Cd^{2+}, Br^-, N and C, and dispersion corrections from *International Tables for X-ray Crystallography* (1974, Vol. IV); full-matrix least-squares refinement with anisotropic thermal parameters (a total of 23 parameters varied). At the final stage of refinement, reflection 100 was removed because of the possibility of extinction effects. $R = 0.035$, $wR = 0.029$, $S = 1.07$, $w = [\sigma^2(F_o)]^{-1}$, $(\Delta/\sigma)_{max} = 0.006$; $(\Delta\rho)_{max} = 0.8$, $(\Delta\rho)_{min} = -0.7$ e Å^{-3}. H atoms could not be found. Computer programs: *UNICS*3 (Sakurai & Kobayashi, 1979). Final atomic parameters are given in Table 1.* Bond lengths and angles are listed in Table 2. The crystal structure is shown in Figs. 1 and 2.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53048 (7 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. The intensities and spacings of 28 reflections for this compound, obtained using powder crystallography, have been reported by Daoud (1976). In the structure analysis of $[N(CH_3)_4][CdCl_3]$ (Morosin, 1972), disorder of the $N(CH_3)_4$ groups was concluded from a difference Fourier synthesis which excluded C atoms; each $N(CH_3)_4$ group takes on two configurations with equal probability, related to each other by mirror symmetry. Such disorder of $N(CH_3)_4$ groups was also confirmed in the present study. The $[N(CH_3)_4][MnCl_3]$ crystal structure (Morosin & Graeber, 1967) is also isomorphous.

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Structure of Bis(μ-methylenediphenylthiophosphinato)-gold(I)mercury(II) Bis(1,1-dicyanoethylene-2,2-dithiolato-*S*,*S*')aurate

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Abstract. $[AuHg(C_{13}H_{12}PS)_2][Au(C_4N_2S_2)_2]$, $M_r = 1337.26$, triclinic, $P\bar{1}$, $a = 12.816$ (6), $b = 12.837$ (7), $c = 14.507$ (7) Å, $\alpha = 92.93$ (4), $\beta = 108.96$ (4), $\gamma = 116.23$ (4)°, $V = 1971$ (2) Å^3, $Z = 2$, $D_x = 2.25$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 120.0$ cm^{-1}, $F(000) = 1240$, $T = 298$ K, final $R = 0.042$, $wR = 0.0574$ for 3919 unique observed reflections. The molecule consists of an Hg^{II}–Au^{I} bimetallic cation with two methylenediphenylthiophosphinate ligands and an Au^{III} anion with two 1,1-dicyanoethylene-2,2-dithiolato ligands. The Hg^{II} and Au^{I} centers are linearly coordinated by two methylene groups and two S atoms, respectively. The Au^{III} is coordinated by four S atoms in a square-planar fashion.

Experimental. The bimetallic compound, $[Au^{I}Hg^{II}(C_{13}H_{12}PS)_2][Au^{III}(C_4N_2S_2)_2]$, was obtained quantitatively by the reaction of $[Au^{I}Hg^{II}(C_{13}H_{12}PS)_2]PF_6$ (Wang & Fackler, 1988) with $[N(n\text{-}C_4H_9)_4][Au(C_4N_2S_2)_2]$ (Khan, Wang & Fackler, 1989) in a 1:1 ratio in CH_2Cl_2 solution. Crystals suitable for X-ray analysis were obtained by recrystallization from a dichloromethane–methanol solution. An orange rectangular crystal of dimensions 0.20 × 0.20 × 0.40 mm was selected and mounted in a random orientation on a glass fiber. Axial dimensions and triclinic symmetry were verified by axial rotation photographs. Unit-cell parameters were obtained from 25 reflections with $8.30 < 2\theta < 23.0°$. Data collection was carried out at room temperature using Wyckoff (ω scan) technique in bisecting geometry (Nicolet *R3m/E* diffractometer, graphite-monochromated Mo $K\alpha$ radiation). 5420 reflections ($-11 \le h \le 0$, $|k| \le 11$, $|l| \le 13$) measured with $4 < 2\theta < 45°$. Scan rate variable, 2.80–29.0° min^{-1}; scan range $-1.0°$ in ω from $K\alpha_1$ to $+1.0°$ from $K\alpha_2$. Background intensities were estimated from a 96-step peak profile. Three standard reflections ($\bar{3}5\bar{5}$, $\bar{4}\bar{1}2$, $\bar{3}\bar{2}2$) were measured every 97 reflections. The data were corrected for absorption, standard variation (<3%), Lorentz and polarization effects. Absorp-

