Ag(3)—S(2)	2.591 (4)	P(2)—C(231)	1.82 (1)
Ag(3)—S(5)	2.698 (4)	P(3)—C(331)	1.80 (1)
Ag(1)—P(1)	2.454 (4)	P(3)—C(321)	1.82 (1)
Ag(2)—P(2)	2.420 (4)	P(3)—C(311)	1.85 (1)
Ag(3)—P(3)	2.454 (4)		
Ag(1)—Mo—Ag(3)	65.33 (4)	C(211)—P(2)—C(231)	104.3 (6)
Ag(2)—Mo—Ag(1)	87.84 (4)	C(221)—P(2)—C(231)	105.8 (6)
Ag(2)—Mo—Ag(3)	73.18 (4)	C(331)—P(3)—C(321)	106.5 (7)
S(2)—Mo—S(1)	112.5 (1)	C(331)—P(3)—C(311)	105.3 (7)
S(3)—Mo—S(1)	113.2 (1)	C(321)—P(3)—C(311)	102.0 (7)
S(3)—Mo—S(2)	107.8 (2)	Mo—S(2)—Ag(1)	77.8 (1)
S(4)—Mo—S(1)	106.6 (2)	Mo—S(2)—Ag(3)	80.4 (1)
S(4)—Mo—S(2)	107.7 (2)	Mo—S(3)—Ag(2)	75.9 (1)
S(4)—Mo—S(3)	108.9 (2)	Ag(1)—S(2)—Ag(3)	79.9 (1)
P(1)—Ag(1)—S(1)	111.2 (1)	Ag(2)—S(1)—Ag(1)	109.6 (1)
P(1)—Ag(1)—S(2)	116.1 (1)	Ag(2)—S(5)—Ag(3)	85.0 (1)
P(1)—Ag(1)—S(6)	110.4 (1)	P—S(5)—Ag(2)	108.2 (2)
S(1)—Ag(1)—S(2)	92.0 (1)	P—S(5)—Ag(3)	95.2 (2)
S(1)—Ag(1)—S(6)	119.3 (1)	P—S(6)—Ag(1)	98.6 (2)
S(2)—Ag(1)—S(6)	106.9 (1)	O(2)—P—O(1)	106.0 (6)
P(2)—Ag(2)—S(1)	127.4 (1)	O(2)—P—S(5)	104.7 (4)
P(2)—Ag(2)—S(3)	121.7 (1)	O(2)—P—S(6)	113.5 (4)
P(2)—Ag(2)—S(5)	109.2 (1)	O(1)—P—S(5)	109.4 (4)
S(1)—Ag(2)—S(3)	93.5 (1)	O(1)—P—S(6)	105.6 (4)
S(1)—Ag(2)—S(5)	105.9 (1)	S(6)—P—S(5)	117.1 (2)
S(3)—Ag(2)—S(5)	93.2 (1)	C(1)—O(1)—P	119 (1)
S(2)—Ag(3)—S(5)	133.0 (1)	C(3)—O(2)—P	124 (1)
P(3)—Ag(3)—S(2)	118.4 (1)	C(2)—C(1)—O(1)	107 (1)
P(3)—Ag(3)—S(5)	103.8 (1)	O(2)—C(3)—C(4)	114 (2)
Mo—S(1)—Ag(1)	77.6 (1)	C(111)—P(1)—Ag(1)	114.5 (5)
Mo—S(1)—Ag(2)	77.1 (1)	C(121)—P(1)—Ag(1)	113.7 (5)
C(321)—P(3)—Ag(3)	118.1 (5)	C(131)—P(1)—Ag(1)	113.2 (5)
C(311)—P(3)—Ag(3)	111.0 (5)	C(211)—P(2)—Ag(2)	112.7 (5)
C(111)—P(1)—C(121)	103.8 (6)	C(221)—P(2)—Ag(2)	114.8 (4)
C(111)—P(1)—C(131)	105.7 (7)	C(231)—P(2)—Ag(2)	114.7 (4)
C(121)—P(1)—C(131)	104.8 (7)	C(331)—P(3)—Ag(3)	112.7 (5)
C(211)—P(2)—C(221)	103.5 (6)		

