

Fig. 2. Stereoview of the unit cell of PtCl<sub>3</sub>(morphinium) hemihydrate acetone solvate. Only the 0–c/2 portion is shown. The atoms are represented by spheres of arbitrary sizes. The H bonds are represented by dashed lines.

O(1)–H...O(2) hydrogen bonds [O(1)–O(2) = 2.70 (2) Å, Table 2]. At the same time, the H attached to O(2) forms an additional H bond with the water molecule O(4). As for the H atoms of water, they act as donors in H bonds with the carbonyl groups O(5) of two acetone molecules. The interactions surrounding O(4) are, however, weaker than those responsible for molecular pairing. The second system of H bonds links these pairs into a helical chain running roughly along the c direction. They involve the ammonium N–H bond and the coordinated Cl atoms, which are the presumed sites of charge concentration in the zwitterion. The N–H bond is pointing roughly halfway between Cl(1) and Cl(2). The N–Cl distances [3.14 (1) and 3.33 (2) Å, respectively] lie well below the upper limit of 3.54 Å proposed by Stout & Jensen (1968) for this type of H bond.

In summary, this structure shows that the PtCl<sub>3</sub> group forms a normal olefinic complex with the morphinium moiety, without direct participation of other potential donor sites, such as the hydroxyl groups, the exocyclic O or the aromatic system.

A generous gift of morphine hydrochloride trihydrate from Professor J. Cros is gratefully acknowledged. The technical assistance of J. Escalier and Marc J. Oliver is appreciated.

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*Acta Cryst.* (1985). **C41**, 862–865

## Structure of Tetraphenylphosphonium Bis(benzenethiolato)aurate(I), [P(C<sub>6</sub>H<sub>5</sub>)<sub>4</sub>][Au(C<sub>6</sub>H<sub>5</sub>S)<sub>2</sub>]

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(Received 29 June 1984; accepted 31 January 1985)

**Abstract.**  $M_r = 754.7$ , hexagonal,  $P6_1$ ,  $a = 9.746$  (3),  $c = 56.552$  (11) Å,  $U = 4652$  Å<sup>3</sup>,  $Z = 6$ ,  $D_m = 1.63$ ,  $D_x = 1.617$  g cm<sup>-3</sup>, Mo  $K\alpha$ ,  $\lambda = 0.71073$  Å,  $\mu =$

$50.7$  cm<sup>-1</sup>,  $F(000) = 2232$ , room temperature,  $R = 0.034$  using 882 data. The coordination about the gold atom is found to be linear with Au–S bond lengths of 2.271 (8) and 2.262 (8) Å. The two S–C bond lengths are 1.79 (1) and 1.80 (1) Å suggesting purely  $\sigma$  nature. The two Au–S–C angles are consistent with tetrahedral S atoms.

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**Introduction.** An investigation into the coordination geometry of gold(I) thiolate complexes led to the synthesis of several simple anionic derivatives of the general formula  $[\text{Au}(\text{SR})_2]^-$ , where  $R = \text{Me}$ ,  $t\text{Bu}$  or  $\text{Ph}$  (Bowmaker & Dobson, 1981). Only one such anion had previously been isolated, where  $R$  was the strongly electron-withdrawing group,  $\text{C}_6\text{F}_5$  (Beck, Stettner, Todros & Schwarzhaus, 1967). These findings contrast with the similar,  $\text{Au}^{\text{I}}$  thiomalate system, where a  $^{13}\text{C}$  NMR study (Isab & Sadler, 1976) of the reaction of thiomalic acid with disodium thiomalatogold(I),  $\text{Na}_2[\text{Au}\{\text{SCHCO}_2(\text{CH}_2\text{CO}_2)\}]$ , concluded that clusters,  $[\text{Au}_4(\text{SR})_7]^{3-}$  ( $\text{RS}^- = \text{thiomalate}$ ), are formed in solution and that further addition of thiomalate does not result in the formation of mononuclear  $[\text{Au}(\text{SR})_2]^-$ . The postulated structure of the tetragold cluster involves three- and four-coordinate gold(I) (Isab & Sadler, 1976). The IR study of the dithiolate complexes  $[\text{Au}(\text{SR})_2]^-$  ( $R = \text{Me}$ ,  $t\text{Bu}$ ,  $\text{Ph}$ ) (Bowmaker & Dobson, 1981) suggests the presence of linearly coordinated gold(I) but for the title compound, where  $R = \text{Ph}$ , the far-IR spectrum is a complicated one, particularly in the region  $230\text{--}260\text{ cm}^{-1}$  where a group of bands, attributed to  $\nu(\text{Au}\text{--}\text{S})$ , is observed. Since the reason for this multiplicity is not obvious the crystals were subjected to X-ray analysis.

