Table 2. *Selected bond distances* (Å) *and angles* (°)

U(1)–O(1)	1·76 (3)	U(2)–O(2)	1·74 (2)
U(1)–Cl(1)	2·66 (3)	U(2)–Cl(2)	2·65 (2)
U(1)–Cl(3)	2·83 (2)	U(2)–Cl(3)	2·81 (2)
U(1)–Cl(4)	2·80 (2)	U(2)–Cl(4)	2·83 (2)
U(1)–O(3)	2·32 (3)	U(2)–O(5)	2·49 (4)
U(1)–O(4)	2·46 (5)	U(2)–O(6)	2·36 (4)
U(1)···U(2)	4·619 (5)		
O(1)–U(1)–O(1)′	179 (1)	O(2)–U(2)–O(2)′	178 (2)

Experimental. Crystal obtained by an unexpected oxidation of bis(acetylacetonato)dichlorouranium(IV) in a THF solution; yellow crystal 0·55 × 0·35 × 0·25 mm; data collected on an Enraf–Nonius CAD-4 diffractometer using graphite-monochromated Mo $K\alpha$ radiation and ω–2θ scanning; cell parameters derived from 25 reflections ($8 < \theta < 12°$); 1354 reflections with $2 < \theta < 23°$ (h 0→10, k 0→12, l 0→24), 470 with $I > 3\sigma(I)$; three standard reflections monitored every hour showed a decay of 1·5% in 12 h; Lorentz and polarization corrections; empirical absorption correction using *DIFABS* (Walker & Stuart, 1983), transmission coefficients: min. 0·839 and max. 1·490; structure solved by the heavy-atom method and refined by full-matrix least squares (F); anisotropic thermal parameters for U and Cl; H atoms introduced in calculated positions (C—H = 0·95 Å, $B = 5$ Å^2), not refined but constrained to ride their C atoms; $R = 0{\cdot}038$, $wR = 0{\cdot}046$ ($w = 1$), $S = 2{\cdot}1$, $(\Delta/\sigma)_{\max} = 0{\cdot}03$, $\Delta\rho_{\max} = 0{\cdot}83$ e Å^{-3}; atomic scattering factors including anomalous-dispersion terms from *International Tables for X-ray Crystallography* (1974); program: Enraf–Nonius *SDP-Plus* (Frenz, 1983); PDP 11/23 Plus; Fig. 1 plotted using *ORTEP* (Johnson, 1976).

The structure of the title compound is shown in Fig. 1. Atomic parameters are given in Table 1, selected bond lengths and angles in Table 2.*

Related literature. This compound is a by-product obtained during the synthesis of [U(acac)$_2$Cl$_2$(THF)$_2$] (Doretti, Zanella, Faleschini & Faraglia, 1973).

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, all bond lengths and angles and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44016 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Mononuclear Gold(I) Complex Containing a Covalently Bound Ylide Ligand. The Structure of Chloro[methyl(methylene)diphenylphosphoranyl-*C*]gold(I)

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Abstract. [AuCl{P(CH$_2$)(CH$_3$)(C$_6$H$_5$)$_2$}], $M_r = 432{\cdot}5$, monoclinic, $P2_1/n$, $a = 15{\cdot}313$ (4), $b = 11{\cdot}726$ (3), $c = 16{\cdot}712$ (4) Å, $\beta = 103{\cdot}62$ (2)°, $V = 2916$ (1) Å^3, $Z = 8$, $D_x = 2{\cdot}03$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 103{\cdot}24$ cm^{-1}, $F(000) = 1616$, $T = 298$ K. Final $R = 0{\cdot}0452$ for 2657 unique observed reflections. The structure of a mononuclear gold(I) complex containing a covalently bound phosphonium ylide ligand is reported. The asymmetric unit contains two crystallographically independent molecules, each consisting of a dimethyldiphenylphosphonium ylide linked by a methylene group to an Au$^{\text{I}}$ center. The Au atoms are two-coordinate, linear, with a chloride ligand *trans* to the ylide group.

Experimental. The gold(I) ylide dimer, [Au(CH$_2$)$_2$P(C$_6$H$_5$)$_2$]$_2$, was prepared by a modification of the literature procedure (Schmidbaur & Franke, 1975). The mononuclear gold(I) complex, ClAu(CH$_2$)P(CH$_3$)(C$_6$H$_5$)$_2$, was prepared in good yield by introducing a

