

(2) The hydrogen malate moiety does not show conformational stability under various packing conditions. In the lithium salt we find a synclinal conformation for the  $^-OOC-C-C-COOH$  chain, in contrast to the expected antiperiplanar form which is found in the other salts.

(3) Kanters *et al.* had claimed that in the  $COO^-$  group of  $\alpha$ -hydroxy acids the C—O bonded to the cation is shorter than the other C—O bonds. Our data show that a firm, statistically significant, statement about the (in)equivalence of  $O_1$  and  $O_2$  is as yet premature.

(4) In the hydrogen malates the bond C(1)—C(2) next to the  $COO^-$  seems elongated with respect to C(3)—C(4) next to the COOH group.

One could speculate that the unusual conformation is coupled to the hydrogen bridge O(3)—H $\cdots$ O(4). Furthermore, the torsion angle O(4)—C(4)—C(3)—C(2) varies between  $-9$  and  $-69^\circ$ , the variations probably being dictated by the way COOH is stacked in the lattice. The only conformational feature common to all compounds is that O(5) is more or less in the plane of the  $COO^-$  group. But, notwithstanding this, the spread

in the values of torsion angle O(1)—C(1)—C(2)—O(5) and consequently also in those of O(1)—C(1)—C(2)—C(3) is rather large.

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## $\mu$ -Chloro-bis(triphenylphosphine)digold(I) Perchlorate Dichloromethane Solvate

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**Abstract.**  $C_{36}H_{30}Au_2ClP_2^+ \cdot ClO_4^- \cdot CH_2Cl_2$ ,  $[Au_2(C_{18}H_{15}P)_2Cl]ClO_4 \cdot CH_2Cl_2$ ,  $M_r = 1138.36$ , triclinic,  $P1$ ,  $a = 11.214$  (3),  $b = 14.675$  (4),  $c = 25.505$  (6) Å,  $\alpha = 85.62$  (2),  $\beta = 85.16$  (2),  $\gamma = 68.25$  (2)°,  $U = 3880$  Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.949$  Mg m<sup>-3</sup>,  $\mu(Mo K\alpha) = 7.9$  mm<sup>-1</sup>; final  $R = 0.072$  for 8642 reflexions. There are two formula units in the asymmetric unit. The Au—Cl—Au angles are 80.7, 82.7° with correspondingly short Au $\cdots$ Au contacts (3.035, 3.085 Å). Au—Cl lengths range from 2.328 to 2.345 Å.

**Introduction.** We have recently discovered a general method of preparing single-bridged binuclear Au<sup>I</sup> complexes with anionic bridging groups (Uson, Laguna

& Castrillo, 1979); only one such complex,  $(Me_3PAu)_2S$ , has hitherto been reported (Schmidbaur, Franke & Eberlein, 1975). A crystal structure determination of  $(Ph_3PAu)_2Cl^+ \cdot ClO_4^-$  was attempted to confirm the assumed bridged structure.

Crystals were obtained as colourless flat blocks from dichloromethane/hexane. 10 712 intensities in the range  $7 < 2\theta < 45^\circ$  were measured on a Stoe four-circle diffractometer with monochromated Mo  $K\alpha$  radiation and a crystal  $0.4 \times 0.3 \times 0.1$  mm. After application of Lp and empirical absorption corrections, averaging equivalent reflexions gave 10 098 unique reflexions, 8642 with  $F > 4\sigma(F)$ . Cell dimensions were obtained from accurate centring of 28 strong reflexions.

