Table 4. *Observed isotropic shifts* (p.p.m.) *and couplings* (Hz)

	δ*	$^1J(^{119}Sn–^{125}Te)$
Te(*a*)	−713	3086, 3145
Te(*b*)	−781	3030, 3161
Te(*c*)	−817	3197, 3227
Sn(*a*)	−185	3150, 3238
Sn(*b*)	−183.4	3089, 3158
Sn(*c*)	−233.6	3043, 3190

* Shifts are with respect to $(CH_3)_4Sn$ (±0.3 p.p.m.) or $(CH_3)_2Te$ (±1 p.p.m.).

Table 5. *Herzfeld–Berger chemical-shift anisotropy parameters,* $\Delta\sigma$ (p.p.m.) *and* ρ

	$\Delta\sigma$	ρ
Te(*a*)	407 ± 2	−0.12 ± 0.03
Te(*b*)	634 ± 5	−0.28 ± 0.02
Re(*c*)	602 ± 4	−0.18 ± 0.02
Sn(*a*)	800 ± 16	−0.79 ± 0.07
Sn(*b*)	792 ± 9	0.66 ± 0.03
Sn(*c*)	463 ± 7	−0.98 ± 0.05

for study by solid-state NMR, given the presence of three inequivalent Sn and Te atoms.

The ^{119}Sn and ^{125}Te solid-state NMR spectra showed the presence of three Sn resonances and three Te resonances which, in each case, were clearly resolved. The ^{119}Sn and ^{125}Te isotropic shifts and $^1J(^{119}Sn–^{125}Te)$ coupling constants are given in Table 4. The ^2J(Sn–Sn) couplings were not resolved but all lie in the range 200–270 Hz. Similarly, the ^2J(Te–Te) couplings were not resolved but lie in the range 140–160 Hz. The magnitudes of the shifts and couplings observed here are similar to those in $[(CH_3)_2SnTe]_3$(tetragonal).

From the Herzfeld–Berger chemical-shift anisotropy parameters listed in Table 5, it can be seen that Sn(*c*) has a shift tensor that is axially symmetric within the experimental e.s.d. Given the presence of a pseudo-twofold axis in the molecule it is possible that Sn(*c*) in the NMR data corresponds to Sn(*c*) in the crystal data.

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References

BATCHELOR, R. J., EINSTEIN, F. W. B. & JONES, C. H. W. (1989). *Acta Cryst.* C**45**, 1813–1815.

CARRUTHERS, J. R. & WATKIN, D. J. (1979). *Acta Cryst.* A**35**, 698–699.

GABE, E. J., LARSON, A. C., LEE, F. L. & LE PAGE, Y. (1984). *NRCVAX Crystal Structure System.* National Research Council, Ottawa, Canada.

GAY, I. D., JONES, C. H. W. & SHARMA, R. D. (1989). *J. Magn. Reson.* **84**, 501–514.

GAY, I. D., JONES, C. H. W. & SHARMA, R. D. (1990). *J. Magn. Reson.* **89**, 657–658.

HERZFELD, J. & BERGER, A. (1980). *J. Chem. Phys.* **73**, 6021–6030.

JOHNSON, C. K. (1965). *ORTEP.* Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

MEAULENAER, J. DE & TOMPA, H. (1965). *Acta Cryst.* **19**, 1014–1018.

WATKIN, D. J., CARRUTHERS, J. R. & BETTERIDGE, P. W. (1985). *CRYSTALS.* Chemical Crystallography Laboratory, Univ. of Oxford, England.

