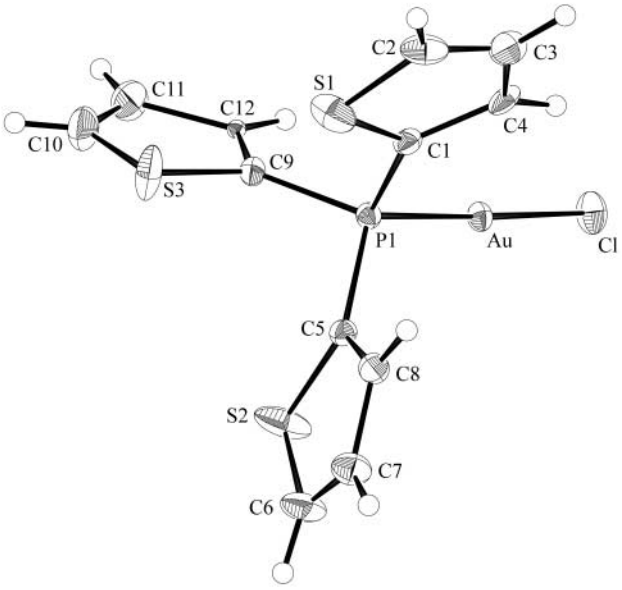
Crystal structure of chloro[tris(2-thienyl)phosphine]gold(I), $C_{12}H_9AuClPS_3$

S. Y. Ho and E. R. T. Tiekink*

The National University of Singapore, Department of Chemistry, Singapore 117543

Received December 16, 2002, accepted and available on-line January 21, 2003; CCDC-No. 1267/997



Abstract

$C_{12}H_9AuClPS_3$, triclinic, $P\bar{1}$ (No. 2), a = 9.0698(5) Å, b = 9.1073(5) Å, c = 10.4612(6) Å, α = 112.926(1)°, β = 102.513(1)°, γ = 98.132(1)°, V = 752.1 Å^3, Z = 2, $R_{gt}(F)$ = 0.031, $wR_{ref}(F^2)$ = 0.079, T = 203 K.

Source of material

(Thienyl)$_3$PAuCl was prepared from the reaction between Na[AuCl$_4$] · 2H$_2$O (Aldrich) and tri(2-thienyl)P (Lancaster Synthesis) in accord with the literature procedure [1]. Colourless crystals were obtained from the slow evaporation of an ethanol solution of the compound (mp 461 K – 462 K). ^{1}H NMR and ^{31}P NMR results are included in the deposited CIF-file.

Experimental details

The H atoms were placed in their geometrically calculated positions and included in the final refinement in the riding model approximation. Disorder was evident in the thienyl rings. One of these was modelled over with two sites for the S3/C12 atoms. The major component had s.o.f. = 0.55(1). The S3 atoms were refined anisotropically but the C12 atoms were not. Alternate sites could not be resolved for the other two rings.

Discussion

Phosphinegold(I) thiolates demonstrate biological activity [2] thereby making their study of some interest. In this context, various phosphinegold(I) thiolates are formed from their phosphinegold(I) chloride precursors. In order to verify the nature of the newly prepared title compound, an X-ray structure determination was conducted.

A linear geometry is found for the Au atom so that d(Au—Cl) = 2.285(1) Å, d(Au—P) = 2.227(1) Å with the angle at Au being 177.99(4)°. The structure is in essential agreement (but, is not isomorphous) with the recently determined 2-furanyl derivative [3]. There are no significant aurophilic interactions in the lattice. The lattice is stabilised by C–H···π interactions so that the distance between C11-H and the ring centroid of the 2-thienyl ring containing the S1 atom is 2.69 Å with an angle of 138° subtended at the H atom; symmetry operation: 1–x, 1–y, 1–z.

Table 1. Data collection and handling.