Table 1. Atom coordinates ( $\times 10^4$ ) and isotropic temperature factors ( $\text{\AA}^2 \times 10^3$ )

	x	y	z	U
Au(1)	340 (1)	1547 (1)	384 (1)	60 (1)*
Au(2)	504 (1)	3096 (1)	-478 (1)	57 (1)*
Au(1')	2818 (1)	568 (1)	4585 (1)	64 (1)*
Au(2')	799 (1)	922 (1)	5495 (1)	60 (1)*
Cl	2103 (3)	1819 (3)	-45 (2)	78 (2)*
P(1)	-1287 (4)	1207 (2)	801 (1)	51 (1)*
P(2)	-991 (4)	4363 (2)	-881 (1)	52 (1)*
Cl'	1871 (4)	-485 (2)	5016 (2)	70 (2)*
P(1')	3813 (4)	1522 (3)	4179 (2)	60 (2)*
P(2')	-331 (4)	2288 (2)	5915 (1)	54 (1)*
C(22)	-742 (9)	6122 (7)	-1288 (4)	81 (5)
C(23)	-145 (9)	6646 (7)	-1620 (4)	93 (6)
C(24)	943 (9)	6147 (7)	-1939 (4)	95 (6)
C(25)	1433 (9)	5123 (7)	-1925 (4)	87 (5)
C(26)	836 (9)	4598 (7)	-1593 (4)	67 (4)
C(21)	-251 (9)	5097 (7)	-1275 (4)	62 (4)
C(32)	-2567 (10)	3414 (6)	-1097 (3)	67 (4)
C(33)	-3404 (10)	3200 (6)	-1398 (3)	73 (4)
C(34)	-3593 (10)	3588 (6)	-1914 (3)	71 (4)
C(35)	-2944 (10)	4190 (6)	-2130 (3)	78 (5)
C(36)	-2106 (10)	4403 (6)	-1829 (3)	59 (4)
C(31)	-1917 (10)	4016 (6)	-1313 (3)	51 (3)
C(12)	-3330 (10)	5780 (7)	-582 (3)	72 (4)
C(13)	-4215 (10)	6431 (7)	-238 (3)	96 (6)
C(14)	-3928 (10)	6434 (7)	283 (3)	89 (5)
C(15)	-2755 (10)	5785 (7)	461 (3)	75 (5)
C(16)	-1870 (10)	5134 (7)	117 (3)	69 (4)
C(11)	-2157 (10)	5132 (7)	-405 (3)	56 (4)
C(42)	-3515 (9)	1008 (7)	555 (3)	70 (4)
C(43)	-4373 (9)	918 (7)	215 (3)	79 (5)
C(44)	-4056 (9)	874 (7)	-325 (3)	77 (5)
C(45)	-2882 (9)	920 (7)	-526 (3)	72 (4)
C(46)	-2024 (9)	1009 (7)	-186 (3)	68 (4)
C(41)	-2341 (9)	1053 (7)	355 (3)	51 (3)
C(52)	373 (9)	8 (6)	1527 (4)	68 (4)
C(53)	893 (9)	-825 (6)	1858 (4)	85 (5)
C(54)	377 (9)	-1561 (6)	1893 (4)	87 (5)
C(55)	-658 (9)	-1462 (6)	1597 (4)	83 (5)
C(56)	-1178 (9)	-629 (6)	1266 (4)	76 (5)
C(51)	-662 (9)	106 (6)	1231 (4)	53 (3)
C(62)	-2670 (11)	1961 (6)	1735 (4)	74 (5)
C(63)	-3512 (11)	2714 (6)	2038 (4)	99 (6)
C(64)	-4007 (11)	3672 (6)	1820 (4)	94 (6)
C(65)	-3660 (11)	3876 (6)	1299 (4)	95 (6)
C(66)	-2818 (11)	3122 (6)	996 (4)	73 (4)
C(61)	-2323 (11)	2165 (6)	1214 (4)	57 (4)
C(12')	-1441 (11)	3077 (6)	4981 (4)	83 (5)
C(13')	-1989 (11)	3832 (6)	4608 (4)	79 (5)
C(14')	-2123 (11)	4793 (6)	4697 (4)	102 (6)
C(15')	-1709 (11)	4997 (6)	5159 (4)	107 (6)
C(16')	-1161 (11)	4241 (6)	5533 (4)	84 (5)
C(11')	-1027 (11)	3281 (6)	5443 (4)	47 (3)
C(22')	-2731 (10)	2955 (5)	6454 (4)	75 (5)
C(23')	-3726 (10)	2818 (5)	6776 (4)	85 (5)
C(24')	-3624 (10)	1875 (5)	6962 (4)	88 (5)
C(25')	-2527 (10)	1068 (5)	6826 (4)	88 (5)
C(26')	-1532 (10)	1205 (5)	6504 (4)	77 (5)
C(21')	-1634 (10)	2148 (5)	6318 (4)	59 (4)
C(32')	1689 (10)	2829 (7)	6109 (3)	80 (5)
C(33')	2399 (10)	3167 (7)	6416 (3)	81 (5)
C(34')	2000 (10)	3370 (7)	6941 (3)	74 (4)
C(35')	891 (10)	3234 (7)	7160 (3)	86 (5)
C(36')	181 (10)	2897 (7)	6854 (3)	78 (5)
C(31')	581 (10)	2694 (7)	6328 (3)	61 (4)

