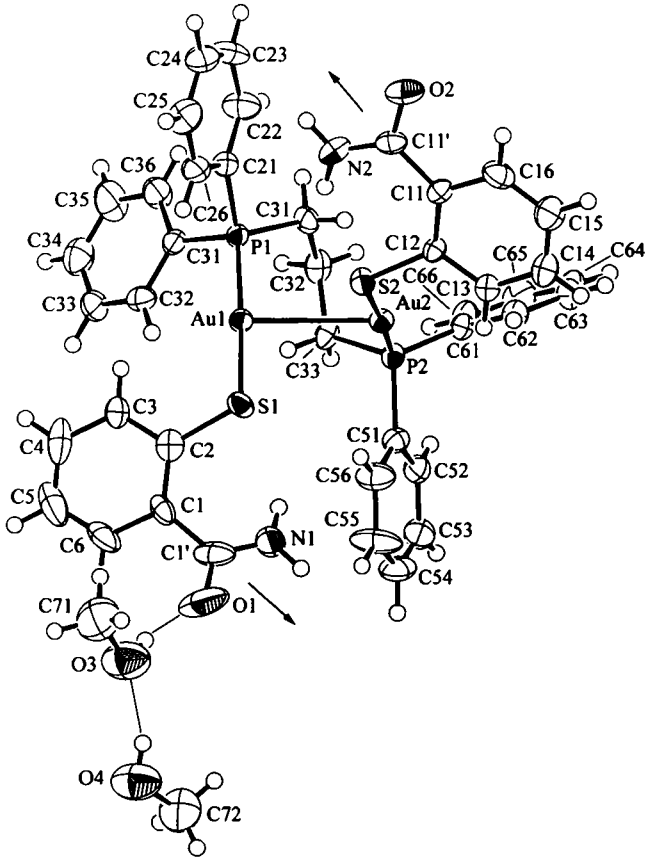
Crystal structure of bis(benzamide-2-thiolato)(μ_2-bis(diphenylphosphino)-propane)digold(I) dimethanol solvate, $C_{43}H_{46}Au_2N_2O_4P_2S_2$

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. 1267/879



Abstract

$C_{43}H_{46}Au_2N_2O_4P_2S_2$, monoclinic, $P12_1/c1$ (No. 14), $a = 12.233(3)$ Å, $b = 15.395(3)$ Å, $c = 23.014(8)$ Å, $\beta = 96.23(3)°$, $V = 4308.6$ Å^3, $Z = 4$, $R_{gt}(F) = 0.033$, $wR_{ref}(F^2) = 0.087$, $T = 173$ K.

Source of material

The title compound was prepared in 81% yield from the 1:2 reaction between $(Ph_2PCH_2CH_2PPh_2)(AuCl)_2$ and 2-mercaptobenzamide [1]. Pale-yellow crystals were obtained from the vapour diffusion of methanol into a dimethylformamide solution of the compound; mp 373 K – 374 K.

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 features an intramolecular Au···Au interaction so that d(Au1···Au2) is 2.9997(7) Å. The gold atoms exist in linear enviroment defined by S (d(Au1, Au2—S) are 2.303(2) Å and 2.300(2) Å) and P (d(Au1, Au2—P) are 2.2665(18) Å and 2.260(2) Å) donor atoms so that ∠S–Au–P are 177.34(7)° and 175.58(7)° for Au1 and Au2 , respectively. The supramolecular structure adopts a chain motif as has been reported recently for related diphosphine systems [2,3]. The amide groups associate in a pseudo head-to-tail fashion so that d(N1–H1a···O2^i) is 1.96 Å, d(N1···O2^i) is 2.822(9) Å and the angle at H1a is 168°; symmetry operation i: x, –0.5–y, 0.5+z. The corresponding d(O1···H2a^i–N2^i) is 3.12 Å owing to the presence of a competing interaction involving the a solvent methanol molecule as illustrated in the Figure. The parameters associated with the O–H···O interactions are d(O3–H3···O1) = 1.86 Å, d(O3···O1) = 2.68(1) Å with an angle of 167° at H3, and d(O4–H4···O3) = 1.93 Å, d(O4···O3) = 2.68(1) Å with an angle of 149° at H4. The H1b and H2b atoms form intramolecular contacts with the S1 and S2 atoms, respectively, so that d(H···S) are 2.38 Å and 2.23 Å, respectively. In this structure the strong preferences of amides to form dimeric hydrogen-bonded motifs has been compromised by the presence of methanol molecules in the lattice. Despite the weak nature of the O1···H2a^i interaction, molecules aggregate to form a chain that is propagated along the c-direction.

