

Fig. 2. The central region of  $[(\eta^5\text{-C}_5\text{H}_5)_2\text{Mo}_2(\mu_4\text{-Te})(\mu_3\text{-Te})_2\text{Co}_2(\text{CO})_8]$  viewed along the Mo—Mo vector; the Cp rings are deleted to enhance clarity.

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## A Dinuclear Gold(II) Ylide Complex Possessing Chloride Ligands and Bridging Methylenethiophosphinate Groups

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**Abstract.** (Dichloro)bis( $\mu$ -methylenediphenylthiophosphinato-*C,S*)digold(II)-dichloromethane (1/1),  $[\text{Au}_2\{\text{CH}_2\text{P}(\text{S})(\text{C}_6\text{H}_5)_2\text{Cl}_2\}]\cdot\text{CH}_2\text{Cl}_2$ ,  $M_r = 1012.3$ , triclinic,  $P\bar{1}$ ,  $a = 9.477(2)$ ,  $b = 12.477(3)$ ,  $c = 14.170(3)$  Å,  $\alpha = 105.55(2)$ ,  $\beta = 100.01(1)$ ,  $\gamma = 101.39(2)^\circ$ ,  $V = 1536.0(5)$  Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 2.189$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 10.38$  mm<sup>-1</sup>,  $F(000) = 952$ ,  $T = 298$  K. The crystal structure of a dinuclear gold(II) methylenethiophosphinate ylide complex containing chloride ligands is reported. The structure consists of an eight-membered heterocyclic ring system that contains Au, S, P and C nuclei in a chair conformation. The Au atoms have square-planar coordination geometries and each form bonds to a second Au center, one Cl<sup>-</sup> ligand, an S atom from one of the bridging ylide ligands and a methylene C from the other. A metal–metal bond is present and the two Au centers are separated by a distance of 2.553(1) Å. The asymmetric unit of this structure consists of one complete dimer and a molecule of dichloromethane. Final conventional  $R$  values of  $R = 0.0409$  and  $wR = 0.0431$  were obtained using 286 variable parameters and 2857 reflections with  $F_o^2 > 3\sigma(F_o^2)$ .

**Experimental.** Orange rectangularly shaped plate of approximate dimensions 0.50 × 0.20 × 0.10 mm obtained following recrystallization of the dinuclear

gold(II) benzoate complex,  $[\text{Au}_2\{\text{CH}_2\text{P}(\text{S})(\text{C}_6\text{H}_5)_2(\text{C}_6\text{H}_5\text{CO}_2)\}]$ , from a dichloromethane/diethyl ether solution. Presumably halogen for benzoate exchange gave the product, but in low yield. 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  $25 < 2\theta < 30^\circ$ . Intensity data collected at room temperature (no symmetry-equivalent reflections were collected) using the  $\omega$ -scanning technique in bisecting geometry (Nicolet R3m/E diffractometer, graphite-monochromated Mo  $K\alpha$  radiation). A total of 4027 unique reflections were measured with  $0 < 2\theta < 45^\circ$  ( $h < 9$ ,  $|k| < 3$ ,  $|l| < 15$ ). Three standard reflections ( $\bar{1}\bar{1}0$ ,  $\bar{1}\bar{1}1$ ,  $\bar{2}0\bar{2}$ ) measured every 100 data. Scan rate variable, 2–30°; scan range,  $-1.0^\circ$  in  $\omega$  from  $K\alpha_1$  to  $+1.0^\circ$  from  $K\alpha_2$ . The data were corrected for standard variation (<0.1%), absorption, Lorentz and polarization effects. Corrections for absorption applied empirically on the basis of azimuthal scans of eight medium intensity reflections spanning a range of  $2\theta$  values. Minimum and maximum transmission 0.167 and 0.268. Background intensities estimated from a 96-step peak profile. Structure solution and refinement carried out using the *SHELXTL* collection of crystallographic software (Sheldrick, 1981). Au positions determined from a sharpened Patterson map; remaining atoms from difference Fourier maps. All non-hydrogen atoms refined anisotropically. Phenyl rings refined as idealized polygons (C–C = 1.395 Å, C–C–C = 120°)