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stoichiometric amount of HCl into a solution of the dinuclear gold(I) ylide in tetrahydrofuran. Crystals suitable for X-ray analysis were obtained by recrystallization from a dichloromethane–diethyl ether solution. A colorless rectangular plate of dimensions 0·10 × 0·96 × 0·80 mm was selected and mounted in a random orientation on a glass fiber. Axial dimensions and monoclinic symmetry verified by axial rotation photographs. Refined cell parameters obtained from the setting angles of 25 high-angle reflections with $30 < 2\theta < 35°$. Non-standard setting chosen in order to avoid a cell with a highly acute angle for β. Data collection carried out at room temperature using the ω-scanning technique in bisecting geometry (Nicolet *R3m/E* diffractometer, graphite-monochromated Mo $K\alpha$ radiation), 4201 reflections ($+h \leq 16$, $|k| \leq 12$, $|l| \leq 17$) measured with $0 < 2\theta < 45°$. Scan rate variable, 2–30° min^{-1}; scan range $-1·0°$ in ω from $K\alpha_1$ to $+1·0°$ from $K\alpha_2$. Background intensities estimated from a 96-step peak profile. Three standard reflections ($01\bar{2}$, 103, 040) measured every 100 data. The data were corrected for absorption, standard variation (<4%), Lorentz and polarization effects. Absorption corrections applied empirically using azimuthal scans of eight medium-intensity reflections spanning a range of 2θ values; minimum and maximum transmission, 0·107 and 0·645 respectively. Structure determination carried out using the *SHELXTL* collection of crystallographic software (Sheldrick, 1978). Au-atom positions determined from a sharpened Patterson map; remaining non-H atoms located using difference Fourier techniques. All non-H atoms refined

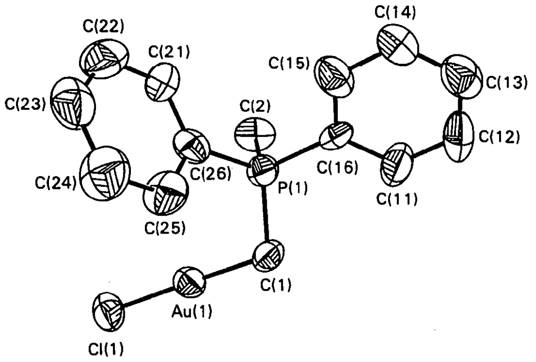

Fig. 1. A perspective view of one of the two mononuclear gold(I) ylides present in the asymmetric unit. Thermal ellipsoids have been drawn at the 50% probability level. H atoms have been omitted for clarity. The atom labels for the second complex in the asymmetric unit: Cl(2)–Au(2)–C(3); C(3) is bonded to P(2). Phenyl rings C(31)–C(36) and C(41)–C(46) are bonded to P(2) through C(36) and C(46).

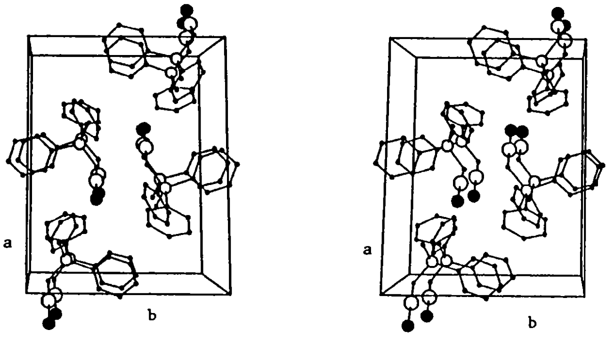

Fig. 2. A stereoview packing diagram of the unit-cell contents projected down the *c* axis. Filled circles represent Cl atoms. Large and small open circles represent Au and P atoms, respectively.

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* (Å$^2 \times 10^3$) *for* $ClAu(CH_2)PCH_3(C_6H_5)_2$

	x	y	z	U_{eq}*
Au(1)	5581 (1)	−6098 (1)	−1901 (1)	51 (1)
Au(2)	4223 (1)	−3541 (1)	−2608 (1)	50 (1)
Cl(1)	6379 (3)	−6116 (3)	−2915 (3)	69 (2)
Cl(2)	3539 (3)	−3507 (4)	−1528 (3)	75 (2)
P(1)	3980 (2)	−7033 (3)	−1246 (2)	44 (1)
P(2)	5744 (2)	−2712 (3)	−3458 (2)	40 (1)
C(1)	4890 (10)	−6060 (12)	−983 (9)	48 (5)
C(2)	3260 (10)	−6635 (13)	−2217 (8)	59 (6)
C(3)	4779 (10)	−3619 (12)	−3598 (10)	56 (6)
C(4)	3476 (10)	−6953 (13)	2493 (8)	54 (6)
C(11)	3161 (7)	−6015 (7)	−121 (6)	70 (7)
C(12)	2613	−5995	437	82 (9)
C(13)	2199	−6993	611	64 (7)
C(14)	2334	−8011	227	58 (6)
C(15)	2882	−8031	−330	64 (7)
C(16)	3296	−7033	−504	41 (5)
C(21)	3814 (6)	−9262 (9)	−1822 (7)	72 (7)
C(22)	4109	−10386	−1820	87 (9)
C(23)	4941	−10699	−1324	86 (9)
C(24)	5478	−9887	−830	97 (10)
C(25)	5183	−8762	−832	75 (8)
C(26)	4351	−8450	−1329	50 (6)
C(31)	5982 (6)	−3648 (8)	−4936 (6)	78 (8)
C(32)	6442	−3792	−5557	90 (9)
C(33)	7240	−3201	−5521	67 (7)
C(34)	7579	−2467	−4865	65 (7)
C(35)	7119	−2324	−4244	63 (6)
C(36)	6321	−2915	−4280	37 (5)
C(41)	4644 (7)	−855 (9)	−3944 (7)	91 (9)
C(42)	4421	299	−3974	122 (11)
C(43)	5008	1080	−3497	95 (10)
C(44)	5817	709	−2992	83 (9)
C(45)	6040	−445	−2962	63 (7)
C(46)	5453	−1227	−3439	43 (5)