Table 1. Data collection and handling.

Crystal:	pale-yellow block, size 0.15 × 0.15 × 0.32 mm
Wavelength:	Mo K_α radiation (0.7107 Å)
μ:	70.40 cm^{-1}
Diffractometer, scan mode:	Rigaku AFC7R, $\omega/2\theta$
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	10702, 1024
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 5684
$N(param)_{refined}$:	496
Programs:	teXsan [4], DIRDIF92 [5], SHELXL-97 [6], DIFABS [7], ORTEPII [8]

* Correspondence author (e-mail: chmtert@nus.edu.sg)
[1] Current address: National University of Singapore, Department of Chemistry, Singapore 117543

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

Atom	Site	x	y	z	U_{iso}
H(3)	4e	0.4464	−0.2222	0.5126	0.091
H(4)	4e	0.4864	−0.3130	0.6047	0.087
H(1a)	4e	0.1347	−0.1653	0.4339	0.079
H(1b)	4e	0.1535	−0.1098	0.3811	0.079
H(2a)	4e	0.1512	−0.1910	0.0146	0.064
H(2b)	4e	0.1114	−0.1822	0.0748	0.064
H(3a)	4e	0.4023	0.0051	0.2716	0.045
H(4a)	4e	0.5509	0.0654	0.3293	0.072
H(5)	4e	0.5836	0.0282	0.4280	0.088
H(6)	4e	0.4664	−0.0673	0.4676	0.072
H(13)	4e	−0.1614	−0.2488	0.1881	0.049
H(14)	4e	−0.3020	−0.3372	0.1454	0.070
H(15)	4e	−0.2857	−0.3994	0.0540	0.065
H(16)	4e	−0.1416	−0.3601	0.0026	0.054
H(22)	4e	0.1015	0.1173	0.0151	0.062
H(23)	4e	0.1685	0.0595	−0.0683	0.067
H(24)	4e	0.3012	−0.0492	−0.0596	0.064
H(25)	4e	0.3710	−0.0971	0.0322	0.065
H(26)	4e	0.3102	−0.0361	0.1156	0.052
H(32)	4e	0.2798	0.1573	0.2418	0.044
H(33)	4e	0.3690	0.2877	0.2665	0.058
H(34)	4e	0.3776	0.3936	0.1961	0.068
H(35)	4e	0.2860	0.3754	0.1032	0.085

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(36)	4e	0.1986	0.2454	0.0771	0.064
H(41a)	4e	0.0069	0.1542	0.0836	0.041
H(41b)	4e	−0.0351	0.0705	0.1155	0.041
H(42a)	4e	0.0063	0.2363	0.1689	0.046
H(42b)	4e	−0.1127	0.1968	0.1503	0.046
H(43a)	4e	−0.0687	0.1944	0.2513	0.037
H(43b)	4e	0.0362	0.1354	0.2445	0.037
H(52)	4e	−0.2358	0.1438	0.3198	0.045
H(53)	4e	−0.2459	0.1332	0.4204	0.057
H(54)	4e	−0.1331	0.0362	0.4757	0.062
H(55)	4e	−0.0241	−0.0593	0.4308	0.087
H(56)	4e	−0.0209	−0.0532	0.3292	0.064
H(62)	4e	−0.3013	−0.0534	0.1874	0.044
H(63)	4e	−0.4813	−0.0281	0.1476	0.051
H(64)	4e	−0.5505	0.1129	0.1421	0.062
H(65)	4e	−0.4367	0.2300	0.1729	0.061
H(66)	4e	−0.2565	0.2052	0.2130	0.050
H(71a)	4e	0.5499	−0.2623	0.4557	0.148
H(71b)	4e	0.5478	−0.3523	0.4904	0.148
H(71c)	4e	0.6402	−0.2813	0.5099	0.148
H(72a)	4e	0.3870	−0.3237	0.6968	0.116
H(72b)	4e	0.3399	−0.2842	0.6348	0.116
H(72c)	4e	0.4147	−0.2256	0.6808	0.116