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Table 1. *Atomic coordinates* ($\times 10^4$) *and isotropic thermal parameters* ($Å^2 \times 10^3$) *with e.s.d.'s in parentheses*

	x	y	z	U_{iso}
Hg	179 (1)	3948 (1)	1706 (1)	43 (1)*
Au(1)	6372 (1)	4870 (1)	1090 (1)	48 (1)*
Au(2)	128 (1)	5543 (1)	3337 (1)	43 (1)*
S(1)	2068 (4)	7087 (3)	3556 (3)	50 (2)*
S(2)	−1871 (3)	4162 (3)	3134 (3)	50 (2)*
S(3)	4922 (4)	2915 (4)	185 (3)	61 (2)*
S(4)	4658 (4)	4341 (4)	1565 (3)	58 (2)*
S(5)	7873 (4)	6850 (4)	1968 (3)	55 (2)*
S(6)	8025 (4)	5337 (4)	558 (3)	58 (2)*
P(1)	2860 (3)	6171 (3)	3158 (2)	40 (2)*
P(2)	−2008 (3)	2604 (3)	2555 (2)	40 (2)*
C(1)	2004 (13)	5269 (12)	1908 (9)	46 (7)*
C(2)	−1669 (13)	2641 (12)	1460 (9)	48 (7)*
C(3)	3886 (14)	2941 (13)	712 (10)	49 (8)*
C(4)	2729 (14)	2077 (12)	536 (11)	52 (8)*
C(5)	2270 (15)	986 (15)	−148 (13)	73 (9)*
C(6)	1964 (15)	2204 (13)	963 (12)	63 (8)*
C(7)	8804 (13)	6746 (13)	1360 (9)	46 (7)*
C(8)	9971 (13)	7583 (15)	1477 (10)	53 (8)*
C(9)	10612 (13)	7330 (13)	944 (9)	48 (7)*
C(10)	10667 (16)	8748 (16)	2216 (14)	65 (9)*
N(1)	1954 (18)	146 (16)	−704 (15)	118 (12)*
N(2)	1308 (14)	2345 (13)	1317 (12)	76 (9)*
N(3)	11081 (12)	7049 (13)	504 (9)	66 (7)*
N(4)	11204 (17)	9618 (18)	2746 (14)	102 (11)*
C(11)	4865 (8)	7255 (8)	2565 (5)	55 (4)
C(12)	6069 (8)	8137 (8)	2691 (5)	70 (4)
C(13)	6806 (8)	9050 (8)	2558 (5)	68 (4)
C(14)	6340 (8)	9081 (8)	4299 (5)	82 (5)
C(15)	5136 (8)	8200 (8)	4172 (5)	64 (4)
C(16)	4399 (8)	7287 (8)	3305 (5)	38 (3)
C(21)	3306 (9)	4333 (8)	3672 (5)	54 (4)
C(22)	3481 (9)	3584 (8)	4298 (5)	66 (4)
C(23)	3371 (9)	3695 (8)	5219 (5)	58 (4)
C(24)	3085 (9)	4555 (8)	5514 (5)	69 (4)
C(25)	2911 (9)	5304 (8)	4888 (5)	57 (4)
C(26)	3021 (9)	5193 (8)	3967 (5)	41 (3)
C(31)	−4267 (11)	1669 (10)	2823 (8)	100 (6)
C(32)	−5517 (11)	819 (10)	2601 (8)	126 (8)
C(33)	−6110 (11)	−204 (10)	1839 (8)	109 (7)
C(34)	−5451 (11)	−376 (10)	1300 (8)	86 (5)
C(35)	−4201 (11)	475 (10)	1523 (8)	69 (4)
C(36)	−3609 (11)	1498 (10)	2285 (8)	49 (3)
C(41)	−379 (9)	1660 (9)	3216 (5)	62 (4)
C(42)	335 (9)	1303 (9)	3940 (5)	70 (4)
C(43)	403 (9)	1472 (9)	4920 (5)	62 (4)
C(44)	−242 (9)	1999 (9)	5176 (5)	67 (4)
C(45)	−956 (9)	2356 (9)	4451 (5)	65 (4)
C(46)	−1025 (9)	2186 (9)	3471 (5)	40 (3)

