Tris[μ-bis(diphenylphosphino)methane-κ^2P:P']-di-μ_3-chloro-trisilver(I) hexafluoroantimonate dichloromethane disolvate

Mei-Mei Wu* and Yong-Hai Qin

State Key Laboratory of Structural Chemistry, Fujian Institute of Research on the Structure of Matter, Fuzhou, Fujian 350002, People's Republic of China
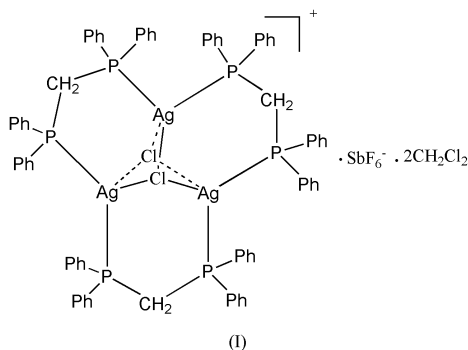
Correspondence e-mail: wmm@ms.fjirsm.ac.cn

The title μ_3-Cl dicapped trisilver compound, [Ag$_3$Cl$_2$-(C$_{25}$H$_{22}$P$_2$)$_3$][SbF$_6$]·2CH$_2$Cl$_2$, exhibits distorted trigonal bipyramidal coordination with two μ_3-Cl atoms at the apices. The Ag^{I} atoms are four-coordinated and exhibit a tetrahedral geometry.

Comment

Bis(diphenylphosphino)methane (dppm) is a good bridging ligand which can form polynuclear complexes with many metal ions (Diez *et al.*, 1987; Jitendra *et al.*, 1999). Trinuclear compounds with group 10 and 11 metals can be stabilized by three bridging dppm ligands, which can form the framework [M$_3$(μ_3-dppm)$_3$] (Diez *et al.*, 1997), examples being [Ag$_3$(μ_3-X)$_2$(μ_3-dppm)$_3$] (X = Cl, Br) (Aly *et al.*, 1978; Franzoni *et al.*, 1989) and [Cu$_3$(μ_3-X)$_2$(μ_3-dppm)$_3$] (X = Cl, Br, I) (Bera *et al.*, 1999). We report here the structure of a new trisilver complex, *viz.* [Ag$_3$(μ_3-Cl)$_2$(μ-dppm)$_3$][SbF$_6$]·2CH$_2$Cl$_2$, (I).

(I)

A perspective drawing of the complex cation with the atomic numbering scheme is shown in Fig. 1 and selected bonding parameters are presented in Table 1. The complex consists of an [Ag$_3$(μ_3-Cl)$_2$(μ_3-dppm)$_3$]$^+$ cation, an SbF$_6^-$ anion, and two dichloromethane solvent molecules. The μ_3-Cl dicapped trisilver complex exhibits distorted trigonal bipyramidal coordination with two μ_3-Cl atoms at the apices, and three Ag atoms in the equatorial sites. The Ag atoms are located in distorted tetrahedral environments with P$_2$Cl$_2$ chromophores. The Ag$\cdots$Ag distances range from 3.3546 (9) to 3.4730 (9) Å, comparable to the sum of the van der Waals radii for silver (3.40 Å). The Ag—Cl distances are in the range 2.690 (2)–2.825 (2) Å, and the Ag—P bond distances are in the range 2.465 (2)–2.506 (2) Å, close to the values found in the similar complex [Ag$_3$(μ_3-Cl)$_2$(μ_3-dppm)$_3$]ClO$_4$ (Zhang *et al.*, 1990).

Experimental

The title compound was isolated from the reaction of [Ag$_2$(μ_2-dppm)$_2$(MeCN)$_2$][SbF$_6$]$_2$ (Diez *et al.*, 1987) and CuCl in a 1:2 ratio in

dichloromethane under anaerobic conditions for 12 h. Well shaped pale-yellow crystals suitable for X-ray diffraction study were grown by slow diffusion of diethyl ether into the dichloromethane solution at room temperature.

Crystal data

$[Ag_3Cl_2(C_{25}H_{22}P_2)_3][SbF_6]\cdot 2CH_2Cl_2$	$D_x = 1.575$ Mg m^{-3}
$M_r = 1953.21$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 6217
$a = 16.0613$ (4) Å	reflections
$b = 18.1719$ (5) Å	$\theta = 1.3$–$25.6°$
$c = 28.3500$ (7) Å	$\mu = 1.39$ mm^{-1}
$\beta = 95.3030$ (10)$°$	$T = 293$ (2) K
$V = 8238.9$ (4) Å^3	Prism, pale yellow
$Z = 4$	$0.60 \times 0.50 \times 0.32$ mm

