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(*N*,*N*-Diethyldithiocarbamato)[tris(*p*-methoxyphenyl)-phosphine]gold(I)

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Sulfur- and phosphorus-donor atoms define an approximately linear geometry about the Au atom in the title compound, $[Au(S_2CNEt_2)\{(p\text{-MeO-}C_6H_4)_3P\}]$. The key geometric parameters are Au—S 2.3350 (8) Å, Au—P 2.2528 (7) Å and an S—Au—P angle of 169.80 (3)°. The deviation from linearity is ascribed to a close intramolecular Au···S contact of 3.0564 (8) Å.

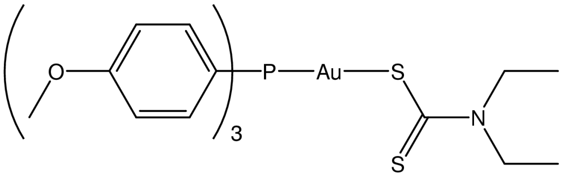
Key indicators

Single-crystal X-ray study
T = 223 K
Mean σ(C–C) = 0.005 Å
R factor = 0.033
wR factor = 0.078
Data-to-parameter ratio = 25.3

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

Comment

Intramolecular Au···S or Au···O interactions are found in structures with the general formula $R_3PAu(S_2COR')$ (R, R' = alkyl, aryl) and are thought to be dictated by global crystal-packing considerations (Siasios & Tiekink, 1993*a*,*b*). Analogous Au···S interactions are found in the related crystal structures of $(C_6H_5)_3PAu(S_2CNEt_2)$, (II) (Wijnhoven *et al.*, 1972), $(c\text{–}C_6H_{11})_3PAu(S_2CNEt_2)$, (III) (Ho & Tiekink, 2001*b*), and dinuclear $(Et_2NCS_2)AuP(C_6H_5)_2CH_2CH_2(C_6H_5)_2PAu(S_2CNEt_2)$, (IV) (Faamau & Tiekink, 1994). In this context, the structure of the title compound, $(p\text{-MeO-}C_6H_4)_3PAu(S_2CNEt_2)$, (I), was determined.



(I)

A linear geometry defined by the sulfur, derived from a monodentate dithiocarbamate ligand, and phosphorus, from the phosphine, is found in (I) (Fig. 1 and Table 1). The Au—S and Au—P separations of 2.3350 (8) and 2.2528 (7) Å, respectively, lie within the range of Au—S and Au—P distances found in the mononuclear structures of (II) and (III) mentioned above. To a first approximation, the geometry is linear, but a significant distortion away from the ideal angle of 180° is noted, as the S—Au—P angle is 169.80 (3)°. This may be correlated with the close approach of the non-coordinating atom S2 to the Au centre so that Au···S2 is 3.0564 (8) Å. This separation lies between the comparable Au···S separations found in (II) and (III), for which the angles subtended at gold were found to be 175.7 (1) and 171.61 (4)°, respectively.

There are two C—H···π contacts less than 3.2 Å. These occur between the methylene C4—H and the centroid of the C6–C11 ring (distance 2.95 Å and angle at H of 125°, with symmetry operation 1+x, y, z) and C17—H with the centroid of the C20–C25 ring (3.12 Å, 152° and x, 1/2−y, −1/2+z).

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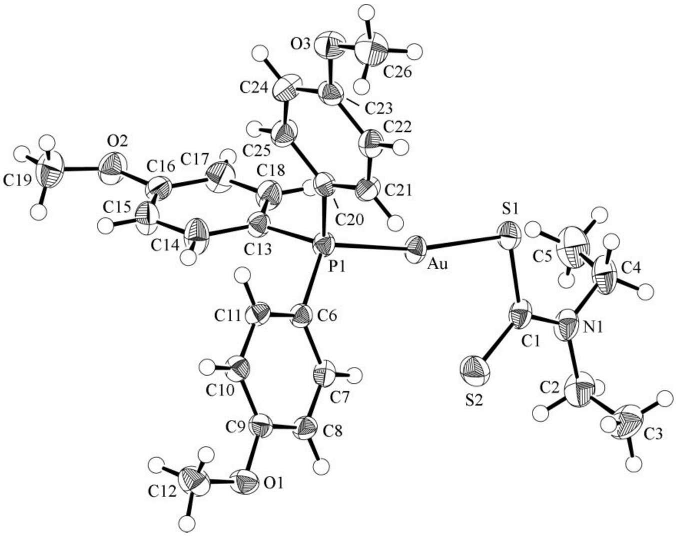

Figure 1

The molecular structure and crystallographic numbering scheme for (I). Displacement ellipsoids are shown at the 50% probability level.