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

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Au(1)	4e	0.18595(2)	−0.00759(2)	0.21525(1)	0.0266(1)	0.0303(1)	0.0276(1)	0.0008(1)	0.0018(1)	0.0066(1)
Au(2)	4e	−0.04791(2)	−0.06945(2)	0.19911(1)	0.0280(1)	0.0243(1)	0.0265(1)	0.0008(1)	0.0048(1)	−0.0027(1)
S(1)	4e	0.2263(2)	−0.1068(1)	0.28934(8)	0.040(1)	0.0321(9)	0.030(1)	−0.0075(8)	−0.0083(8)	0.0080(8)
S(2)	4e	0.0389(2)	−0.1842(1)	0.15942(8)	0.028(1)	0.0306(9)	0.035(1)	0.0025(7)	0.0057(8)	−0.0090(8)
P(1)	4e	0.1539(2)	0.0887(1)	0.14086(8)	0.0234(9)	0.031(1)	0.027(1)	−0.0006(7)	0.0022(7)	0.0062(8)
P(2)	4e	−0.1223(2)	0.0498(1)	0.23643(8)	0.025(1)	0.0256(9)	0.031(1)	−0.0012(7)	0.0065(8)	−0.0043(7)
O(1)	4e	0.3188(6)	−0.1750(6)	0.4742(3)	0.070(5)	0.22(1) ??	0.056(5)	0.058(6)	0.029(4)	0.067(6)
O(2)	4e	0.0127(5)	−0.2910(4)	−0.0261(2)	0.068(4)	0.059(4)	0.030(3)	0.010(3)	0.015(3)	−0.003(3)
O(3)	4e	0.4945(7)	−0.2528(5)	0.5320(3)	0.097(6)	0.105(6)	0.071(5)	0.030(5)	0.011(5)	0.019(5)
O(4)	4e	0.4975(7)	−0.3131(5)	0.6412(3)	0.103(6)	0.097(6)	0.065(5)	0.033(5)	0.012(4)	0.009(4)
N(1)	4e	0.1796(6)	−0.1373(5)	0.4131(3)	0.055(5)	0.091(6)	0.048(5)	−0.031(5)	−0.011(4)	0.035(5)
N(2)	4e	0.1042(6)	−0.2048(5)	0.0395(3)	0.049(5)	0.075(5)	0.040(4)	−0.012(4)	0.018(3)	0.008(4)
C(1)	4e	0.3582(6)	−0.0841(5)	0.3953(3)	0.022(4)	0.052(5)	0.038(4)	0.009(3)	−0.003(3)	−0.018(4)
C(1')	4e	0.2832(8)	−0.1366(6)	0.4296(4)	0.060(6)	0.074(6)	0.029(4)	0.036(5)	0.012(4)	0.002(4)
C(2)	4e	0.3372(6)	−0.0624(4)	0.3356(3)	0.035(4)	0.026(4)	0.049(5)	0.007(3)	0.006(4)	−0.004(3)
C(3)	4e	0.4128(6)	−0.0083(5)	0.3121(4)	0.026(4)	0.038(4)	0.048(5)	0.001(3)	0.005(3)	−0.001(4)
C(4)	4e	0.5028(7)	0.0266(6)	0.3462(5)	0.029(5)	0.048(5)	0.104(9)	−0.005(4)	0.015(5)	−0.021(5)
C(5)	4e	0.5220(8)	0.0049(7)	0.4043(5)	0.038(5)	0.097(9)	0.083(8)	0.000(6)	−0.008(5)	−0.053(7)
C(6)	4e	0.4513(7)	−0.0508(7)	0.4278(4)	0.043(5)	0.100(8)	0.036(5)	0.008(5)	−0.006(4)	−0.031(5)
C(11)	4e	−0.0614(6)	−0.2793(4)	0.0639(3)	0.030(4)	0.024(3)	0.030(4)	0.004(3)	0.008(3)	0.006(3)