* For values with asterisks, the equivalent isotropic U is defined as 1/3 of the trace of the U_{ij} tensor.

Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Hg—Au(2)	3·085 (1)	Hg—C(1)	2·105 (13)
Hg—C(2)	2·105 (13)	Au(1)—S(3)	2·331 (4)
Au(1)—S(4)	2·340 (5)	Au(1)—S(5)	2·353 (3)
Au(1)—S(6)	2·330 (5)	Au(2)—S(1)	2·297 (3)
Au(2)—S(2)	2·288 (4)	S(1)—P(1)	2·027 (7)
S(2)—P(2)	2·039 (6)	S(3)—C(3)	1·745 (20)
S(4)—C(3)	1·774 (14)	S(5)—C(7)	1·742 (19)
S(6)—C(7)	1·748 (14)	P(1)—C(1)	1·790 (12)
P(1)—C(16)	1·785 (9)	P(1)—C(26)	1·794 (11)
P(2)—C(2)	1·775 (16)	P(2)—C(36)	1·795 (12)
P(2)—C(46)	1·796 (12)	C(3)—C(4)	1·329 (19)
C(4)—C(5)	1·430 (24)	C(4)—C(6)	1·377 (30)
C(5)—N(1)	1·135 (28)	C(6)—N(2)	1·187 (30)
C(7)—C(8)	1·348 (19)	C(8)—C(9)	1·414 (27)
C(8)—C(10)	1·489 (23)	C(9)—N(3)	1·147 (26)
C(10)—N(4)	1·097 (25)		

Au(2)—Hg—C(1)	90·7 (5)	Au(2)—Hg—C(2)	89·8 (5)
C(1)—Hg—C(2)	178·1 (5)	S(3)—Au(1)—S(4)	75·0 (2)
S(3)—Au(1)—S(5)	178·1 (2)	S(4)—Au(1)—S(5)	106·8 (2)
S(3)—Au(1)—S(6)	102·8 (2)	S(4)—Au(1)—S(6)	177·7 (1)
S(5)—Au(1)—S(6)	75·3 (2)	Hg—Au(2)—S(1)	92·2 (1)
Hg—Au(2)—S(2)	92·6 (1)	S(1)—Au(2)—S(2)	173·7 (2)
Au(2)—S(1)—P(1)	100·5 (2)	Au(2)—S(2)—P(2)	102·6 (2)
Au(1)—S(3)—C(3)	89·0 (4)	Au(1)—S(4)—C(3)	88·0 (6)
Au(1)—S(5)—C(7)	87·0 (4)	Au(1)—S(6)—C(7)	87·6 (6)
S(1)—P(1)—C(1)	114·3 (6)	S(1)—P(1)—C(16)	104·9 (4)
C(1)—P(1)—C(16)	110·6 (6)	S(1)—P(1)—C(26)	112·0 (5)
C(1)—P(1)—C(26)	106·5 (6)	C(16)—P(1)—C(26)	108·5 (5)
S(2)—P(2)—C(2)	113·1 (6)	S(2)—P(2)—C(36)	104·6 (6)
C(2)—P(2)—C(36)	110·9 (5)	S(2)—P(2)—C(46)	111·4 (4)
C(2)—P(2)—C(46)	110·1 (7)	C(36)—P(2)—C(46)	106·4 (6)
Hg—C(1)—P(1)	111·6 (8)	Hg—C(2)—P(2)	113·3 (5)
S(3)—C(3)—S(4)	107·8 (7)	S(3)—C(3)—C(4)	127·4 (13)
S(4)—C(3)—C(4)	124·7 (16)	C(3)—C(4)—C(5)	117·3 (18)
C(3)—C(4)—C(6)	123·0 (15)	C(5)—C(4)—C(6)	119·7 (13)
C(4)—C(5)—N(1)	176·7 (22)	C(4)—C(6)—N(2)	177·9 (18)
S(5)—C(7)—S(6)	110·1 (7)	S(5)—C(7)—C(8)	127·4 (13)
S(6)—C(7)—C(8)	122·4 (15)	C(7)—C(8)—C(9)	119·9 (14)
C(7)—C(8)—C(10)	122·2 (17)	C(9)—C(8)—C(10)	117·8 (13)
C(8)—C(9)—N(3)	175·6 (15)	C(8)—C(10)—N(4)	177·9 (23)
P(1)—C(16)—C(11)	120·8 (3)	P(1)—C(16)—C(15)	119·2 (3)
P(1)—C(26)—C(21)	118·4 (4)	P(1)—C(26)—C(25)	121·5 (4)
P(2)—C(36)—C(31)	120·1 (5)	P(2)—C(36)—C(35)	119·9 (5)
P(2)—C(46)—C(41)	121·7 (4)	P(2)—C(46)—C(45)	118·3 (4)

