was tried first and confirmed by successful refinement. The structures were solved by direct methods and refined by full-matrix least-squares methods. All non-H atoms were refined with anisotropic temperature factors. In (I) the H atoms on the anion were found in difference maps and refined freely. In (II), all H atoms except those bonded to the C_6 ring located on the mirror plane were inserted at calculated positions. The thermal parameters of the H atoms bonded to the methyl C atoms were constrained to one value which was refined. The thermal parameters of the H atoms on the phenyl group situated off the mirror plane were constrained to one value which was refined. No attempt was made to refine H atoms in (III). A ΔF map revealed a CH_2Cl_2 molecule which was refined with anisotropic thermal parameters for the C and two Cl atoms. $wR = \{[\sum w(|F_o| - |F_c|)^2]/[\sum w(F_o)^2]\}^{1/2}$ was minimized, where $w = \sigma^2(|F_o|^2)^{-1}$ and σ is the standard deviation in a single measurement; the final R values were 0·043, 0·047 and 0·041, wR = 0·068, 0·065 and 0·062, S = 1·585, 1·490 and 1·892 for 132, 162 and 199 variables for (I), (II) and (III), respectively. The largest shift/e.s.d. in the final least-squares cycle was 0·16, 0·028 and 0·08, respectively; the maximum and minimum residual electron densities in the ΔF maps were 0·73 and −0·56, 0·30 and −0·23, and 0·60 and −0·41 e Å^{-3}, respectively.* The molecules and the atomic labeling schemes are shown in Figs. 1, 2 and 3 for complexes (I), (II) and (III), respectively. Final positional and equivalent isotropic thermal parameters are listed in Tables 1, 2 and 3 [for complexes (I), (II) and (III), respectively]; important bond distances and angles are listed in Tables 4, 5 and 6.

Related literature. The IR and electronic spectra of $[Ph_4P][CrCl_4(PR_3)_2]$, the crystal structures of $[N^nPr_4][Cr(cis\text{-}Ph_2PCHCHPPh_2)Cl_4]$ and *mer*-$[Cr\{P(CH_2CH_2PPh_2)_3\}Cl_3]$ have been reported (Bennett, Clark & Goodwin, 1970; Gray, Hale, Levason, McCullough & Webster, 1983, 1984).

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* Lists of structure factors, positional parameters [and H-bond distances and angles for (I)] for H atoms, stereoviews of the unit-cell packing, anisotropic thermal parameters, and intramolecular bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53676 (40 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Chloro(dicyclohexylphenylphosphine)gold(I)

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Abstract. $[AuCl\{P(C_6H_5)(C_6H_{11})_2\}]$, M_r = 506·8, orthorhombic, $P2_12_12_1$, a = 8·476 (5), b = 13·747 (2), c = 15·951 (3) Å, V = 1858·8 (3) Å^3, Z = 4, D_x = 1·81 g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ = 81·24 cm^{-1}, $F(000)$ = 984, T = 296 K, final R = 0·031 for 1860 unique observed reflections. The complex $Cy_2PhPAuCl$ adopts the two-coordinate linear geometry typical of Au^{I} complexes. The P—Au—Cl angle is 178·3 (1)° while Au—P and Au—Cl distances are 2·234 (2) and 2·281 (3) Å, respectively.

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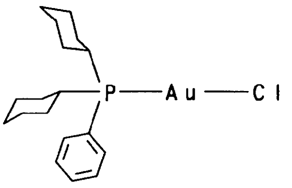
The geometry of the P atom is tetrahedral with an average Au—P—C angle of 112·1 (3)° and an average P—C distance of 1·827 (8) Å.

Experimental. The complex $Cy_2PhPAuCl$, (1), was prepared following the procedure used by Sutton for the preparation of some (alkylphosphine)gold chloride derivatives (Sutton, McGusty, Walz & DiMartino, 1972). A sample containing 1·0487 g (0·0027 mol) of tetrachloroauric acid was dissolved in 40 ml ethanol and reduced to AuCl by reaction with excess 2,2′-thiodiethanol (0·038 mol). The reaction mixture was stirred for 2 h until it became almost colorless and then a stoichiometric amount of dicyclohexylphenylphosphine (Cy_2PhP) (Strem) was added inside a glove bag (Aldrich) purged with nitrogen. The product was removed from the glove bag and isolated by vacuum filtration. The total yield was 1·2302 g (0·0024 mol, 95%). Analysis for $C_{18}H_{27}AuClP$: Calc: C, 42·66; H, 5·37%; found: C, 42·61; H, 5·37%.