C(11')	4e	0.0224(6)	−0.2596(5)	0.0237(3)	0.043(5)	0.033(4)	0.027(4)	0.009(3)	0.006(3)	0.006(3)
C(12)	4e	−0.0669(5)	−0.2475(4)	0.1203(3)	0.025(4)	0.023(3)	0.029(4)	0.000(3)	0.008(3)	−0.002(3)
C(13)	4e	−0.1572(6)	−0.2708(5)	0.1498(3)	0.034(4)	0.055(5)	0.034(4)	−0.011(4)	0.003(3)	0.001(4)
C(14)	4e	−0.2403(7)	−0.3246(6)	0.1252(4)	0.049(6)	0.069(6)	0.058(6)	−0.027(5)	0.006(5)	0.006(5)
C(15)	4e	−0.2318(7)	−0.3595(5)	0.0706(4)	0.060(6)	0.043(5)	0.058(6)	−0.021(4)	0.004(5)	0.008(4)
C(16)	4e	−0.1453(7)	−0.3366(5)	0.0405(4)	0.057(6)	0.040(4)	0.035(5)	−0.008(4)	−0.004(4)	−0.005(4)
C(21)	4e	0.1982(6)	0.0452(5)	0.0735(3)	0.032(4)	0.039(4)	0.030(4)	−0.001(3)	0.002(3)	0.005(3)
C(22)	4e	0.1569(8)	0.0738(6)	0.0190(4)	0.065(6)	0.053(5)	0.038(5)	0.018(5)	0.006(4)	0.004(4)
C(23)	4e	0.1964(8)	0.0389(6)	−0.0307(4)	0.078(7)	0.064(6)	0.024(4)	0.003(5)	−0.005(4)	0.008(4)
C(24)	4e	0.2755(7)	−0.0249(6)	−0.0256(4)	0.053(6)	0.074(6)	0.034(5)	0.000(5)	0.014(4)	−0.010(5)
C(25)	4e	0.3166(7)	−0.0529(6)	0.0284(4)	0.050(6)	0.053(5)	0.061(6)	0.012(4)	0.017(5)	−0.004(5)
C(26)	4e	0.2793(6)	−0.0172(5)	0.0781(4)	0.034(4)	0.063(5)	0.034(4)	0.003(4)	0.005(3)	−0.002(4)
C(31)	4e	0.2281(6)	0.1889(5)	0.1569(3)	0.026(4)	0.035(4)	0.031(4)	−0.001(3)	0.005(3)	0.005(3)
C(32)	4e	0.2810(6)	0.2019(5)	0.2134(3)	0.040(5)	0.041(4)	0.030(4)	0.003(3)	0.008(3)	−0.001(3)
C(33)	4e	0.3348(7)	0.2786(5)	0.2280(4)	0.047(5)	0.056(5)	0.042(5)	−0.021(4)	0.004(4)	−0.018(4)
C(34)	4e	0.3383(8)	0.3413(6)	0.1866(4)	0.053(6)	0.050(5)	0.069(7)	−0.013(4)	0.014(5)	−0.008(5)
C(35)	4e	0.2855(8)	0.3299(6)	0.1311(5)	0.076(7)	0.049(6)	0.082(8)	−0.028(5)	−0.017(6)	0.026(5)
C(36)	4e	0.2324(7)	0.2537(5)	0.1159(4)	0.054(6)	0.050(5)	0.051(5)	−0.022(4)	−0.011(4)	0.022(4)
C(41)	4e	0.0114(6)	0.1230(5)	0.1214(3)	0.026(4)	0.039(4)	0.036(4)	0.004(3)	−0.005(3)	0.009(3)
C(42)	4e	−0.0366(6)	0.1816(4)	0.1662(3)	0.037(4)	0.021(3)	0.057(5)	−0.004(3)	0.003(4)	0.003(3)
C(43)	4e	−0.0402(6)	0.1475(4)	0.2277(3)	0.031(4)	0.022(3)	0.040(4)	−0.005(3)	0.012(3)	−0.005(3)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(51)	4*e*	–0.1288(6)	0.0446(4)	0.3146(3)	0.037(4)	0.029(4)	0.029(4)	–0.003(3)	0.007(3)	–0.003(3)
