Table 3. *Interatomic distances* (Å) *in* [Cu(*pats*)(O*Ac*)]$_2$ *and related compounds*

Compound[a]	[Cu(pats)-(OAc)]$_2$	Cu(dtc)$_2$	Cu(OAc)$_2$	Cu(dmg)$_2$	[Cu(mspd)-(OAc)]$_2$
Reference	This work	(1)	(2)	(3)	(4,5)
Cu···Cu	3·442 (3)	3·38	2·62		3·38
Cu···O	2·422 (2)		1·969	2·45	2·51
	1·946 (2)				2·83
Cu···N	2·052 (2)[b]			1·94[c]	
	1·969 (2)				
Cu···S	2·268 (1)	2·32		2·30	
		2·71		2·31	

(a) dtc = dialkyldithiocarbamato; dmg = dimethylglyoximato; mspd = *N*-methyl-*N*'-salicylidene-1,3-propanediaminato. (b) Cu–N(pyridyl) bond. (c) Mean bond length.

References: (1) Pignedoli & Peyronel (1962); (2) Brown & Chidamabaram (1973); (3) Frasson *et al.* (1959); (4) Hämäläinen *et al.* (1982); (5) Takii *et al.* (1979).

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Structure of a Dinuclear Gold(II) Ylide Complex Containing a Gold–Selenium Bond

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Abstract. [{Au(CH$_2$)$_2$P(C$_6$H$_5$)$_2$}$_2$(Cl)(C$_6$H$_5$Se)], M_r = 1011·9, triclinic, $P\bar{1}$, a = 11·966 (5), b = 14·970 (4), c = 9·466 (3) Å, α = 98·37 (2), β = 97·50 (3), γ = 100·41 (3)°, V = 1628·4 (9) Å^3, D_x = 2·064 Mg m^{-3}, Z = 2, μ = 10·29 mm^{-1}, λ(Mo $K\alpha$) = 0·71073 Å, $F(000)$ = 952, T = 298 K. The X-ray crystal structure of the first gold(III) complex possessing bonds to selenium, benzeneselenolato[bis-μ-(dimethylene)-diphenylphosphoranyl-*C,C*']chlorodigold(II), is described. The asymmetric unit consists of one complete dinuclear gold(II) ylide bridged symmetrically by ylide anion ligands. The gold centers have square-planar coordination geometries and are linked by a 2·655 (1) Å metal–metal bond. Each Au atom forms bonds to two methylene carbons of the ylide ligands, with one bonded to a chloride ligand, the other to the Se atom of a phenyl selenide group. Convergence to

conventional R values of R = 0·0342 and wR = 0·0366 was obtained using 361 variable parameters and 3037 reflections with $F > 3\sigma(F)$.

Introduction. We recently reported the structures of two novel organometallic complexes containing pairs of dinuclear Au^{II} ylide dimers linked together by four- and five-atom polysulfide bridges (Fackler & Porter, 1986). The unusual structures of these large twelve- and thirteen-atom polysulfide rings containing Au^{II} centers piqued our interest in preparing and characterizing other dinuclear Au^{II} ylide complexes containing chalcogenide ligands. As part of this investigation, we examined the reaction of diphenyl diselenide, (C$_6$H$_5$)$_2$-Se$_2$, with the dinuclear Au^{I} ylide, [Au(CH$_2$)P(C$_6$-H$_5$)$_2$]$_2$. Previous work has shown that this complex readily undergoes two-center two-electron oxidative-addition reactions in the presence of a number of halide and alkyl halide substrates leading to the formation of

dinuclear Au^{II} products containing discrete metal–metal bonds (Fackler & Basil, 1984), Au^{III} A-frame species containing bridging methylene groups (Murray, Mazany & Fackler, 1985), as well as mixed valent Au$^{I/III}$ complexes containing chelating ylide anion ligands (Dudis & Fackler, 1985). In a limited number of instances isomerization reactions have been observed (Murray, Porter & Fackler, 1986), as well as cleavage reactions leading to the production of mononuclear products (Porter, Knachel & Fackler, 1986). The coordination chemistry of ylides has been reviewed, and examples of ylide coordination compounds can currently be found for many of the transition metal series of elements, as well as several main group and lanthanide elements (Schmidbaur, 1975, 1985; Kaska, 1983).

