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Key indicators

Single-crystal X-ray study
 T = 100 K
 Mean $\sigma(C-C)$ = 0.013 Å
 R factor = 0.066
 wR factor = 0.099
 Data-to-parameter ratio = 21.9

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

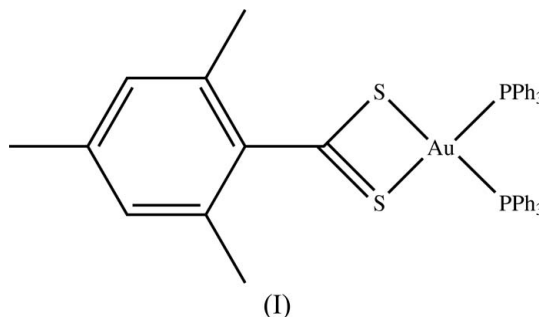
(2,4,6-Trimethyldithiobenzoato- κ^2S,S')-bis(triphenylphosphine- κP)gold(I)

The asymmetric unit of the title compound, $[Au(C_{10}H_8S_2)(C_{18}H_{15}P)_2]$, consists of two independent molecules. The Au coordination environment can be described as a highly distorted tetrahedral arrangement (S_2AuP_2), but is more accurately described as a flattened trigonal pyramid defined by three normal covalent bonds ($SAuP_2$), with a second S atom more distantly bonded to the Au atom.

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Comment

We have recently reported (Macalindong *et al.*, 2006) a structure in which a ring-substituted dithiobenzoate acted as a monodentate ligand, together with one PPh_3 ligand, in a two-coordinate gold(I) complex. The title structure, (I), consists of a ring-substituted dithiobenzoate acting as an unsymmetrical bidentate ligand, together with two PPh_3 ligands, in a quasi-four-coordinate gold(I) complex.



If the dithiobenzoate were a symmetrical bidentate ligand, the S_2AuP_2 coordination polyhedron would have two equal S–Au bonds, and the two S atoms would be equidistant from the P–Au–P plane. However, in each of the two independent molecules of the title structure, significant and consistent deviations from ideality occur.

The statistically (and chemically) equivalent pairs of internuclear distances, with averages in square brackets, are: P1–Au1 and P3–Au2 [2.319 (1) Å], P2–Au1 and P4–Au2 [2.339 (1) Å], and S2–Au1 and S3–Au2 [2.597 (1) Å]. These distances are consistent with normal covalent bonds and define a trigonal–pyramidal coordination, with an Au atom at each apex. Each Au atom is also coordinated by the other S atom of the dithiocarboxylate (S1–Au1 and S4–Au2), but these internuclear distances are significantly longer and more disparate than the other S–Au bonds, suggesting a weaker binding force which is more susceptible to variations in the steric and electrostatic environment.

The coordination polyhedron itself is also quite flexible. Although the magnitudes of the three bond angles in each

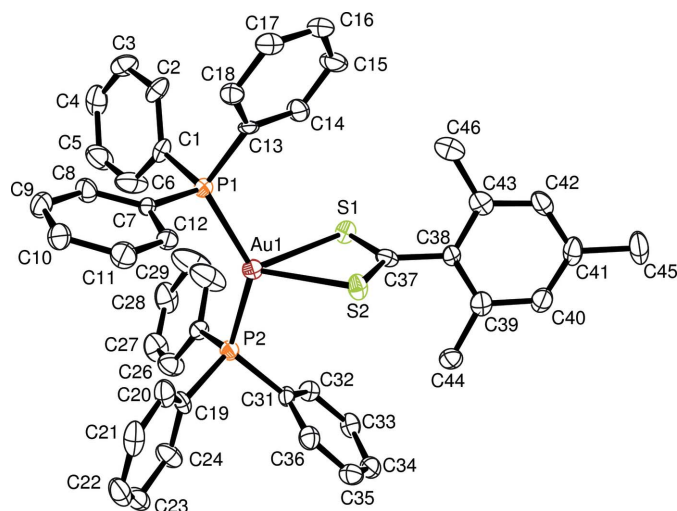


Figure 1

A view of one molecule of the asymmetric unit of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity.

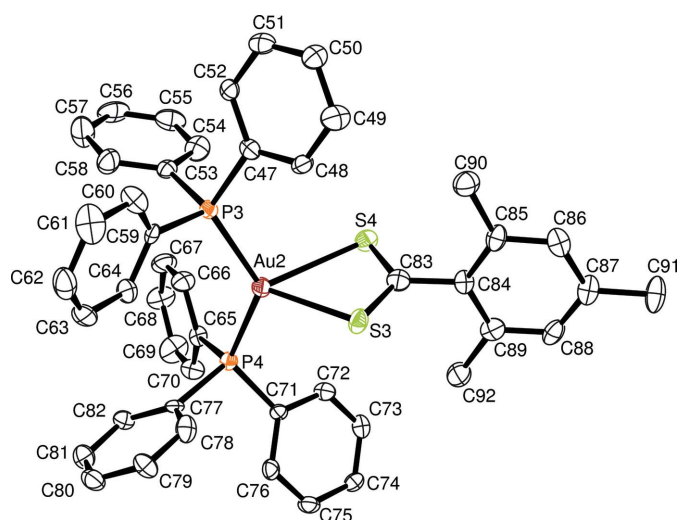


Figure 2

A view of the second molecule of the asymmetric unit of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity.

