

mum $\Delta/\sigma = 0.041$, $\Delta\rho_{\max} = 3.63 \text{ e } \text{\AA}^{-3}$ at a distance of 0.94 \AA from the Pt atom, $\Delta\rho_{\min} = -4.61 \text{ e } \text{\AA}^{-3}$. Scattering factors for all atoms from *SHELXTL PLUS*. Atomic parameters are given in Table 1,* and selected bond distances and bond angles are listed in Table 2. Fig. 1 shows the cation together with the numbering scheme used.

Related literature. The related mononuclear compound $[\text{Pt}\{\text{t-Bu}_2\text{P}(\text{CH}_2)_3\text{P}'\text{Bu}_2\}\text{Cl}_2]$ has been structurally characterized (Harada, Kai, Yasuoka & Kasai,

1979). The expected differences associated with the Pt-ligand parameters on going to the dinuclear, Cl-bridged complex cation are observed. Thus the Pt—P and Pt—Cl distances are lengthened and the Cl—Pt—Cl angle is substantially reduced. Furthermore, the correlation between equivalence of Pt—Cl distances with equivalence of the P(1)—Pt(1)—Cl(1a) and P(2)—Pt(1)—Cl(1) angles (Robertson & Wickramasinghe, 1987) is maintained.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond distances and angles including the anion have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54792 (28 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI10109]

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Structure of Tris(dicyclohexylphenylphosphine)gold(I) Perchlorate

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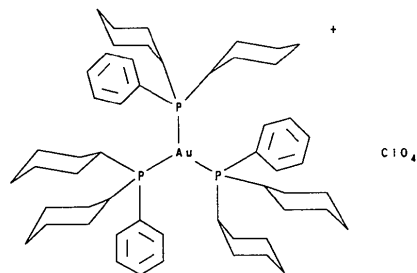
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Abstract. $[\text{Au}\{\text{P}(\text{C}_6\text{H}_{11})_2(\text{C}_6\text{H}_5)\}_3][\text{ClO}_4]$, $M_r = 1119.59$, trigonal, $R3c$, $a = 13.192(5)$, $c = 52.83(2) \text{ \AA}$, $V = 7962(9) \text{ \AA}^3$, $Z = 6$, $D_x = 1.401 \text{ g cm}^{-3}$, $\lambda(\text{Mo } K\alpha) = 0.71073 \text{ \AA}$, $\mu = 29.5 \text{ cm}^{-1}$, $F(000) = 3468$, $T = 296 \text{ K}$, final $R = 0.047$ for 1321 unique observed reflections. The Au atom in the anion and the perchlorate ion in $[\text{Au}(\text{Cy}_2\text{PhP})_3][\text{ClO}_4]$ lie on a threefold axis. The complex has an almost ideal trigonal-planar geometry, with an Au—P distance of $2.421(3) \text{ \AA}$, P—Au—P angles of $119.9(3)^\circ$, and the Au atom only $0.08(2) \text{ \AA}$ out of the plane defined by the three P atoms.

Experimental. To a beaker of 40 ml ethanol were added 0.20 mmol of $[\text{Cy}_2\text{PhPAuCl}]$, prepared following the procedure used by Sutton (Sutton, McGusty, Walz & Di Martino, 1972). A stoichiometric amount of ammonium perchlorate and a sixfold excess of Cy_2PhP (Strem) were then added under nitrogen atmosphere in a glove bag. After stirring for about 20 min the beaker was removed from the glove bag and a white solid (NH_4Cl) was filtered from solution. After some time colorless crystals formed in solution and were isolated by vacuum filtration.

Analysis for $\text{C}_{54}\text{H}_{81}\text{AuClO}_4\text{P}_3$: Calc. C 57.93, H 7.29%; found, C 58.02, H 7.29%.