Acta Cryst. (1993). C**49**, 472–475

Structures of Bis(triphenylphosphine)gold(I) Hexafluorophosphate, $[Au(PPh_3)_2]PF_6$ (1), and Bis(triphenylphosphine)gold(I) Nitrate, $[Au(PPh_3)_2]NO_3$ (2)

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Abstract. $[Au(C_{18}H_{15}P)_2]PF_6$ (1), $M_r = 866.5$, monoclinic, $P2_1/c$, $a = 9.927$ (1) $b = 20.086$ (5), $c = 17.500$ (5) Å, $\beta = 96.72$ (1)°, $V = 3465$ (1) Å^3, $Z = 4$, $D_x = 1.66$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 44.28$ cm^{-1}, $F(000) = 1696$, $T = 295$ K, final $R = 0.0372$, $wR = 0.0380$ for 2818 observed reflections [$F_o^2 > 3\sigma(F_o^2)$]. $[Au(C_{18}H_{15}P)_2]NO_3$ (2), $M_r = 783.5$, monoclinic, $P2_1/n$, $a = 17.502$ (5), $b = 11.020$ (1), $c = 18.012$ (3) Å, $\beta = 112.40$ (2)°, $V = 3212$ (1) Å^3, $Z = 4$, $D_x = 1.62$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 47.04$ cm^{-1}, $F(000) = 1544$, $T = 295$ K, final $R = 0.0331$, $wR = 0.0380$ for 3540 observed reflections. The coordination at the Au^I centres is essentially linear with P—Au—P bond angles of 177.4 (1)° in (1) and 171.1 (2), 168.4 (1)° in (2). The nitrate anion in complex (2) does not interact with the Au^I center.

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It does appear to influence the triphenylphosphine ligands. The bond angles are displaced further from linearity in (2) than in complex (1).

Introduction. We have previously reported the bonding interaction of the nitrate group with the triphenylphosphinegold(I) cation, with Au—O 2.074 Å and P—Au—O 179.2° (Wang, Khan & Fackler, 1989). Messmer & Palenik (1969) reported the nitrate interaction with copper(I) in bis(triphenylphosphine)copper(I) nitrate, where the P—Cu—O bond angle is 126.0°. A silver(I)–oxygen interaction also has been reported for $[Ag(PPh_3)_2]NO_3$, P—Ag—O 117.2° (Barron, Dyason, Healy, Engelhardt, Skelton & White, 1986). Structural features of various bis(triphenylphosphine)gold(I) halides (halides include Cl, Br, I, SCN) have been reported to contain three-coordinate gold(I) (Bowmaker, Dyason, Healy, Engelhardt, Pakawatchai & White, 1987). In an attempt to further evaluate possible interactions associated with the anions and triphenylphosphinegold(I) centers, the structural characterization and comparison of the title complexes, bis(triphenylphosphine)gold(I) hexafluorophosphate, (1), and bis(triphenylphosphine)gold(I) nitrate, (2), was undertaken.

Experimental. Preparation of compound (1), $[Au(PPh_3)_2]PF_6$: PPh_3AuCl (100 mg, 0.202 mmol) and 1 equivalent of PPh_3 (53 mg, 0.202 mmol) were dissolved in a mixture of CH_3CN and CH_2Cl_2 (4 mL + 2 mL). $TlPF_6$ (70.6 mg, 0.202 mmol) in 2 mL CH_3CN was added. TlCl immediately precipitated. After 2.5 h the solution was centrifuged. The supernatant was removed under reduced pressure to an oil. This oil was dissolved in dry CH_2Cl_2 and solid crystallized by slow diffusion of dry diethyl ether to give colorless crystals of the title compound (1).

Preparation of compound (2), $[Au(PPh_3)_2]NO_3$: PPh_3AuCl (100 mg, 0.202 mmol) and 1 equivalent of PPh_3 (53 mg, 0.202 mmol) were dissolved in a mixture of CH_3CN and CH_2Cl_2 (4 mL + 2 mL). $TlNO_3$ (70.6 mg, 0.202 mmol) in 2 mL CH_3CN was added. TlCl immediately precipitated. After 2.5 h the solution was centrifuged. The supernatant was removed under reduced pressure to an oil. This oil was dissolved in dry CH_2Cl_2 and solid crystallized by slow diffusion of dry diethyl ether to give colorless crystals of the title compound (2).

