(Ibers & Hamilton, 1964); the values for f' and f'' were those of Cromer (1974). The diagram was produced using *ORTEP*II (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71642 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: DU1052]

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The Monoclinic Phase [Au2(C25H22P2)2]Cl2.2CH3CN

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Abstract

Bis[μ-bis(diphenylphosphino)methane-*P,P'*]digold(I) dichloride bis(acetonitrile) solvate, $[Au_2(C_{25}H_{22}P_2)_2]Cl_2.2CH_3CN$, crystallized in the monoclinic system. A crystallographic inversion centre lies at the

centre of the molecule. The Au—P distances are 2.299 (3) and 2.332 (3) Å. The Au···Cl distance is 2.951 (4) Å and the Au···Au separation is 2.994 (1) Å. The molecular structure is similar to the previously reported triclinic structure except that a longer Au···Cl distance is observed for the present monoclinic structure.

Comment

In recent years, studies of the metal–metal interactions in the $[Au(dppm)X]_2$ system, where dppm is bis(diphenylphosphino)methane, have increased because of their interesting bonding and luminescent properties (King, Wang, Khan & Fackler, 1989; Khan, King, Heinrich, Fackler & Poter, 1989; Wang, Khan & Fackler, 1989). The structure of $[Au(dppm)Cl]_2$ solvated with acetone has been reported in the triclinic system (Schmidbauer,
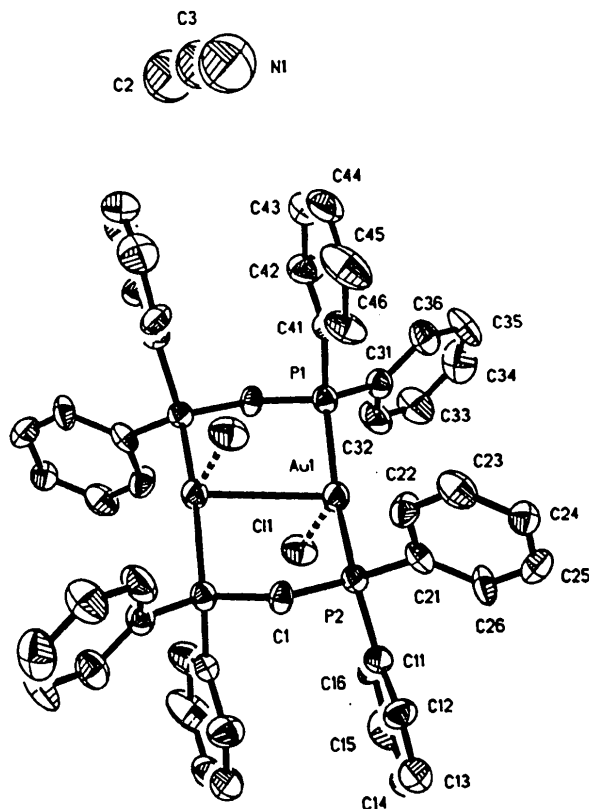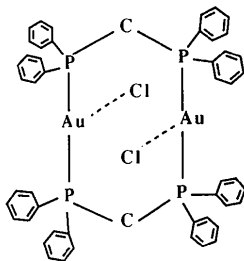

Fig. 1. Molecular drawing of the title compound with 50% probability displacement ellipsoids for the non-H atoms.

Wohlleben, Schubert, Frank & Huttner, 1977). In this paper, we report a monoclinic phase crystal structure (space group $P2_1/c$) of [Au(dppm)Cl]$_2$, (I), solvated with CH$_3$CN. The molecular structure is

similar to the triclinic structure. The only significant difference between the two structures is the Au—Cl distance, which is 2.951 (4) Å for the monoclinic structure and 2.771 (4) Å for the triclinic structure. Fig. 1 shows the molecule and atomic labeling scheme (Sheldrick, 1991).

Experimental

The title compound was synthesized by reacting equimolar amounts of Au(tht)Cl (0.2093 g, 0.65 mmol), where tht is tetrahydrothiophene (Uson, Laguna & Laguna, 1989), and bis(diphenylphosphino)methane (dppm; 0.2515 g, 0.68 mmol) in CH$_2$Cl$_2$ under a nitrogen atmosphere for 1 h. The title compound was formed in almost quantitative yield by adding a large amount of diethyl ether to the reaction solution. Crystals suitable for X-ray analysis were grown by slow diffusion of diethyl ether into a toluene/acetonitrile solution of [Au$_2$(dppm)$_2$]Cl$_2$.

