The disordered perchlorate anion is hydrogen bonded to the apical methanol molecule [2.830 (5) Å for O(23)···O(1)].

The shortest intermolecular Cu···Cu^{i} distance is 6.433 (3) Å [symmetry code: (i) = $-x$, $-y$, $-z$].

Close examples to the structure that we report herein are the complexes $[Cu(terpy)(NO_2)(H_2O)]NO_3.H_2O$ (Savariault, Rojo, Arriortúa & Galy, 1983), $[Cu(dien)(HCO_2)]HCO_2$ (Davey & Stephens, 1971) and $[Cu(dien)(CH_3COO)]ClO_4$ (Towle, Hoffmann, Hatfield, Singh & Chaudhuri, 1988), where dien is diethylenetriamine. The Cu environment is also tetragonally distorted octahedral in these three complexes and three of the four equatorial positions are filled by the N atoms of the tridenate terpy and dien ligands. Formate and acetate act as bridges in an *anti-syn* fashion between Cu^{II} ions yielding one-dimensional chains of metal ions. The aqua complex is monomeric: the nitrite anion acts as an asymmetrically bidentate ligand, its two O atoms occupying one equatorial and one axial position, the remaining axial site being filled by a water molecule. In the title complex the nitrate and methanol play the role of the nitrite and water, respectively. The presence of the semicoordinated CH_3OH and water molecules in $[Cu(terpy)(CH_3OH)(NO_3)]ClO_4$ and $[Cu(terpy)(NO_2)(H_2O)]NO_3.H_2O$ block the one-dimensional μ-bridging found in $[Cu(dien)(HCO_2)]HCO_2$ and $[Cu(dien)(CH_3COO)]ClO_4$. The structures of the nitrito and nitrato complexes show that it is possible to grow single crystals of closely related complexes by using mixed counterions.

Financial support from the Comisión Interministerial de Ciencia y Tecnología, Spain (Project PB88-0490), is gratefully acknowledged. One of us (RR) thanks the Consellería de Cultura, Educación i Ciència de la Generalitat Valenciana for a grant.

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Acta Cryst. (1992). C**48**, 2114–2116

Chloro[diphenyl(diphenylphosphino-κ*P*-methyl)phosphine selenide]gold(I) Dichloromethane Hemisolvate at 178 K

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(*Received* 14 *November* 1991; *accepted* 7 *April* 1992)

Abstract. $[AuCl(C_{25}H_{22}P_2Se)].\frac{1}{2}CH_2Cl_2$, $M_r = 738.2$, monoclinic, $P2_1/c$, $a = 9.790$ (3), $b = 18.050$ (4), $c = 15.118$ (4) Å, $\beta = 105.86$ (3)°, $V = 2570$ Å^3, $Z = 4$, $D_x = 1.91$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 7.49$ mm^{-1}, $F(000) = 1412$, $T = 178$ K. The structure was refined to $R = 0.046$ for 3339 unique observed

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0108-2701/92/122114-03$06.00

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement coefficients* ($\text{Å}^2 \times 10^4$)

$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
Au	7228.1 (3)	1786.8 (2)	6974.7 (2)	274 (1)
Se	3828 (1)	1882.5 (6)	5926.4 (7)	380 (4)
Cl	7024 (2)	1176 (1)	8276 (2)	315 (8)
P(1)	7617 (2)	2351 (1)	5758 (2)	234 (7)
P(2)	4379 (2)	2264 (1)	4755 (2)	269 (8)
C(1)	6024 (8)	2806 (5)	5048 (6)	266 (20)
C(12)	8078 (6)	967 (3)	5075 (4)	354 (22)
C(13)	8509	488	4479	509 (29)
C(14)	9090	772	3804	509 (28)
C(15)	9240	1536	3725	448 (26)
C(16)	8809	2015	4320	349 (22)
C(11)	8228	1731	4996	245 (18)
C(22)	8616 (5)	3826 (3)	5744 (4)	352 (22)
C(23)	9669	4368	5976	469 (27)
C(24)	11036	4180	6492	449 (26)
C(25)	11351	3449	6777	448 (26)
C(26)	10297	2907	6545	375 (23)
C(21)	8930	3096	6029	238 (19)
C(32)	4178 (6)	814 (3)	4119 (4)	404 (24)
C(33)	4372	241	3546	494 (29)
C(34)	5012	384	2845	467 (27)
C(35)	5456	1101	2716	450 (26)
C(36)	5261	1674	3289	318 (22)
C(31)	4622	1531	3990	296 (20)
C(42)	3326 (5)	3249 (3)	3277 (4)	352 (21)
C(43)	2330	3746	2763	412 (24)
C(44)	1098	3902	3023	370 (23)
C(45)	864	3562	3797	432 (25)
C(46)	1860	3065	4311	379 (24)
C(41)	3092	2909	4051	300 (20)
Cl(0)	3990 (3)	439 (2)	333 (2)	648 (13)
C(0)†	5313 (20)	−133 (11)	649 (14)	403 (49)

