Crystal structure of bis(benzamide-2-thiolato)(μ_2 -bis(diphenylphosphino)ethane)digold(I), $C_{40}H_{36}Au_2N_2O_2P_2S_2$

D. R. Smyth and E. R. T. Tiekink*,¹

The University of Adelaide, Department of Chemistry, Australia 5005

Received June 10, 2002 accepted and available on-line August 6, 2002; CCDC-No. 1267/878

Abstract

$C_{40}H_{36}Au_2N_2O_2P_2S_2$, triclinic, $P\bar{1}$ (No. 2), $a = 10.673(2)$ Å, $b = 11.078(2)$ Å, $c = 9.066(2)$ Å, $\alpha = 107.54(2)^\circ$, $\beta = 113.58(2)^\circ$, $\gamma = 84.84(2)^\circ$, $V = 936.2$ Å³, $Z = 1$, $R_{\text{gt}}(F) = 0.026$, $wR_{\text{ref}}(F^2) = 0.065$, $T = 173$ K.

Source of material

The title compound was prepared in 82% yield from the 1:2 reaction between $(Ph_2PCH_2CH_2PPh_2)(AuCl)_2$ and 2-mercapto-benzamide [1]. Pale-yellow crystals were obtained from the vapour diffusion of acetone into a dimethylformamide solution of the compound; mp 513 K – 515 K (decomposition).

Experimental details

The H atoms were placed in their geometrically calculated positions and included in the final refinement in the riding model approximation.

Discussion

The dinuclear molecule is situated about a crystallographic centre of inversion so that the thiolate groups are *anti*. The gold atom exists in a linear environment, $174.92(4)^\circ$, defined by S ($d(\text{Au}—\text{S}) = 2.316(1)$ Å) and P ($d(\text{Au}—\text{P}) = 2.251(1)$ Å) donors. Molecules

aggregate via hydrogen bonding interactions involving the amide groups to form chains along the *a*-direction. Thus, $d(\text{H1a} \cdots \text{O1}^i)$ is 2.03 Å, $d(\text{N1} \cdots \text{O1}^i)$ is 2.883(7) Å and the angle at H1a is 162° for symmetry operation *i*: $-x, 1-y, 1-z$. The second amide hydrogen atom, H1b, forms an intramolecular interaction with the S atom so that $d(\text{H1b} \cdots \text{S2})$ is 2.33 Å, $d(\text{N1} \cdots \text{S2})$ is 3.046(5) Å and the angle at H1b is 138° . Chains are linked by $\text{Au} \cdots \text{S}^{ii}$ interactions (3.403(2) Å) formed between centrosymmetrically molecules resulting in the formation of ($\text{Au} \cdots \text{S}$) rectangles; symmetry operation *ii*: $-x, 1-y, -z$. The structure of the title compound is isomorphous with the carboxylic acid analogue [2].

Table 1. Data collection and handling.

Crystal:	pale-yellow block, size $0.06 \times 0.13 \times 0.21$ mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ :	80.89 cm^{-1}
Diffractionmeter, scan mode:	Rigaku AFC7R, $\omega/2\theta$
$2\theta_{\text{max}}$:	55°
$N(hkl)_{\text{measured}}, N(hkl)_{\text{unique}}$:	4524, 4291
Criterion for $I_{\text{obs}}, N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3477
$N(\text{param})_{\text{refined}}$:	226
Programs:	teXsan [3], DIRDIF92 [4], SHELXL-97 [5], DIFABS [6], ORTEPII [7]

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

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(1a)	2i	−0.0679	0.4773	0.3416	0.051
H(1b)	2i	−0.0607	0.4006	0.1747	0.051
H(3)	2i	0.1390	0.0804	−0.0943	0.042
H(4)	2i	0.2188	−0.0788	0.0332	0.050
H(5)	2i	0.2276	−0.0488	0.3060	0.048
H(6)	2i	0.1498	0.1352	0.4390	0.043
H(12)	2i	0.2785	0.6749	0.0129	0.039
H(13)	2i	0.3667	0.7449	−0.1468	0.053
H(14)	2i	0.6017	0.7677	−0.0610	0.046
H(15)	2i	0.7493	0.7154	0.1781	0.043
H(16)	2i	0.6642	0.6490	0.3401	0.035
H(22)	2i	0.1595	0.6834	0.4036	0.036
H(23)	2i	0.1165	0.8607	0.5917	0.046
H(24)	2i	0.2796	1.0233	0.7501	0.048
H(25)	2i	0.4915	1.0096	0.7304	0.049
H(26)	2i	0.5371	0.8330	0.5410	0.040
H(31a)	2i	0.5638	0.4589	0.3799	0.030
H(31b)	2i	0.6011	0.5912	0.5257	0.030

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

¹ Current address: National University of Singapore, Department of Chemistry, Singapore 117543