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using H atoms placed in idealized positions with fixed isotropic thermal parameters [ $U(H) = 0.08 \text{ \AA}^2$ ]. Refinement based on  $F$  with weights of the form  $w^{-1} = [\sigma^2(|F_o|) + 0.001464|F_o|^2]$ . The absolute value of  $g (1.464 \times 10^{-3})$  was refined by fitting  $(F_o - F_c)^2$  to  $[\sigma^2(F) + |gF^2|]/k$  where  $k$  is the scale factor. Neutral-atom scattering factors, including terms for anomalous dispersion from the *International Tables for X-ray Crystallography* (1974). One intense low-angle reflection (001) suffered from severe extinction and was therefore omitted during the refinement process. Convergence to final conventional  $R$  values of  $R = 0.0409$  and  $wR = 0.0431$  was obtained using 286 variable parameters and 2857 reflections with  $F_o^2 > 3\sigma(F_o^2)$ . Maximum  $\Delta/\sigma$  for final cycle  $-0.032$  with a goodness-of-fit indicator of 0.997. Difference Fourier map calculated after final cycle of refinement essentially featureless with minimum and maximum residual electron densities of  $-2.59$  and  $2.35 \text{ e \AA}^{-3}$  in the vicinities of Au(1) and Au(2), respectively. A perspective drawing of this complex illustrating the atomic numbering scheme is shown in Fig. 1. A stereoview packing diagram of the unit cell is shown in Fig. 2. Atomic positional and equivalent isotropic thermal parameters are presented in Table 1.\* Intramolecular bond distances and angles are summarized in Table 2.

**Related literature.** The organometallic chemistry of ylides has been reviewed (Schmidbaur, 1975, 1983; Kaska, 1983). The structures of the dinuclear gold(I) methylenethiophosphinate ylide starting material, a dinuclear gold(II)/(II) iodide adduct, and a mixed-valent gold(I)/(III) iodide complex have been previously reported (Mazany & Fackler, 1985).

\* Lists of structure factors, anisotropic thermal parameters and hydrogen-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43476 (27 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

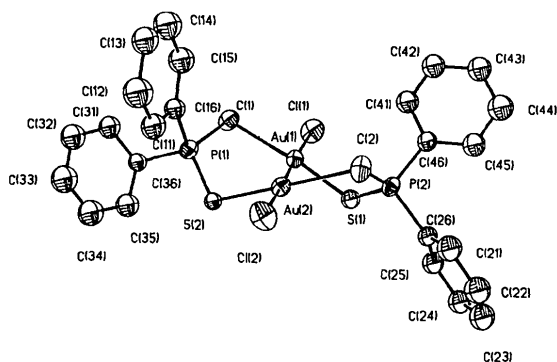


Fig. 1. A perspective drawing of the title complex illustrating the atomic numbering scheme. H atoms have been omitted for clarity.

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Table 1. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic thermal parameters ( $\text{\AA}^2 \times 10^3$ )

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

	$x$	$y$	$z$	$U_{eq}$
Au(1)	-1352 (1)	191 (1)	-1126 (1)	29 (1)
Au(2)	-507 (1)	2095 (1)	-1466 (1)	29 (1)
Cl(1)	-2257 (4)	-1684 (3)	-1016 (3)	51 (2)
Cl(2)	246 (4)	3896 (3)	-1741 (3)	50 (2)
Cl(3)	7108 (5)	2636 (4)	5641 (4)	78 (2)
Cl(4)	6894 (7)	4944 (5)	6506 (5)	103 (3)
S(1)	-3136 (4)	784 (3)	-270 (3)	39 (1)
S(2)	-472 (4)	1127 (3)	-3149 (3)	40 (2)
P(1)	-2392 (4)	2516 (3)	149 (3)	31 (1)
P(2)	294 (4)	-221 (3)	-2966 (3)	31 (1)
C(1)	-561 (12)	2961 (10)	3 (9)	27 (5)
C(2)	408 (15)	-297 (11)	-1719 (9)	37 (6)
C(3)	6830 (23)	3624 (15)	6702 (14)	76 (10)
C(11)	-5066 (9)	2508 (6)	-1054 (8)	58 (7)
C(12)	-6020	3020	-1540	70 (8)
C(13)	-5522	4157	-1521	57 (8)
C(14)	-4071	4782	-1016	66 (9)
C(15)	-3118	4270	-530	49 (7)
C(16)	-3616	3133	-549	37 (6)
C(21)	2562 (9)	501 (8)	-3827 (7)	50 (7)
C(22)	3993	619	-3977	59 (8)
C(23)	4972	119	-3498	54 (7)
C(24)	4519	-498	-2868	46 (7)
C(25)	3087	-616	-2718	42 (6)
C(26)	2109	-117	-3197	33 (5)
C(31)	-1102 (8)	3474 (8)	2205 (7)	50 (7)
C(32)	-1142	3838	3219	62 (8)
C(33)	-2499	3798	3481	76 (10)
C(34)	-3816	3395	2731	64 (9)
C(35)	-3776	3031	1717	44 (7)
C(36)	-2419	3071	1454	30 (5)
C(41)	-428 (8)	-2093 (8)	-4719 (7)	44 (6)
C(42)	-1409	-3044	-5469	61 (8)
C(43)	-2852	-3429	-5383	54 (7)
C(44)	-3314	-2864	-4547	53 (7)
C(45)	-2333	-1913	-3797	44 (6)
C(46)	-889	-1528	-3883	33 (6)

