C(11)	0.4778 (3)	0.3602 (5)	0.1611 (2)	5.8 (2)
C(12)	0.0872 (4)	0.2203 (5)	−0.0201 (2)	6.4 (2)
C(13)	0.3970 (4)	0.2872 (5)	−0.0376 (3)	6.2 (2)
C(14)	0.3508 (6)	0.0245 (5)	0.0819 (3)	8.0 (3)

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

Al—C(14)	1.956 (5)	Al—C(13)	1.962 (4)
Al—C(12)	1.971 (4)	Al—N(1)	2.050 (2)
N(1)—C(4)	1.488 (3)	N(1)—C(1)	1.494 (3)
N(1)—C(11)	1.494 (4)	N(2)—C(5)	1.405 (3)
N(2)—C(3)	1.461 (4)		
C(14)—Al—C(13)	115.1 (2)	C(14)—Al—C(12)	113.6 (2)
C(14)—Al—N(1)	103.6 (2)	C(13)—Al—C(12)	113.9 (2)
C(13)—Al—N(1)	103.6 (1)	C(12)—Al—N(1)	105.4 (1)
C(4)—N(1)—C(1)	106.5 (2)	C(4)—N(1)—C(11)	110.5 (3)
C(4)—N(1)—Al	110.3 (2)	C(1)—N(1)—C(11)	110.0 (2)
C(1)—N(1)—Al	110.2 (2)	C(11—N(1)—Al	108.3 (2)

The single crystal was sealed in a glass capillary under nitrogen. *CAD*-4 *Software* (Enraf–Nonius, 1989) was used for data collection and cell refinement. The structure was solved by direct methods using *MITHRIL* (Gilmore, 1983) and subsequent Fourier techniques, and refined by full-matrix least-squares methods. The H atoms were found by difference Fourier methods. H-atom coordinates were used for correction of the structure. All calculations were performed using *TEXSAN* (Molecular Structure Corporation, 1989). *ORTEP*II (Johnson, 1976) was used to produce the figure.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and least-squares-planes data have been deposited with the IUCr (Reference: KH1055). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Trifluoromethyl(triphenylphosphine)gold(I)

Ulrich Flörke,[a] Hans-Jürgen Haupt[a] and Peter G. Jones[b]

[a]*Anorganische und Analytische Chemie, Universität-GH Paderborn, Warburgerstr. 100, D-33098 Paderborn, Germany, and* [b]*Institut für Anorganische und Analytische Chemie, Technische Universität, Postfach 3329, D-38023 Braunschweig, Germany. E-mail: floe@mvaxac.uni-paderborn.de*

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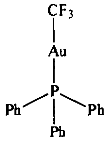
Abstract

The title compound, $[Au(CF_3)\{P(C_6H_5)_3\}]$, is the first crystallographically characterized mononuclear gold(I) complex with an Au—CF$_3$ bond. The Au coordination is nearly linear with P—Au—C 178.56 (11)°, Au—P 2.2859 (9) and Au—C 2.045 (4) Å.

Comment

The structures of linear gold(I) complexes Ph$_3$PAuX have attracted much interest with regard to the type of ligand X and the *trans*-positioned Au—P bond (Ahrland, Dreisch, Norén & Oskarsson, 1987; Barron, Engelhardt, Healy, Oddy & White, 1987; Jones & Lautner, 1988). The complexes with X = Cl, Br, I, CN, SCN and OCOCH$_3$ are effectively isostructural with orthorhombic packing, $P2_12_12_1$, whereas the monoclinic complexes with X = ONO$_2$ and CH$_3$ differ in their packing. We have investigated the related compound, (I), with X = trifluoromethyl, which, according to a search in the Cambridge Structural Database (Allen *et al.*, 1991), is also the first crystallographically characterized example of a mononuclear complex with an Au^I—CF$_3$ bond.



(I)

The title molecule shows the expected linear coordination of the Au^I atom with a P—Au—C angle of 178.56 (11)°. The most interesting geometric features are the Au—P and Au—C bond distances. The former, 2.2859 (9) Å, has the same length as those in the complexes with X = CN [2.278 (2) Å; Jones & Lautner, 1988] and X = CH$_3$ [2.279 (8) Å; Gavens, Guy, Mays & Sheldrick, 1977], although these three ligands differ appreciably in their electronic properties. It has already been stated by Barron *et al.* (1987) that despite the different *trans* influence of *X*, these groups have rather small effects on Au—P distances in the solid state, but show significant effects on the ^{31}P chemical-shift data. Thus, the ^{31}P NMR signal for the title compound is δ = 38.9 p.p.m. and for the methyl compound δ = 48.0 p.p.m. (Bruker AMX 300, standard 85% H_3PO_4). The Au—C distance of 2.045 (4) Å is slightly longer than the corresponding bond in the above-mentioned CN complex [2.003 (7) Å]. Comparison with the Au—C bond distance of the CH_3 compound suffers from the high standard deviation of the reported value, 2.124 (28) Å. The only other known structure with an Au—CF_3 bond is an Au^{II} dimer complex and so the considerably longer reported distance of 2.140 (7) Å (Murray, Fackler, Porter, Briggs, Guerra & Lagow, 1987) is not directly comparable. There are no Au···Au interactions, the shortest Au···Au distance being 5.498 (1) Å, nor have any other unusual intermolecular contacts been found.