† Site occupancy 0.5.

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

Au—Cl	2.311 (3)	Au—P(1)	2.224 (3)
Se—P(2)	2.104 (3)	P(1)—C(1)	1.828 (8)
P(1)—C(11)	1.821 (7)	P(1)—C(21)	1.828 (5)
P(2)—C(1)	1.832 (8)	P(2)—C(31)	1.816 (7)
P(2)—C(41)	1.827 (6)		

Cl—Au—P(1)	175.2 (1)	Au—P(1)—C(1)	112.3 (3)
Au—P(1)—C(11)	113.7 (2)	C(1)—P(1)—C(11)	106.2 (4)
Au—P(1)—C(21)	114.8 (2)	C(1)—P(1)—C(21)	103.6 (3)
C(11)—P(1)—C(21)	105.1 (3)	Se—P(2)—C(1)	112.5 (3)
Se—P(2)—C(31)	113.9 (2)	C(1)—P(2)—C(31)	106.5 (4)
Se—P(2)—C(41)	113.8 (2)	C(1)—P(2)—C(41)	102.8 (3)
C(31)—P(2)—C(41)	106.5 (3)	P(1)—C(1)—P(2)	117.0 (5)
P(1)—C(11)—C(12)	119.5 (2)	P(1)—C(11)—C(16)	120.5 (2)
P(1)—C(21)—C(22)	122.9 (2)	P(1)—C(21)—C(26)	117.1 (2)
P(2)—C(31)—C(32)	119.2 (2)	P(2)—C(31)—C(36)	120.8 (2)
P(2)—C(41)—C(42)	121.5 (2)	P(2)—C(41)—C(46)	118.5 (2)

reflections. The title compound crystallizes with two disordered dichloromethane molecules per unit cell. The coordination geometry at Au is linear. A distorted tetrahedral geometry is observed at P. The P substituents, Se and Au^{I}Cl, display a *cis* configuration with a short non-bonded Au···Se contact of 3.277 Å.

Introduction. Our recent studies of Au^I complexes have involved structural characterizations of complexes with Au—Se bonds (Jones & Thöne, 1990, 1991*a*,*b*,*c*, 1992). Some of these Au^I–Se complexes display short non-bonded intermolecular Au···Se contacts. We have begun to study the role of such interactions as a function of the steric demands of the ligands.

We have described the preparation of the title compound elsewhere (Thöne, 1991). However, a prior method has appeared in the literature (Schmidbaur, Ebner von Eschenbach, Krumberger & Müller, 1990). Single crystals were obtained directly from dichloromethane/diethyl ether and were mounted in inert oil because of ready loss of solvent.

Experimental. A colourless crystal 0.38 × 0.52 × 0.12 mm was used to record 8336 intensities on a Nicolet–Siemens four-circle diffractometer with an LT-2 low-temperature device (monochromated Mo $K\alpha$ radiation, $2\theta_{max} = 50°$, hemisphere $\pm h \pm k - l$ and some $+l$ equivalents). Three check reflections showed no significant intensity change. An absorption correction based on ψ scans was applied; transmissions ranged from 0.37 to 1.00. Merging equivalents gave 4516 unique reflections ($R_{int} = 0.050$, index ranges h −11 to +11, k −20 to +21, l −17 to +6), 3356 of which with $F > 4\sigma(F)$ were used for all calculations performed with program system Siemens *SHELXTL-Plus* (Sheldrick, 1987). Cell constants were refined from setting angles of 50 reflections in the range 20–23°.