Colorless crystals (*ca* 0.4 mm on edge) of $[Au(PPh_3)_2]PF_6$ (1) and $[Au(PPh_3)_2]NO_3$ (2) were mounted on glass fibers in a random orientation. The complexes were shown to be monoclinic on the basis of the symmetry of axial photographs and Delaunay reduction. Refined cell parameters were obtained from setting angles of 25 reflections with 2θ range of 20–30°. Data collection was carried out at room temperature using Wyckoff scanning technique in a bisecting geometry (Nicolet *R3m/E* diffractometer, graphite-monochromated Mo $K\alpha$ radiation). Backgrounds were estimated from a 96-step profile. Scan rate was variable, 2–30° min^{-1}, scan width 1.0°, scan offset 1.0°. 5048 reflections (*hkl* range −11, 0, 0 to 11, 22, 19) for complex (1) and 4627 reflections (*hkl* range 0, 0, −20 to 19, 12, 20) for complex (2) were measured with $3 < 2\theta < 45°$. Three standards were measured every 100 reflections and the data corrected for standard decay, Lorentz and polarization effects. Absorption corrections were applied empirically on the basis of azimuthal scans of five reflections [transmission range 0.693–0.932 for complex (1), 0.435–0.971 for complex (2)]. Structure solution and refinement on F were carried out using the *SHELXTL* collection of crystallographic software (Sheldrick, 1986). Scattering factors, including terms for anomalous dispersion, were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV).

The complexes (1) and (2) were found to be in the space groups $P2_1/c$ and $P2_1/n$, respectively, by systematic absences and successful refinement. Both structures were solved by the Patterson method which provided the positions of Au atoms. Remaining non-H-atom positions were determined by Fourier methods. Phenyl rings of complex (1) were refined as rigid groups with C—C 1.395 Å and C—C—C 120°. Non-H atoms were refined anisotropically. H atoms in both structures were placed in calculated positions (C—H 0.960 Å) with fixed displacement parameters ($U_{iso} = 1.2 \times U_{carbon}$). $\sum w(|F_o| - |F_c|)^2$ was minimized, where $w = [\sigma^2(F_o) + 0.00052F_o^2]^{-1}$. Final parameters for complex (1): $R = 0.0372$, $wR = 0.0380$ for 381 variables and 2818 unique observed reflections [$F_o^2 > 3\sigma(F_o^2)$], with a goodness of fit $S = 1.069$; largest shift/e.s.d. = 0.01; maximum and minimum residual electron densities in the difference Fourier map 0.41 and −0.46 e $Å^{-3}$. Final parameters for complex (2): $R = 0.0331$, $wR = 0.0355$ for 393 variables and 3540 unique observed reflections, with a goodness of fit $S = 1.457$; largest shift/e.s.d. = 0.002; maximum and minimum residual electron densities in the difference Fourier map 1.18 [located next to Au(1)] and −1.30 e $Å^{-3}$.

Discussion. Final positional and thermal parameters for (1) are presented in Table 1,* with bond lengths and angles given in Table 2. Fig. 1 shows the molecule with the atomic labeling scheme. The PF_6^-

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55642 (63 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BR1000]