Small rectangular plate, size and D_m not measured, Enraf-Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$, ω - 2θ scan, scan width $(0.9 + 0.34\tan\theta)^\circ$, $2\theta_{\max} = 57^\circ$, lattice parameters from least-squares fit of 24 reflections in the range $12 < \theta < 15^\circ$. Conditions limiting reflections hkl ($-h + k + l = 3n$); $00l$ ($l = 6n$); with $h \leq 17$, $k \leq 17$, $l \leq 71$; 3 standard reflections, $(3,4,22)$, $(4,7,22)$, $(7,4,16)$, total intensity loss 0.7% during 17.1 h exposure, total reflections measured 2768, no equivalent reflections measured, 2224 of these unique, 1321 with $F^2 > 2\sigma^2(F_o)$, $\sigma(F_o) = \{[\sigma^2(I_{\text{raw}}) + (0.04F_o^2)^2]^{1/2}/2F_o\}$.

Table 1. Atomic coordinates and equivalent isotropic temperature factors (\AA^2) (hexagonal cell) for $[\text{Au}(\text{Cy}_2\text{PhP})_3][\text{ClO}_4]$

$$B_{\text{eq}} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	x	y	z	B_{eq}
Au	1.000	1.000	1.000	2.333 (7)
P1	0.7883 (3)	0.8993 (4)	1.00165 (5)	2.39 (7)
C1	$\frac{1}{3}$	$\frac{1}{3}$	1.0489 (2)	5.3 (2)
O1	$\frac{1}{3}$	$\frac{1}{3}$	1.0766 (7)	10.0
O2	0.431 (2)	0.666 (2)	1.0450 (4)	10.0
C1	0.7215 (9)	0.994 (1)	1.0018 (5)	3.2 (2)
C2	0.623 (1)	0.966 (1)	1.0151 (4)	5.7 (5)
C3	0.574 (2)	1.041 (2)	1.0134 (5)	7.0 (6)
C4	0.619 (2)	1.131 (2)	0.9959 (5)	6.8 (5)
C5	0.720 (1)	1.158 (1)	0.9822 (4)	4.5 (4)
C6	0.762 (1)	1.081 (1)	0.9827 (3)	3.4 (4)
C7	0.711 (1)	0.799 (1)	0.9754 (3)	2.8 (3)
C8	0.578 (1)	0.735 (2)	0.9762 (4)	4.7 (5)
C9	0.523 (2)	0.656 (2)	0.9547 (4)	6.1 (6)
C10	0.564 (2)	0.707 (2)	0.9298 (4)	7.2 (7)
C11	0.700 (1)	0.770 (1)	0.9290 (3)	5.5 (4)
C12	0.757 (1)	0.849 (1)	0.9497 (3)	3.6 (4)
C13	0.728 (1)	0.812 (1)	1.0301 (3)	2.9 (3)
C14	0.753 (1)	0.708 (1)	1.0315 (3)	3.1 (3)
C15	0.710 (2)	0.643 (1)	1.0570 (3)	4.4 (5)
C16	0.764 (2)	0.721 (2)	1.0798 (3)	4.9 (5)
C17	0.739 (2)	0.821 (1)	1.0785 (3)	4.8 (5)
C18	0.785 (1)	0.894 (1)	1.0539 (3)	3.9 (3)

The atomic coordinates and equivalent isotropic temperature factors are listed in Table 1, and selected bond lengths and angles in Table 2.* An ORTEPII (Johnson, 1976) plot of the title compound with the atom numbering is shown in Fig. 1 and the packing of the molecules is shown in a PLUTO (Motherwell & Clegg, 1978) plot in Fig. 2.

Related literature. The trigonal planar geometry of the present $[\text{Au}(\text{Cy}_2\text{PhP})_3]^+$ complex is nearly ideal [Au—P 2.421 (3) Å, P—Au—P 119.9 (3)°, Au 0.09 (2) Å from the P_3 plane], while that of the related compound $[\text{AuCl}(\text{Cy}_2\text{PhP})_2]$ is severely distorted [Au—P 2.300, 2.324 Å, P—Au—P 158.24° (Muir, Cuadrado, Muir & Barnes, 1988)]. By comparison, the complex $[\text{Au}(\text{Ph}_3\text{P})_3]^+$ has close to ideal

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, full bond lengths and angles, and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54807 (30 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST0543]

Table 2. Selected bond lengths (\AA) and angles ($^\circ$) in $[\text{Au}(\text{Cy}_2\text{PhP})_3][\text{ClO}_4]$