Experimental

To a dichloromethane solution (4 ml) of (p-MeO-C_6H_4)$_3$PAuCl (0.20 g, 0.34 mmol; Ho & Tiekink, 2001*a*) was added NaS_2CNEt_2 (Merck; 58 mg, 0.34 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 methanol into a chloroform solution of the compound to yield X-ray quality pale-yellow crystals (168 mg, 71%); m.p. 404 K. ^{1}H NMR ($CDCl_3$): δ 7.57–7.49 (*m*, 6H, Ph), 6.95–6.91 (*m*, 6H, Ph), 3.93 (*q*, 4H, J = 7.5 Hz), 3.82 (*s*, 9H, OCH_3), 1.33 p.p.m. (*t*, 6H, J = 7.5 Hz). ^{31}P{^{1}H} ($CDCl_3$): δ 32.5 p.p.m. ESI–MS: m/z 1247 [(M^+–NEt_2)]$_2$. IR (KBr) 1499 ν(C—N), 1105 and 995 ν(C—S) cm^{-1}.

Crystal data

$[Au(C_5H_{10}NS_2)(C_{21}H_{21}O_3P)]$
M_r = 697.57
Monoclinic, $P2_1/c$
a = 10.4567 (4) Å
b = 15.6672 (7) Å
c = 17.0037 (7) Å
β = 105.576 (1)°
V = 2683.36 (19) Å^3
Z = 4
D_x = 1.727 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 21862 reflections
θ = 1.8–30.0°
μ = 5.73 mm^{-1}
T = 223 (2) K
Block, pale-yellow
0.42 × 0.10 × 0.10 mm

Data collection

Bruker AXS SMART CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*, Bruker, 2000)
T_{min} = 0.218, T_{max} = 0.564
21862 measured reflections
7806 independent reflections
6349 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 30.0°
$h = -14 \rightarrow 14$
$k = -22 \rightarrow 10$
$l = -23 \rightarrow 23$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.078
S = 1.02
7806 reflections
308 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0366P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 2.14 e Å^{-3}
$\Delta\rho_{min}$ = −1.26 e Å^{-3}

Table 1

Selected geometric parameters (Å, °).

Au—P1	2.2528 (7)	O1—C12	1.438 (4)
Au—S1	2.3350 (8)	O2—C16	1.371 (4)
S1—C1	1.760 (4)	O2—C19	1.420 (4)
S2—C1	1.683 (3)	O3—C23	1.362 (4)
P1—C13	1.808 (3)	O3—C26	1.414 (4)
P1—C6	1.814 (3)	N1—C1	1.339 (4)
P1—C20	1.813 (3)	N1—C4	1.471 (5)
O1—C9	1.364 (4)	N1—C2	1.476 (5)
P1—Au—S1	169.80 (3)	C16—O2—C19	116.8 (3)
C1—S1—Au	97.80 (11)	C23—O3—C26	118.0 (3)
C13—P1—C6	105.77 (14)	C1—N1—C4	123.2 (3)
C13—P1—C20	105.36 (15)	C1—N1—C2	120.7 (3)
C6—P1—C20	104.94 (14)	C4—N1—C2	116.1 (3)
C13—P1—Au	112.46 (10)	N1—C1—S2	123.3 (3)
C6—P1—Au	115.23 (11)	N1—C1—S1	116.6 (3)
C20—P1—Au	112.26 (9)	S2—C1—S1	120.05 (18)
C9—O1—C12	117.1 (3)		

The C-bound H atoms were placed in geometrically calculated positions and included in the final refinement in the riding-model approximation, with an overall displacement parameter U_{iso} = U_{eq}(C) for phenyl H, 1.25 × U_{eq}(C) for methylene H and 1.50 × U_{eq}(C) for CH_3 H atoms. The largest 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: *PATTY* in *DIRDIF*92 (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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