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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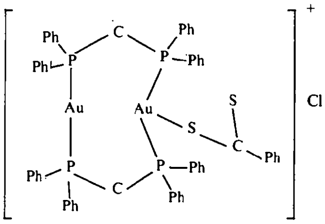
Abstract

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



(I)

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 $Au_2(C_6H_5CS_2)(\mu\text{-dppm})_2Cl$, (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.

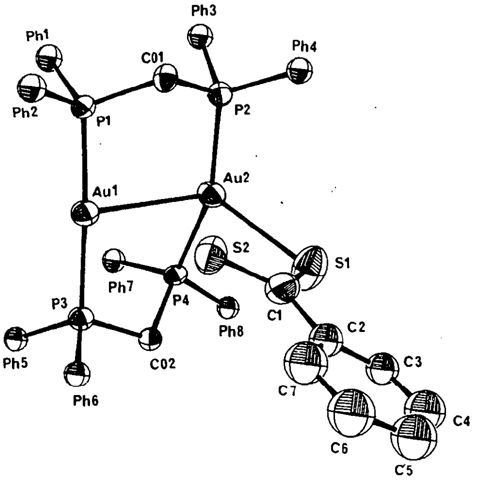


Fig. 1. *ORTEP* drawing of the title compound with 50% probability ellipsoids for non-H atoms. C atoms on the phenyl rings are omitted for clarity, except for those attached to the P atoms.

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

Monoclinic
$P2_1/n$
$a = 11.583$ (1) Å
$b = 19.334$ (2) Å
$c = 23.416$ (8) Å
$\beta = 98.94$ (2)°
$V = 5182.0$ (21) Å^3
$Z = 4$

Cell parameters from 25 reflections
$\theta = 7$–15°
$\mu = 5.93$ mm^{-1}
$T = 298$ K
Irregular
0.3 × 0.2 × 0.15 mm
Green

Data collection

Enraf-Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical
$T_{\min} = 0.72$, $T_{\max} = 1.00$
7427 measured reflections
7020 independent reflections
4613 observed reflections $[I > 3\sigma(I)]$

$R_{\rm int} = 0.032$
$\theta_{\max} = 22.5$°
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 20$
$l = -25 \rightarrow 25$
3 standard reflections
frequency: 60 min
intensity variation: −3.6%

Refinement

Refinement on F
$R = 0.034$
$wR = 0.037$
$S = 1.346$
4613 reflections
325 parameters
H-atom parameters not refined

$w = 4F_o^2/[\sigma^2(I) + (0.02F_o^2)^2]$
$(\Delta/\sigma)_{\max} = 0.04$
$\Delta\rho_{\max} = 1.830$ e Å^{-3}
$\Delta\rho_{\min} = -1.826$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