Au—P1	2.421 (3)	P1—C1	1.85 (2)
C1—O1	1.47 (4)	P1—C7	1.83 (1)
C1—O2	1.31 (2)	P1—C13	1.82 (1)
C—C (phenyl, ave.)	1.39 (3)	C—C (cyclohex, ave.)	1.52 (2)
O1—C1—O2	99 (1)	P1—C1—C6	115 (3)
Au—P1—C1	115.9 (3)	P1—C7—C8	117 (1)
Au—P1—C7	116.2 (5)	P1—C7—C12	114.3 (8)
Au—P1—C13	113.4 (5)	P1—C13—C14	112 (1)
C1—P1—C7	102.0 (8)	P1—C13—C18	108.1 (9)
C1—P1—C13	102.7 (9)	P1—C1—C2	122 (1)
C7—P1—C13	105.0 (6)	P—Au1—P	119.9 (3)
C—C—C (phenyl, ave.)	120 (2)	C—C—C (cyclohex, ave.)	120.0 (3)

Absorption corrections from ψ scans, relative transmission factors 0.910 to 0.999. Structure was solved with the *Enraf-Nonius SDP-Plus Structure Determination Package* (Frenz, 1985), heavy-atom and difference Fourier methods; isotropic then anisotropic refinement on F of all non-H atoms, except the perchlorate O atoms which were refined with fixed temperature factors because of the presence of disorder; H atoms at calculated positions [C—H 0.95 Å, $U(\text{H}) 1.3 \times U(\text{C})$], and then treated as riding on the corresponding C atom; 181 parameters refined, $R = 0.047$, $wR = 0.057$, $w = 1/[\sigma^2(F_o)]$, $S = 1.70$; difference syntheses showed no densities above 0.92 e \AA^{-3} (min -1.45 e \AA^{-3}); max. Δ/σ 0.01. Neutral-atom scattering factors were used (*International Tables for X-ray Crystallography*, 1974, Vol. IV, Tables 2.2.8 and 2.3.1.), and were corrected for anomalous dispersion (Cromer & Liberman, 1970). No correction for secondary extinction.

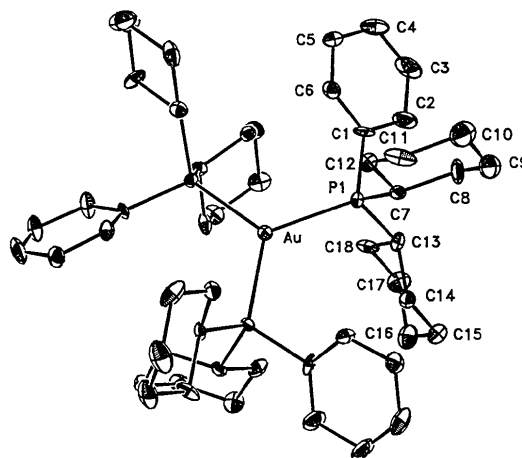


Fig. 1. Labeling of atoms in the $[\text{Au}(\text{Cy}_2\text{PhP})_3]^+$ complex (50% probability ellipsoids).

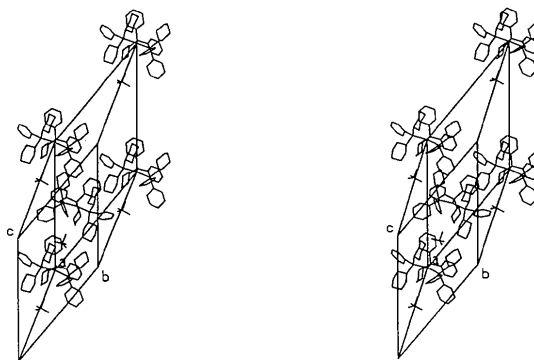


Fig. 2. Packing diagram of $[\text{Au}(\text{Cy}_2\text{PhP})_3][\text{ClO}_4]$ in the unit cell.

trigonal-planar geometry in $[\text{Au}(\text{Ph}_3\text{P})_3][\text{B}_9\text{H}_{19}\text{S}]$ [Au—P 2.345, 2.384, 2.389 Å; P—Au—P 112.3, 121.5, 124.1°, Au 0.20 Å from the P_3 plane (Guggenberger, 1974)] and in $[\text{Au}(\text{Ph}_3\text{P})_3][\text{BPh}_4]$ [Au—P 2.365, 2.384, 2.403 Å, P—Au—P 115.2, 119.3, 125.4°, Au 0.06 Å from the P_3 plane (Jones, 1980)].

