and lithium succinate, 1·525 (2) Å (Klapper & Küppers, 1973). The angle O(1)—C(2)—O(2) of 123·3 (4)° and the distances O(1)—C(2), 1·262 (5), and C(2)—O(2), 1·265 (5) Å, show that the two oxygen atoms are both ionized. The crystal structure determination shows that Na⁺ is coordinated by six O atoms (Fig. 1), five from the water molecules and one from the succinate ion, with distances ranging from 2·346 (4) to 2·563 (4) Å; the bond distances and angles given in Table 2 show that there is a significant distortion from an ideal octahedron. An extended hydrogen-bond system (Fig. 1, Table 2) is present in the crystal. The succinate ions are linked through the water molecules and the Na⁺ ion, giving rise to a strong three-dimensional network.

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Structure of a Mononuclear Gold(I) Complex Containing a Covalently Bound Ylide Ligand

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Abstract. Bromo[methyl(methylene)diphenylphosporanyl-C]gold(I), [AuBr{P(CH₂)(CH₃)(C₆H₅)₂}], M_r = 491·1, monoclinic, $P2_1/n$, $a = 15·746$ (4), $b = 11·930$ (4), $c = 16·564$ (5) Å, $\beta = 105·10$ (2)°, $V = 3004$ (2) Å³, $Z = 8$, $D_x = 2·17$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0·71073$ Å, $\mu = 12·496$ mm⁻¹, $F(000) = 1824$, $T = 298$ K, $R = 0·0477$ and $wR = 0·0489$ for 3081 reflections with $F_o^2 > 3\sigma(F_o^2)$. 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 diphenyldimethylphosphonium ylide linked by a methylene group to an Auᴵ center. The Au atoms are two-coordinate linear, with a Br ligand trans to the ylide group.

Introduction. Ylides have long been regarded as highly versatile and useful synthetic reagents. However, only relatively recently has the coordination chemistry of ylides become well established. The dipolar nature of

ylides enables these compounds to form exceptionally strong metal–carbon bonds. As a result, organometallic species containing ylide ligands are often quite stable, and examples can currently be found for many of the transition-metal series of elements (Schmidbaur, 1978, 1983; Kaska, 1983).

The gold(I) ylide dimer first reported by Schmidbaur & Franke (1975) has been shown to have an extensive reaction chemistry. In many instances the chemistry parallels that observed in other dimeric complexes containing bridging bidentate phosphine ligands. Examples include two-center two-electron oxidative–addition reactions leading to Auᴵᴵ products with discrete metal–metal bonds (Murray, Fackler & Mazany, 1984), as well as the formation of a molecular Auᴵᴵᴵ A-frame species containing bridging methylene groups (Murray, Mazany & Fackler, 1985). In a limited number of instances isomerization reactions have been observed (Dudis & Fackler, 1985). The predominant reaction products obtained, however, are in general those which do not involve any changes in the basic binuclear configuration.

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In this paper we report the preparation and crystal structure of a novel cleavage product obtained from the reaction of the bis[methyl(methylene)diphenylphosphoranyl-C]digold(I) ylide dimer with acetyl bromide.

Experimental. Gold(I) ylide dimer prepared by a modification of the literature procedure (Schmidbaur & Franke, 1975). Gold ylide monomer Au^I(CH$_2$)P(CH$_3$)-(C$_6$H$_6$)$_2$Br resulted from addition of a slight excess of neat acetyl bromide to Au^I ylide dimer in tetrahydrofuran following crystallization from dichloromethane–diethyl ether solution. Colorless regularly shaped plate of approximate dimensions 0·1 × 0·2 × 0·04 mm. Refined cell parameters from setting angles of 20 high-angle reflections with $25 < 2\theta < 30°$. Monoclinic symmetry and axial dimensions confirmed by axial rotation photographs. Data collection at room temperature using θ–2θ scanning technique in bisecting geometry (Nicolet $R3m/E$ diffractometer, graphite-monochromated Mo $K\alpha$ radiation). Intensities ($\pm h$, $+k$, $+l$; $h_{max} = 18$, $k_{max} = 14$, $l_{max} = 19$) measured for 5814 unique reflections with $0 < 2\theta < 45°$. Scan rate variable 4–30° min^{-1}, scan range $-1·0°$ in 2θ from $K\alpha_1$ to $+1·0°$ from $K\alpha_2$. Background intensities from a 96-step peak profile. Three check reflections ($20\bar{2}$, $\bar{3}2\bar{2}$, 320) monitored every 100 data. Data corrected for absorption, standard decay (<3%), Lorentz and polarization effects. Absorption corrections applied empirically on basis of azimuthal scans of 15 low-angle reflections. Min. and max. transmission 0·018 and 0·064 respectively. Structure determination using *SHELXTL* collection of crystallographic software implemented on a Data General Eclipse minicomputer (Sheldrick, 1978). Au-atom positions from sharpened Patterson map; remaining non-hydrogen atoms located using difference Fourier techniques. All atoms refined anisotropically; H atoms not included. Scattering factors,

