A second polymorph of (2-thiazolidinethionato)-(triphenylphosphine)gold(I)

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The title compound, $[Au(C_3H_4NS_2)(C_{18}H_{15}P)]$, contains a thiazolidinethione ligand coordinated to an Au^I atom through the S atom. A triphenylphosphine ligand completes the linear coordination geometry around the Au atom. The compound contains two molecules in the asymmetric unit and is a polymorphic form of a previously reported structure [Grant, Forward & Fackler Jr (1996). *Z. Kristallogr.* **211**, 483–484]. The composite molecules in the two forms exhibit nearly identical bond distances and angles.

Comment

In an ongoing study of utilizing various coordination modes of azoles in gold chemistry (Raubenheimer & Cronje, 2001), it became necessary to include sulfur derivatives of azoles to establish coordination preferences (Cronje *et al.*, 2003). Data collection at 100 K showed that the gold(I) compound, (I), had crystallized also in space group $P\bar{1}$ with two formula units per unit cell; this structure differs significantly from a previously reported crystal structure of this compound (Grant *et al.*, 1996), also obtained in space group $P\bar{1}$ but at 293 K and with a final R value of 0.03. We interpreted this result as indicating either that the structure undergoes a phase transition above 100 K or that we had identified a second polymorph. Unit cell determination of (I) at room temperature agreed with our data determined at 100 K and still differed from the single-crystal X-ray data of Grant and co-workers. We concluded that the structure presented here is a genuine polymorph and not the consequence of a phase transition from the previously reported form as a result of cooling. In a subsequent effort, we grew new crystals and identified an additional triclinic polymorph of (I), described by Horvath & Raubenheimer (2006).

(I)
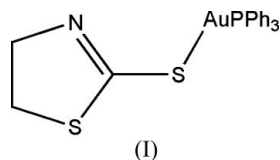
The molecular structure of (I) is shown in Fig. 1. Analogous to the previously reported structure, the Au atom shows linear coordination [176.96 (5)°] by the S atom of the sulfide unit and the P atom of the phosphine ligand. The Au—S bond length, 2.3051 (15) Å, is similar to reported values in other essentially comparable complexes (Cronje *et al.*, 2003). In general, all the bond distances and angles correspond to the values of the previously reported structure (Grant *et al.*, 1996). Crystal packing results from N···H (2.53 Å) and S···H (2.96 Å) contacts between the thione ligand and the phenyl groups in
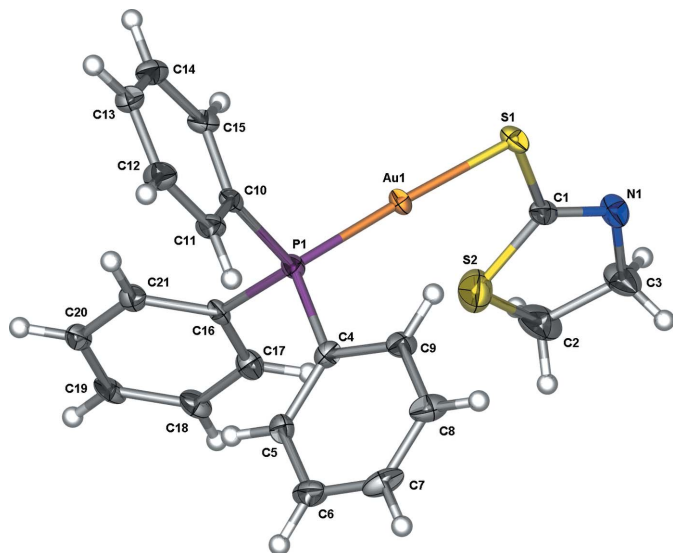
Figure 1
The molecular structure of (I), with atom labels and 50% probability displacement ellipsoids for non-H atoms.
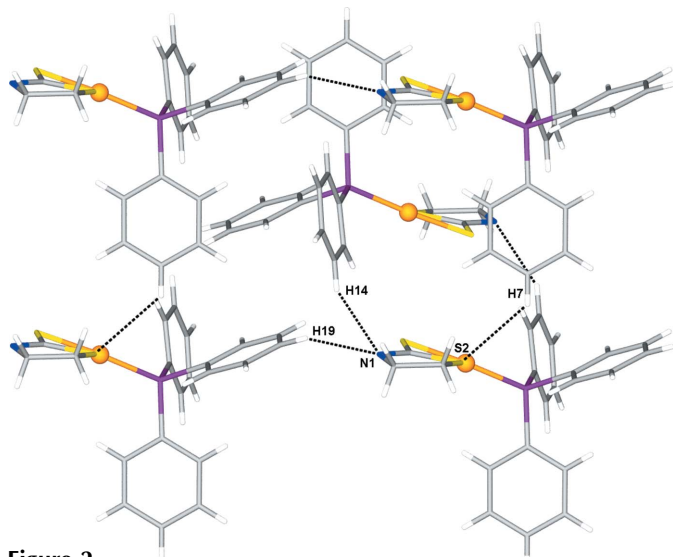
Figure 2
The packing, viewed along the *b* axis. The closest N···H and S···H contacts are shown as dashed lines. The symmetry codes for atoms H7, H14 and H19 are $(x - 1, y, z)$, $(-x, 1 - y, z)$ and $(x, y, z - 1)$, respectively.