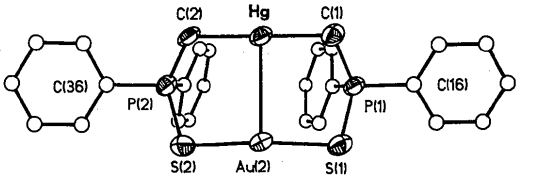

Fig. 1. A perspective view of the $[AuHg(C_{13}H_{12}PS)_2]^+$ cation. Thermal ellipsoids have been drawn at the 50% probability level. H atoms have been omitted for clarity.

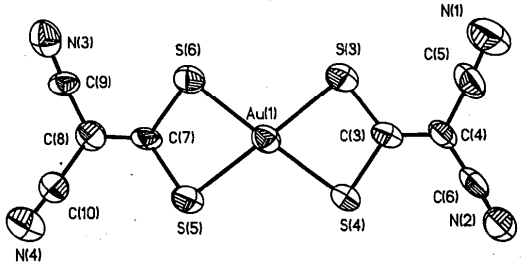

Fig. 2. A perspective view of the $[Au(C_4N_2S_2)_2]^-$ anion. Thermal ellipsoids have been drawn at the 50% probability level.

tion corrections were applied empirically using azimuthal scans of eight medium-intensity reflections spanning a range of 2θ values (10·23–32·04°); minimum and maximum transmission, 0·226 and 0·625, respectively. Structure determination was carried out using the *SHELXTL* collection of crystallographic software (Sheldrick, 1986). Au- and Hg-atom positions were determined from a Patterson map; the remaining non-H atoms were located using difference Fourier techniques. All atoms, besides H and C atoms of the phenyl rings, were refined anisotropically. Phenyl rings were defined as rigid polygons (C—C = 1·35 Å, C—C—C = 120°) using H atoms placed in idealized positions with fixed isotropic thermal parameters [U(H) = 0·08 Å^2]. Scattering factors, including terms for anomalous dispersion, were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV.) Refinement

was based on F with weights of the form $w^{-1} = [\sigma^2(F) + 0{\cdot}00203(F^2)]$. Convergence to conventional R values of $R = 0{\cdot}0442$ and $wR = 0{\cdot}0574$ was obtained using 274 variables and 3919 unique reflections with $F^2 > 3\sigma(F^2)$. In the final cycle, the maximum shift/σ was 0·004 with maximum and minimum residual electron densities of +1·57 and −1·57 e Å^{-3} in the vicinity of the Hg atom. Goodness of fit indicator 1·052. Perspective views of the cation and anion are shown in Figs. 1 and 2, respectively. Atomic positional and equivalent isotropic thermal parameters are given in Table 1.* Bond lengths and angles are summarized in Table 2. Fig. 3 shows the packing.

