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Bis[μ-bis(diphenylphosphino)hexane-*P*:*P*′]-digold(I) Hexafluoroantimonate Acetonitrile Solvate

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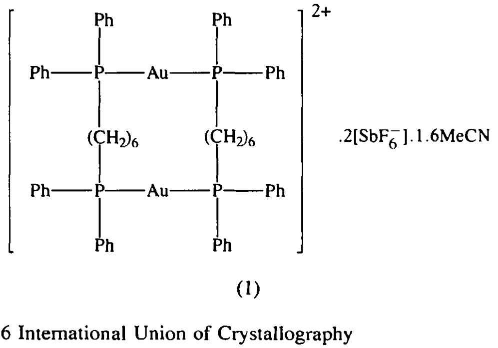
Abstract

In the title compound, $[Au_2(C_6H_{12}\{P(C_6H_5)_2\}_2)_2][SbF_6]_2.1.6CH_3CN$, the $[\{Ph_2P\text{-}(CH_2)_6\text{-}PPh_2\}_2Au_2]^{2+}$ cation lies with its Au atoms on a twofold axis and the $[SbF_6]^-$ anions in general positions. Main bond lengths and angles include Au—P 2.307 (2) and 2.311 (2) Å, P—Au—P 172.7 (1) and 174.2 (1)°. There are no significant intramolecular or intermolecular Au···Au interactions. The C-atom chain of the hexamethylene moiety is maximally extended with C—C—C—C torsion angles in the range −170.4 (7) to −177.7 (7)° and all the Au—P—C—C and P—C—C—C conformations are *gauche*.

Comment

The observation of two-coordinate Au atoms in a bis-diphosphine cation is unusual. Most compounds related to the title compound, (1), contain Au atoms which are coordinated to two P-atom sites but they also have an intramolecular Au···Au interaction, *i.e.* (P_2,Au)-coordination. Examples of such complexes include $[Au_2\{1,2\text{-bis(dimethylphosphino)ethane}\}_2][ClO_4]_2.H_2O$, (2), with P—Au—P and Au—Au 174.7 (2)° and 2.872 (2) Å, respectively (Jaw, Savas, Rogers & Mason, 1989), $[Au_2\{1,1\text{-bis(diphenylphosphino)methane}\}_2][BH_3CN]_2$, (3) [175.2 (2)°, 2.982 (2) Å, respectively; Khan, King, Heinrich, Fackler & Porter, 1989], $[Au_2\{1,2\text{-bis(dicyclohexylphosphino)ethane}\}_2][PF_6]_2$, (4) [160.6 (1)°, 2.935 (1) Å, respectively; Schaefer, Marsh, McCleskey & Gray, 1991]. A further example would appear to be $[Au_2\{1,1\text{-bis(diphenylphosphino)methane}\}_2][Cl_2].CH_3CN$, (5) [164.9 (1)°, 2.994 (1) Å, respectively; Liou, Liu & Wang, 1994], but this compound may be considered to contain four-coordinate Au atoms since there are weakly coordinating Cl^- ions with Au···Cl 2.935 (1) Å. A typical Au—Cl bonded distance is 2.301 Å (Orpen *et al.*, 1994).



(1)

Neutral gold complexes containing two-coordinate gold bonded to one phosphine and one Cl ligand are well known. Examples are $[(AuCl)_2\{1,4\text{-bis(diphenylphosphino)butane}\}]$, (6) (Schmidbaur, Bissinger, Lachmann & Steigelmann, 1992), and $[(AuCl)_2\{1,6\text{-bis(diphenylphosphino)hexane}\}]$, (7) (Van Calcar, Olmstead & Balch, 1995). In both (6) and (7), no intramolecular or intermolecular Au···Au contacts are observed. The structures of (6) and (7) may be contrasted with that of $[(AuCl)_2\{1,5\text{-bis(diphenylphosphino)pentane}\}]$, (8), which has three-coordinate gold (P,Cl,Au) and an intermolecular Au···Au′ distance of 3.323 (1) Å (Schmidbaur, Bissinger, Lachmann & Steigelmann, 1992), and that of $[(AuI)_2\{1,6\text{-bis(diphenylphosphino)hexane}\}]$, (9), which has three-coordinate gold (P,I,Au) and an Au···Au distance of 3.124 (2) Å (Van Calcar, Olmstead & Balch, 1995). It is also worth noting that the three-coordinate (P_2,Cl) gold complex $[AuCl(\text{diphenylphosphinoferrocene})]_n$, (10) (Phang, Hor, Zhou & Mak, 1994), exhibits no intra- and intermolecular Au···Au contacts (6.577 and 12.714 Å, respectively).