adjacent molecules (Fig. 2). The P atom is pseudo-tetrahedrally coordinated but now with a significantly larger angle of 115.7 (2)° for C10—P1—Au1. The C—P—C bond angles, with an average value of 105°, correlate with the result for the known polymorph. The major difference between the molecules in the two polymorphs is that the thiazole ring is slightly rotated in our form, which now has an S2—C1—S1—Au1 torsion angle of 16.7 (4)° compared with an angle of −20.3° in the known structure.

Experimental

A suspension of triphenylphosphinegold(I) chloride (0.287 g, 0.58 mmol), 2-thiazolidinethione (60 mg, 0.58 mmol) and NaOH

(26 mg, 0.65 mmol) in MeOH (20 ml) was stirred for 1 h at room temperature. The mixture was reduced to dryness *in vacuo*. The crude product was extracted with CH_2Cl_2 (15 ml), followed by filtration over Celite and solvent removal *in vacuo* to yield colourless microcrystalline material (0.28 g, 82%). Single crystals suitable for X-ray diffraction were obtained by slow evaporation of a CH_2Cl_2 solution at 243 K (m.p. 328–335 K). FAB–MS confirmed the purity of the compound. IR (KBr, ν_{max}/cm^{-1}): 3068, 3049, 2962, 1686, 1576, 1547, 1479, 1435, 1296, 1261, 1099, 1026, 997, 957, 910, 802, 746, 692; ^{1}H NMR (CD_2Cl_2): δ 7.53 (*m*, 15H), 4.22 (*m*, 2H), 3.81 (*m*, 2H); ^{13}C NMR (CD_2Cl_2): δ 169.0, 134.9 (*d*, *J* = 14.1 Hz), 132.4, 130.5, 129.8 (*d*, *J* = 11.5 Hz), 68.6, 66.2; ^{31}P NMR (CD_2Cl_2): δ 37.4; FAB–MS: *m/z* = 578 (26%) [*M*]$^+$, 459 (82%) [AuPPh$_3$].

Crystal data

[Au(C$_3$H$_4$NS$_2$)(C$_{18}$H$_{15}$P)]	$V = 998.4$ (3) Å^3
$M_r = 577.43$	$Z = 2$
Triclinic, $P\bar{1}$	$D_x = 1.921$ Mg m^{-3}
$a = 8.8944$ (13) Å	Mo $K\alpha$ radiation
$b = 10.6804$ (16) Å	$\mu = 7.66$ mm^{-1}
$c = 11.4533$ (17) Å	$T = 100$ (2) K
$\alpha = 86.763$ (3)°	Block, colourless
$\beta = 87.498$ (2)°	$0.14 \times 0.10 \times 0.07$ mm
$\gamma = 66.828$ (3)°	

Data collection

Bruker APEX CCD area-detector diffractometer	5909 measured reflections
ω scans	4108 independent reflections
	3789 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Blessing, 1995)	$R_{int} = 0.019$
$T_{min} = 0.319$, $T_{max} = 0.585$	$\theta_{max} = 26.8°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.049P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.035$	$+ 4.1532P]$
$wR(F^2) = 0.088$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} < 0.001$
4108 reflections	$\Delta\rho_{max} = 2.39$ e Å^{-3}
235 parameters	$\Delta\rho_{min} = -1.53$ e Å^{-3}
H-atom parameters constrained	

All H atoms were positioned geometrically (C—H = 0.95 and 0.99 Å for aromatic CH and CH$_2$, respectively) and constrained to ride on their parent atoms; U_{iso}(H) values were set at 1.2 times U_{eq}(C). The highest peak is located at 0.82 Å and the deepest hole at 0.91 Å from atom Au1.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Atwood & Barbour, 2003; Barbour, 2001); software used to prepare material for publication: *X-SEED*.

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