(N,N-Diethyldithiocarbamato)(tricyclohexyl-phosphine)gold(I)

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Key indicators

Single-crystal X-ray study

T = 223 K

Mean σ(C–C) = 0.008 Å

R factor = 0.042

wR factor = 0.115

Data-to-parameter ratio = 28.4

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

The Au atom in the title compound, $(c\text{-}C_6H_{11})_3PAu(S_2CNEt_2)$ or $[Au(C_{18}H_{33}P)(C_5H_{10}NS_2)]$, exists in a linear geometry so that Au—S is 2.3340 (11), Au—P is 2.2599 (10) Å and the angle at gold is 171.61 (4)°. The distortion from linearity may be traced to the close approach of the non-coordinating S atom which is separated from the Au atom by 3.0859 (13) Å.

Comment

In connection with previous studies on compounds of the general formula $R_3PAu(S_2COR')$, R and R' = alkyl (Siasios & Tiekink, 1993a,b), which demonstrated interesting flexibility in the mode of coordination of the thiolate ligands, the structure of the title compound, $(c\text{-}C_6H_{11})_3PAu(S_2CNEt_2)$, (I), was investigated.

(I)
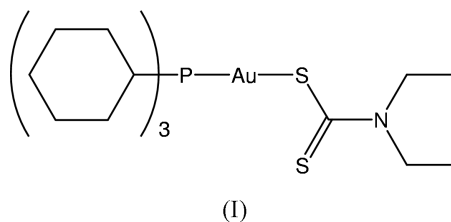
The Au atom in (I) exists in an approximately linear geometry defined by the S atom of the monodentate dithiocarbamate ligand and the P atom of the phosphine ligand (Fig. 1 and Table 1). There is a significant distortion from the ideal 180° angle for P—Au—S that may be correlated with the close approach of atom S2 to the Au centre. Thus, the Au···S2 separation is 3.0859 (13) Å, a distance that is well within the sum of the van der Waals radii, 3.50 Å, for these atoms (Bondi, 1964). Although there are a number of related gold–phosphine–dithiocarbamate structures in the literature, there is, surprisingly, only one directly comparable mononuclear structure available for comparison, namely that of the triphenylphosphine analogue (Wijnhoven et al., 1972). The respective Au—S bond distance in the present structure and that in $(C_6H_5)_3PAu(S_2CNEt_2)$ of 2.3340 (11) and 2.338 (3) Å, respectively, are equal within experimental error. Likewise, the Au—P bond distances of 2.2599 (10) and 2.251 (3) Å, respectively, are also equivalent. However, the distortion from linearity in the cyclohexyl derivative is somewhat greater as the P—Au—S angle in the triphenylphosphine analogue was determined to be 175.7 (1)°. These results show that in the uncongested Au atom environment, bulky phosphines may be accommodated without significant deviations in the geometric parameters about Au. A similar conclusion has been reported
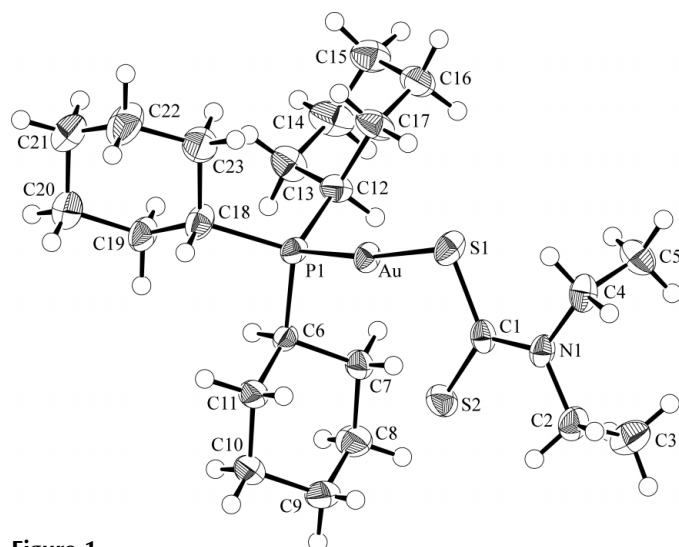
Figure 1
The molecular structure and crystallographic numbering scheme for (I); displacement ellipsoids are shown at the 50% probability level (Johnson, 1976).

previously for the closely related dithiocarbonate structures, *i.e.* $R_3PAu(S_2COR')$ (Siasios & Tiekink, 1993*a,b*). The crystal structure is stabilized by hydrophobic interactions.

