

(4,6-Dimethylpyrimidine-2-thiolato)-
(triphenylphosphine)gold(I)

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Key indicators

Single-crystal X-ray study

$T = 293\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.015\text{ \AA}$

R factor = 0.047

wR factor = 0.100

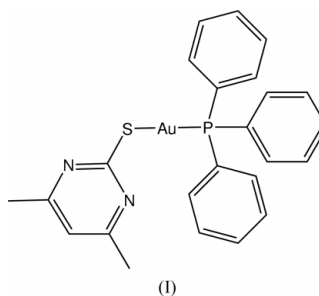
Data-to-parameter ratio = 18.9

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

The title compound, $[\text{Au}(4,6\text{-Me}_2\text{pymS})(\text{PPh}_3)]$ or $[\text{Au}(\text{C}_6\text{H}_7\text{N}_2\text{S})(\text{C}_{18}\text{H}_{15}\text{P})]$, exhibits a linear Au atom S—Au—P geometry, with Au—P = 2.247 (2) Å, Au—S = 2.289 (2) Å and P—Au—S = 178.19 (11)°. There is an intramolecular interaction between one N atom of the 4,6-Me₂pymS ligand and the Au atom, with an Au···N distance of 3.154 (8) Å.

Comment

The preparation of the novel two triphenylphosphinegold(I) complexes of the anions derived from pyridine-2-thione (2-pySH) and 1*H*-pyrimidine-2-thione (2-pymSH) have been recently reported, together with their X-ray crystal structure determinations (Cookson & Tiekink, 1993). In this paper, we report the preparation and structural characterization of the title compound, (I).



The anionic 4,6-Me₂pymS ligand is coordinated *via* the S atom (Fig. 1 and Table 1). The Au—S bond length and S—Au—P angle in (I) are close to those found for the two complexes $[\text{Au}(2\text{-pyS})(\text{PPh}_3)]$ [2.297 (2) Å and 177.9 (1)°, respectively] and $[\text{Au}(2\text{-pymS})(\text{PPh}_3)]$ [2.310 (3) Å and 174.7 (1)°, respectively] (Cookson & Tiekink, 1993). The Au atom shows nearly linear coordination with the S and P atoms, in spite of a fairly close intramolecular interaction with an N atom of the 4,6-Me₂pymS ligand. This interaction, Au1···N42 of 3.154 (8) Å, is slightly less than the sum of the van der Waals radii (Pauling, 1960) of the Au and N atoms, 3.25 Å. Given the linearity of the S—Au—P angle, the 4,6-Me₂pymS ligand appears to be coordinating essentially in the monodentate mode through its S atom. There is no metal–metal interaction in the solid state but weak (Desiraju & Steiner, 1999) intermolecular C—H···N hydrogen bonds are present (Table 2).

In CDCl₃ solution at room temperature, the ¹H NMR data shows inequivalent methyl groups for the anionic 4,6-Me₂pymS ligand. This suggests that the ligand is not permitted to rotate freely in solution, which is consistent with the persistence of the intramolecular interaction between the N atom of the ligand and the Au atom.

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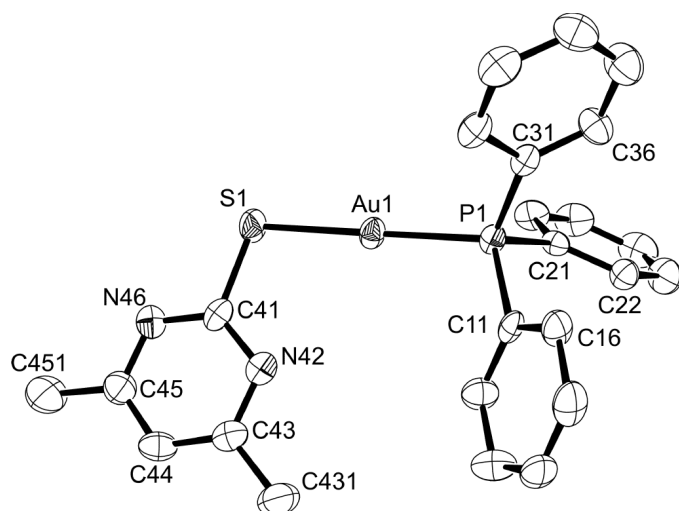


Figure 1
View of molecule (I), with the atomic labeling scheme. Displacement ellipsoids are drawn at the 50% probability level.