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($Å^2 \times 10^3$) *for* $[Au(PPh_3)_2]PF_6$ (1)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Au	706 (1)	7214 (1)	5216 (1)	55 (1)
P(1)	−642 (3)	7915 (1)	4403 (2)	50 (1)
P(2)	2035 (3)	6477 (1)	5986 (1)	52 (1)
C(11)	−2845 (7)	8718 (3)	4646 (4)	72 (5)
C(12)	−4077 (7)	8841 (3)	4929 (4)	77 (5)
C(13)	−4692 (7)	8343 (3)	5322 (4)	76 (5)
C(14)	−4074 (7)	7721 (3)	5431 (4)	89 (6)
C(15)	−2842 (7)	7597 (3)	5148 (4)	70 (5)
C(16)	−2227 (7)	8096 (3)	4756 (4)	48 (3)
C(21)	1140 (7)	8934 (3)	4818 (3)	70 (4)
C(22)	1789 (7)	9539 (3)	4718 (3)	93 (6)
C(23)	1462 (7)	9905 (3)	4044 (3)	102 (6)
C(24)	486 (7)	9667 (3)	3471 (3)	94 (6)
C(25)	−163 (7)	9063 (3)	3571 (3)	71 (5)
C(26)	164 (7)	8696 (3)	4244 (3)	51 (4)
C(31)	4 (6)	7301 (4)	3088 (4)	76 (5)
C(32)	−273 (6)	6969 (4)	2387 (4)	93 (6)
C(33)	−1613 (6)	6871 (4)	2067 (4)	111 (7)
C(34)	−2676 (6)	7106 (4)	2449 (4)	147 (9)
C(35)	−2398 (6)	7437 (4)	3150 (4)	113 (7)
C(36)	−1058 (6)	7535 (4)	3470 (4)	56 (4)
C(41)	3040 (7)	7453 (3)	7003 (4)	66 (4)
C(42)	3616 (7)	7695 (3)	7714 (4)	86 (5)
C(43)	3785 (7)	7272 (3)	8351 (4)	83 (5)
C(44)	3378 (7)	6608 (3)	8277 (4)	82 (5)
C(45)	2802 (7)	6367 (3)	7565 (4)	67 (4)
C(46)	2632 (7)	6789 (3)	6928 (4)	53 (4)
C(51)	3507 (8)	6231 (4)	4781 (4)	79 (5)
C(52)	4661 (8)	6038 (4)	4453 (4)	108 (7)
C(53)	5844 (8)	5870 (4)	4924 (4)	121 (8)
C(54)	5874 (8)	5895 (4)	5723 (4)	124 (8)
C(55)	4719 (8)	6088 (4)	6050 (4)	78 (5)
C(56)	3536 (8)	6256 (4)	5580 (4)	59 (4)
C(61)	−236 (7)	5726 (4)	6204 (6)	166 (10)
C(62)	−918 (7)	5140 (4)	6346 (6)	200 (11)
C(63)	−217 (7)	4537 (4)	6415 (6)	127 (8)
C(64)	1167 (7)	4520 (4)	6340 (6)	153 (10)
C(65)	1849 (7)	5106 (4)	6198 (6)	133 (8)
C(66)	1147 (7)	5709 (4)	6129 (6)	60 (4)
P(3)	4048 (3)	9695 (2)	7035 (2)	87 (2)
F(1)	3374 (10)	9487 (4)	7728 (4)	212 (7)
F(2)	5114 (18)	10069 (12)	7555 (6)	237 (20)
F(3)	4919 (22)	9076 (7)	7115 (11)	177 (17)
F(4)	2980 (18)	9321 (12)	6518 (6)	338 (31)
F(5)	3178 (22)	10314 (7)	6956 (11)	196 (20)
F(6)	4719 (10)	9903 (4)	6341 (5)	212 (7)
F(2*a*)	3968 (30)	8987 (4)	6743 (10)	199 (24)
F(3*a*)	2673 (7)	9838 (15)	6600 (8)	206 (20)
F(4*a*)	4127 (30)	10404 (4)	7328 (10)	201 (21)
F(5*a*)	5422 (7)	9552 (15)	7472 (9)	256 (27)

anion is not shown. The hexafluorophosphate anion is disordered in the equatorial plane. It was modeled successfully with two sets of F atoms in the plane at 50% occupancy, with fixed P—F bond length of 1.511 Å. The bond angle within each set of four F atoms was fixed at 90° and the angle between the two sets is 47.5°. Final positional and thermal parameters for complex (2) are listed in Table 3 with bond lengths and angles given in Table 4. Fig. 2 shows the two molecules with the atomic labeling scheme, one nitrate anion is not shown for clarity. Complex (2) crystallized with the Au atom located on the twofold axis. The nitrate anion also sits along the twofold axis. This affects the thermal ellipsoids; therefore refinement of the N—O bond length was required, which gave a final value of 1.153 (1) Å.