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($\mathring{A}^2 \times 10^3$) *for* BrAu-(CH$_2$)PCH$_3$(C$_6H_5$)$_2$

	x	y	z	U^*
Au(1)	581 (1)	1090 (1)	3077 (1)	57 (1)
Au(2)	4226 (1)	6546 (1)	7354 (1)	57 (1)
Br(1)	1363 (1)	1094 (1)	1994 (1)	72 (1)
Br(2)	3567 (1)	6598 (1)	8519 (1)	77 (1)
P(1)	−974 (3)	1991 (3)	3730 (2)	54 (1)
P(2)	5711 (2)	7338 (3)	6518 (2)	53 (1)
C(1)	−74 (9)	1080 (11)	4013 (9)	57 (5)
C(2)	4763 (9)	6452 (14)	6339 (9)	65 (6)
C(3)	−1696 (10)	1562 (13)	2738 (9)	70 (6)
C(4)	6471 (9)	7040 (12)	7521 (8)	60 (5)
C(16)	−1611 (9)	2003 (11)	4473 (8)	54 (5)
C(15)	−1735 (12)	997 (13)	4864 (10)	74 (7)
C(14)	−2320 (13)	937 (16)	5410 (10)	84 (8)
C(13)	−2714 (10)	1963 (18)	5573 (9)	72 (7)
C(12)	−2590 (10)	2946 (14)	5198 (11)	69 (7)
C(11)	−2056 (9)	2991 (13)	4659 (10)	67 (6)
C(26)	6297 (9)	7082 (11)	5732 (8)	53 (5)
C(25)	7113 (11)	7623 (14)	5777 (10)	75 (7)
C(24)	7562 (11)	7454 (17)	5207 (11)	86 (8)
C(23)	7226 (13)	6679 (15)	4547 (11)	85 (8)
C(22)	6404 (13)	6152 (15)	4464 (10)	84 (8)
C(21)	5971 (12)	6373 (13)	5051 (11)	78 (7)
C(36)	−625 (9)	3403 (12)	3653 (10)	58 (6)
C(35)	147 (12)	3743 (14)	4170 (12)	86 (8)
C(34)	473 (14)	4864 (15)	4165 (15)	109 (11)
C(33)	−54 (14)	5629 (16)	3630 (14)	96 (10)
C(32)	−840 (15)	5286 (14)	3117 (14)	99 (10)
C(31)	−1147 (11)	4167 (15)	3076 (12)	90 (8)
C(46)	5427 (9)	8808 (11)	6485 (8)	54 (5)
C(45)	4621 (14)	9191 (14)	5991 (11)	102 (9)
C(44)	4433 (15)	10311 (17)	5967 (15)	118 (11)
C(43)	5017 (17)	11066 (16)	6405 (15)	104 (11)
C(42)	5805 (13)	10678 (15)	6918 (14)	94 (9)
C(41)	6037 (10)	9550 (12)	6976 (11)	70 (7)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

including terms for anomalous dispersion, from *International Tables for X-ray Crystallography* (1974). Refinement based on F with $w^{-1} = [\sigma^2(F) + 0·0018(F^2)]$. Convergence to conventional R values of $R = 0·0477$ and $wR = 0·0489$ using 3081 unique reflections with $F_o^2 > 3\sigma(F_o^2)$ and 261 variable parameters. For final cycle max. shift/$\sigma = -0·119$. Min. and max. residual electron density of $+1·21$ and $-1·73$ e $\mathring{A}^{-3}$ on final difference Fourier map.

