

## Chloro(triethylphosphine)gold(I)

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**Abstract.** [AuCl(C<sub>6</sub>H<sub>15</sub>P)],  $M_r = 350.6$ , monoclinic,  $P2_1$ ,  $a = 7.106$  (1),  $b = 19.016$  (2),  $c = 7.735$  (1) Å,  $\beta = 106.31$  (1)°,  $U = 1003$  (1) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 2.322$  Mg m<sup>-3</sup>, Mo  $K\alpha$  radiation,  $\lambda = 0.7107$  Å,  $\mu = 14.952$  mm<sup>-1</sup>,  $F(000) = 648$ ,  $T = 295$  (2) K,  $R = 0.048$  for 1165 observed reflections. There are two molecules in the asymmetric unit arising from minor differences in ethyl-group orientations. For each molecule, the Au atom is in the expected linear geometry with Au–Cl 2.305 (8) [2.306 (8) Å for molecule 2], Au–P 2.232 (9) [2.231 (8) Å] and Cl–Au–P 178.5 (3) [178.9 (3)°]. The closest Au...Au contact in the crystal lattice is 3.615 (2) Å.

**Experimental.** The compound was prepared by G. A. Koutsantonis by the established method (Davies, 1987) and crystals obtained by the slow evaporation of an ethanol solution held at 277 K. Enraf–Nonius CAD-4F diffractometer controlled by a PDP8/A computer, graphite-monochromated Mo  $K\alpha$  radiation;  $\omega:2\theta$  scan technique. Cell parameters on crystal with faces (and distances from centroid)  $(\bar{1}00)$  0.125;  $\pm(010)$  0.063;  $(031)$  0.075;  $(0\bar{3}1)$  0.075; and  $(130)$  0.125 mm by least squares on 25 reflections ( $7 \leq \theta \leq 12^\circ$ ) (de Boer & Duisenberg, 1984). Analytical absorption correction applied; max. and min. transmission factors 0.820 and 0.073 (Sheldrick, 1976). Total of 2765 reflections ( $1.2 \leq \theta \leq 25.0^\circ$ ) measured in the range  $-8 \leq h \leq 8$ ,  $0 \leq k \leq 22$ ,  $-9 \leq l \leq 3$ ; some Friedel pairs also included. No significant variation in the net intensities of three reference reflections ( $\bar{2}22$ ,  $\bar{2}61$ ,  $\bar{2}13$ ) measured every 7200 s. 1883 unique reflections ( $R_{int}$  0.070) and 1165 satisfied  $I \geq 2.5\sigma(I)$ . Structure solved by Patterson method, full-matrix least-squares refinement of 104 parameters based on  $F$  (Sheldrick, 1976). Anisotropic thermal parameters for Au, Cl and P; other non-H atoms refined isotropically. Owing to high thermal motion two ethyl groups were refined with constrained bond distances. No evidence found for multiple sites for ethyl groups (Tiekink, Hoskins & Kurucsev, 1989). H atoms included at their calculated positions. Satisfactory refinement was not achieved in the  $P2_1/m$  space group; further support for  $P2_1$  was found in the distribution of  $E$  statistics, *i.e.*  $\langle |E^2 - 1| \rangle = 0.74$  (Stout & Jensen, 1968; Snow & Tiekink, 1988). The

Table 1. Fractional atomic coordinates and  $B_{eq}$  values (Å<sup>2</sup>)
$$B_{eq} = 8\pi^2(U_{11} + U_{22} + U_{33})/3.$$

	<i>x</i>	<i>y</i>	<i>z</i>	$B_{eq}$
Au(1)	0.4529 (2)	$\frac{1}{2}$	0.4111 (2)	4.91
Au(2)	-0.0539 (2)	0.2744 (1)	0.3778 (1)	5.12
Cl(1)	0.4732 (13)	0.3698 (4)	0.4587 (12)	5.93
Cl(2)	0.0170 (11)	0.2008 (5)	0.6247 (10)	5.70
P(1)	0.4409 (14)	0.1337 (5)	0.3706 (13)	5.90
P(2)	-0.1224 (16)	0.3473 (5)	0.1424 (11)	5.79
C(11)	0.1984 (66)	0.0972 (28)	0.2653 (58)	9.51
C(12)	0.1312 (64)	0.1271 (28)	0.0810 (50)	9.11
C(13)	0.6188 (58)	0.1022 (21)	0.2675 (50)	7.10
C(14)	0.6079 (56)	0.0250 (24)	0.2291 (50)	8.04
C(15)	0.4338 (104)	0.0728 (33)	0.5463 (67)	18.43
C(16)	0.5745 (107)	0.1038 (52)	0.7098 (91)	21.32
C(21)	-0.0877 (112)	0.4402 (9)	0.1820 (57)	14.70
C(22)	-0.0305 (65)	0.4673 (29)	0.3715 (50)	9.81
C(23)	0.0444 (60)	0.3364 (23)	-0.0019 (50)	7.68
C(24)	0.0014 (60)	0.3880 (25)	-0.1597 (52)	8.34
C(25)	-0.3622 (72)	0.3431 (28)	0.0066 (63)	9.64
C(26)	-0.4468 (57)	0.2762 (23)	-0.0631 (43)	7.43

