

μ -cis-1,2-Bis(diphenylphosphino)ethylene-bis[chlorogold(I)]

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Abstract. $C_{26}H_{22}Au_2Cl_2P_2$, $M_r = 861.25$, monoclinic, *Cc*. The cell constants change appreciably on irradiation. Those used for calculations were from an irradiated crystal: $a = 13.293$ (6), $b = 12.863$ (6), $c = 15.497$ (7) Å, $\beta = 101.30$ (4)°, $U = 2598$ Å³, $Z = 4$, $\mu(\text{Mo } K\alpha) = 11.6$ mm⁻¹. $R = 0.044$ for 4118 unique observed reflexions. The ligand coordinates two AuCl moieties, with a short Au...Au distance of 3.05 (1) Å.

Introduction. The title compound was obtained as one of several products from the reaction of the ligand with carbonylchlorogold(I) in benzene. Colourless blade-shaped crystals (elongated along [101]) were obtained from dichloromethane/diisopropyl ether. A crystal 0.4 × 0.15 × 0.15 mm was used to collect profile-fitted data (Clegg, 1981) on a Stoe four-circle diffractometer, with monochromated Mo $K\alpha$ radiation. 4761 reflexions, including a full set of Friedel pairs, were collected in the range $7^\circ < 2\theta < 50^\circ$. After L_p and empirical absorption corrections, averaging equivalent reflexions gave 4480 unique reflexions, 4119 with $F > 4\sigma(F)$.

During data collection it was noted that many reflexions were badly off-centre. It was thus suspected that the cell constants might be altering as a result of irradiation. This was checked by measuring cell constants of four crystals. Two which had been previously irradiated gave a , b , c (Å), β (°) = 13.293 (6), 12.863 (6), 15.497 (7), 101.30 (4); 13.299 (10), 12.868 (9), 15.494 (10), 101.27 (5). Two previously unirradiated gave 13.244 (10), 12.854 (9), 15.405 (10), 101.09 (5); 13.253 (6), 12.854 (6), 15.412 (7), 101.15 (4). No further crystals of suitable quality were available. It thus seems that radiation damage, the exact nature of which is uncertain, causes the cell constants to increase. The cell constants used in the calculations are those of the crystal used to collect data, after irradiation.

The structure was solved by the heavy-atom method in space group *Cc*; the other space group consistent with the systematic absences is *C2/c*, but intensity statistics and Harker peaks (and eventual successful refinement) indicated the noncentrosymmetric space group. In the final stages of refinement Au, P and Cl

atoms were anisotropic, C and H isotropic with $U(\text{Hn}) = 1.2U(\text{Cn})$; rigid phenyl groups with C–C 1.395, C–H 0.96 Å and all angles 120° were employed. The weighting scheme was $w^{-1} = \sigma^2(F) + 0.0007F^2$, giving a final $R_w = 0.045$, $R = 0.044$. [The alternative structure, with all coordinates changed in sign, was rejected ($R = 6.2\%$, $R_w = 6.6\%$).] Final atomic coordinates are given in Table 1, bond lengths and

Table 1. Atom coordinates ($\times 10^4$) and isotropic temperature factors (Å² $\times 10^3$)

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i>
Au(1)	5000	9042 (1)	5000	50 (1)*
Au(2)	5107 (1)	9778 (1)	6886 (1)	50 (1)*
P(1)	3351 (3)	8600 (3)	4652 (2)	51 (1)*
Cl(1)	6715 (3)	9437 (4)	5175 (3)	71 (1)*
P(2)	4372 (2)	8219 (3)	6940 (2)	46 (1)*
Cl(2)	5812 (4)	11407 (3)	6988 (3)	77 (1)*
C(12)	1543 (9)	9371 (7)	3615 (8)	87 (5)
C(13)	852 (9)	10166 (7)	3306 (8)	110 (7)
C(14)	1096 (9)	11194 (7)	3547 (8)	100 (6)
C(15)	2030 (9)	11427 (7)	4098 (8)	104 (6)
C(16)	2721 (9)	10632 (7)	4407 (8)	84 (5)
C(11)	2477 (9)	9604 (7)	4166 (8)	51 (3)
C(22)	2673 (7)	6607 (7)	3984 (5)	62 (4)
C(23)	2575 (7)	5831 (7)	3344 (5)	65 (4)
C(24)	2987 (7)	5978 (7)	2591 (5)	70 (4)
C(25)	3496 (7)	6901 (7)	2479 (5)	73 (4)
C(26)	3594 (7)	7677 (7)	3118 (5)	62 (4)
C(21)	3182 (7)	7530 (7)	3871 (5)	54 (3)
C(1)	2679 (10)	8223 (10)	5528 (8)	51 (3)
C(2)	3017 (10)	8138 (10)	6392 (8)	47 (3)
C(32)	5008 (6)	8393 (5)	8740 (5)	45 (3)
C(33)	5105 (6)	8080 (5)	9615 (5)	51 (3)
C(34)	4571 (6)	7211 (5)	9821 (5)	59 (3)
C(35)	3941 (6)	6653 (5)	9154 (5)	68 (4)
C(36)	3844 (6)	6966 (5)	8279 (5)	61 (4)
C(31)	4377 (6)	7836 (5)	8073 (5)	49 (3)
C(42)	4370 (6)	6269 (8)	6158 (7)	65 (4)
C(43)	4848 (6)	5405 (8)	5872 (7)	82 (5)
C(44)	5908 (6)	5401 (8)	5928 (7)	120 (8)
C(45)	6491 (6)	6262 (8)	6270 (7)	94 (6)
C(46)	6013 (6)	7126 (8)	6556 (7)	70 (4)
C(41)	4953 (6)	7130 (8)	6500 (7)	53 (3)

* Equivalent isotropic *U* calculated from anisotropic *U*.