The structures and properties of gold complexes containing sulfur ligands have recently become the focus of a considerable amount of attention since several of these systems have been shown to be useful in the treatment of arthritis (crysotherapy). In contrast, relatively little is known about the structures of gold complexes possessing bonds to selenium, although selenium itself is of considerable significance in biological systems (Klayman & Guenther, 1973). In this paper we report the preparation and X-ray crystal structure of a dinuclear Au^{II} ylide phenyl selenide complex, the first example of an Au^{II} complex containing a covalent bond to selenium.

Experimental. The dinuclear Au^I ylide [Au(CH₂)₂P(C₆H₅)₂]₂, was prepared by a modification of the literature procedure (Schmidbaur & Franke, 1975). The Au^{II} diphenyl selenide complex [Au(CH₂)₂P(C₆H₅)₂]₂(C₆H₅)₂Se₂, was prepared by adding an equimolar amount of (C₆H₅)₂Se₂ to the Au^I dimer in THF. The title compound was obtained following recrystallization of the bis(phenyl selenide) adduct from a chloroform/diethyl ether solution. Single red rectangular plate of dimensions 0·350 × 0·201 × 0·034 mm bounded by the faces (100), (010), (001), (1̄00), (01̄0), (001̄) mounted approximately on the (010) face on the end of a glass fiber. Triclinic symmetry suggested on the basis of interaxial angles and confirmed by a Delaunay reduction. Refined cell parameters obtained from the setting angles of 25 reflections with $30 < 2\theta < 35°$. Data collection carried out at room temperature on a Nicolet $R3m/E$ diffractometer (graphite-monochromated Mo $K\alpha$ radiation) using the ω-scanning technique in bisecting geometry. Scan rate variable, 2–30° min⁻¹; scan range −1·0° in ω from $K\alpha_1$ to +1·0° from $K\alpha_2$. Intensities measured for 4357 unique reflections with $|h| \leq 12$, $|k| \leq 15$, $l \leq 10$, measured with $0 \leq 2\theta \leq 45°$. Three low-angle standards (21̄1, 02̄2, 001) measured every 100 data. Backgrounds estimated for a 96-step peak profile. The data were corrected for standard variation (<2%), absorption, Lorentz and

polarization effects. Absorption corrections applied using a Gaussian quadrature method following careful measurement of crystal dimensions and assignment of indices to the crystal faces (minimum and maximum transmission factors 0·244 and 0·858). Structure solution and refinement 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 anisotropically; H atoms not included. Scattering factors, including terms for anomalous dispersion, taken from *International Tables for X-ray Crystallography* (1974). Refinement based on F; weights of the form $w^{-1} = [\sigma^2(F) + 0\cdot00068(F^2)]$. Convergence to conventional R values of $R = 0\cdot0342$ and $wR = 0\cdot0366$ obtained using 361 variable parameters and 3037 reflections with $F > 3\sigma(F)$. For final cycle, maximum shift/$\sigma = 0\cdot019$. Difference Fourier map calculated after last cycle of refinement essentially featureless with minimum and maximum residual electron densities of +1·09 and −1·13 e Å⁻³ in the vicinities of Au(1) and Au(2) respectively.
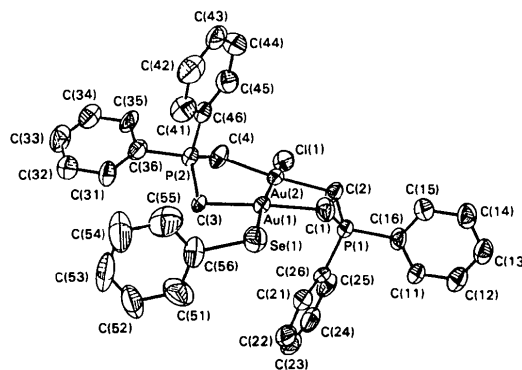

