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(*O*-Ethyldithiocarbonato)[tris(*p*-methoxyphenyl)phosphine]gold(**I**)

Soo Yei Ho and
Edward R. T. Tiekink*

Department of Chemistry, National University of Singapore, Singapore 117543

Correspondence e-mail: chmtert@nus.edu.sg

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Key indicators

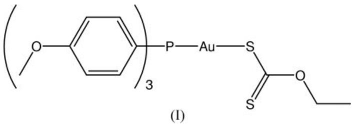
Single-crystal X-ray study
T = 208 K
Mean σ(C–C) = 0.004 Å
R factor = 0.028
wR factor = 0.068
Data-to-parameter ratio = 25.1

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, (p-MeOC$_6$H$_4$)$_3$PAu(S$_2$COEt) or [Au(C$_3$H$_5$OS$_2$)(C$_{21}$H$_{21}$O$_3$P)], exists in a linear geometry, such that the Au—S bond length is 2.3004 (8) Å and Au—P is 2.2505 (7) Å, and the angle at gold is 175.87 (3)°.

Comment

Dithiocarbonate (*i.e.* xanthate or $^{-}$S$_2$COR) ligands invariably coordinate to the metal centre in their phosphinegold(I) complexes *via* one of the S atoms, thereby leading to a linear P—Au—S arrangement (Tiekink, 1985; Siasios & Tiekink, 1992, 1993*a*,*b*). Weak intramolecular Au$\cdots X$ associations are also present and these can involve either the second S atom or the O atom. The requirements of efficient crystal packing are thought to play a major role in determining the nature of the weaker intramolecular interactions to Au in these complexes. In this context, the molecular structure of (p-MeOC$_6$H$_4$)$_3$PAu(S$_2$COEt), (I) (Fig. 1 and Table 1), was investigated. The Au atom exists within a linear geometry [175.87 (3)°] defined by S [2.3004 (8) Å] and P [2.2505 (7) Å] donor atoms. The orientation of the xanthate ligand is such that the S2 atom is in close proximity [3.4239 (11) Å] to the Au atom, as is normally found in these systems. The dihedral angles formed between pairs of aromatic rings are 59.01 (14), 86.03 (14) and 77.46 (14)°. The crystal structure is stabilized by C—H$\cdots\pi$ interactions and weaker O/S$\cdots$H contacts. Each of the aromatic rings appears to be involved in a C—H$\cdots\pi$ contact. C17—H17*C* is 2.94 Å from the centroid of C4$^{\rm i}$—C9$^{\rm i}$ with an angle at H17*C* of 163° [symmetry code: (i) $1 - x, y, \frac{1}{2} - z$], C10—H10*B* is 2.92 Å from the centroid of C11$^{\rm ii}$—C16$^{\rm ii}$ with an angle of 156° at H10*B* [symmetry code: (ii) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$], and C3—H3*A* is 2.97 Å from the centroid of C18$^{\rm iii}$—C23$^{\rm iii}$ with an angle of 154° at H3*A* [symmetry code: (iii) $-\frac{1}{2} + x, \frac{1}{2} - y, -\frac{1}{2} + z$].


(I)

Experimental

The title compound was prepared in 65% yield from the reaction between (p-MeOC$_6$H$_4$)$_3$PAuCl (Ho & Tiekink, 2001) and K[S$_2$COEt], using established procedures (Tiekink, 1985). Yellow crystals were obtained from the vapour diffusion of diethyl ether into a chloroform solution of the compound; m.p. 409–410 K. ^{1}H NMR (CDCl$_3$): δ 7.48–7.28 (*m*, 6H), 6.98–6.95 (*m*, 6H), 4.54 (*q*, 2H, *J* = 7.1 Hz), 3.84 (*s*, 9H),