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

Table 2. *Bond lengths* (Å) *and angles* (°) *for* $ClAu(CH_2)PCH_3(C_6H_5)_2$

Au(1)–Cl(1)	2·313 (5)	Au(1)–C(1)	2·060 (16)
Au(2)–Cl(2)	2·293 (6)	Au(2)–C(3)	2·033 (18)
P(1)–C(1)	1·774 (14)	P(1)–C(2)	1·796 (14)
P(1)–C(16)	1·803 (12)	P(1)–C(26)	1·775 (11)
P(2)–C(3)	1·792 (15)	P(2)–C(4)	1·809 (13)
P(2)–C(36)	1·816 (11)	P(2)–C(46)	1·803 (11)
C(4)–P(2)	1·809 (13)		
Cl(1)–Au(1)–C(1)	178·8 (3)	Cl(2)–Au(2)–C(3)	177·2 (4)
C(1)–P(1)–C(2)	109·6 (7)	C(1)–P(1)–C(16)	112·3 (6)
C(2)–P(1)–C(16)	106·8 (6)	C(1)–P(1)–C(26)	112·1 (6)
C(2)–P(1)–C(26)	108·3 (6)	C(3)–P(2)–C(4)	109·7 (7)
C(3)–P(2)–C(36)	111·0 (6)	C(3)–P(2)–C(46)	112·2 (6)
Au(1)–C(1)–P(1)	108·2 (7)	Au(2)–C(3)–P(2)	111·0 (8)
P(1)–C(16)–C(11)	119·3 (3)	P(1)–C(16)–C(15)	120·7 (3)
P(1)–C(26)–C(21)	122·2 (3)	P(1)–C(26)–C(25)	117·7 (3)

anisotropically. Phenyl rings refined as rigid polygons (C—C = 1·35 Å, C—C—C = 120°) using H atoms placed in idealized positions with fixed isotropic thermal parameters [U(H) = 0·08 Å^2]. Scattering factors, including terms for anomalous dispersion, taken from *International Tables for X-ray Crystallography* (1974). Refinement based on F with weights of the form $w^{-1} = [\sigma^2(F) + 0\cdot001402(F^2)]$. Convergence to conventional R values of $R = 0\cdot0452$ and $wR = 0\cdot0491$ obtained using 256 variable parameters and 2657 unique reflections with $F^2 > 3\sigma(F^2)$. One intense low-angle reflection ($\bar{2}02$) suffered from severe extinction and was therefore omitted during the refinement process. For final cycle maximum shift/σ = 0·013 with maximum and minimum residual electron densities of +0·97 and −0·91 e Å^{-3} in the vicinities of Au(1) and Au(2). Slope of normal probability plot equal to 1·08 with a goodness-of-fit indicator of 1·273. Two crystallographically independent molecules are present in the asymmetric unit. A perspective view of one is shown in Fig. 1. Atomic positional and equivalent isotropic thermal parameters for both molecules are presented in Table 1.* Bond angles and distances are summarized in Table 2. Fig. 2 shows the packing.

Related literature. The structure of the gold(I) bromide derivative has been previously reported (Porter, Knachel & Fackler, 1986). The preparation and X-ray crystal structures of mononuclear trimethylgold(III) complexes containing sulfoxonium and phosphonium ylide ligands have also been described (Fackler & Paparizos, 1977; Stein, Fackler, Paparizos & Chen, 1981).

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Structure of (Methanesulfonato)(*meso*-tetraphenylporphinato)iron(III)–Chloroform (1/1)

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Abstract. [Fe($C_{44}H_{26}N_4$)(CH_3O_3S)].$CHCl_3$, M_r = 883·06, orthorhombic, *Pbca*, a = 15·035 (5), b = 25·888 (5), c = 21·007 (4) Å, V = 8176 (6) Å^3, Z = 8, D_x = 1·435 g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ = 6·599 cm^{-1}, F(000) = 3624, T = 293 K, R = 0·075 for 1510 unique observed reflections. One O atom from $CH_3SO_3^-$ and the four pyrrole N atoms coordinate to Fe to form the five-coordinated complex. The Fe atom is displaced 0·412 (2) Å from the porphinato plane. The average Fe—N bond distance is 2·05 (2) Å, while the Fe—O distance is 1·95 (1) Å. The Fe—N bond lengths indicate that Fe is in a high-spin state.

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