Experimental

The compound 4,6-dimethylpyrimidine-2(1*H*)-thione (4,6-Me₂-pymSH) was prepared according to the literature method (Nigam *et al.*, 1983) and the title complex was synthesized by the following procedure: 0.28 g (2 mmol) of 4,6-Me₂pymSH was dissolved in 30 ml of THF (freshly distilled) in a Schlenk tube kept under an N₂ atmosphere. To the solution, 0.84 ml (6 mmol) of triethylamine, NEt₃, was added and the resulting mixture was stirred for 30 min. To this solution, 0.99 g (2 mmol) of [AuCl(PPh₃)] dissolved in 10 ml of THF was added, over a period of 10 min, and the mixture was stirred for about 3 h. A white flocculent precipitate was filtered off and identified as triethylamine hydrochloride, [(HNEt₃)Cl]. Slow evaporation of the solvent led to the appearance of a white microcrystalline solid [yield 1.0 g (92%)]. Well developed crystals of (I), suitable for X-ray analysis, were obtained by recrystallization from a 1:1 (*v/v*) mixture of dichloromethane and *n*-hexane (m.p. 470–472 K).

Crystal data

[Au(C₆H₇N₂S)(C₁₈H₁₅P)]
 $M_r = 598.43$
 Triclinic, $P\bar{1}$
 $a = 8.8282$ (17) Å
 $b = 11.144$ (2) Å
 $c = 12.318$ (3) Å
 $\alpha = 97.104$ (19)°
 $\beta = 105.586$ (18)°
 $\gamma = 95.05$ (2)°
 $V = 1148.9$ (4) Å³
 $Z = 2$
 $D_x = 1.730$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 25 reflections
 $\theta = 7.3$ – 15.2 °
 $\mu = 6.58$ mm⁻¹
 $T = 293$ (2) K
 Block, colorless
 0.20 × 0.15 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: ψ scan (ABSPSI routine in PLATON; Spek, 1995)
 $T_{\min} = 0.318$, $T_{\max} = 0.518$
 8437 measured reflections
 5002 independent reflections
 2962 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.089$
 $\theta_{\text{max}} = 27.0$ °
 $h = -11 \rightarrow 11$
 $k = -14 \rightarrow 14$
 $l = -15 \rightarrow 15$
 3 standard reflections every 300 reflections
 frequency: 3600 min
 intensity decay: 12.4%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.047$
 $wR(F^2) = 0.100$
 $S = 0.98$
 5002 reflections
 264 parameters
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0366P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.016$
 $\Delta\rho_{\text{max}} = 0.88$ e Å⁻³
 $\Delta\rho_{\text{min}} = -1.43$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Au1–P1	2.247 (2)	Au1–N42	3.154 (8)
Au1–S1	2.289 (2)	S1–C41	1.749 (10)
P1–Au1–S1	178.19 (11)	S1–Au1–N42	56.27 (14)
P1–Au1–N42	123.97 (14)	C41–S1–Au1	104.4 (3)

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C14–H14 ⁱ ···N46 ⁱ	0.93	2.65	3.547 (13)	162
C33–H33 ⁱ ···N46 ⁱⁱ	0.93	2.63	3.426 (14)	144

Symmetry codes: (i) $x, 1 + y, z$; (ii) $1 - x, -y, 1 - z$.

The H atoms were constrained to geometric positions fixed to the parent atoms with distances of 0.93 and 0.96 Å for aromatic and methyl H atoms, respectively. The isotropic displacement parameters were fixed to 102 and 150% of that of the parent atom for the aromatic and methyl H atoms, respectively.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *SET4* in *CAD-4 Software*; data reduction: *HELENA* (Spek, 1995); 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: *SHELXL97*.

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