

Fig. 1. The anion in  $(\text{NH}_4)_4[\text{H}_6\text{CuMo}_6\text{O}_{24}]\cdot 4\text{H}_2\text{O}$ . Thermal ellipsoids are scaled to enclose 30% probability.

(max. 1.23 and min. 0.87). Positions of Cu and Mo atoms from Patterson map, N and O atoms from Fourier syntheses; full-matrix least-squares refinement on  $F$ ; H atoms not located; all atoms anisotropic, final  $R = 0.060$ ,  $wR = 0.075$  and  $S = 1.40$  for 1734 observed reflections [ $|F| > 3\sigma(F)$ ], 187 parameters,  $w = 1/\sigma^2(F)$ ,  $(\Delta/\sigma)_{\text{max}} = 0.03$ , maximum  $\Delta\rho = 1.41 \text{ e } \text{\AA}^{-3}$  possibly at site fractionally occupied by disordered  $\text{H}_2\text{O}$ . All calculations using *TEXSAN* (Swepston, 1986) with scattering factors from *International Tables for X-ray Crystallography* (1974). Atomic parameters

are listed in Table 1\* and interatomic distances are given in Table 2. Fig. 1 shows an *ORTEP* (Johnson, 1976) drawing of the structure.

**Related literature.** In  $[\text{H}_6\text{CrMo}_6\text{O}_{24}]^{3-}$  Cr–O and Mo–O are in the ranges 1.97–1.99 and 1.70–2.35 Å, respectively (Perloff, 1970). In  $[\text{TeMo}_6\text{O}_{24}]^{6-}$  Te–O and Mo–O are in the ranges 1.93–1.94 and 1.69–2.32 Å, respectively (Evans, 1974). In  $[\text{IMo}_6\text{O}_{24}]^{7-}$  I–O and Mo–O are in the ranges 1.88–1.89 and 1.70–2.37 Å, respectively (Kondo, Kobayashi & Sasaki, 1980).

\* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51676 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

#### References

- EVANS, H. T. JR (1974). *Acta Cryst.* **B30**, 2095–2100.  
*International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)  
 JOHNSON, C. K. (1976). *ORTEP*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.  
 KONDO, H., KOBAYASHI, A. & SASAKI, Y. (1980). *Acta Cryst.* **B36**, 661–664.  
 PERLOFF, A. (1970). *Inorg. Chem.* **9**, 2228–2239.  
 ROSENHEIM, A. (1916). *Z. Anorg. Chem.* **96**, 139–181.  
 SWEPSTON, P. N. (1986). *TEXSAN* Software. Molecular Structure Corporation, College Station, Texas, USA.

*Acta Cryst.* (1989). **C45**, 947–949

## Structure of the Bis[bis(diphenylphosphino)methane]digold(I) Cation in $[\text{Au}_2(\text{dppm})_2](\text{BF}_4)_2$

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(Received 9 September 1988; accepted 14 November 1988)

**Abstract.** Bis- $\mu$ -[bis(diphenylphosphino)methane-*P,P'*]-digold(I) bis(tetrafluoroborate),  $[\text{Au}_2(\text{C}_{25}\text{H}_{22}\text{P}_2)_2](\text{BF}_4)_2$ ,  $[\text{Au}_2\{\text{C}_6\text{H}_5\text{PCH}_2\text{P}(\text{C}_6\text{H}_5)_2\}_2](\text{BF}_4)_2$ ,  $M_r = 1336.4$ , monoclinic,  $P2_1/a$ ,  $a = 15.047(5)$ ,  $b = 17.462(3)$ ,  $c = 10.532(2)$  Å,  $\beta = 117.59(2)^\circ$ ,  $V = 2452(1)$  Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 1.810 \text{ g cm}^{-3}$ ,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 61.56 \text{ cm}^{-1}$ ,  $F(000) = 1288$ ,  $T = 298 \text{ K}$ . Final  $R = 0.040$  for 2587 unique observed

reflections. This is the first structure of an  $\text{Au}_2(\text{dppm})_2^{2+}$  cation in which the anion is not coordinated to an Au atom. The Au...Au separation is 2.931(1) Å.