Fig. 1. A perspective view of the structure of the title compound illustrating the atomic numbering scheme. Thermal ellipsoids have been drawn at the 50% probability level.
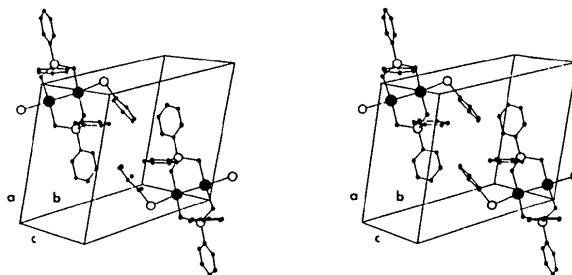

Fig. 2. A stereoview of packing diagram. Gold atoms are shown as filled circles; phosphorus, chlorine and selenium atoms as open circles.

Discussion. The unit cell of this complex contains two complete dinuclear Au^{II} dimers and displays no unusual intermolecular contacts. A perspective drawing of the structure illustrating the atomic numbering scheme is shown in Fig. 1. A stereoview packing diagram is shown in Fig. 2. Atomic positional and equivalent isotropic thermal parameters are presented in Table 1.* Bond distance and angles are summarized in Table 2.

The overall structure of this complex is that of an eight-membered heterocyclic ring in a chair conformation with bonds at axial positions to chloride and phenyl selenide ligands. The Au^{II} atoms have square-planar coordination geometries and are bridged symmetrically by the ylide anion ligands. The Au^{II} atoms are separated by a distance of 2·636 (1) Å, consistent with the presence of two Au^{II} centers linked by a single metal–metal bond. Interaxial angles around the two Au atoms in this structure range from a minimum of

83·1 (4) to a maximum of 92·9 (3)°, with bonds to methylene carbons ranging from 2·097 (11) to 2·123 (11) Å. The Se–Au–Au–Cl atoms in this complex are essentially collinear, with bonds to Se(1) and Cl(1) that measure 2·469 (2) and 2·476 (3) Å respectively.

The P centers in the bridging ylide have tetrahedral coordination geometries and form bonds to both methylene and phenyl carbons. For both atoms the deviation from ideal tetrahedral geometry is minimal; the average C–P–C bond angle measures 110·0° with a mean variation of less than 4°. P–C bond lengths in these groups range from 1·771 (13) to 1·846 (13) Å, with bonds to phenyl groups that are, on the average, slightly longer than those to methylene carbons (Bart, 1968, 1969).

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43283 (28 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic coordinates* $(\times 10^4)$ *and isotropic thermal parameters* $(Å^2 \times 10^3)$