Table 1 (cont.)

	x	y	z	U
C(42')	4657 (11)	2950 (7)	4469 (4)	80 (5)
C(43')	5125 (11)	3397 (7)	4823 (4)	104 (6)
C(44')	5336 (11)	3000 (7)	5337 (4)	103 (6)
C(45')	5080 (11)	2157 (7)	5497 (4)	103 (6)
C(46')	4612 (11)	1710 (7)	5142 (4)	79 (5)
C(41')	4401 (11)	2107 (7)	4629 (4)	70 (4)
C(52')	5035 (10)	84 (8)	3481 (5)	90 (5)
C(53')	6039 (10)	-450 (8)	3135 (5)	107 (6)
C(54')	7173 (10)	-258 (8)	3076 (5)	104 (6)
C(55')	7303 (10)	468 (8)	3363 (5)	114 (7)
C(56')	6299 (10)	1002 (8)	3709 (5)	99 (6)
C(51')	5165 (10)	810 (8)	3768 (5)	63 (4)
C(62')	1535 (10)	3052 (7)	3958 (4)	69 (4)
C(63')	730 (10)	3826 (7)	3649 (4)	76 (5)
C(64')	1175 (10)	4064 (7)	3150 (4)	102 (6)
C(65')	2424 (10)	3527 (7)	2959 (4)	146 (9)
C(66')	3229 (10)	2754 (7)	3268 (4)	96 (6)
C(61')	2784 (10)	2516 (7)	3767 (4)	64 (4)
Cl(11)	4014 (5)	1890 (3)	8331 (2)	85 (2)*
O(11)	5382 (16)	1526 (11)	8306 (7)	142 (6)
O(12)	3909 (38)	900 (26)	8559 (15)	337 (18)
O(13)	3557 (25)	1741 (18)	7908 (11)	219 (10)
O(14)	3449 (30)	2463 (21)	8711 (12)	263 (12)
Cl(21)	5992 (6)	4432 (3)	3315 (2)	97 (3)*
O(21)	5581 (20)	4954 (14)	3769 (9)	171 (7)
O(22)	5837 (26)	4960 (18)	2870 (11)	216 (10)
O(23)	7358 (35)	4435 (22)	3255 (13)	282 (14)
O(24)	6418 (21)	3427 (15)	3410 (9)	177 (7)
Cl(31)	-214 (8)	1427 (4)	3841 (2)	134 (4)*
Cl(32)	-84 (7)	1321 (4)	2707 (3)	136 (3)*
C(3)	-484 (20)	2082 (14)	3232 (8)	100 (6)
Cl(41)	3053 (12)	1148 (10)	1330 (5)	296 (19)*
Cl(42)	4191 (21)	1971 (15)	1776 (9)	202 (5)
Cl(43)	2849 (20)	1010 (13)	2257 (8)	202 (5)
C(4)	4058 (30)	809 (20)	1905 (13)	162 (10)

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

The Au atoms were located by direct methods and other atoms by successive difference syntheses (after suitable refinement). Two molecules of dichloromethane were identified; both these and the perchlorate anions show high temperature factors and substantial distortions from ideal geometry, and are probably disordered. A disordered model [half-occupied Cl(42) and Cl(43)] was adopted for one CH<sub>2</sub>Cl<sub>2</sub> molecule.