**Experimental.** Crystals of  $[\text{PPh}_4][\text{Au}(\text{SPh})_2]$  supplied by Dr G. A. Bowmaker & Mr B. C. Dobson of the University of Auckland.  $D_m$  measured by flotation in aqueous zinc bromide, CAD-4 diffractometer,  $\text{Mo K}\alpha$  radiation. The 1783 reflections measured yielded, on averaging, 1462 unique reflections ( $R_{\text{int}} = 0.0267$ ), 882 with  $I > 3\sigma(I)$  used;  $\omega/2\theta$  scan mode;  $hkl$  range  $0\text{--}8$ ,  $0\text{--}8$ ,  $0\text{--}54$ ;  $\theta$  limit  $20^\circ$ ; scan range  $\theta = (0.7 + 0.347 \tan \theta)^\circ$  was extended by 25% on either side of the peak to record backgrounds; horizontal aperture  $(1.7 + 1.2 \tan \theta)$  mm, vertical aperture 4 mm; scan speed chosen to return a constant  $\sigma(I) = 0.02I$  with a maximum scan time set at 120 s. Data were collected in shells of increasing  $\theta$  but the collection was stopped at  $\theta = 20^\circ$  since, despite the relatively long maximum scan time allowed, only a small number of observed reflections were being recorded. Subsequent calculations over  $\theta$  intervals of  $1^\circ$  showed that a substantial fall-off in the number of data, for which  $F^2 > 3\sigma(F^2)$ , occurred in the range  $15\text{--}20^\circ$ . From 61% observed data measured over  $\theta = 15\text{--}16^\circ$  the number had fallen to only 27% for the  $19\text{--}20^\circ$   $\theta$  interval. Three standard reflections recorded every 7200 s and three orientation control reflections after every 100 measurements; variations in standards were less than 3%. Standard deviations determined from counting statistics. Crystal: thin hexagonal plate with faces of form  $\{100\}$ ,  $\{\bar{1}00\}$ ,  $\{110\}$ ,  $\{\bar{1}\bar{1}0\}$ ,  $\{010\}$ ,  $\{0\bar{1}0\}$ ,  $\{001\}$  and  $\{00\bar{1}\}$ , dimensions  $0.30 \times 0.30 \times 0.04$  mm, calculated volume  $0.00312\text{ mm}^3$ . Absorption corrections

applied [program *ABSCOR* in Enraf–Nonius (1980) *SDP* package]; grid size  $6 \times 6 \times 2$ , maximum transmission factor 0.814, minimum value 0.268. (The  $R$  factor calculated for the averaging process on data not corrected for absorption was 0.0615.) Systematic absences  $00l$  ( $l \neq 6n$ ). Structure solved by Patterson and Fourier methods. Least-squares refinement [calculations with Enraf–Nonius *SDP* package and *SHELX* (Sheldrick, 1976)] of 114 variables of atomic positions and thermal parameters brought  $R$  to 0.034 and  $wR$  to 0.031. (Final structure factor calculation assuming space group  $P6_5$  returned  $R$  as 0.045.) Atoms of phenyl rings treated as rigid group ( $\text{C}\text{--}\text{C}$ , 1.395;  $\text{C}\text{--}\text{H}$ , 0.96 Å) with isotropic thermal parameters (individual values refined for C; H values refined as group). Other atoms given anisotropic thermal parameters. Function minimized  $\sum w(|F_o| - |F_c|)^2$  with  $w = 1.135/[\sigma^2(F) + 0.000391F^2]$ ,  $(\Delta/\sigma)_{\text{max}} = 0.1$ . Atom scattering factors and anomalous-dispersion coefficients from Cromer & Mann (1968) and Cromer & Liberman (1970). Final  $\Delta\rho$  map showed the largest peak of  $ca\ 0.5\text{ e \AA}^{-3}$  within 1.4 Å of Au. The deepest trough noted was of  $ca\ 0.3\text{ e \AA}^{-3}$ .

**Discussion.** Atomic coordinates are listed in Table 1.\* Listings of bond distances and bond angles are given in Table 2.