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Two New Crystal Morphologies for Tetraphenyldibismuthine, Bi_2Ph_4

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Abstract. Tetraphenyldibismuthine, Bi_2Ph_4 , $M_r = 726.38$. (1) monoclinic, $P2_1/n$, $a = 11.531$ (2), $b = 5.801$ (3), $c = 16.691$ (2) Å, $\beta = 104.87$ (2)°, $V = 1079.2$ (6) Å³, $Z = 2$, $D_x = 2.24$ g cm⁻³, $\text{Mo K}\alpha$, $\lambda = 0.7107$ Å, $\mu = 162.42$ cm⁻¹, $F(000) = 660$, $T = 296$ K, $R = 0.034$ for 1468 reflections with $I > 3\sigma(I)$. Two identical pyramidal Ph_2Bi fragments, related by an inversion center, are joined with a Bi—Bi bond [2.988 (1) Å]. The average Bi—C bond distance is 2.26 Å with Bi—Bi—C_{ave} 93.6° and C—Bi—C 93.3 (4)°. (2) Tetragonal, $I4_1/a$, $a = 28.07$ (1), $c = 10.83$ (9) Å, $V = 8533$ (4) Å³, $Z = 16$, $D_x = 2.26$ g cm⁻³, $\text{Mo K}\alpha$, $\lambda = 0.7107$ Å, $\mu = 164.15$ cm⁻¹, $F(000) = 5280$, $T = 296$ K, $R = 0.038$ for 1287 reflections with $I > 3\sigma(I)$. The structure is essentially as for the monoclinic form with $d_{\text{Bi—Bi}} = 2.984$ (2) Å, $d_{\text{Bi—C(ave)}} = 2.24$ Å, Bi—Bi—C_{ave} 92.7 and C—Bi—C_{ave} 96.7°.

Experimental. Bi_2Ph_4 was obtained as the by-product of the reaction of $\text{Fe}(\text{CO})_5$ with $\text{Na}^+\text{BiPh}_2^-$ in NH_3 (1). Crystals which grew from hexane solution gave crystals with two new cell morphologies: monoclinic and tetragonal. Data on the monoclinic form were measured from a needle-like crystal of dimensions $0.1 \times 0.1 \times 0.5$ mm using a Rigaku AFC5S fully

automated single-crystal X-ray diffractometer over the range h 0 to 14, k 0 to 7, l -21 to 20. The unit cell was determined from 23 reflections over the range $9.9 \leq 2\theta \leq 17.3^\circ$ and later refined using reflections in the range $23 \leq 2\theta \leq 29^\circ$. 2849 reflections were collected of which 2720 were unique ($R_{\text{int}} = 3.4\%$) and 1468 were classified as observed [$I > 3\sigma(I)$]. The standards showed significant decay during data collection (40%). The structure was solved using the program *MITHRIL* (Gilmore, 1983) which located the Bi atoms, followed by full-matrix least squares and Fourier syntheses to find the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic U values while H atoms were included in calculated positions but not refined. The data were corrected for Lorentz-polarization effects and absorption (ψ scans, transmission range 0.8167–1.000) (*DIFABS*; Walker & Stuart, 1983). Refinement of 118 variables (Molecular Structure Corporation, 1988) was carried out on F with final $R = 0.034$, $wR = 0.040$, $S = 1.02$ and a maximum Δ/σ of 0.0003. The final difference map showed peaks $\Delta\rho_{\text{min}} = -1.56$, $\Delta\rho_{\text{max}} = 1.12$ e Å⁻³. Scattering factors were taken from *International Tables for X-ray Crystallography* (Cromer & Waber, 1974). The molecule sits on a crystallographic inversion center lying at the midpoint of the Bi—Bi bond and consequently only half of the molecule is

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