The Au···Au separations in both complexes are quite large at 8.8255 (5) and 8.7592 (6) Å, respectively. This suggests that there is no attractive Au···Au interaction in the solid state. The phosphine ligands appear to be too bulky to allow the Au atoms to associate.

Table 2. *Bond lengths* (Å) *and angles* (°) *for* $[Au(PPh_3)_2]PF_6$ (1)

Au—P(1)	2.314 (2)	Au—P(2)	2.309 (2)
P(1)—C(16)	1.793 (7)	P(1)—C(26)	1.797 (7)
P(1)—C(36)	1.807 (8)	P(2)—C(46)	1.799 (7)
P(2)—C(56)	1.780 (9)	P(2)—C(66)	1.808 (9)
P(1)—Au—P(2)	177.4 (1)	Au—P(1)—C(16)	112.3 (2)
Au—P(1)—C(26)	113.0 (2)	C(16)—P(1)—C(26)	107.5 (3)
Au—P(1)—C(36)	110.7 (3)	C(16)—P(1)—C(36)	106.2 (3)
C(26)—P(1)—C(36)	106.8 (3)	Au—P(2)—C(46)	114.5 (2)
Au—P(2)—C(56)	112.0 (3)	C(46)—P(2)—C(56)	104.6 (3)
Au—P(2)—C(66)	111.9 (3)	C(46)—P(2)—C(66)	106.5 (4)
C(56)—P(2)—C(66)	106.7 (4)	P(1)—C(16)—C(11)	121.5 (2)
P(1)—C(16)—C(15)	118.5 (2)	P(1)—C(26)—C(21)	118.2 (2)
P(1)—C(26)—C(25)	121.8 (2)	P(1)—C(36)—C(31)	118.0 (2)
P(1)—C(36)—C(35)	121.9 (2)	P(2)—C(46)—C(41)	118.7 (2)
P(2)—C(46)—C(45)	121.1 (2)	P(2)—C(56)—C(51)	119.3 (2)
P(2)—C(56)—C(55)	120.7 (2)	P(2)—C(66)—C(61)	119.6 (3)
P(2)—C(66)—C(65)	120.4 (3)		

Table 3. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($Å^2 \times 10^3$) *for* $[Au(PPh_3)_2]NO_3$ (2)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Au(1)	2500	3109 (1)	7500	36 (1)
P(1)	2931 (2)	2945 (3)	8879 (2)	33 (1)
C(11)	2615 (8)	1503 (12)	9170 (8)	40 (5)
C(12)	2287 (10)	1393 (15)	9738 (9)	56 (7)
C(13)	2081 (5)	265 (7)	9952 (5)	79 (4)
C(14)	2192 (12)	−741 (18)	9568 (13)	84 (11)
C(15)	2509 (11)	−658 (14)	8987 (12)	71 (9)
C(16)	2737 (5)	469 (8)	8774 (5)	53 (4)
C(21)	4052 (5)	2993 (7)	9402 (4)	35 (3)
C(22)	4488 (5)	3749 (8)	9104 (5)	46 (3)
C(23)	5330 (5)	3880 (9)	9497 (5)	58 (4)
C(24)	5738 (6)	3255 (9)	10201 (6)	60 (4)
C(25)	5300 (6)	2506 (10)	10510 (6)	62 (4)
C(26)	4461 (4)	2368 (7)	10101 (4)	53 (3)
C(31)	2542 (4)	4118 (5)	9344 (4)	36 (2)
C(32)	1927 (4)	4888 (6)	8876 (4)	45 (3)
C(33)	1616 (4)	5783 (6)	9223 (4)	57 (3)
C(34)	1923 (5)	5913 (7)	10042 (4)	55 (3)
C(35)	2535 (4)	5160 (6)	10513 (4)	53 (3)
C(36)	2847 (4)	4275 (6)	10178 (3)	45 (3)
Au(2)	7500	2767 (1)	7500	48 (1)
P(2)	6087 (1)	2978 (2)	7112 (1)	44 (1)
C(41)	5519 (4)	2745 (6)	6057 (4)	44 (3)
C(42)	4900 (4)	3526 (7)	5596 (4)	63 (3)
C(43)	4438 (5)	3274 (9)	4792 (5)	83 (4)
C(44)	4603 (5)	2250 (8)	4438 (5)	77 (4)
C(45)	5239 (5)	1478 (7)	4892 (4)	71 (4)
C(46)	5685 (4)	1742 (6)	5699 (4)	58 (3)
C(51)	5843 (4)	4488 (6)	7342 (3)	43 (2)
C(52)	5183 (4)	4733 (7)	7567 (4)	56 (3)
C(53)	5018 (5)	5893 (8)	7762 (5)	68 (4)
C(54)	5525 (6)	6820 (7)	7733 (4)	70 (4)
C(55)	6179 (5)	6614 (7)	7509 (5)	71 (4)
C(56)	6339 (4)	5456 (7)	7311 (4)	60 (3)
C(61)	5632 (4)	1952 (6)	7607 (4)	47 (3)
C(62)	6082 (5)	1585 (7)	8381 (4)	64 (3)
C(63)	5725 (5)	848 (8)	8783 (5)	83 (4)
C(64)	4929 (5)	471 (7)	8417 (5)	82 (4)
C(65)	4490 (5)	799 (9)	7646 (6)	99 (5)
C(66)	4814 (5)	1545 (9)	7234 (5)	80 (4)
N(2)	7500	−390 (1)	7500	109 (6)
N(1)	2500	6425 (1)	7500	82 (5)
O(11)	2500	7472 (5)	7500	325 (19)
O(12)	3088 (3)	5900 (7)	7886 (4)	131 (4)
O(21)	7500	−1436 (5)	7500	428 (26)
O(22)	7093 (7)	148 (11)	6940 (5)	272 (9)