Table 2. Bond lengths ( $\text{\AA}$ ) and angles ( $^\circ$ )

Au(1)—Au(2)	2.553 (1)	Au(1)—Cl(1)	2.384 (4)
Au(1)—S(1)	2.367 (4)	Au(1)—C(2)	2.115 (15)
Au(2)—Cl(2)	2.375 (4)	Au(2)—S(2)	2.377 (3)
Au(2)—C(1)	2.087 (12)	Cl(3)—C(3)	1.767 (20)
Cl(4)—C(3)	1.734 (22)	S(1)—P(1)	2.021 (5)
S(2)—P(2)	2.018 (6)	P(1)—C(1)	1.779 (12)
P(1)—C(16)	1.810 (11)	P(1)—C(36)	1.800 (10)
P(2)—C(2)	1.780 (15)	P(2)—C(26)	1.792 (10)
P(2)—C(46)	1.800 (8)		
Au(2)—Au(1)—Cl(1)	173.3 (1)	Au(2)—Au(1)—S(1)	94.6 (1)
Cl(1)—Au(1)—S(1)	87.9 (1)	Au(2)—Au(1)—C(2)	86.6 (4)
Cl(1)—Au(1)—C(2)	91.6 (4)	S(1)—Au(1)—C(2)	173.2 (3)
Au(1)—Au(2)—Cl(2)	178.2 (1)	Au(1)—Au(2)—S(2)	91.7 (1)
Cl(2)—Au(2)—S(2)	90.0 (1)	Au(1)—Au(2)—C(1)	88.9 (4)
Cl(2)—Au(2)—C(1)	89.5 (4)	S(2)—Au(2)—C(1)	179.1 (4)
Au(1)—S(1)—P(1)	101.5 (2)	Au(2)—S(2)—P(2)	101.2 (2)
S(1)—P(1)—C(1)	112.2 (4)	S(1)—P(1)—C(16)	111.4 (3)
C(1)—P(1)—C(16)	109.4 (6)	S(1)—P(1)—C(36)	107.4 (4)
C(1)—P(1)—C(36)	109.6 (5)	S(2)—P(2)—C(2)	111.5 (5)
S(2)—P(2)—C(26)	111.1 (4)	C(2)—P(2)—C(26)	107.7 (6)
S(2)—P(2)—C(46)	109.2 (4)	C(2)—P(2)—C(46)	110.9 (6)
Au(2)—C(1)—P(1)	108.3 (5)	Au(1)—C(2)—P(2)	112.4 (8)
Cl(3)—C(3)—Cl(4)	110.4 (12)	P(1)—C(16)—C(11)	120.8 (3)
P(1)—C(16)—C(15)	119.1 (2)	P(2)—C(26)—C(21)	120.0 (4)
P(2)—C(26)—C(25)	120.0 (4)	P(1)—C(36)—C(31)	120.5 (3)
P(1)—C(36)—C(35)	119.4 (3)	P(2)—C(46)—C(41)	120.8 (3)
P(2)—C(46)—C(45)	118.9 (3)		

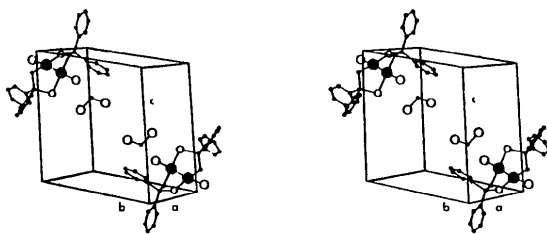


Fig. 2. A stereoview packing diagram of the unit cell. Au atoms are shown as filled circles; P, S and Cl atoms as open circles. H atoms have not been included.