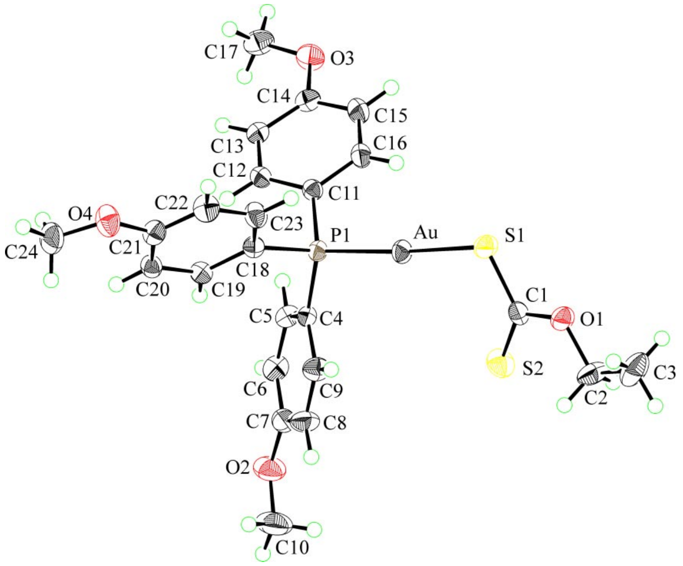


Figure 1
The molecular structure and crystallographic numbering scheme for (I). Displacement ellipsoids are shown at the 50% probability level (Johnson, 1976).

1.39 p.p.m. (*t*, 3H, $J = 7.1$ Hz). $^{31}P\{^1H\}$ NMR ($CDCl_3$): δ 33.7 p.p.m. ESI–MS: m/z = 902 $[\{(p\text{-MeOC}_6H_5)_3P\}_2Au]^+$ and 1220 $[\{(p\text{-MeOC}_6H_5)_3P\}_2Au_2(S_2COEt)]^+$. IR (KBr): 1182 ($\nu$C—O) and 1047 cm^{-1} ($\nu$C—S).

Crystal data

$[AuC_3H_5OS_2)(C_{21}H_{21}O_3P)]$
$M_r = 670.50$
Monoclinic, $C2/c$
$a = 14.7524$ (8) Å
$b = 15.2702$ (8) Å
$c = 22.7615$ (14) Å
$\beta = 102.634$ (2)°
$V = 5003.4$ (5) Å^3
$Z = 8$

$D_x = 1.780$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7220 reflections
$\theta = 2.3$–29.4°
$\mu = 6.14$ mm^{-1}
$T = 208$ (2) K
Block, yellow
0.29 × 0.26 × 0.12 mm

Data collection

Bruker AXS SMART CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.232$, $T_{max} = 0.479$
20818 measured reflections

7255 independent reflections
5989 reflections with $I > 2\sigma(I)$
$R_{int} = 0.039$
$\theta_{max} = 30.0°$
$h = -14 \rightarrow 20$
$k = -21 \rightarrow 19$
$l = -31 \rightarrow 31$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.068$
$S = 1.00$
7255 reflections
289 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0316P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.15$ e Å^{-3}
$\Delta\rho_{min} = -0.80$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Au—P1	2.2505 (7)	P1—C11	1.805 (3)
Au—S1	2.3004 (8)	P1—C18	1.808 (3)
S1—C1	1.724 (3)	O1—C1	1.331 (3)
S2—C1	1.650 (3)	O1—C2	1.449 (4)
P1—C4	1.810 (3)		
P1—Au—S1	175.87 (3)	Au—P1—C11	112.51 (10)
Au—S1—C1	104.25 (10)	Au—P1—C18	113.85 (9)
C4—P1—C11	107.44 (13)	C1—O1—C2	119.1 (3)
C4—P1—C18	104.06 (13)	S1—C1—S2	127.03 (17)
C11—P1—C18	105.69 (12)	S1—C1—O1	108.9 (2)
Au—P1—C4	112.62 (9)	S2—C1—O1	124.1 (2)

H atoms were placed in calculated positions and included in the final refinement in the riding-model approximation. The residual electron-density peak was located in the vicinity of the Au atom.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT* and *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: SHEXLTL.

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References

Beurskens, P. T., Admiraal, G., Beurskens, G., Bosman, W. P., Garcia-Granda, S., Gould, R. O., Smits, J. M. M. & Smykalla, C. (1992). *The DIRDIF Program System*. Technical Report. Crystallography Laboratory, University of Nijmegen, The Netherlands.
Bruker (2000). *SMART, SAINT* and *SHELXTL* (Versions V5.6) and *SADABS* (Version 2.01). Bruker AXS Inc., Madison, Wisconsin, USA.
Ho, S. Y. & Tiekink, E. R. T. (2001). *Acta Cryst.* E**57**, m549–m550.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Siasios, G. & Tiekink, E. R. T. (1992). *Z. Kristallogr.* **198**, 139–141.
Siasios, G. & Tiekink, E. R. T. (1993*a*). *Z. Kristallogr.* **199**, 95–105.
Siasios, G. & Tiekink, E. R. T. (1993*b*). *Z. Kristallogr.* **199**, 261–270.
Tiekink, E. R. T. (1985). *Z. Kristallogr.* **173**, 243–247.