Table 4. *Bond lengths* (Å) *and angles* (°) *for* $[Au(PPh_3)_2]NO_3$ (2)

Au(1)—P(1)	2.312 (4)	P(1)—C(11)	1.824 (15)
P(1)—C(21)	1.825 (8)	P(1)—C(31)	1.808 (8)
C(11)—C(12)	1.355 (26)	C(11)—C(16)	1.403 (17)
C(12)—C(13)	1.390 (19)	C(13)—C(14)	1.359 (24)
C(14)—C(15)	1.363 (36)	C(15)—C(16)	1.401 (20)
C(21)—C(22)	1.370 (13)	C(21)—C(26)	1.372 (10)
C(22)—C(23)	1.377 (11)	C(23)—C(24)	1.380 (13)
C(24)—C(25)	1.380 (16)	C(25)—C(26)	1.378 (11)
C(31)—C(32)	1.378 (8)	C(31)—C(36)	1.399 (8)
C(32)—C(33)	1.384 (11)	C(33)—C(34)	1.373 (10)
C(34)—C(35)	1.365 (9)	C(35)—C(36)	1.366 (10)
Au(2)—P(2)	2.311 (2)	P(2)—C(41)	1.797 (6)
P(2)—C(51)	1.804 (7)	P(2)—C(61)	1.803 (8)
C(41)—C(42)	1.385 (9)	C(41)—C(46)	1.366 (10)
C(42)—C(43)	1.390 (10)	C(43)—C(44)	1.380 (13)
C(44)—C(45)	1.392 (11)	C(45)—C(46)	1.393 (10)
C(51)—C(52)	1.389 (11)	C(51)—C(56)	1.390 (10)
C(52)—C(53)	1.385 (12)	C(53)—C(54)	1.367 (13)
C(54)—C(55)	1.370 (15)	C(55)—C(56)	1.381 (11)
C(61)—C(62)	1.374 (9)	C(61)—C(66)	1.403 (10)
C(62)—C(63)	1.386 (13)	C(63)—C(64)	1.359 (11)
C(64)—C(65)	1.355 (12)	C(65)—C(66)	1.369 (15)
N(2)—O(21)	1.153 (5)	N(2)—O(22)	1.152 (9)
N(2)—O(22*a*)	1.152 (9)	N(1)—O(11)	1.153 (5)
N(1)—O(12)	1.154 (6)	N(1)—O(12*a*)	1.154 (6)
P(1)—Au(1)—P(1*a*)	171.1 (2)	Au(1)—P(1)—C(11)	111.4 (4)
Au(1)—P(1)—C(21)	113.5 (3)	C(11)—P(1)—C(21)	105.1 (5)
Au(1)—P(1)—C(31)	114.6 (2)	C(11)—P(1)—C(31)	106.3 (6)
C(21)—P(1)—C(31)	105.1 (3)	P(1)—C(11)—C(12)	123.8 (11)
P(1)—C(11)—C(16)	116.1 (11)	C(12)—C(11)—C(16)	120.1 (13)
C(11)—C(12)—C(13)	121.2 (14)	C(12)—C(13)—C(14)	119.1 (15)