In the final cycles Au, P and Cl atoms were refined anisotropically, phenyl rings as rigid groups (C—C 1.395, C—H 0.96 Å, all angles 120°) with isotropic C atoms and with H-atom temperature factors fixed at 0.1 Å<sup>2</sup>. The final  $R' = \sum w^{1/2} \Delta / \sum w^{1/2} |F_o|$  was 0.074, with  $R = 0.072$ . The weighting scheme was  $w^{-1} = \sigma^2(F) + 0.001F^2$ . Final coordinates are given in Table 1, bond lengths and angles in Tables 2 and 3.\*

\* 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 35152 (55 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond lengths (Å)

Au(1)—Cl	2.328 (5)	Au(1)—P(1)	2.230 (5)
Au(2)—Cl	2.340 (5)	Au(2)—P(2)	2.239 (4)
Au(1')—Cl'	2.342 (5)	Au(1')—P(1')	2.240 (6)
Au(2')—Cl'	2.345 (5)	Au(2')—P(2')	2.232 (4)
C(21)—P(2)	1.792 (13)	C(31)—P(2)	1.795 (13)
C(11)—P(2)	1.819 (10)	C(41)—P(1)	1.791 (12)
C(51)—P(1)	1.824 (11)	C(61)—P(1)	1.800 (10)
C(11')—P(2')	1.797 (10)	C(21')—P(2')	1.781 (13)
C(31')—P(2')	1.801 (14)	C(41')—P(1')	1.784 (14)
C(51')—P(1')	1.792 (12)	C(61')—P(1')	1.813 (11)

Table 3. Bond angles (°)

Cl—Au(1)—P(1)	177.1 (2)	Cl—Au(2)—P(2)	177.6 (3)
Cl'—Au(1')—P(1')	177.3 (2)	Cl'—Au(2')—P(2')	176.1 (3)
Au(1)—Cl—Au(2)	82.7 (2)	Au(1)—P(1)—C(41)	112.5 (4)
Au(1)—P(1)—C(51)	109.8 (5)	C(41)—P(1)—C(51)	110.0 (6)
Au(1)—P(1)—C(61)	113.7 (5)	C(41)—P(1)—C(61)	104.2 (6)
C(51)—P(1)—C(61)	106.4 (6)	Au(2)—P(2)—C(21)	110.2 (4)
Au(2)—P(2)—C(31)	114.3 (4)	C(21)—P(2)—C(31)	106.6 (6)
Au(2)—P(2)—C(11)	111.0 (4)	C(21)—P(2)—C(11)	108.9 (6)
C(31)—P(2)—C(11)	105.5 (6)	Au(1')—Cl'—Au(2')	80.7 (2)
Au(1')—P(1')—C(41')	112.8 (5)	Au(1')—P(1')—C(51')	110.5 (5)
C(41')—P(1')—C(51')	107.1 (7)	Au(1')—P(1')—C(61')	113.5 (5)
C(41')—P(1')—C(61')	105.0 (6)	C(51')—P(1')—C(61')	107.4 (6)
Au(2')—P(2')—C(11')	109.7 (4)	Au(2')—P(2')—C(21')	111.4 (4)
C(11')—P(2')—C(21')	106.5 (6)	Au(2')—P(2')—C(31')	115.0 (4)
C(11')—P(2')—C(31')	106.7 (6)	C(21')—P(2')—C(31')	107.1 (6)
P(2)—C(21)—C(22)	123.1 (4)	P(2)—C(21)—C(26)	116.8 (4)
P(2)—C(31)—C(32)	117.3 (4)	P(2)—C(31)—C(36)	122.3 (4)
P(2)—C(11)—C(12)	118.1 (4)	P(2)—C(11)—C(16)	121.7 (4)
P(1)—C(41)—C(42)	118.7 (4)	P(1)—C(41)—C(46)	121.2 (4)
P(1)—C(51)—C(52)	117.9 (5)	P(1)—C(51)—C(56)	122.1 (5)
P(1)—C(61)—C(62)	121.5 (4)	P(1)—C(61)—C(66)	118.5 (4)
P(2')—C(11')—C(12')	118.6 (5)	P(2')—C(11')—C(16')	121.4 (5)
P(2')—C(21')—C(22')	121.6 (3)	P(2')—C(21')—C(26')	118.4 (3)
P(2')—C(31')—C(32')	118.6 (4)	P(2')—C(31')—C(36')	121.4 (4)
P(1')—C(41')—C(42')	120.5 (5)	P(1')—C(41')—C(46')	119.5 (5)
P(1')—C(51')—C(52')	118.2 (6)	P(1')—C(51')—C(56')	121.7 (6)
P(1')—C(61')—C(62')	119.0 (4)	P(1')—C(61')—C(66')	121.0 (4)