The crystal structure consists of monomeric units of bis(benzenethiolato)aurate(I) anions and tetraphenylphosphonium cations; the arrangement of these in the unit cell can be seen in the stereoview in Fig. 1. The aurate(I) anion with its atom-numbering scheme is shown in Fig. 2. In the cation the four phenyl rings are labelled C(13)–C(18), C(19)–C(24), C(25)–C(30) and C(31)–C(36). The coordination at the gold atom is linear as is expected for  $\text{Au}^{\text{I}}$  complexes with few intra- or intermolecular steric pressures. The  $\text{S}(1)\text{--}\text{Au}\text{--}\text{S}(2)$  angle of  $179.0(3)$  may be compared with those observed in the similar complexes  $\text{AuClPPh}_3$  [ $\angle\text{Cl}\text{--}\text{Au}\text{--}\text{P}$ ,  $179.68(8)^\circ$ ] (Baenziger, Bennett & Soboroff, 1976) and  $\text{AuClP(OPh)}_3$  [ $\angle\text{P}\text{--}\text{Au}\text{--}\text{P}$ ,  $178.5(2)^\circ$ ] (Hitchcock & Pye, 1977). The two  $\text{Au}\text{--}\text{S}$  distances of 2.271(8) and 2.262(8) Å lie within the range observed (2.223–2.297 Å) in a number of other  $\text{Au}^{\text{I}}$  complexes with two S donors (Hesse & Jennische, 1972; Guy, Jones & Sheldrick, 1976; Ruben, Zalkin, Faltens & Templeton, 1974; Lawton, Rohrbaugh & Kokotailo, 1972). They are a little shorter than expected for purely  $\sigma$  bonds [the sum of the covalent radii is 2.36 Å (Blundell & Powell, 1971; Pauling, 1960)] and, as already suggested (Drew & Riedl, 1973), probably

\* Lists of structure factors, anisotropic thermal parameters for the non-group atoms and hydrogen-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42034 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

involve a small measure of  $d\pi-d\pi$  bonding. The two S—C bond lengths of 1.79 (1) and 1.80 (1) Å agree with those found in the molecules  $\text{Me}_2\text{S}$  (1.81 Å) (Maier, 1961) and  $\text{Me}_2\text{SO}$  (1.80 Å) (Thomas, Shoemaker & Eriks, 1966) suggesting purely  $\sigma$  nature.

Table 1. Fractional atomic coordinates and isotropic thermal parameters ( $\times 10^4$ ; for C  $\times 10^3$ ) with their e.s.d.'s for  $[\text{PPh}_4][\text{Au}(\text{SPh})_2]$

	x	y	z	$U_{\text{eq}}$ or $U(\text{\AA}^2)$
Non-group atoms				
Au	0.5447 (1)	0.0713 (1)	0*	718 (6)†
S(1)	0.5715 (8)	0.3015 (9)	-0.0142 (1)	956 (53)†
S(2)	0.5174 (9)	-0.1596 (9)	0.0135 (1)	919 (54)†
P	0.0666 (6)	0.0268 (7)	0.0632 (1)	448 (34)†
Rigid-group atoms				
C(1)	0.3773 (15)	0.2755 (21)	-0.0172 (3)	77 (8)
C(2)	0.2436 (15)	0.1266 (21)	-0.0204 (3)	74 (8)
C(3)	0.0947 (15)	0.1126 (21)	-0.0229 (3)	59 (7)
C(4)	0.0795 (15)	0.2474 (21)	-0.0221 (3)	67 (8)
C(5)	0.2132 (15)	0.3963 (21)	-0.0188 (3)	81 (8)
C(6)	0.3621 (15)	0.4103 (21)	-0.0164 (3)	77 (8)
C(7)	0.4830 (19)	-0.1722 (22)	0.0448 (2)	76 (8)
C(8)	0.4023 (19)	-0.3205 (22)	0.0556 (2)	100 (10)
C(9)	0.3801 (19)	-0.3316 (22)	0.0800 (2)	98 (10)
C(10)	0.4385 (19)	-0.1944 (22)	0.0937 (2)	102 (10)
C(11)	0.5193 (19)	-0.0460 (22)	0.0828 (2)	90 (10)
C(12)	0.5415 (19)	-0.0349 (22)	0.0584 (2)	58 (7)
C(13)	0.2704 (12)	0.1578 (14)	0.0680 (3)	47 (6)
C(14)	0.3675 (12)	0.2331 (14)	0.0485 (3)	51 (6)
C(15)	0.5224 (12)	0.3563 (14)	0.0520 (3)	59 (7)
C(16)	0.5802 (12)	0.4041 (14)	0.0749 (3)	84 (9)
C(17)	0.4831 (12)	0.3288 (14)	0.0943 (3)	64 (8)
C(18)	0.3282 (12)	0.2057 (14)	0.0908 (3)	49 (7)
C(19)	-0.0163 (16)	0.1520 (13)	0.0601 (3)	46 (6)
C(20)	-0.0317 (16)	0.2017 (13)	0.0376 (3)	60 (7)
C(21)	-0.0623 (16)	0.3263 (13)	0.0352 (3)	82 (8)
C(22)	-0.0774 (16)	0.4013 (13)	0.0552 (3)	56 (7)
C(23)	-0.0620 (16)	0.3516 (13)	0.0777 (3)	66 (7)
C(24)	-0.0314 (16)	0.2269 (13)	0.0801 (3)	55 (7)
C(25)	-0.0189 (17)	-0.1068 (13)	0.0874 (2)	41 (6)
C(26)	-0.1721 (17)	-0.1562 (13)	0.0954 (2)	38 (6)
C(27)	-0.2394 (17)	-0.2713 (13)	0.1130 (2)	55 (7)
C(28)	-0.1534 (17)	-0.3370 (13)	0.1227 (2)	37 (6)
C(29)	-0.0002 (17)	-0.2875 (13)	0.1147 (2)	38 (6)
C(30)	0.0671 (17)	-0.1725 (13)	0.0971 (2)	62 (7)
C(31)	0.0286 (16)	-0.0970 (13)	0.0379 (2)	29 (5)
C(32)	-0.1225 (16)	-0.1684 (13)	0.0280 (2)	45 (6)
C(33)	-0.1637 (16)	-0.2814 (13)	0.0101 (2)	66 (8)
C(34)	-0.0537 (16)	-0.3230 (13)	0.0023 (2)	55 (6)
C(35)	0.0974 (16)	-0.2517 (13)	0.0122 (2)	59 (7)
C(36)	0.1386 (16)	-0.1387 (13)	0.0301 (2)	48 (6)