C(13)—C(14)—C(15)	121.0 (17)	C(14)—C(15)—C(16)	120.8 (16)
C(11)—C(16)—C(15)	117.8 (14)	P(1)—C(21)—C(22)	117.7 (5)
P(1)—C(21)—C(26)	122.8 (7)	C(22)—C(21)—C(26)	119.4 (7)
C(21)—C(22)—C(23)	120.6 (8)	C(22)—C(23)—C(24)	119.9 (10)
C(23)—C(24)—C(25)	119.8 (8)	C(24)—C(25)—C(26)	119.5 (8)
C(21)—C(26)—C(25)	120.8 (8)	P(1)—C(31)—C(32)	120.1 (5)
P(1)—C(31)—C(36)	121.9 (4)	C(32)—C(31)—C(36)	118.0 (6)
C(31)—C(32)—C(33)	120.8 (6)	C(32)—C(33)—C(34)	120.0 (6)
C(33)—C(34)—C(35)	119.8 (7)	C(34)—C(35)—C(36)	120.8 (6)
C(31)—C(36)—C(35)	120.6 (5)	P(2)—Au(2)—P(2*a*)	168.4 (1)
Au(2)—P(2)—C(41)	113.7 (3)	Au(2)—P(2)—C(51)	110.1 (2)
C(41)—P(2)—C(51)	106.9 (3)	Au(2)—P(2)—C(61)	114.2 (2)
C(41)—P(2)—C(61)	105.2 (3)	C(51)—P(2)—C(61)	106.1 (3)
P(2)—C(41)—C(42)	122.5 (5)	P(2)—C(41)—C(46)	118.9 (5)
C(42)—C(41)—C(46)	118.6 (6)	C(41)—C(42)—C(43)	120.8 (7)
C(42)—C(43)—C(44)	120.3 (7)	C(43)—C(44)—C(45)	119.2 (7)
C(44)—C(45)—C(46)	119.4 (8)	C(41)—C(46)—C(45)	121.7 (6)
P(2)—C(51)—C(52)	122.9 (5)	P(2)—C(51)—C(56)	119.6 (6)
C(52)—C(51)—C(56)	117.6 (6)	C(51)—C(52)—C(53)	122.1 (7)
C(52)—C(53)—C(54)	118.6 (9)	C(53)—C(54)—C(55)	121.0 (8)
C(54)—C(55)—C(56)	120.2 (8)	C(51)—C(56)—C(55)	120.6 (8)
P(2)—C(61)—C(62)	119.7 (5)	P(2)—C(61)—C(66)	121.7 (6)
C(62)—C(61)—C(66)	118.5 (8)	C(61)—C(62)—C(63)	120.3 (7)
C(62)—C(63)—C(64)	120.6 (7)	C(63)—C(64)—C(65)	119.5 (9)
C(64)—C(65)—C(66)	121.7 (8)	C(61)—C(66)—C(65)	119.4 (7)
O(21)—N(2)—O(22)	121.0 (6)	O(21)—N(2)—O(22*a*)	121.0 (6)
O(22)—N(2)—O(22*a*)	118.1 (11)	O(11)—N(1)—O(12)	120.1 (4)
O(11)—N(1)—O(12*a*)	120.1 (4)	O(12)—N(1)—O(12*a*)	119.8 (7)