Related literature. The structures of the $[AuHg(C_{13}H_{12}PS)_2]^+$ cation and the $[Au(C_4N_2S_2)_2]^-$ anion are similar to the corresponding ones in $[AuHg(C_{13}H_{12}PS)_2]PF_6$ and $[N(n\text{-}C_4H_9)_4][Au(C_4N_2S_2)_2]$ reported previously.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53036 (35 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

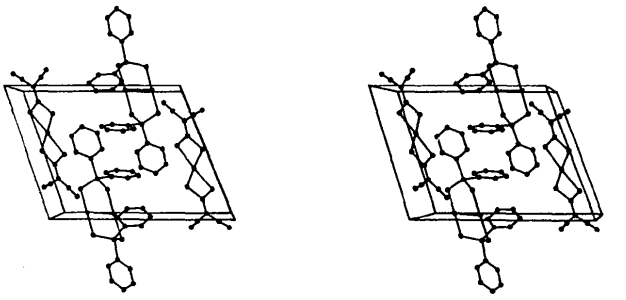

Fig. 3. A stereoview packing diagram of the unit-cell contents projected along the b axis.

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Tetraphenylarsonium Diisopropyldithiophosphinate

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Abstract. $[As(C_6H_5)_4][PS_2(C_3H_7)_2]$, $M_r = 564{\cdot}63$, triclinic, $P\bar{1}$, $a = 11{\cdot}849\ (3)$, $b = 12{\cdot}345\ (3)$, $c = 21{\cdot}153\ (4)$ Å, $\alpha = 91{\cdot}64\ (2)$, $\beta = 105{\cdot}63\ (2)$, $\gamma = 102{\cdot}78\ (2)°$, $V = 2893{\cdot}2$ Å^3, $Z = 4$, $D_x = 1{\cdot}30$ g cm^{-3}, λ(Mo $K\alpha$) = 0·70930 Å, $\mu = 13{\cdot}8$ cm^{-1}, $F(000) = 1176$, $T = 294\ (1)$ K, $R_F = 0{\cdot}034$ for 6201 observed unique reflections. There are two essentially identical molecules in the asymmetric unit. Both the cation and the anion have tetrahedral geometry. All P—S bond lengths are identical (1·989–1·995 Å) and significantly longer than observed for analogs with electron-withdrawing substituents at phosphorus.

Experimental. The title compound was obtained from $NH_4[S_2P(C_3H_7)_2]$ and $[As(C_6H_5)_4]Cl$ in ethanol, followed by evaporation, extraction with CH_2Cl_2, evaporation, and crystallization from 2-propanol/ether as colorless rods. Colorless cuboid crystal, 0·26 × 0·19 × 0·17 mm, was mounted on a glass fiber. Enraf–Nonius CAD-4 diffractometer, graphite monochromator, unit-cell constants from the setting angles of 25 reflections ($24 < 2\theta < 28°$). 10745 total reflections were collected, 10163 unique, 3962 unobserved, 6201 with $F_o^2 > 3{\cdot}0\sigma(F_o^2)$, maximum $2\theta = 50{\cdot}0°$, $0 < h < 14$, $-14 < k < 14$, $-25 < l < 25$, using θ–2θ scans. Corrections: Lorentz and polarization, linear decay (from 0·915–1·130 on I), reflection averaging (agreement on $I = 2{\cdot}6\%$ for 815 reflections), empirical absorption (from 0·926 to 1·000 on I). Centrosymmetric space group judged correct based on successful refinement. Solution by Patterson and Fourier methods. Refinement by full-matrix least squares. H atoms refined as riding atoms (calculated with C—H = 0·95 Å and ideal

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