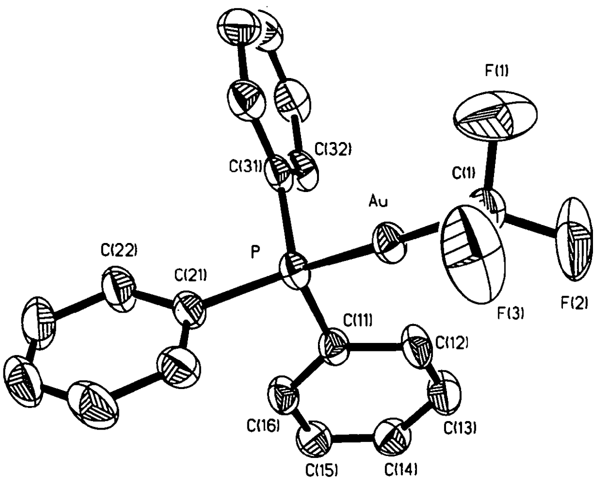


Fig. 1. Molecular structure of the title compound with H atoms omitted. Displacement ellipsoids are plotted at the 50% probability level.

Experimental

The compound was isolated as a by-product from the reaction of triphenylphosphine with $Re_2(HgCF_3)(AuPPh_3)(\mu\text{-}OC_4H_9)$ in thf solution at 296 K (Siefert, 1994).

Crystal data

$[Au(CF_3)(C_{18}H_{15}P)]$
M_r = 528.25
Monoclinic
$P2_1/c$
a = 9.1029 (14) Å
b = 11.395 (2) Å
c = 17.490 (2) Å
β = 104.953 (10)°
V = 1752.8 (4) Å^3
Z = 4
D_x = 2.002 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 59 reflections
θ = 5–12.5°
μ = 8.511 mm^{-1}
T = 173 (2) K
Prism
0.52 × 0.26 × 0.25 mm
Pale amber

Data collection

Siemens *P*4 diffractometer
ω scans
Absorption correction: ψ scans (North, Phillips & Matthews, 1968)
T_{min} = 0.682, T_{max} = 0.937
8307 measured reflections
4013 independent reflections
3266 observed reflections [$I > 2\sigma(I)$]

R_{int} = 0.0265
θ_{max} = 27.49°
$h = -10 \rightarrow 11$
$k = -14 \rightarrow 14$
$l = -21 \rightarrow 22$
3 standard reflections monitored every 247 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.0227
$wR(F^2)$ = 0.0514
S = 0.924
4013 reflections
218 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.0289P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.885 e Å^{-3}
$\Delta\rho_{min}$ = −0.757 e Å^{-3}

Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.00113 (11)
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}
Au	0.67740 (2)	0.438407 (12)	0.400805 (8)	0.03033 (6)
P	0.58455 (11)	0.26670 (8)	0.43719 (6)	0.0300 (2)
C1	0.7643 (5)	0.5897 (3)	0.3672 (2)	0.0346 (8)
F1	0.8052 (6)	0.5860 (3)	0.3002 (2)	0.116 (2)
F2	0.8875 (4)	0.6311 (3)	0.4190 (2)	0.0891 (11)
F3	0.6733 (4)	0.6819 (3)	0.3565 (3)	0.0987 (12)
C11	0.6521 (4)	0.2336 (3)	0.5414 (2)	0.0317 (8)
C12	0.8012 (4)	0.2638 (4)	0.5800 (2)	0.0419 (10)
C13	0.8636 (5)	0.2298 (4)	0.6577 (2)	0.0477 (10)
C14	0.7774 (5)	0.1645 (3)	0.6970 (2)	0.0417 (9)
C15	0.6287 (5)	0.1359 (3)	0.6596 (2)	0.0402 (9)
C16	0.5659 (4)	0.1702 (3)	0.5821 (2)	0.0367 (8)
C21	0.3790 (4)	0.2620 (3)	0.4137 (2)	0.0322 (8)
C22	0.2986 (4)	0.1582 (4)	0.3938 (3)	0.0449 (10)
C23	0.1422 (5)	0.1571 (5)	0.3795 (3)	0.0547 (12)
C24	0.0661 (5)	0.2587 (5)	0.3856 (3)	0.0566 (12)
C25	0.1436 (5)	0.3627 (5)	0.4042 (3)	0.0570 (12)
C26	0.3012 (5)	0.3656 (4)	0.4177 (2)	0.0433 (10)
C31	0.6453 (4)	0.1431 (3)	0.3872 (2)	0.0324 (8)
C32	0.7366 (5)	0.0530 (3)	0.4278 (3)	0.0397 (9)
C33	0.7860 (5)	−0.0350 (4)	0.3857 (3)	0.0462 (10)
C34	0.7474 (5)	−0.0328 (4)	0.3038 (3)	0.0534 (12)
C35	0.6600 (5)	0.0562 (4)	0.2639 (3)	0.0494 (10)
C36	0.6076 (5)	0.1445 (3)	0.3052 (2)	0.0403 (9)