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

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Au	2i	0.20603(2)	0.46782(2)	0.14226(2)	0.0232(1)	0.02330(9)	0.02429(9)	0.00083(6)	0.00903(7)	0.00690(6)
S(2)	2i	0.0320(1)	0.3158(1)	−0.0271(2)	0.0279(6)	0.0304(6)	0.0266(6)	−0.0032(5)	0.0071(5)	0.0059(5)
P(1)	2i	0.3877(1)	0.6014(1)	0.3106(2)	0.0219(6)	0.0207(5)	0.0227(6)	0.0028(4)	0.0093(5)	0.0083(4)
O(1)	2i	0.0860(5)	0.3488(4)	0.5103(5)	0.061(3)	0.054(3)	0.034(2)	0.021(2)	0.021(2)	0.012(2)
N(1)	2i	−0.0361(5)	0.4131(4)	0.2833(6)	0.042(3)	0.047(3)	0.031(2)	0.021(2)	0.015(2)	0.008(2)
C(1)	2i	0.0961(5)	0.2205(5)	0.2543(6)	0.024(2)	0.030(2)	0.030(2)	−0.000(2)	0.011(2)	0.005(2)
C(2)	2i	0.0918(5)	0.2046(5)	0.0927(6)	0.021(2)	0.028(2)	0.036(3)	−0.002(2)	0.012(2)	0.006(2)
C(3)	2i	0.1401(5)	0.0916(5)	0.0143(7)	0.034(3)	0.036(3)	0.041(3)	0.001(2)	0.025(2)	0.003(2)
C(4)	2i	0.1885(6)	−0.0025(5)	0.0896(8)	0.034(3)	0.031(3)	0.062(4)	0.008(2)	0.024(3)	0.010(3)
C(5)	2i	0.1928(6)	0.0150(5)	0.2511(8)	0.037(3)	0.032(3)	0.052(3)	0.006(2)	0.015(3)	0.016(2)
C(6)	2i	0.1469(5)	0.1245(5)	0.3297(7)	0.034(3)	0.039(3)	0.035(3)	−0.003(2)	0.011(2)	0.012(2)
C(7)	2i	0.0480(5)	0.3359(5)	0.3582(6)	0.026(3)	0.033(3)	0.031(3)	0.000(2)	0.012(2)	0.004(2)
C(11)	2i	0.4619(5)	0.6556(4)	0.1937(6)	0.026(2)	0.021(2)	0.025(2)	0.001(2)	0.010(2)	0.007(2)
C(12)	2i	0.3746(5)	0.6839(5)	0.0480(6)	0.028(3)	0.038(3)	0.032(3)	0.005(2)	0.011(2)	0.014(2)
C(13)	2i	0.4270(7)	0.7254(5)	−0.0473(7)	0.060(4)	0.043(3)	0.034(3)	0.003(3)	0.018(3)	0.021(2)
C(14)	2i	0.5659(6)	0.7381(5)	0.0030(7)	0.058(4)	0.030(3)	0.041(3)	−0.001(2)	0.035(3)	0.006(2)
C(15)	2i	0.6533(6)	0.7083(5)	0.1454(7)	0.038(3)	0.036(3)	0.036(3)	0.000(2)	0.022(2)	0.004(2)
C(16)	2i	0.6027(5)	0.6683(5)	0.2409(6)	0.029(3)	0.035(3)	0.025(2)	0.002(2)	0.012(2)	0.009(2)
C(21)	2i	0.3530(5)	0.7406(4)	0.4554(5)	0.025(2)	0.025(2)	0.021(2)	0.005(2)	0.010(2)	0.008(2)
C(22)	2i	0.2273(5)	0.7494(5)	0.4698(6)	0.028(3)	0.030(2)	0.030(3)	0.000(2)	0.012(2)	0.006(2)
C(23)	2i	0.2019(6)	0.8547(5)	0.5807(7)	0.044(3)	0.037(3)	0.043(3)	0.004(2)	0.030(3)	0.008(2)
C(24)	2i	0.2988(6)	0.9507(5)	0.6752(7)	0.051(4)	0.035(3)	0.040(3)	0.004(2)	0.032(3)	0.000(2)
C(25)	2i	0.4242(6)	0.9432(5)	0.6629(7)	0.051(4)	0.031(3)	0.043(3)	−0.008(2)	0.025(3)	0.001(2)
C(26)	2i	0.4516(5)	0.8382(5)	0.5516(6)	0.031(3)	0.032(3)	0.035(3)	−0.001(2)	0.016(2)	0.004(2)
C(31)	2i	0.5265(5)	0.5275(4)	0.4484(6)	0.023(2)	0.029(2)	0.028(2)	0.005(2)	0.013(2)	0.013(2)

Acknowledgments. The Australian Research Council and The University of Adelaide are thanked for support.

References

1. Smyth, D. R.: Structural studies of phosphinegold(I) thiolates with potential medicinal applications. Ph. D. Thesis. The University of Adelaide, 2001.
2. Smyth, D. R.; Vincent, B. R.; Tiekink, E. R. T.: Solvent mediated disruption of intermolecular association in phosphinegold(I) thiolates: the relative importance of carboxylic acid dimer formation on crystal structure. *CrystEngComm* (2000) Art. No. 21 (6 pages).
3. teXsan: Single Crystal Structure Analysis Software. Version 1.05. Molecular Structure Corporation. The Woodlands, TX, USA 1997.
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. Sheldrick, G. M.: SHELXL-97. Program for crystal structure refinement. University of Göttingen, Germany 1997.
6. Walker, N.; Stuart, D.: An empirical method for correcting diffractometer data for absorption effects. *Acta Crystallogr. A* **39** (1983) 158–166.
7. Johnson, C. K.: ORTEP. Report ORNL-5138, Oak Ridge National Laboratory, Tennessee, USA 1976.