# metal-organic papers

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#### **Key indicators**

Single-crystal X-ray study T = 293 K Mean  $\sigma$ (C–C) = 0.006 Å R factor = 0.047 wR factor = 0.131 Data-to-parameter ratio = 17.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

# $\mu$ -1,4-Bis(diphenylphosphino)butane- $\kappa^2 P:P'$ bis[(2,2'-bipyridyl)silver(I)] ditetrafluoroborate

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Pink crystals of the title dinuclear complex,  $[Ag_2(C_{10}H_8N_2)_2(C_{28}H_{28}P_2)](BF_4)_2$ , were obtained from a methanol solution of equivalent amounts of AgBF<sub>4</sub>, 2,2'-bipyridyl (bipy) and 1,4-bis(diphenylphosphino)butane (dppb). The  $[Ag_2(bipy)_2(dppb)]^{2+}$  cation lies across a crystal-lographic inversion centre and the coordination environment around Ag<sup>I</sup> is distorted trigonal.

#### Comment

Diphosphine ligands  $Ph_2P(CH_2)_nPPh_2$  have been widely applied in coordination chemistry because of their versatile coordination modes, such as chelating (McLauchlan & Ibers, 2000),  $\mu_2$ -bridging (Yam *et al.*, 2002) or a combination of both coordination modes (Zhuravel & Glueck, 1999). Among  $Ph_2P(CH_2)_nPPh_2$  ligands, bis(diphenylphosphino)methane (n = 1) is widely used owing to its rigid backbone with the shortest spacer. Recently, diphosphine ligands have been applied to synthesize macrocyclic and two-dimensional network structures, with the participation of another bridging ligand (Puddephatt, 2001). In the past, we have obtained a series of Ag<sup>I</sup> complexes containing both diphosphine and thiolate ligands (Deng et al., 2000; Kang et al., 2002; Zhang et al., 2003), from which we found that the backbone length of the diphosphine ligands is critical for the formation of the complexes. We also discovered that, the higher the value of nfor the  $(CH_2)_n$  backbone, the more difficult it is to obtain single crystals of the complex. Here we report the crystal and molecular structure of  $[Ag_2(bipy)_2(dppb)](BF_4)_2$ , (I), which was prepared with the flexible dppb ligand. This work complements and extends our structural characterization of compounds of highly flexible diphosphine ligands.



The asymmetric unit of (I) consists of one-half of the  $[Ag_2(bipy)_2(dppb)]^{2+}$  cation and a  $BF_4^-$  anion. The other half of the cation is generated by a crystallographic inversion centre (Fig. 1). The mid-point of the C2-C2<sup>i</sup> bond [symmetry code: (i) 2 - x, 2 - y, -z] is found to coincide with the crystallographic inversion centre. The Ag<sup>I</sup> atom has a distorted

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Figure 1

View of (I), showing 50% probability displacement ellipsoids and atomnumbering scheme for the contents of the asymmetric unit.

trigonal coordination environment which is made up of two N atoms from the bipy ligand and a P atom from the dppb ligand. The bipy ligand chelates the Ag<sup>I</sup> atom to form a fivemembered ring with an angle N1-Ag1-N2 of 72.08 (10)°. The dppb ligand acts as a  $\mu_2$ -bridge linking two Ag<sup>I</sup> atoms. The  $BF_4^-$  anion is linked to the cation only through  $C-H \cdots F$ hydrogen bonds (Table 2). The other short contacts observed in the structure are Ag1···N2(1 - x, 2 - y, -z) of 3.320 (5) Å and Ag1···Ag1(1 - x, 2 - y, -z) of 3.5227 (8) Å.

## **Experimental**

The synthesis of (I) was carried out by the reaction of AgBF<sub>4</sub> (0.019 g, 0.1 mmol), 2,2'-dipyridyl (0.016 g, 0.1 mmol) and 1,4-bis(diphosphino)butane (0.043 g, 0.1 mmol) in MeOH solution (10 ml) at room temperature for 0.5 h. The solution was then filtered. Pink crystals of (I) were obtained by evaporation of the pale-red solution.