Crystal data

[Au$_2$(C$_{25}$H$_{22}$P$_2$)$_2$]Cl$_2$.- 2CH$_3$CN
M_r = 1305.6
Monoclinic
$P2_1/c$
a = 12.341 (2) Å
b = 19.580 (2) Å
c = 11.464 (3) Å
β = 113.50 (2)°
V = 2540.3 (7) Å^3
Z = 2

D_x = 1.707 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 36 reflections
θ = 9–14°
μ = 6.037 mm^{-1}
T = 298 K
Irregular
0.25 × 0.2 × 0.2 mm
Colorless

Data collection

Siemens P4 diffractometer
$\theta/2\theta$ scans
Absorption correction: empirical (ψ scan)
T_{min} = 0.092, T_{max} = 0.146
4830 measured reflections
4446 independent reflections
2506 observed reflections
[$I > 3\sigma(I)$]

R_{int} = 0.0274
θ_{max} = 25°
h = 0 → 14
k = 0 → 24
l = −15 → 15
3 standard reflections monitored every 500 reflections
intensity variation: 2%

Refinement

Refinement on F
R = 0.0362
wR = 0.0433
S = 0.61
2501 reflections
229 parameters
H-atom parameters not refined

w = 1/[$\sigma^2(F_o)$ + 0.0026F^2]
$\Delta\rho_{max}$ = 1.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.82 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Au1	0.10221 (4)	0.04283 (2)	0.08287 (4)	0.0363 (2)
Cl1	0.1656 (3)	0.04996 (16)	−0.1393 (3)	0.0504 (12)
P1	−0.0103 (2)	0.14013 (13)	−0.0035 (3)	0.0320 (10)
P2	0.1937 (2)	−0.04906 (14)	0.2083 (2)	0.0304 (10)
C1	0.0977 (9)	−0.1251 (5)	0.1759 (9)	0.032 (4)
C12	0.3993 (6)	−0.1289 (4)	0.2877 (6)	0.042 (5)
C13†	0.5018	−0.1535	0.2787	0.060 (6)
C14	0.5358	−0.1288	0.1842	0.061 (6)
C15	0.4673	−0.0796	0.0986	0.060 (7)
C16	0.3648	−0.0550	0.1076	0.044 (5)
C11	0.3308	−0.0796	0.2021	0.034 (4)
C22	0.1347 (4)	−0.0344 (4)	0.4167 (6)	0.043 (4)
C23	0.1573	−0.0215	0.5439	0.051 (6)
C24	0.2725	−0.0087	0.6307	0.039 (4)
C25	0.3651	−0.0089	0.5902	0.050 (5)
C26	0.3426	−0.0218	0.4630	0.045 (5)
C21	0.2274	−0.0346	0.3762	0.033 (4)
C32	0.1272 (7)	0.2232 (3)	−0.0876 (7)	0.058 (6)
C33	0.1887	0.2828	−0.0890	0.066 (7)
C34	0.1912	0.3370	−0.0092	0.070 (7)
C35	0.1323	0.3317	0.0721	0.064 (7)
C36	0.0709	0.2721	0.0735	0.049 (5)
C31	0.0684	0.2179	−0.0064	0.038 (4)
C42	−0.2025 (7)	0.2130 (4)	0.0011 (6)	0.055 (6)
C43	−0.2819	0.2327	0.0538	0.064 (6)
C44	−0.2736	0.2045	0.1687	0.064 (7)
C45	−0.1858	0.1565	0.2310	0.079 (9)
C46	−0.1064	0.1369	0.1783	0.057 (6)
C41	−0.1147	0.1651	0.0634	0.037 (4)
N1	−0.5446 (18)	0.3304 (10)	−0.127 (2)	0.151 (7)
C3	−0.5770 (18)	0.3447 (10)	−0.233 (2)	0.104 (6)
C2	−0.5979 (19)	0.3561 (11)	−0.360 (2)	0.127 (7)

† An atom whose coordinates do not have e.s.d.'s was refined as part of a rigid group.