**Experimental.** Addition of  $\text{AgBF}_4$  to  $[\text{Au}_2(\text{dppm})_2]\text{Cl}_2$  in tetrahydrofuran gave a precipitate which, on dissolving in  $\text{CH}_3\text{CN}$  and removing the solvent, gave  $[\text{Au}_2(\text{dppm})_2](\text{BF}_4)_2$  in 93% yield. Analysis: C 45.29 (calculated 44.94), H 3.47 (3.32%). M.p. 493 K with decomposition. A colorless crystal, obtained by re-

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crystallization from CH<sub>3</sub>CN/diethyl ether, 0.17 × 0.16 × 0.24 mm, was mounted in a random orientation on a glass fiber. Axial dimensions and monoclinic symmetry were verified by axial rotation photographs. Refined cell parameters were obtained from the setting angles of 25 reflections with 30 < 2θ < 35°. Data were collected on a Nicolet R3m/E diffractometer with graphite-monochromated Mo Kα radiation using the ω-scanning technique in bisecting geometry. 3471 reflections (+h ≤ 17, +k ≤ 19, |l| ≤ 12) measured with 0 < 2θ < 45°. Scan rate variable, 2–30° min<sup>-1</sup>; scan range -0.8° in ω from Kα<sub>1</sub> to +0.8° from Kα<sub>2</sub>. Background intensities estimated from a 96-step peak profile. Three standard reflections (111̄, 020, 110) measured every 100 data. The data were corrected for standard variation (< 1%) and Lorentz and polarization effects. Absorption corrections were applied using a Gaussian quadrature method following careful measurement of crystal dimensions and assignment of indices to the crystal faces (minimum and maximum transmission factors of 0.203 and 0.441). Structure determination used *SHELXTL* (Sheldrick, 1978). Scattering factors, including terms for anomalous dispersion, taken from *International Tables for X-ray Crystallography* (1974). Au-atom positions determined from a sharpened Patterson map; remaining non-H atoms located using difference Fourier techniques. All non-H atoms refined anisotropically, except for the F atoms. Phenyl rings refined as rigid hexagons (C–C = 1.39 Å; C–C–C = 120°); H atoms were placed in idealized positions with fixed thermal parameters [*U*(H) = 0.08 Å<sup>2</sup>]. Least-squares refinement based on minimization of  $\sum w(|F_o| - |F_c|)^2$  with weights of the form  $w^{-1} = [\sigma^2(F_o) + 0.002033(F_o^2)]$ . Convergence to conventional *R* values of *R* = 0.040 and *wR* = 0.045 obtained using 243 parameters and 2587 unique reflections with  $F^2 > 3\sigma(F^2)$ . For final cycle maximum shift/σ = 0.043. The BF<sub>4</sub> ion is

Table 1. Atomic coordinates (× 10<sup>4</sup>) and isotropic thermal parameters (Å<sup>2</sup> × 10<sup>3</sup>) for [Au<sub>2</sub>(dppm)<sub>2</sub>](BF<sub>4</sub>)<sub>2</sub>