Experimental

To a dichloromethane solution (4 ml) of $(C_6H_{11})_3PAuCl$ (0.20 g, 0.39 mmol) was added NaS_2CNEt_2 (67 mg, 0.39 mmol). The colourless solution immediately turned yellow, indicating the formation of the product, and was stirred for 2 h. The yellow solution was filtered through Celite and concentrated to approximately 1 ml to yield the product. The product was recrystallized by the vapour diffusion of hexane into a dichloromethane solution to yield X-ray quality yellow crystals of (I). ^{1}H NMR (CDCl$_3$): δ 3.91 (q, 4 H), 2.02–1.54 (m, 33 H), 1.30 (t, 6 H). ^{13}C{^{1}H} NMR (CDCl$_3$): δ 216.3 (s, CS$_2$), 48.9 (s, ethyl–CH$_2$), 33.5–25.9 (m, P(C$_6$H$_{11}$)$_3$), 12.1 (s, CH$_3$). ^{31}P{^{1}H} (CDCl$_3$): δ 55.3 p.p.m. ESI–MS: *m/z* 626 (M^+). IR (KBr): 1476 ν(C—N), 1083 and 991 ν(C—S) cm^{-1}.

Crystal data

$[Au(C_5H_{10}NS_2)(C_{18}H_{33}P)]$	$Z = 2$
$M_r = 625.64$	$D_x = 1.639$ Mg m^{-3}
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 10.1697 (5)$ Å	Cell parameters from 10496
$b = 11.7231 (6)$ Å	reflections
$c = 12.3281 (6)$ Å	$\theta = 1.8$–30.0°
$\alpha = 71.710 (1)°$	$\mu = 6.04$ mm^{-1}
$\beta = 77.917 (1)°$	$T = 223 (2)$ K
$\gamma = 65.803 (1)°$	Block, pale yellow
$V = 1267.36 (11)$ Å^3	$0.39 \times 0.31 \times 0.21$ mm

Data collection

Bruker AXS SMART CCD	7203 independent reflections
diffractometer	6682 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.036$
Absorption correction: empirical	$\theta_{max} = 30.0°$
($SADABS$; Bruker, 2000)	$h = -10 \rightarrow 14$
$T_{min} = 0.112$, $T_{max} = 0.297$	$k = -10 \rightarrow 16$
10496 measured reflections	$l = -17 \rightarrow 17$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o{}^2) + (0.0897P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.042$	where $P = (F_o{}^2 + 2F_c{}^2)/3$
$wR(F^2) = 0.115$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.05$	$\Delta\rho_{max} = 2.08$ e Å^{-3}
7203 reflections	$\Delta\rho_{min} = -2.91$ e Å^{-3}
254 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Au—P1	2.2599 (10)	P1—C12	1.858 (5)
Au—S1	2.3340 (11)	P1—C18	1.874 (4)
S1—C1	1.745 (4)	N1—C1	1.348 (5)
S2—C1	1.699 (4)	N1—C4	1.464 (6)
P1—C6	1.836 (4)	N1—C2	1.464 (6)
P1—Au—S1	171.61 (4)	C18—P1—Au	112.27 (15)
C1—S1—Au	98.38 (14)	C1—N1—C4	121.5 (4)
C6—P1—C12	105.79 (19)	C1—N1—C2	121.2 (4)
C6—P1—C18	106.16 (19)	C4—N1—C2	116.8 (4)
C12—P1—C18	112.0 (2)	N1—C1—S2	122.2 (3)
C6—P1—Au	112.06 (13)	N1—C1—S1	117.4 (3)
C12—P1—Au	108.40 (14)	S2—C1—S1	120.5 (2)

The C-bound H atoms were placed in their geometrically calculated positions and included in the final refinement in the riding-model approximation with an overall displacement parameter, U_{iso}, with $1.0U_{iso}$ for methine-H, $1.25U_{iso}$ for methylene-H and $1.5U_{iso}$ for CH$_3$. The residual electron-density peak is located in the vicinity of the Au atom.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SHELXTL* (Bruker, 2000); program(s) used to solve structure: *DIRDIF*92 *PATTY* (Beurskens *et al.*, 1992); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXTL*.

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