Data collection was performed using *CONTROL* (Molecular Structure Corporation, 1986) software. The scan speed varied between 2.4 and 8° min^{-1} (in ω) on the basis of *SEARCH* intensity. The scan width was (1.418 + 0.350tanθ)° with maximum (sinθ)/λ = 0.5946 $Å^{-1}$. The structure was solved by direct methods using *MITHRIL* (Gilmore, 1983). The heavy atoms, Mo and Ag, were located in the *E* map and the remaining non-H atoms were located using the *DIRDIF* program (Beurskens, 1984). H atoms were placed in geometrically calculated positions (C—H 0.95 Å), but not included in the refinement. The structure was refined by full-matrix least-squares techniques with anisotropic displacement parameters for all Mo, Ag, S, P and O atoms and isotropic displacement parameters for all C atoms. All calculations were performed on a VAX 785 computer using the *TEXSAN* (Molecular Structure Corporation, 1985) program package. The view of the molecule (Fig. 1) was produced using *ORTEP*II (Johnson, 1976).

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71691 (25 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL1055]

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Bis(2,6-di-*tert*-butylphenolato-κO)tin

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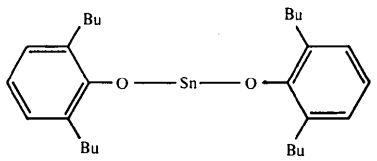
Abstract

The title compound, $[Sn(C_{14}H_{21}O)_2]$ (I), contains a two-coordinate tin metal center with Sn—O distances of 2.003 (3) and 2.044 (3) Å, and an O—Sn—O angle of 88.8 (1)°.

Comment

The related complex $Sn(O\text{-}2,6\text{-}^tBu_2\text{-}4\text{-}Me\text{-}C_6H_2)_2$, prepared from the reaction of tin(II) chloride with lithium 2,6-di-*tert*-butyl-4-methylphenoxide, has

been crystallographically characterized (Çetinkaya, Gümrükçü, Lappert, Atwood, Rogers & Zaworotko, 1980).



(1)

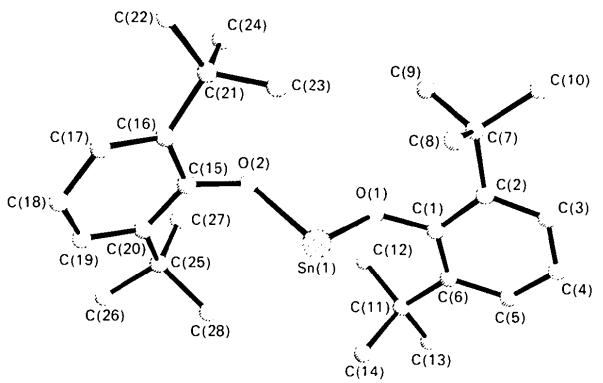


Fig. 1. Molecular structure and labeling scheme for the title compound $[Sn(C_{14}H_{21}O)_2]$.

Experimental

The reaction between SnI_4 and 4 equivalents of K(O-2,6-^{t}Bu$_2$-C_6H_3) in tetrahydrofuran produced the yellow tin(II) complex [Sn(O-2,6-^{t}Bu$_2$-C_6H_3)$_2$] in moderate yield. Suitable crystals were grown by the slow evaporation of a toluene solution under an inert atmosphere.

Crystal data

$[Sn(C_{14}H_{21}O)_2]$
M_r = 529.3
Monoclinic
$P2_1/n$
a = 13.767 (2) Å
b = 13.849 (2) Å
c = 13.865 (2) Å
β = 94.76 (1)°
V = 2634.4 (10) Å^3
Z = 4
D_x = 1.335 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 24 reflections
θ = 14–18°
μ = 0.990 mm^{-1}
T = 203 K
Parallelepiped
0.3 × 0.1 × 0.1 mm
Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
2θ–θ scans
Absorption correction: empirical
T_{min} = 0.883, T_{max} = 0.997
2675 measured reflections
2253 independent reflections
2001 observed reflections [$F > 4.0\sigma(F)$]
R_{int} = 0.012
θ_{max} = 25°
h = −11 → 11
k = 0 → 12
l = 0 → 16
2 standard reflections
frequency: 120 min
intensity variation: 1%