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> <sub>iso</sub>
Au(1)	367 (1)	78 (1)	1548 (1)	32 (1)*
P(1)	-916 (2)	-697 (1)	1423 (2)	31 (1)*
P(2)	1675 (2)	860 (1)	1788 (2)	33 (1)*
F(1)	-544 (6)	2913 (5)	-564 (9)	108 (3)
F(2)	-2076 (10)	2478 (7)	-1190 (15)	85 (4)
F(3)	-677 (10)	1712 (8)	74 (14)	113 (5)
F(4)	-1048 (11)	2671 (9)	1027 (15)	125 (6)
F(2a)	-1877 (21)	2569 (16)	-232 (30)	137 (10)
F(3a)	-1071 (13)	1742 (10)	-1061 (19)	78 (6)
F(4a)	-469 (14)	1961 (11)	1038 (21)	91 (7)
B(1)	-1064 (11)	2403 (7)	-229 (19)	79 (9)*
C(1)	1369 (6)	1352 (5)	90 (8)	32 (4)*
C(11)	-2773 (4)	-528 (3)	1363 (7)	47 (5)*
C(12)	-3605 (4)	-108 (3)	1214 (7)	60 (6)*
C(13)	-3641 (4)	681 (3)	989 (7)	74 (6)*
C(14)	-2845 (4)	1051 (3)	912 (7)	74 (7)*
C(15)	-2014 (4)	631 (3)	1061 (7)	60 (6)*
C(16)	-1977 (4)	-158 (3)	1286 (7)	37 (4)*
C(21)	1706 (5)	2351 (3)	2822 (5)	52 (5)*
C(22)	1917 (5)	2880 (3)	3917 (5)	72 (7)*
C(23)	2406 (5)	2644 (3)	5344 (5)	76 (7)*
C(24)	2684 (5)	1879 (3)	5676 (5)	67 (6)*
C(25)	2473 (5)	1350 (3)	4581 (5)	57 (5)*
C(26)	1984 (5)	1586 (3)	3153 (5)	35 (4)*
C(31)	-168 (5)	-2020 (3)	3138 (6)	58 (6)*
C(32)	203 (5)	-2409 (3)	4441 (6)	61 (6)*
C(33)	223 (5)	-2048 (3)	5636 (6)	63 (6)*
C(34)	-127 (5)	-1299 (3)	5528 (6)	72 (7)*
C(35)	-498 (5)	-911 (3)	4225 (6)	65 (6)*
C(36)	-518 (5)	-1271 (3)	3030 (6)	32 (4)*
C(41)	3697 (5)	739 (3)	2520 (8)	65 (6)*
C(42)	4586 (5)	335 (3)	2914 (8)	75 (6)*
C(43)	4614 (5)	-455 (3)	3128 (8)	88 (8)*
C(44)	3752 (5)	-841 (3)	2950 (8)	95 (9)*
C(45)	2862 (5)	-437 (3)	2556 (8)	73 (6)*
C(46)	2835 (5)	352 (3)	2342 (8)	45 (4)*

\* The equivalent isotropic *U* is defined as 1/3 of the trace of the *U*<sub>*ij*</sub> tensor.

disordered. A second orientation is obtained from the first by a C<sub>6</sub> rotation about the B(1)–F(1) axis. Occupancies refined to 60 (1)% for one orientation [F(2), F(3), F(4)] and 40 (1)% for the other [F(2a), F(3a), F(4a)]. Maximum residual electron density of +0.96 e Å<sup>-3</sup> is 0.90 Å from F(4), indicating that the disorder model is imperfect. Slope of normal probability plot equals 0.925 with a goodness-of-fit indicator of 1.032. The compound, shown in Fig. 1, has a crystallographic center of symmetry. Atomic coordinates and equivalent isotropic thermal parameters are listed in Table 1; bond angles and distances are listed in Table 2. Fig. 2 shows the crystal packing.\*

**Related literature.** The structures of [Au<sub>2</sub>(dppm)<sub>2</sub>]Cl<sub>2</sub> (Schmidbaur, Wohleben, Wagner, Orama & Huttner, 1977), and [Au<sub>2</sub>(dppm)<sub>2</sub>]Br<sub>2</sub> and [Au<sub>2</sub>(dppm)<sub>2</sub>]I<sub>2</sub> (Shain & Fackler, 1987) have been reported. The preparation of [Au<sub>2</sub>(dppm)<sub>2</sub>](BPh<sub>4</sub>)<sub>2</sub> has been described (Schmidbaur *et al.*, 1977).