Our X-ray analysis of (1) shows that the cation lies with its Au atoms on a twofold axis and that each Au atom is two-coordinate with the P—Au—P angles slightly non-linear [172.7 (1) and 174.2 (1)°] presumably to accommodate the ligand. (Fig. 1 and Table 2). The distance between the Au atoms in the cation is 6.911 (2) Å; hence there is no significant intramolecular transannular Au···Au interaction. The shortest in-

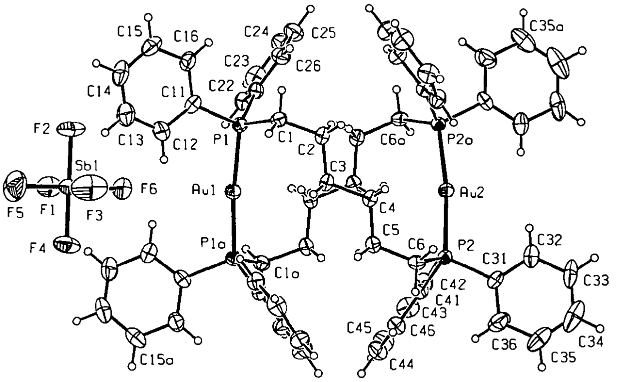


Fig. 1. A view of the cation and anion of (1) with our numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. The twofold axis on which both Au atoms lie is horizontal in this diagram. The atom labels which terminate with an 'a' refer to atoms at equivalent position $1 - x, y, \frac{1}{2} - z$.

termolecular Au···Au separation in (1) is 7.338 (2) Å along the twofold axis; there are no intermolecular gold–phosphine bonds.

The Au—P bond lengths in (1) are normal [2.311 (2) and 2.307 (2) Å] and comparable with typical values of 2.272 Å for two-coordinate gold in $Au(PPh_3)_2$-containing species and 2.331 Å in a variety of $Au(PPh_2Me)$ complexes (Orpen *et al.*, 1994).

All of the P—C and C—C distances in (1) are normal and the P—C—C—C—C—C—C—P chains are fully extended (Fig. 2). The SbF_6^- anion is in a general position and has normal octahedral geometry, with Sb—F distances in the range 1.837 (7)–1.865 (5) Å.

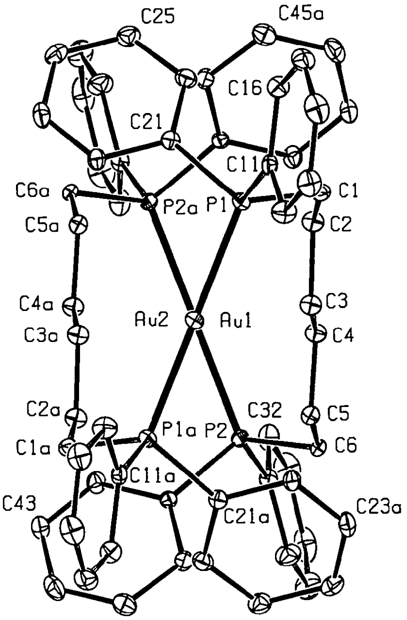


Fig. 2. The cation in (1) viewed along the twofold axis (the Au···Au vector; atom Au2 is below Au1). For clarity, displacement ellipsoids are drawn at the 10% probability level and H atoms are omitted.

Experimental

The reaction between equimolar quantities of 1,6-bis(diphenylphosphino)hexane, $[AuCCC(CH_3)(OH)C_2H_5)]_2$ (Kelleher, 1991) and $Ag[SbF_6]$ in ethanol at room temperature for 24 h afforded the title compound in 60% yield. Recrystallization from acetonitrile gave colourless needle crystals suitable for X-ray analysis.