Crystal:	colourless block, size 0.13 × 0.16 × 0.36 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	104.59 cm^{-1}
Diffractometer, scan mode:	Bruker AXS SMART CCD, ω
$2\theta_{max}$:	60°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6379, 4315
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3943
$N(param)_{refined}$:	171
Programs:	DIRDIF92 [4], teXsan [5], SHELXL-97 [6], ORTEPII [7], PLATON [8]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	x	y	z	U_{iso}
C(12a)	2*i*	0.55(1)	0.393(2)	0.595(2)	0.335(2)	0.051(5)
H(12a)	2*i*	0.55	0.4380	0.5594	0.2584	0.062
C(12b)	2*i*	0.45	0.224(3)	0.612(3)	0.461(2)	0.040(5)
H(12b)	2*i*	0.45	0.1308	0.5922	0.4846	0.048
H(2)	2*i*		0.1399	–0.0413	0.4802	0.047
H(3)	2*i*		0.1947	–0.1874	0.2604	0.046
H(4)	2*i*		0.2071	–0.0332	0.1089	0.036
H(6)	2*i*		–0.3841	0.4393	0.0756	0.052
H(7)	2*i*		–0.4204	0.1951	0.1107	0.043
H(8)	2*i*		–0.1733	0.1264	0.1843	0.032
H(10)	2*i*		0.3762	0.8597	0.6361	0.058
H(11)	2*i*		0.5508	0.8350	0.4942	0.060

* Correspondence author (e-mail: chmtert@nus.edu.sg)

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Au	2*i*		0.22290(2)	0.24112(2)	0.00247(2)	0.0252(1)	0.02001(9)	0.01933(9)	0.00710(6)	0.00912(6)	0.00773(6)
Cl	2*i*		0.3129(2)	0.1743(2)	–0.1956(1)	0.0473(7)	0.0328(6)	0.0295(6)	0.0107(5)	0.0230(5)	0.0113(5)
S(1)	2*i*		0.1225(2)	0.1891(2)	0.4353(2)	0.0547(9)	0.0651(9)	0.0476(8)	0.0365(8)	0.0328(7)	0.0392(8)
S(2)	2*i*		–0.1098(2)	0.4816(2)	0.1362(2)	0.0286(7)	0.0534(8)	0.106(1)	0.0169(6)	0.0197(8)	0.061(1)
S(3a)	2*i*	0.55(1)	0.2175(5)	0.6061(5)	0.4964(4)	0.062(2)	0.033(1)	0.037(2)	–0.005(1)	0.022(2)	–0.008(1)
S(3b)	2*i*	0.45	0.4254(5)	0.5918(5)	0.3208(7)	0.026(2)	0.025(2)	0.075(3)	–0.006(1)	0.020(2)	–0.004(1)
P(1)	2*i*		0.1437(1)	0.3105(1)	0.2005(1)	0.0195(5)	0.0189(5)	0.0184(5)	0.0054(4)	0.0066(4)	0.0074(4)
C(1)	2*i*		0.1530(5)	0.1611(5)	0.2717(4)	0.023(2)	0.024(2)	0.018(2)	0.005(2)	0.003(2)	0.009(2)
C(2)	2*i*		0.1473(6)	0.0013(7)	0.4125(6)	0.028(2)	0.052(3)	0.053(3)	0.010(2)	0.010(2)	0.039(3)
C(3)	2*i*		0.1785(7)	–0.0811(6)	0.2876(6)	0.042(3)	0.025(2)	0.042(3)	0.002(2)	–0.003(2)	0.018(2)
C(4)	2*i*		0.1854(6)	0.0054(5)	0.1985(5)	0.039(3)	0.019(2)	0.020(2)	–0.002(2)	–0.010(2)	0.012(2)
C(5)	2*i*		–0.0538(5)	0.3260(5)	0.1741(4)	0.022(2)	0.020(2)	0.021(2)	0.004(2)	0.004(2)	0.009(2)
C(6)	2*i*		–0.3021(6)	0.3952(7)	0.1039(7)	0.023(2)	0.051(3)	0.063(4)	0.016(2)	0.011(2)	0.031(3)
C(7)	2*i*		–0.3224(6)	0.2581(6)	0.1233(6)	0.025(2)	0.043(3)	0.045(3)	0.010(2)	0.013(2)	0.022(2)
C(8)	2*i*		–0.1799(5)	0.2182(5)	0.1650(5)	0.025(2)	0.025(2)	0.029(2)	0.006(2)	0.007(2)	0.011(2)
C(9)	2*i*		0.2592(5)	0.5075(5)	0.3387(5)	0.022(2)	0.020(2)	0.023(2)	0.005(2)	0.005(2)	0.005(2)
C(10)	2*i*		0.3615(8)	0.7643(7)	0.5506(7)	0.061(4)	0.026(2)	0.042(3)	0.008(3)	0.006(3)	0.004(2)
C(11)	2*i*		0.4586(7)	0.7557(7)	0.4706(7)	0.031(3)	0.033(3)	0.060(4)	–0.003(2)	0.000(3)	0.003(3)

Acknowledgment. The National University of Singapore is thanked for support (R-143-000-139-112).

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