\* Fixed to determine origin.

†  $U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$ .

Table 2. Bond distances (Å) and bond angles ( $^\circ$ ) for  $[\text{PPh}_4][\text{Au}(\text{SPh})_2]$

Standard deviations are given in parentheses.

Au—S(1)	2.271 (8)	P—C(13)	1.76 (1)
Au—S(2)	2.262 (8)	P—C(19)	1.78 (1)
S(1)—C(1)	1.79 (1)	P—C(25)	1.78 (1)
S(2)—C(7)	1.80 (1)	P—C(31)	1.79 (1)
S(1)—Au—S(2)	179.0 (3)	C(19)—P—C(25)	111.7 (7)
Au—S(1)—C(1)	107.6 (7)	C(19)—P—C(31)	111.9 (7)
Au—S(2)—C(7)	108.9 (6)	C(25)—P—C(31)	104.9 (6)
S(1)—C(1)—C(2)	122.1 (6)	P—C(13)—C(14)	118.9 (5)
S(1)—C(1)—C(6)	117.9 (6)	P—C(13)—C(18)	120.3 (5)
S(2)—C(7)—C(8)	119.5 (6)	P—C(19)—C(20)	119.5 (5)
S(2)—C(7)—C(12)	120.4 (6)	P—C(19)—C(24)	118.9 (5)
C(13)—P—C(19)	104.6 (7)	P—C(25)—C(26)	121.9 (5)
C(13)—P—C(25)	110.9 (7)	P—C(25)—C(30)	117.8 (5)
C(13)—P—C(31)	112.9 (7)	P—C(31)—C(32)	118.0 (5)
		P—C(31)—C(36)	121.4 (5)

The two Au—S—C angles of 107.6 (7) and 108.9 (6) $^\circ$  are consistent with tetrahedral S atoms; steric pressures within the anion are relieved by rotations about the Au—S and S—C bonds. Thus the dihedral angle between the two planes containing Au, S and C atoms is 79 $^\circ$ , an angle which is larger than that observed between the two Au—S—S planes (67 $^\circ$ ) in the complex  $\text{Au}(\text{S}_2\text{O}_3)_2$  (Ruben *et al.*, 1974), the difference being due no doubt to the greater bulk of the phenyl rings. Rotations about the two S—C bonds give rise to torsion angles of 155 $^\circ$  for both Au—S(1)—C(1)—C(6) and Au—S(2)—C(7)—C(8). The above rotations result in a dihedral angle between the two phenyl rings for the anion of 89 $^\circ$ . As a consequence the two shortest intramolecular contacts, Au...H(2) and Au...H(12) of 2.88 and 2.87 respectively are equivalent to the sum of the van der Waals radii (2.90 Å) (Huheey, 1975).