The structure was solved by the heavy-atom method and refined anisotropically (Au, Se, P, Cl) on F to $R = 0.046$, $wR = 0.055$. A solvent molecule disordered over a symmetry centre was identified. Idealized phenyl groups were employed. Methylene H atoms were included in the refinement with a riding model. The weighting scheme was $w^{-1} = \sigma(F) + 0.0008F^2$. 111 parameters; $S = 1.2$; max. shift/e.s.d. = 0.001; max. residual electron density within ± 2.2 e Å^{-3} near Au. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Final atomic coordinates are given in Table 1,* with derived bond lengths and angles in Table 2.

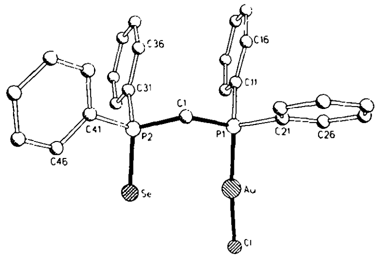

Fig. 1. Molecular structure of title compound with arbitrary radii and H atoms omitted.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55369 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA0101]

Discussion. The current structure (Fig. 1) consists of the two differently substituted organophosphorus fragments $Ph_2P(Se)CH_2$— and $Ph_2P(AuCl)CH_2$—. The fragments are comparable to the known structures of [Se(dppm)] (Colton, Hoskins & Panagiotidou, 1987) and [($AuCl_2$)(dppm)] (Schmidbaur, Wohlleben, Wagner, Orama & Huttner, 1977), respectively [dppm = bis(diphenylphosphino)methane]. The two substituents are mutually *cis* [torsion angle Se—P(2)···P(1)—Au = 8°]. In the first fragment, the P—Se bond length is 2.104 (3) Å. This value is identical with the observed P—Se bond length in [Se(dppm)]. Similar values were found in the crystal structures of Ph_3PSe (Codding & Kerr, 1979) and Me_3PSe (Cogne, Grand, Langier, Robert & Wiesenfeld, 1980). They are typical of a P═Se bond length. The Au—Cl and Au—P bond lengths in the second fragment [2.311 (3) and 2.224 (3) Å] are comparable with those in [$AuCl_2$(dppm)] [2.288 (1) and 2.238 (1) Å]. The Au—Cl bond is somewhat longer than the typical range 2.26–2.29 Å found in many chlorogold complexes (Jones, 1981, 1983, 1986). The Au atom displays the usual linear geometry with only slight deviations [175.2 (1)°]. The title molecule displays an intramolecular non-bonded Au–Se distance of 3.277 (1) Å. Comparable intermolecular non-bonded Au–Se distances are found in [$Ph_3PAuSePh$] [3.460 (1) and 3.381 (1) Å (Jones & Thöne, 1990)], $[(Ph_3PAu)_2Se(p\text{-}C_6F_4Cl)]^+.SbF_6^-$ [3.516 (1) and 3.616 (1) Å (Jones & Thöne, 1992)] and $[Me_2PhPSeAuPPh_3]^+.SbF_6^-$ [3.353 (1) Å (Jones & Thöne, 1991*b*)]. These weak interactions probably play a significant role in the crystal packing.

We thank the Fonds der Chemischen Industrie for financial support.

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Acta Cryst. (1992). C**48**, 2116–2120

Structures of (L-Methionyl-L-methioninato)dimethyltin(IV) and (L-Alanyl-L-histidinato)dimethyltin(IV). A Class of Potential Antitumour Agents

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(*Received* 13 *March* 1991; *accepted* 3 *March* 1992)

Abstract. $C_{12}H_{24}N_2O_3S_2Sn$ [Me_2Sn(MetMet)], (I), $M_r = 427.17$, orthorhombic, $P2_12_12_1$, $a = 15.022$ (2), $b = 12.558$ (2), $c = 9.417$ (2) Å, $V = 1776.5$ (3) Å^3, $Z = 4$, $D_x = 1.60$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 15.44$ cm^{-1}, $F(000) = 864$, $T = 293$ K, $R = 0.032$ for 2243 unique reflections. $C_{11}H_{18}N_4O_3Sn.CH_3OH$ [Me_2Sn(AlaHis) MeOH solvate], (II), $M_r = 405.02$, orthorhombic, $P2_12_12_1$, $a = 14.486$ (2), $b = 12.928$ (2), $c = 8.812$ (1) Å, $V = 1650.3$ (3) Å^3, $Z = 4$, $D_x = 1.63$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, μ =

0108-2701/92/122116-05\$06.00