Table 2. Interatomic distances (Å) and bond angles (°)

Au(1)–Cl(1)	2.305 (8)	Au(2)–Cl(2)	2.306 (8)
Au(1)–P(1)	2.232 (9)	Au(2)–P(2)	2.231 (8)
P(1)–C(11)	1.82 (5)	P(2)–C(21)	1.80 (1)*
P(1)–P(13)	1.78 (4)	P(2)–C(23)	1.85 (4)
P(1)–P(15)	1.80 (1)*	P(2)–C(25)	1.74 (5)
Cl(1)–Au(1)–P(1)	178.5 (3)	Cl(2)–Au(2)–P(2)	178.9 (3)
Au(1)–P(1)–C(11)	116 (2)	Au(2)–P(2)–C(21)	119 (2)
Au(1)–P(1)–C(13)	113 (1)	Au(2)–P(2)–C(23)	113 (1)
Au(1)–P(1)–C(15)	123 (3)	Au(2)–P(2)–C(25)	115 (2)
C(11)–P(1)–C(13)	112 (2)	C(21)–P(2)–C(23)	98 (3)
C(11)–P(1)–C(15)	83 (3)	C(21)–P(2)–C(25)	102 (3)
C(13)–P(1)–C(15)	107 (3)	C(23)–P(2)–C(25)	108 (2)

\* Bonds constrained to 1.80 (1) Å.

absolute structure of the crystal was determined from differences in Friedel pairs included in the data set. At convergence  $R = 0.048$ ,  $wR = 0.048$ ,  $w = 2.45/[\sigma^2(F) + 0.0014F^2]$ ,  $S = 2.10$ ,  $(\Delta/\sigma)_{max} \leq 0.04$ ,  $(\Delta\rho)_{max} = 1.77$ ,  $(\Delta\rho)_{min} = -1.80$  e Å<sup>-3</sup>; no extinction correction. Scattering factors for H, C, P and Cl given in *SHELX76* (Sheldrick, 1976) and those for neutral Au corrected for  $f'$  and  $f''$  (Hamilton, & Ibers, 1974). All calculations on a SUN4/280 computer system. Atomic parameters given in Table 1, selected param-

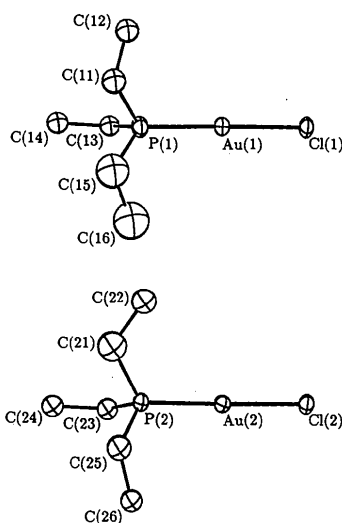


Fig. 1. Molecular structure and numbering scheme for the two molecules in the asymmetric unit of  $\text{Et}_3\text{PAuCl}$  (ORTEPII, Johnson, 1971).

eters in Table 2\* and the numbering scheme used is shown in Fig. 1, which was drawn with ORTEPII (Johnson, 1971) at 15% probability ellipsoids.

**Related literature.** Metal phosphines, in particular gold phosphine complexes, have useful medicinal properties

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, and all bond distances and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51779 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

(Berners-Price & Sadler, 1987; Parish & Cottrill, 1987).  $\text{Et}_3\text{PAuCl}$ , which is known to interact with DNA (Mirabelli, Sung, Zimmerman, Hill, Mong & Crooke, 1986), has a similar coordination geometry to that reported for the triphenylphosphine analogue  $\text{Ph}_3\text{PAuCl}$  (Baenziger, Bennett & Soboroff, 1976).

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## 2,6-Bis(diphenylphosphorylmethyl)pyridine Ethanol Solvate 2·5-Hydrate

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**Abstract.**  $\text{C}_{31}\text{H}_{27}\text{NO}_2\text{P}_2 \cdot \text{C}_2\text{H}_6\text{O} \cdot 2.5\text{H}_2\text{O}$ ,  $M_r = 598.62$ , monoclinic,  $P2_1/c$ ,  $a = 9.392(3)$ ,  $b = 18.903(6)$ ,  $c = 17.736(6)$  Å,  $\beta = 96.81(3)^\circ$ ,  $V = 3127(2)$  Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.272$  g cm<sup>-3</sup>,  $\lambda(\text{Mo K}\alpha) = 0.71069$  Å,  $\mu$

$= 1.75$  cm<sup>-1</sup>,  $F(000) = 1268$ ,  $T = 299$  K, final  $R = 0.073$ ,  $wR = 0.077$  for 1569 reflections. The title compound results from the reaction of the corresponding phosphine with  $\text{Au}_2\text{Cl}_6$ . The bond lengths are normal.

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**Experimental.** The ligand 2,6-bis(diphenylphosphino-methyl)pyridine, PCpyCP, was prepared by a modification of the synthesis reported by Dahlhoff & Nelson