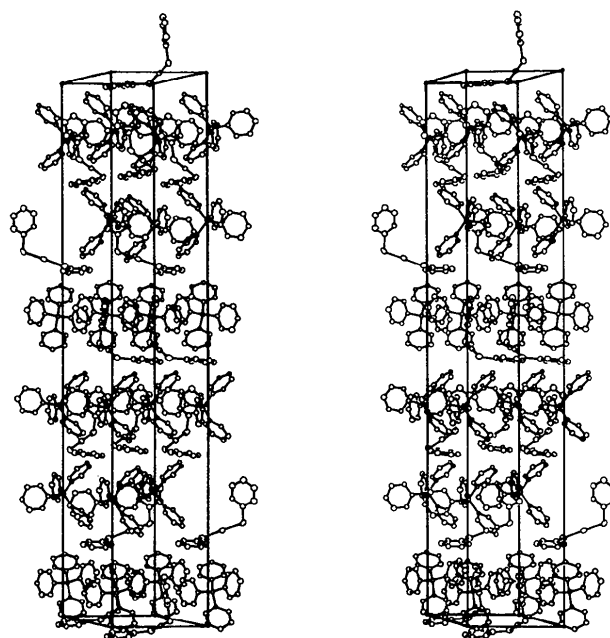


Fig. 1. Stereoview of the unit-cell contents of  $[\text{PPh}_4][\text{Au}(\text{SPh})_2]$ . Hydrogen atoms have been omitted for clarity.

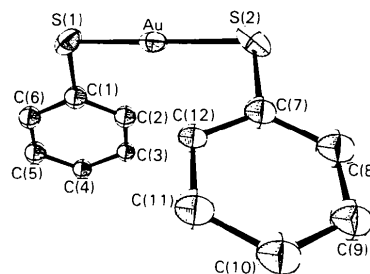


Fig. 2. The complex anion of  $[\text{PPh}_4][\text{Au}(\text{SPh})_2]$  showing the linear coordination geometry. Thermal ellipsoids have been drawn at the 20% probability level.

There are none of the short Au...Au contacts (the shortest is 9.7 Å) that appear to be an indispensable factor in the stability of a number of gold(I) complexes where inter- and intramolecular Au...Au contacts in the range 2.76–3.40 Å have been observed (Hesse & Jennische, 1972; Lawton *et al.*, 1972; Drew & Riedl, 1973; Crane & Beall, 1978; Jones, Sheldrick, Uson & Laguna, 1980).

The geometry observed for the cation, [PPh<sub>4</sub>]<sup>+</sup>, is as expected. The P–C bond lengths (mean 1.78 Å) do not differ significantly from literature values (Coucovanis, Stremple, Simhon, Swenson, Baenziger, Draganjac, Chan, Simopoulos, Papaefthymiou, Kostikas & Petrouleas, 1983; Ciani, Manassero & Seroni, 1980); similarly the C–P–C angles, which lie in the range 104.6 (7)–112.9 (7)°, are unexceptional.

The authors thank Dr G. A. Bowmaker and Mr B. C. Dobson, University of Auckland, for supplying crystals and the University Grants Committee for funding for the purchase of equipment.

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*Acta Cryst.* (1985). **C41**, 865–869

## The Structure of *cis*-Dichloro(25-ethoxycarbonyl-2,3,7,8,12,13,17,18-octaethyl-23,24-methano-23*H*,24*H*-porphyrin-*N*<sup>21</sup>,*N*<sup>22</sup>,*H*)palladium(II) Chloroform Solvate, [PdCl<sub>2</sub>(C<sub>40</sub>H<sub>50</sub>N<sub>4</sub>O<sub>2</sub>)]·CHCl<sub>3</sub>

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(Received 18 September 1984; accepted 4 February 1985)

*Dedicated to Professor Dorothy Hodgkin on the occasion of her 75th birthday*

**Abstract.**  $M_r = 915.56$ , orthorhombic, *Pbca*,  $a = 16.577$  (4),  $b = 26.325$  (6),  $c = 19.951$  (5) Å,  $U =$

$8706.4$  Å<sup>3</sup>,  $Z = 8$ ,  $D_m = 1.38$  (by flotation in aqueous KI),  $D_x = 1.397$  g cm<sup>-3</sup>, Mo  $K\alpha$ ,  $\lambda = 0.71069$  Å,  $\mu = 6.95$  cm<sup>-1</sup>,  $F(000) = 3776.4$ ,  $T = 295$  K,  $R = 0.065$ ,  $wR = 0.053$  for 2442 observed reflections [ $F_o \geq 3\sigma(F_o)$ ]. The PdCl<sub>2</sub> and ethyl acetate groups are bonded to the two opposite pairs of *cis*-N pyrrole atoms and not to the same pair as postulated from chemical

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