	x	y	z	U_{eq}*
Au(1)	1179 (1)	2771 (1)	454 (1)	36 (1)
Au(2)	1522 (1)	1161 (1)	−743 (1)	38 (1)
Se(1)	662 (1)	4242 (1)	1401 (2)	59 (1)
Cl(1)	1916 (3)	−256 (2)	−2114 (4)	58 (1)
P(1)	−945 (3)	1629 (2)	−1752 (3)	41 (1)
P(2)	3637 (3)	2485 (2)	1543 (3)	40 (1)
C(1)	−611 (10)	2295 (9)	51 (12)	49 (5)
C(2)	−235 (10)	691 (8)	−1685 (13)	49 (5)
C(3)	3274 (10)	1478 (9)	200 (12)	51 (5)
C(4)	2947 (9)	3348 (8)	875 (12)	42 (4)
C(11)	−3076 (10)	1334 (9)	−3662 (13)	54 (5)
C(12)	−4261 (11)	1065 (10)	−3960 (15)	70 (6)
C(13)	−4898 (10)	734 (9)	−2940 (15)	55 (5)
C(14)	−4349 (10)	676 (9)	−1586 (15)	58 (6)
C(15)	−3149 (10)	952 (9)	−1232 (13)	51 (5)
C(16)	−2495 (9)	1285 (8)	−2279 (12)	44 (4)
C(21)	−199 (10)	3269 (9)	−2733 (13)	55 (5)
C(22)	178 (11)	3783 (11)	−3778 (15)	66 (6)
C(23)	281 (12)	3308 (13)	−5127 (17)	73 (7)
C(24)	22 (11)	2359 (12)	−5472 (15)	71 (7)
C(25)	−351 (10)	1816 (10)	−4447 (12)	55 (5)
C(26)	−423 (9)	2299 (8)	−3075 (12)	44 (5)
C(31)	5671 (11)	3860 (11)	2226 (15)	70 (6)
C(32)	6859 (12)	4154 (11)	2628 (16)	73 (7)
C(33)	7557 (11)	3494 (12)	2808 (15)	71 (7)
C(34)	7097 (12)	2556 (11)	2619 (14)	63 (6)
C(35)	5884 (9)	2243 (10)	2221 (13)	54 (5)
C(36)	5209 (10)	2899 (9)	2050 (12)	48 (5)
C(41)	3301 (12)	3000 (10)	4308 (15)	67 (6)
C(42)	3000 (13)	2812 (13)	5687 (15)	78 (7)
C(43)	2632 (11)	1922 (13)	5896 (16)	75 (7)
C(44)	2515 (13)	1161 (12)	4746 (18)	76 (7)
C(45)	2786 (11)	1345 (10)	3401 (15)	61 (6)
C(46)	3166 (9)	2241 (7)	3200 (11)	37 (4)
C(51)	2400 (15)	5669 (12)	804 (17)	89 (8)
C(52)	3415 (13)	6391 (11)	1228 (22)	89 (8)
C(53)	3979 (13)	6558 (13)	2633 (23)	100 (9)
C(54)	3640 (15)	6072 (15)	3667 (19)	106 (9)
C(55)	2649 (16)	5403 (12)	3346 (15)	91 (8)
C(56)	2041 (11)	5189 (10)	1913 (15)	65 (6)

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

Table 2. *Bond lengths* (Å) *and angles* (°)