Table 2. *Selected geometric parameters* (Å, °)

Au1···Au1'	2.994 (1)	Au1—P1	2.332 (3)
Au1—P2	2.299 (3)	Au1···Cl1	2.951 (4)
P1—C41	1.810 (10)	P1—C31	1.812 (8)
P2—C1	1.846 (10)	P2—C11	1.822 (9)
P2—C21	1.823 (7)	N1—C3	1.15 (3)
C3—C2	1.39 (4)		
P1—Au1—P2	164.9 (1)	P1—Au1···Au1'	88.9 (1)
P2—Au1···Au1'	92.2 (1)	Au1—P1—C31	117.5 (3)
Au1—P1—C41	116.4 (3)	C31—P1—C41	104.9 (4)
Au1—P1—Cl'	108.5 (3)	C41—P1—Cl'	106.5 (4)
C31—P1—Cl'	101.6 (4)	Au1—P2—C1	112.9 (3)
Au1—P2—C11	117.7 (3)	C1—P2—C11	105.0 (4)
Au1—P2—C21	112.8 (3)	C1—P2—C21	101.3 (4)
C11—P2—C21	105.5 (3)	P2—C11—C12	120.8 (2)
P2—C11—C16	119.2 (2)	P2—C21—C22	118.7 (2)
P2—C21—C26	121.3 (2)	P1—C31—C32	120.0 (3)
P1—C31—C36	120.0 (3)	P1—C41—C42	120.3 (2)
P1—C41—C46	119.7 (2)	N1—C3—C2	170 (3)

The structure was solved by Patterson methods using *SHELXTL-Plus* (Sheldrick, 1991). The C atoms of the phenyl rings were refined as rigid groups (idealized hexagons) using the *AFIX* command in *SHELXTL-Plus*. H atoms were placed in idealized positions and included in the structure-factor calculations but were not included in the least-squares calculations.

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Diamminebis[5-methylpyrimidine-2,4(1H,3H)-dionato(1 −)-N³]copper(II) Trihydrate

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Abstract

The Cu atom in [Cu(C₅H₅N₂O₂)(NH₃)₂].3H₂O has square-planar coordination geometry. The methylpyrimidinedione ligands are *trans* with respect to each other, with Cu—N(1) and Cu—N(3) distances of 2.03 (1) and 1.99 (1) Å, respectively, while the amine Cu—N distances are 2.00 (1) and 1.96 (1) Å. Both methylpyrimidinedione ligands are essentially planar with their mean planes being inclined at 4.0°. The structure is stabilized by a network of hydrogen bonds involving amino-N and O atoms.

Comment

Reported structures of metal-ion complexes containing neutral uracil or thymine are relatively rare. Coordination of metals to either of the two heterocyclic N atoms, which are protonated at neutral pH, is unlikely; exocyclic O(2) and O(4) atoms, however, are available for complexation. Structural examples include bis(uracil)mercury(II) chloride (Carrabine & Sundaralingam, 1971) and bis(1,3-dimethyluracil)-copper(II) chloride (Cartwright, Goodgame, Johns & Skapski, 1978), with both complexes exhibiting metal binding to the O(4) atom.

At elevated pH, ring N atoms are deprotonated and thus are available for metal coordination. Complexes containing the monoanion of uracil or thymine include diamminediaquabis(uracilato)-nickel(II) (Lumme & Mutikainen, 1980) and aqua-(diethylenetriamine)(thyminato)copper(II) bromide dihydrate (Kistenmacher, Sorrell & Marzilli, 1975), with metal coordination at N(1), and triaquabis-(uracilato)cadmium(II) (Mutikainen & Lumme, 1980) and bis(1-methylthyminato)mercury(II) (Kosturko, Folzer & Stewart, 1974), which exhibit metal binding at N(3).

Because of the importance of metal complexes of uracil and thymine in nucleic acid processes as well as in the development of anticancer therapies, we have synthesized several new compounds and herein report the crystal structure of the title compound (1).
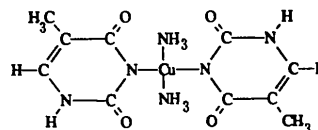
(1)

There is a possible center of symmetry in the molecule at 0.2652, 0.0041, 0.8756, while the three water molecules lie independent of this center and are responsible for the non-centrosymmetric space group. The methylpyrimidinedione ligands and water molecules are hydrogen bonded with O···N and O···O distances in the range 2.74 (1)–2.90 (1) Å. It is interesting to note that the ammonia N atoms are involved in rather longer hydrogen-bonded interactions [2.91 (1)–3.46 (1) Å] with O atoms.