$B_{\rm eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	$B_{\rm eq}$
Au1	0.66915 (3)	0.69869 (2)	0.26967 (2)	2.893 (8)
Au2	0.43371 (3)	0.72733 (2)	0.30459 (2)	2.875 (8)
Cl	0.1968 (3)	0.1452 (2)	0.4740 (1)	4.00 (7)
S1	0.3051 (3)	0.8304 (2)	0.2470 (2)	5.68 (9)
S2	0.5590 (3)	0.8469 (2)	0.2450 (1)	4.34 (7)
P1	0.7573 (2)	0.7207 (1)	0.3629 (1)	2.22 (5)
P2	0.5199 (2)	0.7633 (1)	0.3956 (1)	2.21 (5)
P3	0.5936 (2)	0.6773 (1)	0.1744 (1)	2.37 (6)
P4	0.3743 (2)	0.6541 (1)	0.2262 (1)	2.17 (5)
C01	0.6743 (7)	0.7849 (5)	0.3963 (4)	2.3 (2)
C02	0.4348 (8)	0.6846 (5)	0.1631 (4)	2.4 (2)
C1	0.418 (1)	0.8718 (6)	0.2249 (5)	4.2 (3)
C2*	0.3870 (9)	0.9351 (6)	0.1858 (5)	3.6 (2)
C3*	0.2740 (9)	0.9550 (5)	0.1675 (4)	3.5 (2)
C4*	0.249 (1)	1.0115 (6)	0.1373 (5)	5.1 (3)
C5*	0.333 (1)	1.0522 (7)	0.1180 (6)	5.8 (3)
C6*	0.448 (1)	1.0319 (7)	0.1316 (6)	6.3 (3)
C7*	0.477 (1)	0.9719 (7)	0.1658 (6)	5.6 (3)
C11*	0.7770 (8)	0.6451 (5)	0.4094 (4)	2.7 (2)
C12*	0.8234 (9)	0.6517 (6)	0.4676 (5)	4.2 (2)
C13*	0.847 (1)	0.5902 (6)	0.5008 (5)	5.2 (3)
C14*	0.822 (1)	0.5288 (7)	0.4757 (5)	5.5 (3)
C15*	0.776 (1)	0.5216 (7)	0.4199 (6)	5.6 (3)
C16*	0.7524 (9)	0.5798 (6)	0.3860 (5)	4.1 (2)
C21*	0.9049 (8)	0.7539 (5)	0.3665 (4)	2.7 (2)
C22*	0.9553 (9)	0.7978 (6)	0.4108 (5)	3.8 (2)
C23*	1.074 (1)	0.8144 (6)	0.4136 (5)	4.2 (2)
C24*	1.137 (1)	0.7888 (6)	0.3747 (5)	4.8 (3)
C25*	1.087 (1)	0.7485 (6)	0.3315 (6)	5.2 (3)
C26*	0.9709 (9)	0.7287 (6)	0.3265 (5)	3.9 (2)
C31*	0.5208 (8)	0.7007 (5)	0.4536 (4)	2.6 (2)
C32*	0.5878 (8)	0.7104 (5)	0.5089 (4)	3.2 (2)
C33*	0.5886 (9)	0.6617 (6)	0.5508 (5)	4.1 (2)
C34*	0.5256 (9)	0.6010 (6)	0.5393 (5)	4.2 (2)
C35*	0.4599 (9)	0.5905 (6)	0.4861 (5)	4.2 (2)
C36*	0.4572 (8)	0.6403 (5)	0.4426 (4)	3.0 (2)
C41*	0.4592 (8)	0.8415 (5)	0.4217 (4)	2.4 (2)
C42*	0.4602 (9)	0.9016 (5)	0.3899 (5)	3.9 (2)
C43*	0.413 (1)	0.9634 (6)	0.4074 (5)	5.0 (3)
C44*	0.363 (1)	0.9623 (6)	0.4564 (5)	4.8 (3)
C45*	0.356 (1)	0.9043 (6)	0.4881 (6)	5.2 (3)
C46*	0.4075 (9)	0.8442 (6)	0.4705 (5)	3.7 (2)
C51*	0.6316 (8)	0.5941 (5)	0.1462 (4)	2.3 (2)
C52*	0.5898 (9)	0.5747 (5)	0.0895 (5)	3.6 (2)
C53*	0.6285 (9)	0.5136 (6)	0.0681 (5)	4.1 (2)
C54*	0.708 (1)	0.4730 (6)	0.1021 (5)	4.8 (3)
C55*	0.746 (1)	0.4916 (6)	0.1593 (5)	4.2 (2)
C56*	0.7070 (9)	0.5518 (5)	0.1808 (5)	3.4 (2)
C61*	0.6427 (8)	0.7390 (5)	0.1264 (4)	2.4 (2)
C62*	0.7511 (9)	0.7697 (6)	0.1415 (5)	4.2 (2)
C63*	0.796 (1)	0.8146 (6)	0.1032 (5)	4.9 (3)
C64*	0.732 (1)	0.8282 (6)	0.0505 (5)	4.8 (3)
C65*	0.623 (1)	0.7999 (6)	0.0344 (5)	5.2 (3)
C66*	0.580 (1)	0.7542 (6)	0.0729 (5)	4.0 (2)
C71*	0.4203 (8)	0.5645 (5)	0.2332 (4)	2.4 (2)
C72*	0.4870 (9)	0.5424 (6)	0.2836 (5)	3.7 (2)
C73*	0.526 (1)	0.4741 (6)	0.2875 (5)	5.3 (3)
C74*	0.499 (1)	0.4313 (8)	0.2417 (6)	6.7 (4)
C75*	0.429 (1)	0.4519 (7)	0.1934 (6)	6.8 (4)
C76*	0.391 (1)	0.5205 (6)	0.1866 (5)	5.0 (3)
C81*	0.2180 (8)	0.6483 (5)	0.2014 (4)	2.1 (2)
C82*	0.1517 (9)	0.6061 (6)	0.2295 (5)	4.0 (2)
C83*	0.030 (1)	0.6004 (6)	0.2117 (5)	5.2 (3)
C84*	−0.020 (1)	0.6360 (6)	0.1658 (5)	4.5 (3)
C85*	0.041 (1)	0.6781 (7)	0.1362 (6)	6.5 (3)
C86*	0.163 (1)	0.6844 (6)	0.1546 (5)	5.2 (3)

* Refined isotropically.