\* Lists of structure factors, H-atom coordinates and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51608 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

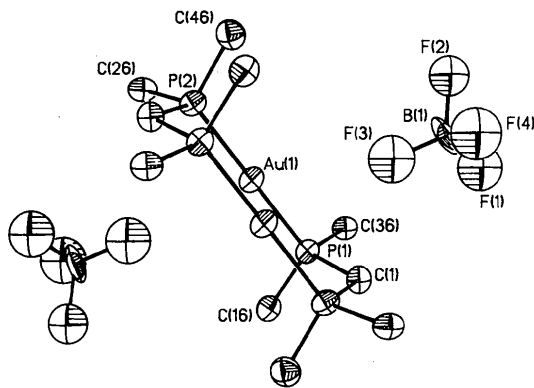


Fig. 1. View of [Au<sub>2</sub>(dppm)<sub>2</sub>](BF<sub>4</sub>)<sub>2</sub>. Thermal ellipsoids drawn at the 50% probability level. Only the *ipso* C atoms of the phenyl rings are shown. H atoms are omitted. Only one orientation of the disordered BF<sub>4</sub><sup>-</sup> is shown.

Table 2. Bond lengths (Å) and angles (°) for  $[\text{Au}_2(\text{dppm})_2](\text{BF}_4)_2$ 

Au(1)–P(1)	2.311 (3)	Au(1)–P(2)	2.310 (3)
Au(1)–Au(1')	2.931 (1)	P(1)–C(16)	1.801 (7)
P(1)–C(36)	1.814 (6)	P(1)–C(1')	1.819 (8)
P(2)–C(1)	1.839 (8)	P(2)–C(26)	1.810 (6)
P(2)–C(46)	1.797 (7)	F(1)–B(1)	1.336 (20)
F(2)–B(1)	1.391 (17)	F(3)–B(1)	1.313 (19)
F(4)–B(1)	1.393 (27)	F(2a)–B(1)	1.256 (39)
F(3a)–B(1)	1.446 (25)	F(4a)–B(1)	1.439 (23)
P(1)–Au(1)–P(2)	177.3 (1)	P(1)–Au(1)–Au(1')	90.4 (1)
P(2)–Au(1)–Au(1')	92.3 (1)	Au(1)–P(1)–C(16)	112.7 (2)
Au(1)–P(1)–C(36)	111.4 (2)	C(16)–P(1)–C(36)	105.1 (4)
Au(1)–P(1)–C(1')	112.5 (4)	C(16)–P(1)–C(1')	107.4 (3)
C(36)–P(1)–C(1')	107.3 (3)	Au(1)–P(2)–C(1)	110.6 (3)
Au(1)–P(2)–C(26)	113.2 (3)	C(1)–P(2)–C(26)	107.6 (3)
Au(1)–P(2)–C(46)	113.2 (2)	C(1)–P(2)–C(46)	108.3 (5)
C(26)–P(2)–C(46)	103.5 (3)	F(1)–B(1)–F(2)	108.8 (13)
F(1)–B(1)–F(3)	115.4 (16)	F(2)–B(1)–F(3)	118.5 (12)
F(1)–B(1)–F(4)	105.8 (12)	F(2)–B(1)–F(4)	101.0 (15)
F(3)–B(1)–F(4)	105.5 (15)	F(1)–B(1)–F(2a)	122.3 (17)
F(2)–B(1)–F(2a)	40.5 (15)	F(3)–B(1)–F(2a)	122.3 (20)
F(4)–B(1)–F(2a)	60.8 (17)	F(1)–B(1)–F(3a)	102.3 (17)
F(2)–B(1)–F(3a)	86.3 (11)	F(3)–B(1)–F(3a)	44.9 (10)
F(4)–B(1)–F(3a)	146.7 (16)	F(2a)–B(1)–F(3a)	116.7 (17)
F(1)–B(1)–F(4a)	114.4 (13)	F(2)–B(1)–F(4a)	136.6 (18)
F(3)–B(1)–F(4a)	42.8 (11)	F(4)–B(1)–F(4a)	64.5 (14)
F(2a)–B(1)–F(4a)	108.2 (23)	F(3a)–B(1)–F(4a)	87.8 (13)
P(2)–C(1)–P(1')	112.4 (4)	P(1)–C(16)–C(11)	120.5 (2)
P(1)–C(16)–C(15)	119.5 (2)	P(2)–C(26)–C(21)	122.4 (2)
P(2)–C(26)–C(25)	117.6 (2)	P(1)–C(36)–C(31)	122.9 (2)
P(1)–C(36)–C(35)	116.9 (2)	P(2)–C(46)–C(41)	120.5 (2)
P(2)–C(46)–C(45)	119.5 (2)		

These studies were supported by the Welch Foundation, the National Science Foundation NSF-8708625, and by the Regents of Texas A&M University through the AUF-sponsored Materials Science and Engineering Program.