trigonal pyramid display the same internal pattern, there is no statistical equivalence between corresponding angles. Similar coordinations and patterns of distortion are found in previously reported S_2AuP_2 structures (Lanfredi *et al.*, 1992; Assefa *et al.*, 1994; Razak *et al.*, 2000; Jian *et al.*, 2000; Jian *et al.*, 2003).

Experimental

The preparation of the title compound is fully described by Schuerman (1988). To a stirred solution of $[(n-C_3H_7)_4N][[(CH_3)_3C_6H_2CS_2]]$ (0.5 g) in dimethylformamide (20 ml) was added $NaAuCl_4 \cdot 2H_2O$ (0.260 g) dissolved in dimethylformamide (10 ml). After 24 h, reddish-brown needles of $Au_2[(CH_3)_3C_6H_2CS_2]_2$ formed. This dimer (0.02 g) was dissolved in a CS_2-CHCl_3 mixture (1:1,

10 ml), to which was added PPh_3 (0.0270 g) dissolved in a CS_2-CHCl_3 mixture (1:1, 2 ml). The resulting pink solution was evaporated and the residue was washed with ethanol-cyclohexane (3:2) and air-dried. Recrystallization from boiling diethyl ether yielded crystals of (I) suitable for analysis.

Crystal data

$[Au(C_{10}H_8S_2)(C_{18}H_{15}P)_2]$
 $M_r = 916.81$
 Monoclinic, $P2_1$
 $a = 12.277$ (3) Å
 $b = 18.803$ (4) Å
 $c = 17.132$ (4) Å
 $\beta = 93.922$ (9)°
 $V = 3945.6$ (16) Å³

$Z = 4$
 $D_x = 1.543$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 3.95$ mm⁻¹
 $T = 100$ K
 Fragment, pale red
 $0.19 \times 0.17 \times 0.13$ mm

Data collection

Bruker Nonius KappaCCD area-detector diffractometer
 ω scans with κ offsets
 Absorption correction: multi-scan (*HKL SCALEPACK*; Otwinowski & Minor 1997)
 $T_{min} = 0.551$, $T_{max} = 0.601$

35432 measured reflections
 20259 independent reflections
 15115 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.064$
 $\theta_{max} = 29.1^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.066$
 $wR(F^2) = 0.099$
 $S = 1.05$
 20259 reflections
 927 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + 14.6708P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.007$
 $\Delta\rho_{max} = 3.28$ e Å⁻³
 $\Delta\rho_{min} = -1.54$ e Å⁻³
 Absolute structure: Flack (1983), with 9362 Friedel pairs
 Flack parameter: 0.146 (5)

Table 1

Selected geometric parameters (Å, °).

C37—S1	1.666 (9)	Au1—S2	2.602 (2)
C37—S2	1.703 (9)	Au1—S1	2.862 (2)
C83—S4	1.673 (9)	Au2—P3	2.323 (2)
C83—S3	1.693 (10)	Au2—P4	2.340 (2)
Au1—P1	2.314 (2)	Au2—S3	2.592 (2)
Au1—P2	2.338 (2)	Au2—S4	2.910 (2)
S1—C37—S2	121.2 (5)	P3—Au2—S3	117.81 (8)
S4—C83—S3	121.0 (5)	P4—Au2—S3	108.63 (8)
P1—Au1—P2	137.40 (7)	P3—Au2—S4	107.42 (7)
P1—Au1—S2	113.57 (7)	P4—Au2—S4	110.81 (7)
P2—Au1—S2	107.36 (8)	S3—Au2—S4	64.05 (7)
P1—Au1—S1	109.74 (7)	C37—S1—Au1	83.0 (3)
P2—Au1—S1	97.69 (7)	C37—S2—Au1	90.9 (3)
S2—Au1—S1	64.78 (7)	C83—S3—Au2	92.6 (3)
P3—Au2—P4	129.10 (8)	C83—S4—Au2	82.4 (3)

Refinement of the Flack (1983) parameter with 9362 Friedel pairs led to a value of 0.146 (5), implying an inversion twin with unequal components. The major component is shown. All carbon-bound H atoms were placed in calculated positions, with C—H distances of 0.95 (phenyl) or 0.98 Å (methyl), and with $U_{iso}(H) = 1.2$ (phenyl) or 1.5 (methyl) U_{eq} of the parent C atom, and thereafter treated as riding. A torsional parameter was refined for each methyl group. The deepest hole is 0.06 Å from Au2, and the minimum peak is 0.53 Å from Au2.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data

reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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