(1)

Small rectangular plate, 0·3 × 0·4 × 0·5 mm, D_m not determined, Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$, ω–2θ scan, scan width $(0{\cdot}9 + 0{\cdot}34\tan\theta)°$, $2\theta_{\max} = 53°$, lattice parameters from least-squares fit of 24 reflections in the range $10 < \theta < 12°$. Systematic absences $h00$ ($h = 2n + 1$); $0k0$ ($k = 2n + 1$); $00l$ ($l = 2n + 1$) with $h \leq 10$, $k \leq 17$, $l \leq 20$; three standard reflections (423, $\bar{1}65$ and $22\bar{8}$), intensities constant within experimental error; total reflections measured 2350, 2224 of these unique, 1860 with $F^2 > 2\sigma^2(F_o)$, $\sigma(F_o) = \{[\sigma^2(I_{\rm raw}) + (0{\cdot}04F_o^2)^2]^{1/2}/2F_o\}$. Absorption corrections from ψ scans, relative transmission factors 0·7337 to 0·9992. Structure was solved with the Enraf–Nonius *Structure Determination Package* (Frenz, 1986), heavy-atom and difference-Fourier methods; isotropic then anisotropic refinement on F of all non-H atoms, H atoms at calculated positions [C—H 0·95 Å, U(H) 1·3U(C)], 190 parameters refined, $R = 0{\cdot}031$, $wR = 0{\cdot}032$, $w = 1/[\sigma^2(F_o)]$, $S = 1{\cdot}0$; difference syntheses showed no densities above 0·695 e Å^{-3} (min. −0·635 e Å^{-3}); max. $\Delta/\sigma = 0{\cdot}01$. Neutral-atom scattering factors were used [*International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2B and 2.3.1)] and were corrected for anomalous dispersion (Cromer & Liberman, 1970). No correction for secondary extinction.

Table 1. *Atomic coordinates and equivalent isotropic temperature factors* (Å^2) *for* $Cy_2PhPAuCl$

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

	x	y	z	B_{eq}
Au	0·85810 (4)	0·40008 (2)	0·77541 (2)	3·437 (5)
Cl	0·9808 (4)	0·3137 (2)	0·6722 (2)	6·88 (7)
P	0·7450 (3)	0·4862 (2)	0·8779 (1)	2·66 (4)
C1	0·581 (1)	0·5605 (6)	0·8394 (5)	3·2 (2)
C2	0·446 (1)	0·4960 (7)	0·8074 (6)	4·3 (2)
C3	0·313 (1)	0·5561 (9)	0·7700 (7)	5·4 (2)
C4	0·374 (1)	0·6254 (8)	0·7011 (6)	5·3 (2)
C5	0·505 (1)	0·6896 (7)	0·7319 (6)	4·7 (2)
C6	0·641 (1)	0·6304 (6)	0·7709 (6)	4·0 (2)
C7	0·6713 (8)	0·4117 (6)	0·9633 (5)	2·8 (2)
C8	0·730 (1)	0·3182 (6)	0·9714 (6)	4·3 (2)
C9	0·690 (1)	0·2631 (8)	1·0424 (7)	6·5 (3)
C10	0·592 (1)	0·2997 (9)	1·1023 (6)	6·4 (3)
C11	0·535 (1)	0·3927 (9)	1·0934 (6)	5·8 (2)
C12	0·569 (1)	0·4480 (7)	1·0238 (6)	3·9 (2)
C13	0·8887 (9)	0·5667 (6)	0·9286 (5)	2·9 (2)
C14	1·029 (1)	0·5069 (6)	0·9623 (6)	4·0 (2)
C15	1·148 (1)	0·5737 (8)	1·0055 (7)	5·4 (2)
C16	1·076 (1)	0·6367 (7)	1·0721 (6)	4·7 (2)
C17	0·945 (1)	0·6960 (6)	1·0372 (6)	4·5 (2)
C18	0·818 (1)	0·6327 (6)	0·9972 (6)	4·4 (2)