*Acta Cryst.* (1989). **C45**, 949–951

## Absolute Structure of Di- $\mu$ -sulfoacetato-bis{[(1*R*,2*R*)-1,2-diaminocyclohexane]-platinum(II)} Trihydrate

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(Received 24 October 1988; accepted 30 November 1988)

**Abstract.**  $[\text{Pt}_2(\text{C}_2\text{H}_2\text{O}_5\text{S})_2(\text{C}_6\text{H}_{14}\text{N}_2)_2] \cdot 3\text{H}_2\text{O}$ ,  $M_r = 948.80$ , orthorhombic,  $P2_12_12_1$ ,  $a = 19.186$  (4),  $b = 14.577$  (2),  $c = 9.876$  (1) Å,  $V = 2762$  (1) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 2.28$ ,  $D_m = 2.25$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu = 10.859$  mm<sup>-1</sup>,  $F(000) = 1816$ ,  $T = 298$  K,  $R = 0.033$ ,  $wR = 0.031$  for 2455 independent reflections. Formal local charges on Pt atoms and SO<sub>3</sub> groups are not compensated and the molecule has a divalent zwitterionic character. The geometric requirement imposed by the bridging carboxyl group causes the relatively short Pt–Pt distance [3.0435 (8) Å] but it

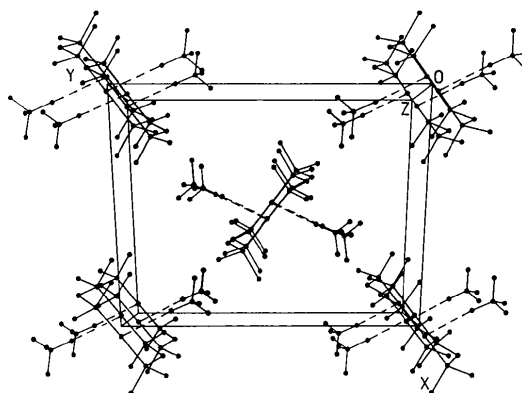


Fig. 2. Packing diagram of  $[\text{Au}_2(\text{dppm})_2](\text{BF}_4)_2$ . The shortest  $\text{BF}_4^- \cdots \text{Au}$  separation, 3.28 Å,  $\text{Au}(1) \cdots \text{F}(3)$ , is indicated by the dashed line. Only one orientation of the disordered  $\text{BF}_4^-$  is shown.

### References

- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- SCHMIDBAUR, H., WOHLLEBEN, A., WAGNER, R., ORAMA, O. & HUTTNER, G. (1977). *Chem. Ber.* **110**, 1748–1754.
- SHAIN, J. & FACKLER, J. P. JR (1987). *Inorg. Chim. Acta*, **131**, 157–158.
- SHELDRIK, G. M. (1978). *SHELXTL*, version 5.1. *An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data*. Univ. of Göttingen, Federal Republic of Germany.

is slightly too long to be regarded as a metal–metal bond. Coordination planes around Pt are not parallel because of the steric repulsion between the cyclohexane rings (dihedral angle between the coordination planes is 42.9°).

**Experimental.** The title compound was synthesized from a reaction of dihydroxyl[(1*R*,2*R*)-1,2-diaminocyclohexane]platinum(II) and sulfoacetic acid and was recrystallized from H<sub>2</sub>O. Yellow prism crystals; 0.23 × 0.15 × 0.10 mm;  $D_m$  by flotation in C<sub>2</sub>H<sub>4</sub>Br<sub>2</sub>–CH<sub>2</sub>I<sub>2</sub>.