Crystal data

$[Ag_{2}(C_{10}H_{8}N_{2})_{2}(C_{28}H_{28}P_{2})](BF_{4})_{2}$ $M_{r} = 1128.17$ Monoclinic, $P2_{1}/c$ a = 10.7951 (19) Å b = 14.402 (3) Å c = 15.118 (3) Å $\beta = 95.274$ (3)° V = 2340.5 (8) Å <sup>3</sup> Z = 2 Data collection	$D_x = 1.601 \text{ Mg m}^{-3}$ Mo K $\alpha$ radiation Cell parameters from 7492 reflections $\theta = 1.9-27.1^{\circ}$ $\mu = 0.98 \text{ mm}^{-1}$ T = 293 (2)  K Block, pink $0.50 \times 0.46 \times 0.44 \text{ mm}$
Bruker SMART CCD	4318 reflections with $I > 2\sigma(I)$
diffractometer	$R_{int} = 0.024$
ω scans	$\theta_{max} = 27.1^{\circ}$
Absorption correction: none	$h = -12 \rightarrow 13$
12917 measured reflections	$k = -18 \rightarrow 13$
5086 independent reflections	$l = -19 \rightarrow 19$

Refinement	
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Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0851P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.047$	+ 0.6154P]
$wR(F^2) = 0.131$	where $P = (F_o^2 + 2F_c^2)/3$
S = 1.07	$(\Delta/\sigma)_{\rm max} = 0.001$
5086 reflections	$\Delta \rho_{\rm max} = 0.53 \ {\rm e} \ {\rm \AA}^{-3}$
299 parameters	$\Delta \rho_{\rm min} = -1.40 \text{ e } \text{\AA}^{-3}$
H-atom parameters constrained	Extinction correction: SHELXL97
-	Extinction coefficient: 1.45(3)

### Table 1

Selected geometric parameters (Å, °).

Ag1-N1	2.277 (3)	Ag1-P1	2.3579 (8)
Ag1-N2	2.334 (3)		
N1-Ag1-N2	72.08 (10)	C19-P1-C1	102.65 (13)
N1-Ag1-P1	144.12 (7)	C13-P1-Ag1	115.68 (10)
N2-Ag1-P1	139.32 (7)	C19-P1-Ag1	111.26 (10)
C13-P1-C19	103.80 (14)	C1-P1-Ag1	116.56 (11)
C13-P1-C1	105.37 (14)		

Table 2	_	
Hydrogen-bonding geometry	(Å,	°).

$D - H \cdots A$	D-H	$H \cdot \cdot \cdot A$	$D \cdots A$	$D - \mathbf{H} \cdots A$
$C1-H1A\cdots F3^{i}$	0.97	2.50	3.460 (5)	170
$C24-H24\cdots F1^{i}$	0.93	2.38	3.042 (8)	128
$C21 - H21 \cdots F1^{ii}$	0.93	2.44	3.111 (8)	129

Symmetry codes: (i) 2 - x, 2 - y, -z; (ii)  $x, \frac{5}{2} - y, \frac{1}{2} + z$ .

The H atoms were geometrically positioned and were treated as riding atoms on the parent C atoms, with C-H distances in the range 0.93–0.97 Å. H atoms were placed in calculated positions and refined using a riding model.

Data collection: SMART (Bruker, 1998); cell refinement: SMART; data reduction: SAINT (Bruker, 1999); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 1998); software used to prepare material for publication: SHELXTL.

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### References

Bruker (1998). SMART (Version 5.0) and SHELXTL (Version 5.1). Bruker AXS Inc., Madison, Wisconsin, USA,

Bruker (1999). SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.

- Deng, L. R., Wang, X. J., Xiao, W., Zhang, H. X., Su, C. Y. & Kang, B. S. (2000). Chem. Res. Chin. Univ. 16, 375-377.
- Kang, B. S., Zhang, L., Deng, L. R., Wang, X. J., Zhang, H. X. & Liu, H. Q. (2002). Sci. China Ser. B, 45, 487-493.
- McLauchlan, C. C. & Ibers, J. A. (2000). Inorg. Chem. pp. 1046-1048.
- Puddephatt, R. J. (2001). Coord. Chem. Rev. pp. 216-217, 313-332.
- Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.
- Yam, V. W. W., Hui, C. K., Wong, K. M. C., Zhu, N. Y. & Cheung, K. K. (2002). Organometallics, 21, 4326-4334.
- Zhang, L., Zhang, H. X., Chen, C. L., Deng, L. R. & Kang, B. S. (2003). Inorg. Chim. Acta. In the press
- Zhuravel, M. A. & Glueck, D. S. (1999). Organometallics, 18, 4673-4676.