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

Au1···Au2	3.0176 (5)	C2—C7	1.40 (1)
Au1—P1	2.301 (2)	C4—C5	1.39 (1)
Au1—P3	2.305 (2)	Au2—P4	2.335 (2)
Au2—P2	2.317 (2)	S2—C1	1.70 (1)
Au2—S1	2.718 (3)	P1—C11	1.816 (8)
S1—C1	1.68 (1)	P2—C01	1.834 (8)
P1—C01	1.818 (8)	P2—C41	1.813 (8)
P1—C21	1.816 (9)	P3—C51	1.818 (8)
P2—C31	1.817 (8)	P4—C02	1.829 (8)
P3—C02	1.823 (8)	P4—C81	1.818 (8)
P3—C61	1.792 (8)	C2—C3	1.37 (1)
P4—C71	1.813 (8)	C3—C4	1.31 (1)
C1—C2	1.54 (1)		
Au2—Au1—P1	89.74 (6)	C02—P3—C51	108.2 (4)
Au2—Au1—P3	94.14 (6)	C02—P3—C61	105.6 (4)
P1—Au1—P3	175.99 (8)	C51—P3—C61	104.1 (4)
Au1—Au2—S1	116.66 (7)	Au2—P4—C02	109.7 (3)
Au1—Au2—P2	91.48 (6)	Au2—P4—C71	117.5 (3)
Au1—Au2—P4	81.03 (5)	Au2—P4—C81	116.5 (3)
S1—Au2—P2	111.18 (9)	C02—P4—C71	103.7 (4)
S1—Au2—P4	89.01 (9)	C02—P4—C81	104.4 (4)
P2—Au2—P4	159.66 (8)	C71—P4—C81	103.5 (4)
Au2—S1—C1	96.4 (4)	S1—C1—S2	122.7 (6)
Au1—P1—C01	110.4 (3)	S1—C1—C2	116.5 (7)
Au1—P1—C11	114.6 (3)	S2—C1—C2	120.9 (8)
Au1—P1—C21	112.7 (3)	P1—C01—P2	114.9 (4)
C01—P1—C11	108.5 (4)	C01—P1—C21	107.2 (4)
C11—P1—C21	103.1 (4)	Au2—P2—C01	110.9 (3)
P3—C02—P4	111.2 (4)	Au2—P2—C31	116.0 (3)
Au2—P2—C41	115.2 (3)	C01—P2—C31	104.7 (4)
C01—P2—C41	103.7 (4)	C31—P2—C41	105.2 (4)
Au1—P3—C02	110.4 (3)	Au1—P3—C51	115.7 (3)
Au1—P3—C61	112.2 (3)		

Computations were performed using *SDP* software (B. A. Frenz & Associates, Inc., 1989).

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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 71716 (35 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HU1048]

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Acta Cryst. (1994). C**50**, 706–708

trans-Bis(2-hydroxyiminopropionato-*N*,*O*)-dimethanolnickel(II) Dimethanol Solvate, $[Ni\{CH_3C(NOH)COO\}_2(CH_3OH)_2].2CH_3OH$

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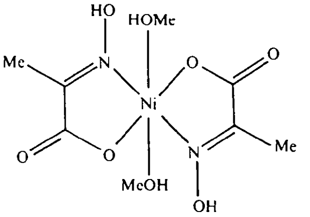
Abstract

$[Ni(C_3H_4NO_3)_2(CH_3OH)_2].2CH_3OH$ contains Ni in a six-coordinated state and decomposes rapidly in air by loss of methanol. The Ni coordination environment is a slightly distorted octahedron. The anions of the 2-hydroxyiminopropionic acids lie in a plane in a *trans* position to each other. Octahedral coordination is completed by two CH_3OH molecules.

Comment

trans-Bis(2-hydroxyiminopropionato-*N*,*O*)dimethanolnickel(II) dimethanol solvate, $[Ni\{CH_3C(NOH)COO\}_2(CH_3OH)_2].2CH_3OH$, was prepared during our work on the synthesis and systematic study of complexes of 2-oximinocarboxylic acids with divalent metal ions (Lampeka, Skopenko, Sliva & Hennig, 1988; Skopenko, Lampeka, Sliva & Stakhov, 1990). Some anionic complexes have been analysed by X-ray diffraction (Simonov, Sliva, Mazus, Dvorkin & Lampeka, 1989; Dvorkin, Simonov, Sliva, Lampeka, Mazus & Skopenko, 1989). In these compounds the oximino groups are mutually *cis* and form the usual intramolecular hydrogen bond. The mutal arrangement of the oximino groups in neutral complexes is of interest. The present paper reports the structure of one example from this family of compounds. The geometry and atom numbering of the complex (I) are illustrated in Fig. 1.



(I)

The complex unit has an inversion centre at the Ni atom. Ni displays slightly distorted octahedral coordination. Each of the two bidentate 2-oximinopropionic anions is bound to Ni through the oxime N atom and carboxylic O atom. Both anions lie in the same plane with the oxime groups on opposite sides of the Ni ion. This renders formation of an intramolecular hydrogen bond impossible. Two methanol molecules are bonded to the metal atom above and below the equatorial $Ni[CH_3C(NOH)COO]_2$ plane, thus completing the octahedral coordination at the Ni atom.

Bond distances for the 2-oximinopropionate ions are close to those reported in the literature (Simonov, Sliva, Mazus, Dvorkin & Lampeka, 1989;

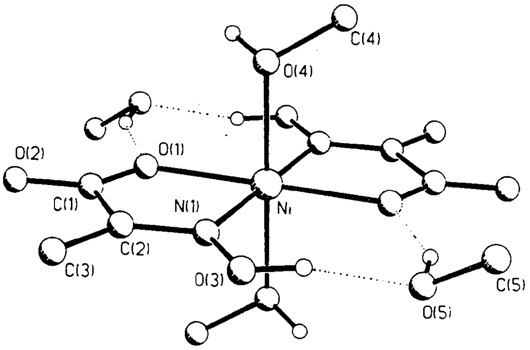


Fig. 1. The geometry and atom-numbering scheme of the complex molecule (dashed lines show the hydrogen bonds).

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