Table 2. Bond lengths (Å)

Au(1)–P(1)	2.226 (4)	Au(1)–Cl(1)	2.299 (5)
Au(2)–P(2)	2.239 (5)	Au(2)–Cl(2)	2.289 (5)
C(11)–P(1)	1.800 (12)	C(21)–P(1)	1.818 (10)
C(1)–P(1)	1.831 (16)	C(1)–C(2)	1.331 (18)
C(2)–P(2)	1.838 (13)	C(31)–P(2)	1.821 (9)
C(41)–P(2)	1.797 (12)		

Table 3. Bond angles (°)

P(1)–Au(1)–Cl(1)	172.5 (2)	P(2)–Au(2)–Cl(2)	173.3 (2)
Au(1)–P(1)–C(11)	116.3 (5)	Au(1)–P(1)–C(21)	110.0 (5)
C(11)–P(1)–C(21)	106.2 (6)	Au(1)–P(1)–C(1)	119.3 (5)
C(11)–P(1)–C(1)	97.6 (7)	C(21)–P(1)–C(1)	106.2 (6)
Au(2)–P(2)–C(2)	115.3 (5)	Au(2)–P(2)–C(31)	111.0 (4)
C(2)–P(2)–C(31)	104.4 (6)	Au(2)–P(2)–C(41)	117.4 (5)
C(2)–P(2)–C(41)	103.7 (6)	C(31)–P(2)–C(41)	103.6 (6)
P(1)–C(11)–C(12)	121.7 (4)	P(1)–C(11)–C(16)	118.1 (4)
P(1)–C(21)–C(22)	124.1 (4)	P(1)–C(21)–C(26)	115.9 (4)
P(1)–C(1)–C(2)	130.5 (12)	P(2)–C(2)–C(1)	124.6 (12)
P(2)–C(31)–C(32)	117.7 (3)	P(2)–C(31)–C(36)	122.1 (3)
P(2)–C(41)–C(42)	121.0 (4)	P(2)–C(41)–C(46)	118.9 (4)

angles in Tables 2 and 3.* The quoted e.s.d.'s take no account of errors in cell parameters, the uncertainties of which in this case would suggest e.s.d.'s considerably higher (about 0.01 Å for bonds involving Au).

Discussion. The potentially chelating ligand is found to coordinate two AuCl moieties (Fig. 1), reflecting the preference of Au^I for linear coordination. The Au–P and Au–Cl lengths fall in the expected range; cf. 2.238 (5), 2.288 (7) Å respectively in Ph₂PCH₂PPh₂·(AuCl)₂ (Schmidbaur, Wohlleben, Wagner, Orama & Huttner, 1977). The angles at Au deviate somewhat from linearity (172.5, 173.3°).

The Au atoms approach each other quite closely [Au···Au = 3.05 (1) Å]. This is a common structural feature of Au^I compounds (e.g. Jones, Sheldrick & Hädicke, 1980), although the factors governing the occurrence of such contacts are not well understood. In the methylene complex (Schmidbaur *et al.*, 1977) the Au···Au distance is considerably longer (3.341 Å),

* Lists of structure factors, anisotropic thermal parameters and H atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35444 (28 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

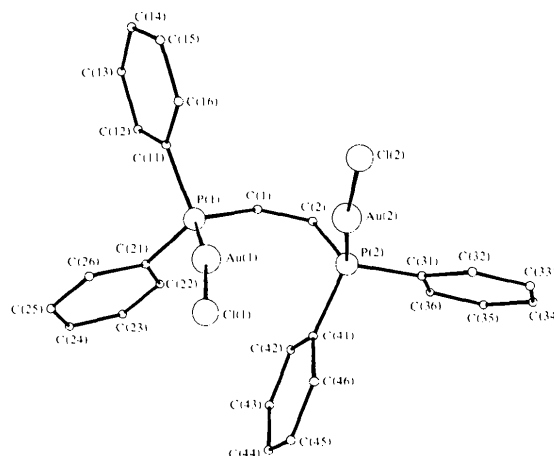


Fig. 1. The title compound, showing the atom numbering (H atoms omitted).

although a small rotation about the P–CH₂ bond would have brought the Au atoms closer. Presumably there is a delicate balance between crystal packing forces and weak Au···Au attractions.

The lack of twofold symmetry in the present compound is illustrated by the differing dispositions of the Au atoms with respect to the PCCP plane. Au(1) lies in this plane [r.m.s. deviation of these five atoms from the mean plane = 0.06 Å; torsion angles Au(1)–P(1)–C(1)–C(2) –1 (2)°, P(1)–C(1)–C(2)–P(2) –9 (2)°] but Au(2) 1.58 Å out of it [Au(2)–P(2)–C(2)–C(1) 67 (2)°]. The Au(1)···P(2) distance is 3.44 (1) Å, too long for any appreciable bonding interaction. Identical Au···P distances occur in the methylene analogue (Schmidbaur *et al.*, 1977).

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