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

Au—C1	2.045 (4)	P—C31	1.817 (4)
Au—P	2.2859 (9)	C1—F1	1.318 (5)
P—C11	1.806 (4)	C1—F3	1.321 (5)
P—C21	1.810 (4)	C1—F2	1.333 (5)

C1—Au—P	178.56 (11)	C11—P—Au	113.92 (12)
C11—P—C21	106.6 (2)	C21—P—Au	113.38 (13)
C11—P—C31	104.9 (2)	C31—P—Au	110.45 (12)
C21—P—C31	107.0 (2)		

Program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1991). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1330). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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$[Au(PPh_3)_2]^+[BF_4]^-$

JU-CHUN WANG

Department of Chemistry, Soochow University, Taipei, Taiwan 11102, Republic of China

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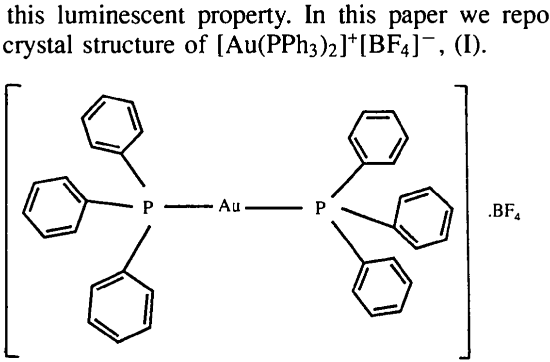
Abstract

The synthesis and structural characterization of the title compound, bis(triphenylphosphine)gold(I) tetrafluoroborate, $[Au(PPh_3)_2]^+[BF_4]^-$, are reported. The BF_4^- anion does not interact with the Au centre. The coordination geometry about Au is close to linear, with Au—P distances of 2.321 (3) and 2.322 (3) Å, and a P1—Au—P2 angle of 167.3 (1)°. The range of P—C distances is 1.787 (10)–1.823 (9) Å.

Comment

A number of mononuclear (King, Khan, Staples & Fackler, 1992, and references therein) and binuclear (Wang, Garzon, King, Wang & Fackler, 1989; King, Wang, Khan & Fackler, 1989) gold(I) compounds are luminescent. Structural information is essential for understanding this luminescent property. In this paper we report the crystal structure of $[Au(PPh_3)_2]^+[BF_4]^-$, (I).



(I)

The coordination number of the central Au atom is two, with two P atoms linearly bound to it. All four F atoms in the BF_4^- anion are disordered. Two other $[Au(PPh_3)_2]^+X^-$-type structures have been reported recently (X = NO_3, PF_6; Staples, King, Khan, Winpenny & Fackler, 1993). Fig. 1 shows a drawing of the molecule with the atom-labelling scheme.

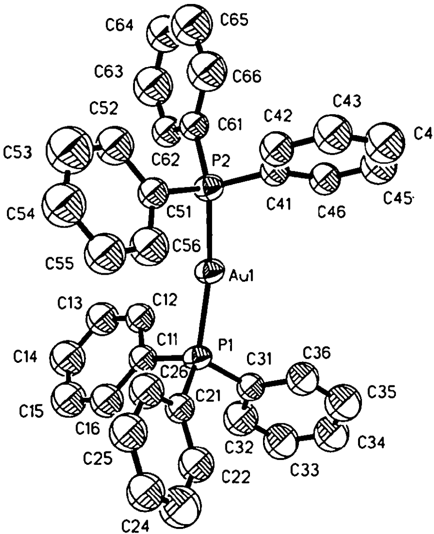


Fig. 1. Drawing (*SHELXTL-Plus*; Sheldrick, 1991) of the $[Au(PPh_3)_2]^+$ cation showing 50% probability ellipsoids for non-H atoms.

Experimental

The title compound was synthesized by reacting $[Au(PPh_3)_2]Cl$ with $AgBF_4$ in CH_2Cl_2 (DCM) and leaving overnight. After filtering off the precipitate, $[Au(PPh_3)_2]BF_4$ powders were formed by adding a large amount of diethyl ether (Et_2O) to the reaction solution. Colourless crystals of $[Au(PPh_3)_2]BF_4$ were grown from DCM/Et_2O mixed solvents.