Crystal data

$[Au_2(C_{30}H_{32}P_2)_2][SbF_6]_2.$-
1.6CH$_3$CN
M_r = 1840.11
Monoclinic
$C2/c$
a = 22.264 (4) Å
b = 14.249 (4) Å
c = 22.004 (3) Å
β = 93.562 (15)°

Mo $K\alpha$ radiation
λ = 0.7107 Å
Cell parameters from 48 reflections
θ = 15.0–20.4°
μ = 5.127 mm^{-1}
T = 294 (1) K
Needle
0.40 × 0.16 × 0.16 mm

V = 6967.2 (27) Å^3
Z = 4
D_x = 1.754 Mg m^{-3}
D_m not measured

Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: four ψ scans at 10° steps (North, Phillips & Mathews, 1968)
T_{min} = 0.817, T_{max} = 0.998
6124 measured reflections
6115 independent reflections
3860 observed reflections [$I > 2\sigma(I)$]

R_{int} = 0.005
θ_{max} = 24.93°
$h = -26 \rightarrow 26$
$k = 0 \rightarrow 16$
$l = 0 \rightarrow 26$
3 standard reflections monitored every 500 reflections
intensity decay: 3.5% (corrected for by appropriate scaling)

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0372$
$wR(F^2) = 0.0982$
S = 0.985
6115 reflections
362 parameters
H atoms riding [*SHELXL*93 (Sheldrick, 1993) defaults, C—H 0.93–0.97 Å]

$w = 1/[\sigma^2(F_o^2) + (0.0501P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max}$ = 0.962 e Å^{-3}
$\Delta\rho_{min}$ = −0.667 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Au1	1/2	0.02521 (3)	1/4	0.04533 (14)
Au2	1/2	0.51024 (3)	1/4	0.04101 (13)
P1	0.59664 (9)	0.03340 (14)	0.29448 (10)	0.0423 (5)
P2	0.45496 (9)	0.49987 (13)	0.15295 (8)	0.0384 (4)
C1	0.6465 (3)	0.1094 (5)	0.2544 (3)	0.047 (2)
C2	0.6249 (4)	0.2093 (5)	0.2413 (4)	0.051 (2)
C3	0.5740 (4)	0.2188 (5)	0.1924 (4)	0.050 (2)
C4	0.5601 (4)	0.3182 (5)	0.1727 (4)	0.050 (2)
C5	0.5081 (3)	0.3246 (5)	0.1264 (3)	0.045 (2)
C6	0.4933 (3)	0.4237 (5)	0.1025 (3)	0.041 (2)
C11	0.6342 (3)	−0.0785 (6)	0.3002 (3)	0.046 (2)
C12	0.6145 (5)	−0.1518 (6)	0.2638 (4)	0.068 (3)
C13	0.6456 (5)	−0.2364 (7)	0.2666 (5)	0.078 (3)
C14	0.6929 (5)	−0.2497 (6)	0.3060 (6)	0.082 (3)
C15	0.7137 (4)	−0.1776 (7)	0.3425 (5)	0.071 (3)
C16	0.6840 (4)	−0.0910 (6)	0.3407 (4)	0.057 (2)
C21	0.5932 (3)	0.0805 (5)	0.3709 (3)	0.044 (2)
C22	0.5409 (4)	0.0617 (6)	0.4019 (4)	0.051 (2)
C23	0.5357 (4)	0.0998 (7)	0.4591 (4)	0.065 (3)
C24	0.5795 (5)	0.1550 (7)	0.4866 (4)	0.065 (3)
C25	0.6306 (4)	0.1721 (7)	0.4559 (4)	0.067 (3)
C26	0.6378 (4)	0.1353 (6)	0.3985 (4)	0.058 (2)
C31	0.4468 (3)	0.6095 (5)	0.1125 (4)	0.047 (2)
C32	0.4717 (4)	0.6904 (6)	0.1369 (5)	0.065 (3)
C33	0.4633 (6)	0.7758 (7)	0.1077 (7)	0.096 (4)
C34	0.4289 (6)	0.7800 (10)	0.0526 (7)	0.106 (5)
C35	0.4066 (6)	0.7019 (10)	0.0267 (6)	0.096 (4)
C36	0.4136 (4)	0.6146 (8)	0.0565 (4)	0.079 (3)
C41	0.3789 (3)	0.4552 (5)	0.1573 (3)	0.040 (2)
C42	0.3483 (4)	0.4756 (6)	0.2084 (4)	0.056 (2)
C43	0.2906 (4)	0.4459 (7)	0.2137 (4)	0.068 (3)