Refinement

Refinement on F^2
R = 0.0241
wR = 0.0312
S = 1.51
2001 reflections
281 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F) + 0.0002F^2]$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Extinction correction: $F^* = F[1 + (0.002 \times \chi F^2/\sin 2\theta)]^{-1/4}$
Extinction coefficient: χ = 0.00015 (4)
Atomic scattering factors from *SHELXTL-Plus* (Sheldrick, 1991)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

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

	x	y	z	U_{eq}
Sn(1)	0.10350 (2)	0.91357 (2)	0.54452 (2)	0.0209 (2)
O(1)	0.2115 (2)	0.8257 (2)	0.59530 (17)	0.0215 (10)
C(1)	0.2949 (3)	0.8079 (3)	0.5504 (3)	0.0175 (16)
C(2)	0.2945 (3)	0.7334 (3)	0.4802 (3)	0.0206 (16)
C(3)	0.3761 (4)	0.7260 (3)	0.4272 (3)	0.0272 (18)
C(4)	0.4554 (4)	0.7855 (4)	0.4453 (3)	0.0280 (18)
C(5)	0.4578 (3)	0.8496 (3)	0.5209 (3)	0.0240 (17)
C(6)	0.3798 (3)	0.8621 (3)	0.5766 (3)	0.0169 (16)
C(7)	0.2098 (3)	0.6612 (3)	0.4596 (3)	0.0254 (17)
C(8)	0.1298 (3)	0.7066 (3)	0.3920 (3)	0.0361 (18)
C(9)	0.1697 (4)	0.6245 (4)	0.5540 (3)	0.0413 (19)
C(10)	0.2426 (4)	0.5705 (4)	0.4070 (4)	0.047 (2)
C(11)	0.3888 (3)	0.9320 (3)	0.6637 (3)	0.0230 (17)
C(12)	0.3504 (4)	0.8851 (4)	0.7537 (3)	0.0359 (18)
C(13)	0.4945 (3)	0.9603 (4)	0.6922 (3)	0.0362 (19)
C(14)	0.3357 (3)	1.0281 (4)	0.6372 (3)	0.0340 (18)
O(2)	0.0251 (2)	0.8700 (2)	0.65562 (17)	0.0199 (10)
C(15)	−0.0625 (4)	0.9145 (3)	0.6641 (3)	0.0156 (17)
C(16)	−0.1489 (4)	0.8712 (3)	0.6216 (3)	0.0183 (17)
C(17)	−0.2332 (4)	0.9277 (4)	0.6163 (3)	0.0230 (18)
C(18)	−0.2343 (3)	1.0191 (4)	0.6562 (3)	0.0243 (18)
C(19)	−0.1527 (4)	1.0531 (3)	0.7098 (3)	0.0230 (17)
C(20)	−0.0654 (3)	1.0011 (3)	0.7176 (3)	0.0159 (16)
C(21)	−0.1527 (3)	0.7660 (3)	0.5867 (3)	0.0223 (16)
C(22)	−0.2572 (4)	0.7333 (4)	0.5576 (4)	0.059 (2)
C(23)	−0.0950 (4)	0.7492 (4)	0.4986 (4)	0.061 (2)
C(24)	−0.1123 (4)	0.6991 (4)	0.6687 (3)	0.047 (2)
C(25)	0.0208 (3)	1.0380 (3)	0.7848 (3)	0.0209 (16)
C(26)	−0.0073 (4)	1.1228 (4)	0.8471 (4)	0.054 (2)
C(27)	0.0583 (4)	0.9585 (4)	0.8550 (3)	0.048 (2)
C(28)	0.1036 (4)	1.0736 (4)	0.7287 (3)	0.057 (2)

Table 2. *Selected geometric parameters* (Å, °)

Sn(1)—O(2)	2.044 (3)	Sn(1)—O(1)	2.003 (3)
O(2)—C(15)	1.368 (6)	O(1)—C(1)	1.373 (5)
O(1)—Sn(1)—O(2)	88.8 (1)	Sn(1)—O(2)—C(15)	117.2 (2)
Sn(1)—O(1)—C(1)	125.0 (2)		