Discussion. The asymmetric unit of this structure consists of two crystallographically independent molecules. The structures of both molecules are essentially the same, therefore a perspective drawing of only one of them is shown here (Fig. 1). Atomic positional and equivalent isotropic thermal parameters for both molecules are presented in Table 1.* Intramolecular bond angles and distances are summarized in Table 2. A packing diagram of the cell viewed down the b axis is shown in Fig. 2.
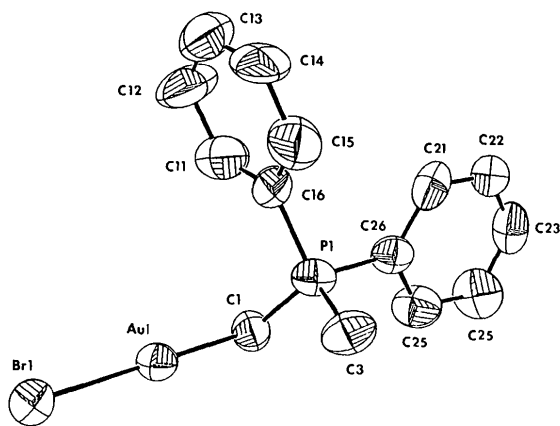

Fig. 1. A perspective view of one of the monomeric gold(I) ylide complexes illustrating the atomic numbering scheme. Thermal ellipsoids are drawn at the 50% probability level.

The overall structures of the two independent molecules display features typical of organometallic gold(I) complexes (Muir, Muir, Pulgar, Jones & Sheldrick, 1985). The gold centers are two-coordinate linear, and each forms bonds to one bromine atom and a methylene carbon of the ylide ligand. The Br—Au—C atoms are collinear within 2° [Br(1)—Au(1)—C(1)