Table 2. *Selected bond lengths* (Å) *and angles* (°) *in* $Cy_2PhPAuCl$

Au—Cl	2·281 (3)	P—C13	1·834 (8)
Au—P	2·234 (2)	C— (Ph, ave.)	1·38 (1)
P—C1	1·831 (9)	C—C (Cy, ave.)	1·52 (1)
P—C7	1·815 (8)		

Cl—Au—P	178·3 (1)	P—C1—C6	109·6 (6)
Au—P—C1	112·1 (3)	P—C7—C8	118·0 (6)
Au—P—C7	113·4 (3)	P—C7—C12	122·2 (6)
Au—P—C13	110·9 (3)	P—C13—C14	110·1 (5)
C1—P—C7	107·8 (4)	P—C13—C18	114·3 (6)
C1—P—C13	108·4 (4)	C—C—C (Ph, ave.)	120·0 (9)
C7—P—C13	103·7 (4)	C—C—C (Cy, ave.)	111·6 (8)
P—C1—C2	110·8 (6)		

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

Related literature. The structure of the complex $Cy_2PhPAuCl$ is very similar to other two-coordinated gold(I) complexes such as Ph_3PAuCl (Baenziger, Bennett & Soboroff, 1976) and Cy_3PAuCl (Muir, Muir, Pulgar, Jones & Sheldrick, 1985). The structure of the three-coordinated

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* Lists of structure factors, anisotropic thermal factors, H-atom parameters and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53690 (29 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

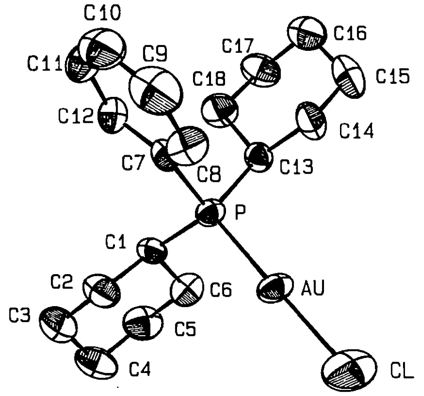

Fig. 1. Labeling of atoms in $Cy_2PhPAuCl$ (50% probability ellipsoids).

complex $(Cy_2PhP)_2AuCl$ has been determined and shows a large increase in the Au—Cl distance [2·744 (2) Å] (Muir, Cuadrado, Muir & Barnes, 1988) compared to $Cy_2PhPAuCl$ [Au—Cl 2·281 (3) Å].

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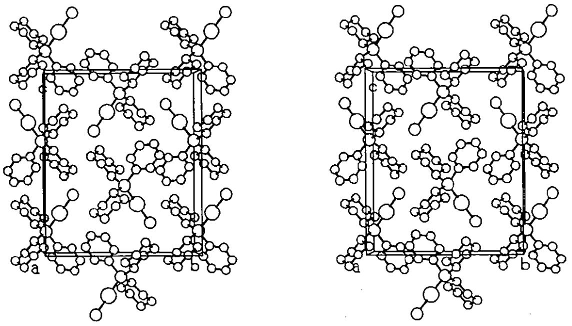

Fig. 2. Stereo packing diagram of $Cy_2PhPAuCl$ in the unit cell.

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Structure of Hexaaquadichloropraseodymium(III) Chloride

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Abstract. $[PrCl_2(H_2O)_6]Cl$, $M_r = 355·4$, monoclinic, $P2/n$, $a = 8·019$ (3), $b = 6·599$ (3), $c = 9·729$ (2) Å, $\beta = 93·75$ (2)°, $V = 513·7$ (7) Å^3, $Z = 2$, $D_x = 2·297$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 54·86$ cm^{-1}, $F(000) = 340$, room temperature, $R = 0·051$ for 1233 observed reflections. The complex is isostructural with Nd, Sm, Gd, Dy and Lu analogues. The cation has C_2 symmetry and the geometry around the eight-coordinated Pr^{III} atom, located on a twofold axis, is a square antiprism. The Pr—Cl (coordinated) distance and the average Pr—O distances are 2·834 (2) and 2·472 Å, respectively.

Experimental. The compound was crystallized from the solution in which a tetranuclear Pr^{III} complex (Xu, Lin, Wu, Xu & Chen, 1991) was synthesized. A