sutton, E. McGusty, D. T. Walz and M. J. DiMartino, *J. Med. Chem., 15*, 1095 (1972).
2 R. C. Blodgett, M. A. Heuer and R. G. Pietrusko, *Semin. Arthritis Rheum., 13*, 255 (1984); M. Chaffman, R. N. Brogden, R. C. Heel, T. M. Speight and G. S. Avery, *Drugs, 27*, 378 (1984).
3 P. J. Sadler, *Struc. Bonding (Berlin), 29*, 171 (1976); C. F. Shaw III, *Inorg. Perspect. Biol. Med., 2*, 298 (1979); D. H. Brown and W. E. Smith, *Chem. Soc. Rev., 9*, 217 (1980); A. J. Lewis and D. T. Walz, *Prog. Med. Chem., 19*, 1 (1982).
4 C. E. Blank and J. C. Dabrowiak, *J. Inorg. Biochem., 21*, 21 (1984).
5 U. S. Nandi, *Biopolymers, 16*, 1863 (1977); C. K. Mirabelli, C. M. Sung, H. E. Bartus and S. T. Crooke, *Proc. Am. Assoc. Cancer Res., 2*, Abstr. 1157 (1983); C. K. S. Pillai and U. S. Nandi, *Biopolymers, 17*, 709 (1978).
6 P. J. Sadler, M. Nasar and V. L. Narayanan, in M. P. Hacker, E. B. Douple and I. H. Krakoff (eds.), 'Platinum Coordination Complexes in Cancer Chemotherapy', Martinus Nijhoff, Boston, 1984, p. 290.
7 C. K. Mirabelli and D. T. Hill (S.K. & F. Laboratories), to be published.
8 C. K. Mirabelli (S.K. & F. Laboratories), personal communication.
9 R. K. Johnson, C. K. Mirabelli, L. F. Faucette, F. L. McCabe, B. M. Sutton, D. L. Bryan, G. R. Girard and D. T. Hill, 'Abstr., American Association for Cancer Research', Am. Assoc. Cancer Res., Houston, Tex., May 1985.
10 D. S. Eggleston, D. F. Chodosh, D. T. Hill and G. R. Girard, *Acta Crystallogr., Sect. C:, 40*, 1357 (1984); D. T. Hill and B. M. Sutton, *Cryst. Struct. Commun., 9*, 681 (1980).
11 M. K. Cooper, L. E. Mitchell, K. Kenrick, M. McPartlin and A. Scott, *Inorg. Chim. Acta, 84*, L9 (1984).
12 S. J. Berners-Price, M. A. Mazid and P. J. Sadler, *J. Chem. Soc., Dalton Trans.*, 969 (1984); P. A. Bates and J. M. Waters, *Inorg. Chim. Acta, 81*, 151 (1984).
13 P. G. Jones, *Acta Crystallogr., Sect. B:, 36*, 2775 (1980).
14 F. Cariati, L. Naldini, G. Simonetta and L. Malatesta, *Inorg. Chim. Acta, 1*, 315 (1967).
15 'International Tables for X-ray Crystallography, Vol. IV', Kynoch Press, Birmingham, 1974, p. 72.
16 P. G. Jones, *Gold Bull., 14*, 102 (1981).
17 P. A. Bates and J. M. Waters, *Inorg. Chim. Acta, 98*, 125 (1985).

Note Added in Proof

The crystal structure of polymorph I of μ-[1,2-bis(diphenylphosphino)ethane]bis[chlorogold(I)] was recently reported [17].