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### 1,3,7,9-Tetra-*tert*-butyl-2,2,8,8-tetramethyl-5,10-diphenyl-1,3,5,7,9,10-hexaaza-2,8-disila-4,6-distannadisp[3.1.3.1]decane

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**Abstract.**  $C_{32}H_{58}N_6Si_2Sn_2$ ,  $M_r = 820.41$ , monoclinic,  $P2_1/c$ ,  $a = 9.361$  (9),  $b = 14.006$  (8),  $c = 15.814$  (10) Å,  $\beta = 105.41$  (6)°,  $V = 1999$  (3) Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 1.363$  Mg m<sup>-3</sup>,  $\lambda(\text{Ag } K\alpha) = 0.5608$  Å,  $\mu = 0.7$  mm<sup>-1</sup>,  $F(000) = 840$ ,  $T = 292$  (1) K, final  $R = 0.038$  for 2042 unique observed diffractometer data and 191 variables. The central part of the centrosymmetric molecule is a four-membered Sn–N–Sn–N ring [Sn–N: 2.032 (5), 2.041 (5) Å, N–Sn–N: 80.8 (2)°, Sn–N–Sn: 99.2 (2)°]. A phenyl group is bound to each N atom of this ring and each Sn atom of this central ring is also a member of an outer four-membered Sn–N–Si–N ring [Sn–N: 2.005 (5), 2.027 (5), Si–N: 1.746 (5), 1.733 (5) Å, N–Sn–N: 76.8 (2)°, Sn–N–Si: 95.6 (2), 95.3 (2)°, N–Si–N: 92.2 (2)°]. The dihedral angle between the plane through the central ring and the least-squares plane through the outer ring is 90.3 (3)°. There are no short intermolecular contacts. The compound is the first and unexpected example of this linear dispiro ring system.

**Experimental.** The substance (Neumann & Obloh, 1986) has been obtained from the known stannylene (Veith, 1975) and phenyl azide at 298 K. Colourless crystals from boiling THF by slow cooling to 273 K.

Crystal size 0.45 × 0.18 × 0.16 mm, enclosed in a capillary,  $\omega/2\theta$  scan, scan speed 1.8–6.7° min<sup>-1</sup> in  $\theta$ , Nonius CAD-4 diffractometer, graphite-

monochromated Ag  $K\alpha$ ; lattice parameters from least-squares fit with 25 reflections up to  $2\theta = 24.3^\circ$ ; four standard reflections recorded every 2.5 h, only random deviations; 8044 reflections measured;  $1 \leq \theta \leq 20^\circ$ ,  $-11 \leq h \leq 11$ ,  $0 \leq k \leq 17$ ,  $-19 \leq l \leq 19$ , max.  $(\sin\theta)/\lambda = 0.61$  Å<sup>-1</sup>; after averaging ( $R_{\text{int}} = 0.033$ ): 3961 unique reflections, 2042 with  $I > 1.96\sigma(I)$ ; Lorentz-polarization correction and absorption correction *via*  $\psi$  scans; systematic absences  $(h0l)$   $l = 2n + 1$ ,  $(0k0)$   $k = 2n + 1$ ; space group  $P2_1/c$ ; structure solution *via* direct methods,  $\Delta F$  syntheses and full-matrix least-squares refinement with anisotropic temperature factors for all non-H atoms and a common isotropic temperature factor for H atoms, which were placed in geometrically calculated positions (C–H 0.95 Å); refinement on  $F$  with 2042 reflections and 191 refined parameters;  $w = 4F_o^2/[\sigma^2(F_o^2) + 0.08F_o^2]$ ;  $S = 0.80$ ,  $R = 0.038$ ,  $wR = 0.046$ ,  $(\Delta/\sigma)_{\text{max}} = 0.02$ ; no extinction correction; largest peak in final  $\Delta F$  map  $\pm 0.5$  (2) e Å<sup>-3</sup>; complex neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974); programs: Enraf–Nonius *SDP* (Frenz, 1981), *ORTEPII* (Johnson, 1976), *MULTAN80* (Main *et al.*, 1980), *POPI* (van de Waal, 1976).

The molecule and the numbering scheme are shown in Fig. 1 and a stereoscopic view of the unit cell in Fig. 2. Positional parameters and the equivalent values of the anisotropic temperature factors for the non-H