Table 2. *Bond lengths (Å) and angles (°) for* BrAu-(CH₂)PCH₃(C₆H₅)₂

Au(1)–Br(1)	2·426 (2)	Au(1)–C(1)	2·073 (17)
Au(2)–Br(2)	2·418 (2)	Au(2)–C(2)	2·071 (16)
P(1)–C(1)	1·750 (14)	P(1)–C(3)	1·811 (14)
P(1)–C(16)	1·781 (16)	P(1)–C(36)	1·787 (15)
P(2)–C(2)	1·790 (15)	P(2)–C(4)	1·812 (12)
P(2)–C(26)	1·808 (16)	P(2)–C(46)	1·806 (14)
C(16)–C(15)	1·400 (21)	C(16)–C(11)	1·444 (22)
C(15)–C(14)	1·452 (28)	C(14)–C(13)	1·429 (28)
C(13)–C(12)	1·365 (26)	C(12)–C(11)	1·377 (26)
C(26)–C(25)	1·423 (22)	C(26)–C(21)	1·396 (20)
C(25)–C(24)	1·334 (28)	C(24)–C(23)	1·424 (25)
C(23)–C(22)	1·412 (28)	C(22)–C(21)	1·351 (29)
C(36)–C(35)	1·353 (21)	C(36)–C(31)	1·418 (22)
C(35)–C(34)	1·434 (26)	C(34)–C(33)	1·386 (28)
C(33)–C(32)	1·369 (29)	C(32)–C(31)	1·415 (25)
C(46)–C(45)	1·396 (23)	C(46)–C(41)	1·400 (19)
C(45)–C(44)	1·367 (27)	C(44)–C(43)	1·354 (30)
C(43)–C(42)	1·388 (29)	C(42)–C(41)	1·391 (23)
Br(1)–Au(1)–C(1)	179·3 (4)	Br(2)–Au(2)–C(2)	178·0 (4)
C(1)–P(1)–C(3)	110·0 (7)	C(1)–P(1)–C(16)	112·5 (7)
C(3)–P(1)–C(16)	107·2 (7)	C(1)–P(1)–C(36)	111·2 (7)
C(3)–P(1)–C(36)	109·5 (7)	C(16)–P(1)–C(36)	106·3 (7)
C(2)–P(2)–C(4)	111·0 (7)	C(2)–P(2)–C(26)	109·9 (7)
C(4)–P(2)–C(26)	106·7 (7)	C(2)–P(2)–C(46)	112·3 (7)
C(4)–P(2)–C(46)	108·5 (6)	C(26)–P(2)–C(46)	108·1 (7)
Au(1)–C(1)–P(1)	108·8 (7)	Au(2)–C(2)–P(2)	109·4 (7)
P(1)–C(16)–C(15)	118·9 (12)	P(1)–C(16)–C(11)	123·1 (11)
C(15)–C(16)–C(11)	117·9 (15)	C(16)–C(15)–C(14)	121·1 (15)
C(15)–C(14)–C(13)	116·9 (16)	C(14)–C(13)–C(12)	122·0 (16)
C(13)–C(12)–C(11)	120·8 (16)	C(16)–C(11)–C(12)	121·2 (15)
P(2)–C(26)–C(25)	120·6 (11)	P(2)–C(26)–C(21)	122·4 (12)
C(25)–C(26)–C(21)	117·0 (15)	C(26)–C(25)–C(24)	122·1 (15)
C(25)–C(24)–C(23)	118·8 (17)	C(24)–C(23)–C(22)	120·8 (19)
C(23)–C(22)–C(21)	117·6 (16)	C(26)–C(21)–C(22)	123·5 (17)
P(1)–C(36)–C(35)	118·6 (11)	P(1)–C(36)–C(31)	121·3 (11)
C(35)–C(36)–C(31)	120·1 (14)	C(36)–C(35)–C(34)	122·7 (16)
C(35)–C(34)–C(33)	117·5 (17)	C(34)–C(33)–C(32)	119·6 (18)
C(33)–C(32)–C(31)	123·6 (17)	C(36)–C(31)–C(32)	116·3 (15)
P(2)–C(46)–C(45)	121·0 (11)	P(2)–C(46)–C(41)	118·0 (10)
C(45)–C(46)–C(41)	121·0 (13)	C(46)–C(45)–C(44)	119·6 (16)
C(45)–C(44)–C(43)	121·5 (20)	C(44)–C(43)–C(42)	118·7 (18)
C(43)–C(42)–C(41)	122·8 (17)	C(46)–C(41)–C(42)	116·3 (14)
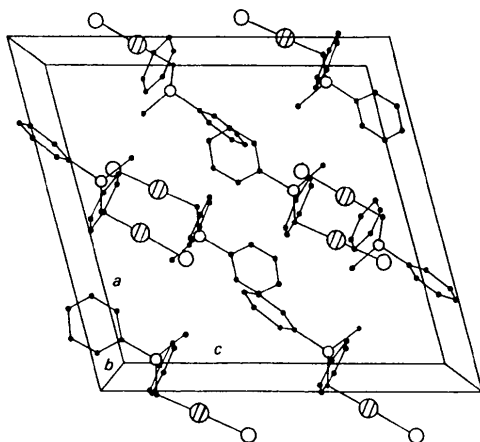
Fig. 2. A packing diagram showing the cell contents projected down the *b* axis.

= 179·3 (4); Br(2)—Au(2)—C(2) = 178·0 (4)°] with Au—Br bond lengths of 2·426 (2) and 2·418 (2) Å for Au(1)—Br(1) and Au(2)—Br(2), respectively. Bonds to the methylene carbons for the two independent molecules are 2·073 (17) and 2·071 (16) Å, with respective Au—CH₂—P bond angles of 108·8 (7) and 109·4 (7)°.

The phosphorus centers in the two independent molecules have tetrahedral coordination geometries and form bonds to methyl, methylene and phenyl carbons. For both adducts the deviation from ideal tetrahedral geometry is minimal; the average C—P—C bond angle is 109·4 (7)°, with an average deviation of less than 3°. The phosphorus–carbon lengths range from 1·750 (14) to 1·812 (12) Å; bonds to methylene C atoms are, on the average, shorter by 0·011 (14) Å compared with bonds to either methyl or phenyl C atoms. The pattern of bond lengths associated with the phosphorus centers in the structures of the two independent molecules reported here is similar to those observed in other dinuclear gold(II) and gold(III) complexes. In phosphorus ylides, both stabilized and unstabilized, a short phosphorus–methylene bond provides some structural evidence in support of the dipolar nature of these compounds (Bart, 1968, 1969).