C(52)	4*e*	–0.1936(6)	0.1011(5)	0.3421(3)	0.033(4)	0.043(4)	0.037(4)	0.000(3)	0.005(3)	–0.004(4)
C(53)	4*e*	–0.1979(7)	0.0962(5)	0.4020(4)	0.051(5)	0.050(5)	0.044(5)	–0.006(4)	0.008(4)	–0.014(4)
C(54)	4*e*	–0.1328(8)	0.0377(5)	0.4344(4)	0.077(7)	0.052(5)	0.029(4)	–0.013(5)	0.014(5)	–0.012(4)
C(55)	4*e*	–0.0681(9)	–0.0180(6)	0.4082(4)	0.115(9)	0.071(7)	0.031(5)	0.043(6)	0.001(5)	0.015(5)
C(56)	4*e*	–0.0664(8)	–0.0141(6)	0.3476(4)	0.068(6)	0.056(5)	0.037(5)	0.017(5)	0.011(4)	–0.002(4)
C(61)	4*e*	–0.2609(5)	0.0735(4)	0.2040(3)	0.025(4)	0.029(3)	0.033(4)	0.001(3)	0.005(3)	0.000(3)
C(62)	4*e*	–0.3288(6)	0.0043(5)	0.1845(3)	0.034(4)	0.036(4)	0.039(4)	–0.003(3)	0.005(3)	0.000(4)
C(63)	4*e*	–0.4357(6)	0.0192(5)	0.1612(4)	0.031(4)	0.052(5)	0.045(5)	–0.005(4)	0.001(4)	–0.001(4)
C(64)	4*e*	–0.4764(7)	0.1028(6)	0.1575(4)	0.023(4)	0.081(7)	0.051(6)	0.004(4)	0.009(4)	0.005(5)
C(65)	4*e*	–0.4090(7)	0.1723(6)	0.1763(4)	0.043(5)	0.050(5)	0.061(6)	0.018(4)	0.014(5)	0.008(4)
C(66)	4*e*	–0.3021(6)	0.1577(5)	0.1998(4)	0.030(4)	0.035(4)	0.060(6)	0.006(3)	0.008(4)	0.004(4)
C(71)	4*e*	0.563(1)	–0.2899(8)	0.4942(5)	0.086(9)	0.12(1)	0.09(1)	–0.001(8)	0.028(8)	–0.021(8)
C(72)	4*e*	0.4025(9)	–0.2845(7)	0.6652(5)	0.080(8)	0.068(7)	0.088(8)	0.008(6)	0.023(7)	–0.027(6)

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. Smyth, D. R.; Hester, J.; Young, V. G.; Tiekink, E. R. T.: Tuning aurophilic interactions in dinuclear phosphinegold(I) thiolates. CrystEngComm, submitted.
4. teXsan: Single Crystal Structure Analysis Software. Version 1.05. Molecular Structure Corporation. The Woodlands, TX, USA 1997.
5. 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 Nertherlands 1992.
6. Sheldrick, G. M.: SHELXL-97. Program for crystal structure refinement. University of Göttingen, Germany 1997.
7. Walker, N.; Stuart, D.: An empirical method for correcting diffractometer data for absorption effects. Acta Crystallogr. A**39** (1983) 158-166.
8. Johnson, C. K.: ORTEPII. Report ORNL-5138, Oak Ridge National Laboratory, Tennessee, USA 1976.