Au(1)–Au(2)	2·636 (1)	Au(1)–Se(1)	2·469 (2)
Au(1)–C(1)	2·097 (11)	Au(1)–C(4)	2·097 (10)
Au(2)–Cl(1)	2·476 (3)	Au(2)–C(2)	2·123 (11)
Au(2)–C(3)	2·108 (11)	Se(1)–C(56)	1·927 (12)
P(1)–C(1)	1·802 (11)	P(1)–C(2)	1·771 (13)
P(1)–C(16)	1·815 (11)	P(1)–C(26)	1·819 (13)
P(2)–C(3)	1·772 (11)	P(2)–C(4)	1·800 (13)
P(2)–C(36)	1·846 (11)	P(2)–C(46)	1·798 (11)
C(11)–C(12)	1·381 (17)	C(11)–C(16)	1·417 (16)
C(12)–C(13)	1·398 (21)	C(13)–C(14)	1·383 (20)
C(14)–C(15)	1·401 (17)	C(15)–C(16)	1·432 (18)
C(21)–C(22)	1·409 (21)	C(21)–C(26)	1·409 (18)
C(22)–C(23)	1·399 (21)	C(23)–C(24)	1·379 (25)
C(24)–C(25)	1·419 (21)	C(25)–C(26)	1·413 (16)
C(31)–C(32)	1·394 (18)	C(31)–C(36)	1·423 (20)
C(32)–C(33)	1·419 (24)	C(33)–C(34)	1·389 (23)
C(34)–C(35)	1·425 (17)	C(35)–C(36)	1·393 (20)
C(41)–C(42)	1·451 (22)	C(41)–C(46)	1·400 (17)
C(42)–C(43)	1·376 (27)	C(43)–C(44)	1·429 (23)
C(44)–C(45)	1·407 (23)	C(45)–C(46)	1·384 (18)
C(51)–C(52)	1·441 (21)	C(51)–C(56)	1·424 (23)
C(52)–C(53)	1·377 (27)	C(53)–C(54)	1·362 (30)
C(54)–C(55)	1·374 (24)	C(55)–C(56)	1·416 (19)
Au(2)–Au(1)–Se(1)	174·0 (1)	Au(2)–Au(1)–C(1)	91·6 (3)
Se(1)–Au(1)–C(1)	83·1 (4)	Au(2)–Au(1)–C(4)	92·4 (3)
Se(1)–Au(1)–C(4)	92·8 (3)	C(1)–Au(1)–C(4)	175·7 (5)
Au(1)–Au(2)–Cl(1)	173·0 (1)	Au(1)–Au(2)–C(2)	92·9 (3)
Cl(1)–Au(2)–C(2)	87·8 (3)	Au(1)–Au(2)–C(3)	92·5 (3)
Cl(1)–Au(2)–C(3)	87·2 (3)	C(2)–Au(2)–C(3)	173·8 (5)
Au(1)–Se(1)–C(56)	108·9 (5)	C(1)–P(1)–C(2)	105·6 (6)
C(1)–P(1)–C(16)	109·6 (6)	C(2)–P(1)–C(16)	113·9 (5)
C(1)–P(1)–C(26)	111·7 (5)	C(2)–P(1)–C(26)	109·6 (6)
C(16)–P(1)–C(26)	106·5 (5)	C(3)–P(2)–C(4)	107·9 (5)
C(3)–P(2)–C(36)	112·3 (6)	C(4)–P(2)–C(36)	111·7 (6)
C(3)–P(2)–C(46)	110·4 (5)	C(4)–P(2)–C(46)	110·4 (6)
C(36)–P(2)–C(46)	104·1 (5)	Au(1)–C(1)–P(1)	106·2 (6)
Au(2)–C(2)–P(1)	110·9 (5)	Au(2)–C(3)–P(2)	113·2 (7)
Au(1)–C(4)–P(2)	105·8 (5)	C(12)–C(11)–C(16)	119·1 (12)
C(11)–C(12)–C(13)	121·6 (12)	C(12)–C(13)–C(14)	120·3 (11)
C(13)–C(14)–C(15)	120·0 (13)	C(14)–C(15)–C(16)	119·8 (11)
C(11)–C(16)–C(15)	119·2 (10)	P(1)–C(16)–C(15)	117·5 (8)
C(21)–C(22)–C(23)	118·6 (14)	C(22)–C(21)–C(26)	118·6 (12)
C(23)–C(24)–C(25)	120·7 (13)	C(22)–C(23)–C(24)	122·5 (16)
P(1)–C(26)–C(21)	119·0 (9)	C(24)–C(25)–C(26)	116·4 (13)
C(21)–C(26)–C(25)	123·0 (12)	P(1)–C(26)–C(25)	117·8 (9)
C(31)–C(32)–C(33)	119·5 (14)	C(32)–C(31)–C(36)	117·7 (14)
C(33)–C(34)–C(35)	118·9 (14)	C(32)–C(33)–C(34)	122·3 (12)
P(2)–C(36)–C(31)	118·9 (10)	C(34)–C(35)–C(36)	118·2 (12)
C(31)–C(36)–C(35)	123·4 (11)	P(2)–C(36)–C(35)	117·7 (9)
C(41)–C(42)–C(43)	121·0 (14)	C(42)–C(41)–C(46)	117·0 (13)
C(43)–C(44)–C(45)	118·4 (15)	C(42)–C(43)–C(44)	120·6 (15)
P(2)–C(46)–C(41)	116·1 (9)	C(44)–C(45)–C(46)	120·8 (12)
C(41)–C(46)–C(45)	122·2 (11)	P(2)–C(46)–C(45)	121·5 (9)
C(51)–C(52)–C(53)	119·2 (17)	C(52)–C(51)–C(56)	116·1 (14)
C(53)–C(54)–C(55)	119·7 (15)	C(52)–C(53)–C(54)	123·8 (15)
Se(1)–C(56)–C(51)	117·3 (10)	C(54)–C(55)–C(56)	119·1 (16)
C(51)–C(56)–C(55)	122·0 (13)	Se(1)–C(56)–C(55)	120·6 (12)