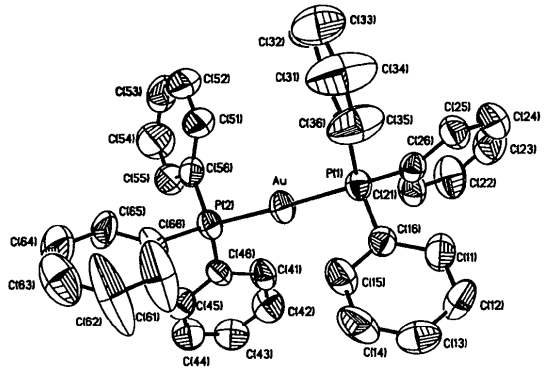

Fig. 1. A drawing showing a view of $[Au(PPh_3)_2]PF_6$, without the PF_6 anion. Thermal ellipsoids have been drawn at 50% probability.

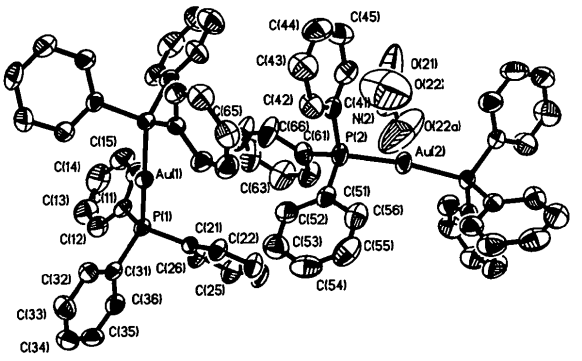

Fig. 2. A drawing showing a view of the two $[Au(PPh_3)_2]NO_3$ molecules; one nitrate anion is not shown for clarity. Thermal ellipsoids have been drawn at 50% probability.

Although the nitrate group does not appear to bond with the Au^I center in complex (2) [Au(1)—O(12) 3.23, Au(2)—O(22) 3.05 Å], the structure is still slightly distorted from linearity with P—Au—P 171.1 and 168.4°. These angles are to be compared with the value obtained for complex (1), P—Au—P 177.4°, where the PF_6^- anion shows no bonding interactions with the cation. The Au—P bond lengths in both structures are essentially indentical, 2.314 and 2.309 Å in (1) and 2.311 and 2.312 Å in (2), well within the range reported for other bis(triphenylphosphine)gold(I) complexes. The slight distortion in (2) appears to be a result of the nitrate anion interacting with the phenyl groups of the phosphine, pushing the P atom away from linearity.

The lack of a gold(I)–oxygen bonding is not entirely unexpected. These observations are further evidence of the coordination differences for these Group 11 (*Ib*) elements in their 1+ oxidation state wherein the additional ligand coordination for a bisphosphine ML_2^+ complex follows the trend Ag > Cu ≫ Au.

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References

BARRON, P. F., DYASON, J. C., HEALY, P. C., ENGELHARDT, L. M., SKELTON, B. W. & WHITE, A. H. (1986). *J. Chem. Soc. Dalton Trans.* pp. 1965–1970.

BOWMAKER, G. A., DYASON, J. C., HEALY, P. C., ENGELHARDT, L. M., PAKAWATCHAI, C. & WHITE, A. H. (1987). *J. Chem. Soc. Dalton Trans.* pp. 1089–1097.

MESSMER, G. G. & PALENIK, G. J. (1969). *Inorg. Chem.* **8**, 2750–2754.

SHELDRICK, G. M. (1986). *SHELXTL. An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data.* Nicolet XRD Corporation, Madison, Wisconsin, USA.

WANG, J.-C., KHAN, M. N. I. & FACKLER, J. P. JR (1989). *Acta Cryst.* C**45**, 1008–1010.