Crystal structure of chloro[tris(2-thienyl)phosphine]gold(I), $C_{12}H_9AuClP_3S_3$

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

$C_{12}H_9AuClP_3S_3$, triclinic, $P\bar{1}$ (No. 2), $a = 9.0698(5)$ Å, $b = 9.1073(5)$ Å, $c = 10.4612(6)$ Å, $\alpha = 112.926(1)^\circ$, $\beta = 102.513(1)^\circ$, $\gamma = 98.132(1)^\circ$, $V = 752.1$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.031$, $wR_{\text{ref}}(F^2) = 0.079$, $T = 203$ K.

Source of material

(Thienyl)₃PAuCl was prepared from the reaction between $Na[AuCl_4] \cdot 2H_2O$ (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). ¹H NMR and ³¹P 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 $d(\text{Au}—\text{Cl}) = 2.285(1)$ Å, $d(\text{Au}—\text{P}) = 2.227(1)$ Å with the angle at Au being $177.99(4)^\circ$. The structure is in essential agreement (but, is not isomorphous) with the recently determined 2-furanyl derivative [3]. There are no significant auophilic 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 $0.13 \times 0.16 \times 0.36$ mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ :	104.59 cm^{-1}
Diffractometer, scan mode:	Bruker AXS SMART CCD, ω 60°
$2\theta_{\text{max}}$:	$6379, 4315$
$N(hkl)_{\text{measured}}, N(hkl)_{\text{unique}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}}), 3943$
Criterion for $I_{\text{obs}}, N(hkl)_{\text{gt}}$:	171
$N(\text{param})_{\text{refined}}$:	
Programs:	DIRDIF92 [4], teXsan [5], SHELXL-97 [6], ORTEPII [7], PLATON [8]

Table 2. Atomic coordinates and displacement parameters (in Å²).

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

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

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	Occ.	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Au	2i		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	2i		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)	2i		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)	2i		−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)	2i	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)	2i	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)	2i		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)	2i		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)	2i		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)	2i		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)	2i		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)	2i		−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)	2i		−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)	2i		−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)	2i		−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)	2i		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)	2i		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)	2i		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).

References

1. Al-Sa'ady, A. K.; McAuliffe, C. A.; Parish, R. V.; Sandbank, J. A.: A general synthesis for gold(I) complexes. *Inorg. Synth.* **23** (1985) 191-194.
2. Tiekink, E. R. T.: Gold derivatives for the treatment of cancer. *Crit. Rev. Oncol/Hemato.* **42** (2002) 225-248.
3. Ho, S. Y.; Tiekink, E. R. T.: Crystal structure of chloro[tris(2-furanyl)-phosphine]gold(I), C₁₂H₉AuClO₃P. *Z. Kristallogr. NCS* **217** (2002) 589-590.
4. Beurskens, P. T.; Admiraal, G.; Beurskens, G.; Bosman, W. P.; García-Granda, S.; Smits, J. M. M.; Smykalla, C.: The DIRDIF program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands 1992.
5. teXsan: Single Crystal Structure Analysis Software. Version 1.04. Molecular Structure Corporation. The Woodlands, TX, USA 1997.
6. Sheldrick, G. M.: SHELXL-97. Program for crystal structure refinement. University of Göttingen, Germany 1997.
7. Johnson, C. K.: ORTEPII. Report ORNL-5138, Oak Ridge National Laboratory, Tennessee, USA 1976.
8. Spek, A.: PLATON. Utrecht University, The Netherlands 2000.