The structure of the adduct described here is similar to a series of trimethylgold(III) sulfoxonium and phosphonium ylides (Fackler & Paparizos, 1977; Stein, Fackler, Paparizos & Chen, 1981), and most closely resembles a copper(I) chloride adduct containing a methanetetraylhexaphenyldiphosphorane double ylide (Schmidbaur, Zybill, Muller & Kruger, 1983). The title complex is isostructural with a chloride derivative prepared using HCl, and has also been identified as the major product produced from the direct reaction of the Au¹ ylide dimer with HBr (Heinrich & Knachel, 1985).

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Structure of the First Example of an Organometallic Dinuclear Gold(II) Complex Possessing Bonds to Oxygen

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Abstract. Bis(benzoato-O)bis-μ-(dimethylenediphenyl-phosphoranyl-C,C')-digold(II)(Au–Au)–tetrahydrofuran (1/1), [Au$_2$(C$_7$H$_5$O$_2$)$_2${P(CH$_2$)$_2$(C$_6$H$_5$)$_2$}$_2$].C$_4$H$_8$O, $M_r = 1134\cdot8$, triclinic, P$\bar{1}$, $a = 14\cdot464$ (7), $b = 15\cdot927$ (8), $c = 10\cdot853$ (4) Å, $\alpha = 99\cdot88$ (3), $\beta = 110\cdot95$ (3), $\gamma = 63\cdot60$ (3)°, $V = 2081$ (1) Å^3, $Z = 2$, $D_x = 1\cdot81$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 7\cdot146$ mm^{-1}, $F(000) = 1100$, $T = 298$ K, $R = 0\cdot0495$ and $wR = 0\cdot0472$ for 2986 reflections with $F_o^2 > 3\sigma(F_o^2)$. The structure consists of discrete dinuclear gold(II) ylide dimers containing unidentate benzoate ligands. The gold atoms have square-planar coordination geometries and are symmetrically bridged by ylide anion ligands. The asymmetric unit contains two crystallographically independent half-dimers and a molecule of solvent. In each dimer a metal–metal bond is present.

Introduction. The emergence of phosphorus ylides as ligands capable of forming strong metal–carbon bonds has led to the recent preparation of a large number of novel ylide coordination compounds. Examples of complexes containing ylide ligands can currently be found for most of the transition-metal series of elements, including several main-group and lanthanide elements (Schmidbaur, 1975, 1983; Kaska, 1983). Because of the importance of organometallic complexes in homogeneous catalysis, both the structures and the chemical reactivities of ylide complexes are of considerable interest.

The dimeric gold(I) ylide [Au(CH$_2$)$_2$P(C$_6$H$_5$)$_2$]$_2$ has been shown to have an extensive reaction chemistry. The chemistry, in general, is characterized by

oxidative–addition reactions of the type frequently observed in binuclear organometallic complexes containing bridging bidentate phosphine ligands. Examples include two-center two-electron oxidative–addition reactions leading to dinuclear gold(II) products with discrete metal–metal bonds (Fackler & Basil, 1984) as well as the formation of molecular gold(III) A-frame species containing bridging methylene groups (Murray, Mazany & Fackler, 1985). Less frequently observed reactions include isomerizations (Dudis & Fackler, 1985) and cleavage reactions leading to mononuclear products (Porter, Knachel & Fackler, 1986).

As part of an on-going project aimed at exploring the reaction chemistry of dinuclear gold ylides, we recently examined the reaction of the dinuclear gold(I) ylide with benzoyl peroxide. In this paper we report the crystal structure of a dinuclear gold(II) ylide complex containing oxygen-bound carboxylate ligands and an exceptionally short gold–gold bond.

Experimental. The Au^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 benzoate adduct was obtained by adding an approximately equimolar amount of benzoyl peroxide (MCB Manufacturing Chemicals, Inc.) to a solution of the gold(I) dimer in benzene. Crystals suitable for X-ray analysis were obtained by crystallization from a tetrahydrofuran/diethyl ether solution. Single multifaceted red crystal of approximate dimensions 0·3 × 0·3 × 0·25 mm. Triclinic symmetry suggested on basis of interaxial angles and confirmed by Delaunay reduction. Axial lengths checked by comparison with interlayer spacings observed in axial photographs. Refined cell parameters from setting angles of 20 reflections with

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