C44 .	0.2633 (4)	0.3884 (7)	0.1701 (5)	0.075 (3)
C45	0.2931 (4)	0.3646 (7)	0.1203 (5)	0.068 (3)
C46	0.3516 (3)	0.3984 (6)	0.1127 (4)	0.050 (2)
Sb1	0.15948 (3)	0.10900 (4)	0.09726 (3)	0.0542 (2)
F1	0.1044 (2)	0.0793 (4)	0.1543 (2)	0.085 (2)
F2	0.2199 (2)	0.1141 (5)	0.1598 (2)	0.093 (2)
F3	0.2138 (3)	0.1412 (7)	0.0404 (3)	0.143 (3)
F4	0.0989 (2)	0.1043 (5)	0.0355 (2)	0.092 (2)
F5	0.1738 (4)	−0.0164 (5)	0.0852 (4)	0.141 (3)
F6	0.1438 (3)	0.2347 (4)	0.1088 (3)	0.107 (2)

Table 2. *Selected geometric parameters (Å, °)*

Au1—P1	2.311 (2)	P2—C31	1.801 (8)
Au2—P2	2.307 (2)	P2—C41	1.818 (7)
Au1···Au2	6.911 (2)	C1—C2	1.525 (10)
P1—C1	1.818 (8)	C2—C3	1.519 (10)
P1—C11	1.802 (8)	C3—C4	1.507 (10)
P1—C21	1.817 (8)	C4—C5	1.497 (10)
P2—C6	1.804 (7)	C5—C6	1.535 (10)
P1—Au1—P1^i	174.2 (1)	C11—P1—C21	108.0 (3)
P2—Au2—P2^i	172.7 (1)	Au2—P2—C6	114.4 (2)
Au1—P1—C1	113.9 (3)	Au2—P2—C31	115.3 (3)
Au1—P1—C11	113.6 (3)	Au2—P2—C41	109.1 (2)
Au1—P1—C21	108.6 (3)	C6—P2—C31	104.8 (3)
C1—P1—C11	105.5 (3)	C6—P2—C41	107.4 (3)
C1—P1—C21	106.9 (4)	C31—P2—C41	105.3 (3)
P1^i—Au1—P1—C1	−50.9 (3)	C2—C3—C4—C5	−177.7 (7)
P2^i—Au2—P2—C6	−50.8 (3)	C3—C4—C5—C6	−177.6 (6)
Au1—P1—C1—C2	52.9 (6)	C4—C5—C6—P2	−75.9 (8)
P1—C1—C2—C3	−72.0 (9)	Au2—P2—C6—C5	51.2 (6)
C1—C2—C3—C4	−170.4 (7)		

Symmetry code: (i) $1 - x, y, \frac{1}{2} - z$.

It became obvious from examination of $\Delta\rho$ maps that there were disordered solvent molecules in the crystal lattice. The crystals prepared for the analysis had been grown from pure acetonitrile, which is thus the only choice, but the geometry pattern of the 'solvent' peaks in the difference map did not correspond with any ordered orientation of acetonitrile molecules. Accordingly, the contribution of the density of the disordered solvent molecules was subtracted from the measured structure factors using the *SQUEEZE* option (Spek, 1994) in *PLATON*95 (Spek, 1995*a*). This indicated that the occupancy of the disordered acetonitrile had a value of 0.8 in the asymmetric unit with a rough error estimate of <0.1. Subsequent refinement then converged with slightly lower *R* factors than when the disordered solvent had been allowed for as a set of 'carbon' peaks; there were no significant changes in the geometry of the gold-containing cation.

Data collection: *DIFRAC* with profile analysis (Gabe & White, 1993). Cell refinement: *DIFRAC*. Data reduction: *DATRD*2 in *NRCVAX*94 (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *SOLVER* in *NRCVAX*94. Program(s) used to refine structure: *NRCVAX*94; *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *NRCVAX*94; *ORTEP*II (Johnson, 1976) in *PLATON* (Spek, 1995*a*); *PLUTON* (Spek, 1995*b*). Software used to prepare material for publication: *NRCVAX*94; *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1344). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2-Ferrocenyl-3-phenylquinoxaline

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Abstract

In the title molecule, $[Fe(C_5H_5)(C_{19}H_{13}N_2)]$, the heterocyclic ring is almost coplanar with the cyclopentadienyl ring to which it is bonded [dihedral angle 4.1 (1)°]; the phenyl ring is almost orthogonal to the heterocyclic ring [dihedral angle 87.6 (1)°].

Comment

Following the observation of a very wide range of hydrogen-bonding patterns in adducts of ferrocenyl

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