The Au—Cl bond length in the complex described here is considerably longer than the 2·388 (8) Å Au—Cl bond length reported in the structure of the dinuclear Au^{II} complex $[Au(CH_2)_2P(C_6H_5)_2]_2Cl_2$, but is almost identical with the 2·466 (3) Å Au—Cl bond length observed in the structure of $[Au(CH_2)_2P(C_6H_5)_2]_2$-$(Cl)(CCl_3)$, a dinuclear Au^{II} complex containing a chloride ligand *trans* to a trichloromethyl group (Murray, Fackler, Porter & Mazany, 1986). The Au—Se bond in the complex reported here is shorter by 0·08 (3) Å than the 2·551 (3) Å Au—Se bond length observed in the centrosymmetric $[Au(CH_2)_2P(C_6$-$H_5)_2]_2(C_6H_5)_2Se_2$ structure (Porter, Kahn & Fackler, 1986); however, it is much longer than the 2·371 (2) Å Au—Se bond length observed in the structure of $[Au(Cl)\{P(C_6H_5)_3Se\}]$, a mononuclear Au^I complex containing a neutral selenium ligand (Hussain, 1986). Information concerning the structures of Au^{III} complexes possessing bonds to selenium is limited, although the preparation of Au^{III} complexes containing bridging SCN^- and $SeCN^-$ ligands has been described (Stocco, Stocco, Scovell & Tobias, 1971; Schmidbaur & Dash, 1973). Accurate bond length data for Au^{III}—Se complexes, however, remain scarce and the crystal structures of these complexes have not yet been reported.

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3,3-Bis(trifluormethyl)-2,2-bis(triphenylphosphan)-1-thia-2λ⁴-palladacyclopropan

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Abstract. $C_{39}H_{30}F_6P_2PdS$, $M_r = 812·72$, monoclinic, $P2_1/c$, $a = 9·443$ (2), $b = 19·925$ (4), $c = 18·903$ (3) Å, $\beta = 95·34$ (2)°, $V = 3541·2$ Å³, $Z = 4$, $D_x = 1·525$ Mg m⁻³, $\lambda(Mo\,K\alpha) = 0·71069$ Å, $\mu = 0·72$ mm⁻¹, $F(000) = 1640$, room temperature (293 K), $R = 0·0385$ for 3608 observed reflections. The bonding in the three-membered M—(CS) ring ($M = $ metal) cannot be adequately described by a single-valence-bond structure. The structure shows a slight lengthening of the C=S double bond and a weak *trans* influence of the $C(CF_3)_2S$ ligand.

Einleitung. Bei bisher bekannten M—(CS)-Dreiring-Komplexen (Miki, Kai, Yasuoka & Kasai, 1981; Mason & Rae, 1970) lassen sich die Bindungsverhältnisse des Ringes kaum eindeutig beschreiben. Nach Untersuchung des $[Pt\{C(CF_3)_2S\}(PPh_3)_2]$-Komplexes (Jones, Roesky, Gries, Meyer-Bäse & Sheldrick, 1986)