**Discussion.** The structure confirms the bridging nature of the Cl atom (Fig. 1). The two independent cations display some differences in ring orientation (corresponding Au—P—C—C torsion angles differ by up to 19°) but are otherwise very similar. The most striking features are the small Au—Cl—Au angles (80.7, 82.7°) and short Au...Au contacts [3.085 (2), 3.035 (2) Å]. Such short contacts are frequently observed in Au<sup>I</sup> derivatives, even when the Au atoms are in separate molecules (e.g. 3.301 Å in C<sub>5</sub>H<sub>11</sub>AuClN; Guy, Jones, Mays & Sheldrick, 1977). The shortest such contact is 2.76 Å in the Au<sup>I</sup> dipropylthiocarbamate dimer, [(C<sub>3</sub>H<sub>7</sub>)<sub>2</sub>NCS<sub>2</sub>Au]<sub>2</sub> (Hesse & Jennische, 1972), in which a weak Au—Au bond has been indicated by Raman spectroscopy (Farrell & Spiro, 1971). The ease of packing of molecules with linearly coordinated metal atoms has also been suggested as a

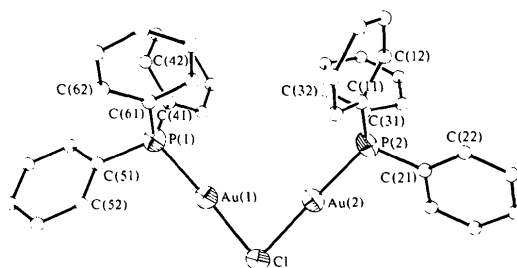


Fig. 1. One (Ph<sub>3</sub>PAu)<sub>2</sub>Cl<sup>+</sup> cation showing the atom numbering. (The second cation is distinguished by the addition of primes to the atom names.)

reason for close Au...Au approaches (Hesse & Jennische, 1972). In the present structure, some slight bonding interaction between Au atoms is indicated by the Au—Cl—Au angles' being considerably less than 90°.

The coordination at Au is essentially linear (P—Au—Cl 177.1, 177.6°). The Au—Cl lengths (2.328, 2.340, 2.342, 2.345 Å) are, as expected, somewhat longer than for terminal Au—Cl [e.g. 2.279 Å for Ph<sub>3</sub>PAuCl (Baenziger, Bennett & Soboroff, 1976); an Au—Cl bond of 2.33 Å in Cl<sub>3</sub>PAuCl has been quoted (Arai, 1962) but calculation from published coordinates and cell constants gives 2.29 Å]. In AuCl itself, which consists of infinite zigzag chains of alternating Au and Cl atoms, the Au—Cl bond is also long (2.36 Å); the Au—Cl—Au angle is, however, 92°, with a correspondingly longer Au...Au distance of 3.22 Å (Janssen, Folmer & Wiegers, 1974). A shorter Au...Au approach would entail Cl...Cl distances less than the observed 3.22 Å.

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