The crystal used for data collection was coated in Nujol and mounted on a fiber in the nitrogen cold stream of the diffractometer. Data reduction was carried out using the Enraf–Nonius *MolEN* (Fair, 1990) system. The structure was solved by Patterson methods using the *SHELXTL-Plus* (Sheldrick, 1991) program, with full-matrix least-squares refinement on F^2. All non-H atoms were refined anisotropically and H atoms were placed in calculated positions.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71673 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR1074]

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$[Au_2(C_6H_5CS_2)(\mu\text{-dppm})_2]Cl$

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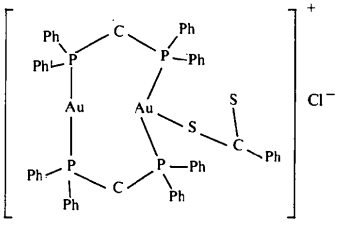
Abstract

Bis[μ-bis(diphenylphosphino)methane-1κP:2$\kappa P'$]-(dithiobenzoato-1κS)digold(I) chloride, $[Au_2(C_6H_5CS_2)(\mu\text{-dppm})_2]Cl$, where dppm = $C_{25}H_{22}P_2$, was synthesized and structurally characterized. The Au···Au separation is 3.0176 (5) Å. The coordination geometries of the two Au centers differ. The bonds across the Au1 center are nearly linear with Au—P distances of 2.301 (2) and 2.305 (2) Å. The coordination number of the Au2 center is four; the two Au—P distances are 2.317 (2) and 2.335 (2) Å and the Au—S distance is 2.718 (3) Å. The dithiobenzoate ligand is coordinated to only one metal center (Au2) through one of its S atoms. The two S—C distances in this ligand are equal.

Comment

The luminescent and bonding properties of binuclear gold(I) complexes have been gaining attention recently (Khan, Fackler, King, Wang & Wang, 1988; King, Wang, Khan & Fackler, 1989). In this paper we report the crystal structure of



(I)

$Au_2(C_6H_5CS_2)(\mu\text{-dppm})_2Cl$, (I). A short Au···Au contact [3.0176 (5) Å], similar to that in other $Au_2(dppm)_2X_2$ complexes [X = Cl (Schmidbaur, Wohlleben, Schubert, Frank & Huttner, 1977), NO_3 (Wang, Khan & Fackler, 1989), BH_3CN and S_2CNEt (Khan, King, Heinrich, Fackler & Porter, 1989)] is observed. Unlike the Au centers in the other $Au_2(dppm)_2X_2$ compounds, the two Au centers in the title compound are asymmetric; the overall geometry about Au1 is close to linear and the dithiobenzoate is coordinated to only one metal center through one S atom, with an Au2—S1 distance of 2.718 (3) Å. The linearity of P2—Au2—P4 [159.66 (8)°] is perturbed by the interaction between the Au2 and S1 atoms. Thus, the overall coordination geometry of Au2 can be viewed as distorted tetrahedral. The two S—C bond distances in the dithiobenzoate ligand are not significantly different. Fig. 1 shows an *ORTEP* (Johnson, 1970) drawing of the molecule.

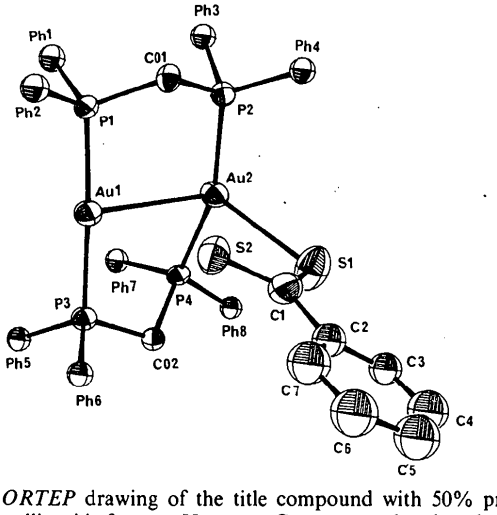


Fig. 1. *ORTEP* drawing of the title compound with 50% probability ellipsoids for non-H atoms. C atoms on the phenyl rings are omitted for clarity, except for those attached to the P atoms.

Experimental

Crystal data

$[Au_2(C_6H_5CS_2)(C_{25}H_{22}P_2)_2]Cl$
M_r = 1351.44
D_x = 1.732 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å