Data collection

Bruker SMART CCD	14352 independent reflections
diffractometer	10131 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.057$
Absorption correction: multi-scan	$\theta_{max} = 25.6°$
(SADABS; Sheldrick, 1996)	$h = -19 \rightarrow 9$
$T_{min} = 0.438$, $T_{max} = 0.641$	$k = -16 \rightarrow 21$
28476 measured reflections	$l = -32 \rightarrow 34$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0294P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.072$	$+ 55.193P]$
$wR(F^2) = 0.171$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.22$	$(\Delta/\sigma)_{max} = 0.094$
14352 reflections	$\Delta\rho_{max} = 1.54$ e Å^{-3}
892 parameters	$\Delta\rho_{min} = -1.06$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Ag3—P3	2.477 (2)	Ag2—Cl1	2.690 (2)
Ag3—P2	2.488 (2)	Ag2—Cl2	2.852 (2)
Ag3—Cl1	2.762 (2)	Ag1—P6	2.484 (2)
Ag3—Cl2	2.781 (2)	Ag1—P1	2.506 (2)
Ag3···Ag2	3.3544 (9)	Ag1—Cl2	2.705 (2)
Ag2—P5	2.465 (2)	Ag1—Cl1	2.794 (2)
Ag2—P4	2.475 (2)		
P3—Ag3—P2	126.79 (7)	P6—Ag1—P1	131.40 (8)
P3—Ag3—Cl1	122.55 (7)	P6—Ag1—Cl2	112.40 (7)
P2—Ag3—Cl2	110.31 (7)	P6—Ag1—Cl1	102.58 (7)
Cl1—Ag3—Cl2	88.13 (6)	Cl2—Ag1—Cl1	88.98 (7)
P3—Ag3···Ag2	86.27 (5)	C02—P2—Ag3	113.0 (3)
Cl1—Ag3···Ag2	51.06 (5)	C211—P2—Ag3	114.9 (3)
P5—Ag2—P4	125.59 (8)	Ag1—Cl2—Ag3	78.54 (6)
P4—Ag2—Cl1	116.33 (8)	Ag2—Cl1—Ag3	75.94 (6)
P5—Ag2—Cl2	106.01 (7)	C611—P6—Ag1	113.6 (3)
Cl1—Ag2—Cl2	88.10 (7)	C411—P4—Ag2	109.4 (3)
P5—Ag2···Ag3	147.05 (6)	C311—P3—Ag3	116.2 (3)
Cl1—Ag2···Ag3	53.00 (5)	C121—P1—Ag1	113.5 (3)
Cl2—Ag2···Ag3	52.48 (5)		

H atoms were placed in calculated positions, with C—H distances of 0.93 (phenyl) and 0.97 Å (methylene), and included in the riding-model approximation, with $U_{iso} = 1.2U_{eq}(C)$. The dichloromethane solvent molecules were restrained to ideal geometries and appear to be loosely held in the lattice, giving the atoms of these molecules larger than normal displacement parameters. The highest electron-density peak in the final difference Fourier map is 1.83 Å from atom P3 and the deepest hole is 0.95 Å from atom Ag3.
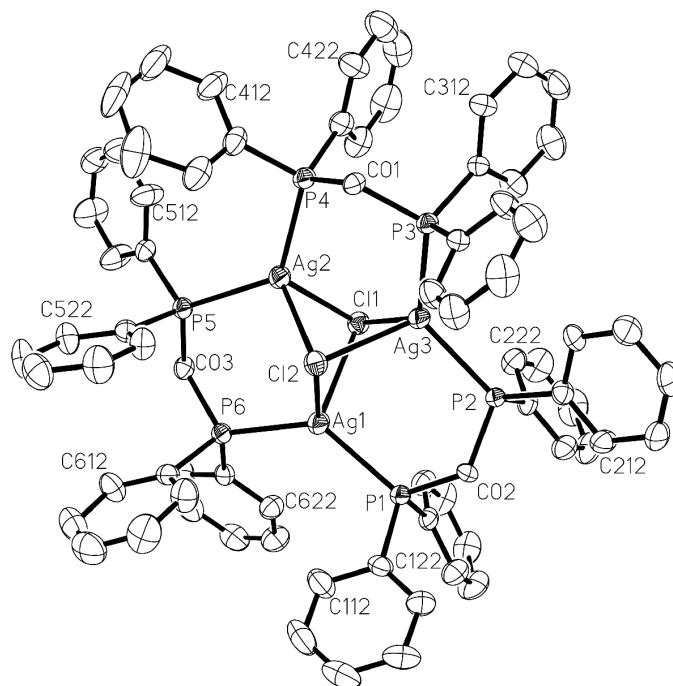

Figure 1
A view of the cation of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted.

Data collection: SMART (Siemens, 1996); cell refinement: SMART and SAINT (Siemens, 1994); data reduction: XPREP (Siemens, 1994); program(s) used to solve structure: SHELXTL (Siemens, 1994); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

References

Aly, A. A. M., Neugebauer, D., Orama, O., Schubert, U. & Schmidbaur, H. (1978). *Angew. Chem. Int. Ed. Engl.* **17**, 125–126.
Bera, J. K., Nethaji, M. & Samuelson, A. G. (1999). *Inorg. Chem.* **38**, 218–228.
Diez, J., Gamasa, M. P., Gimeno, J., Aguirre, A. & Garcia-Granda, S. (1997). *Organometallics*, **16**, 3684–3689.
Diez, J., Gamasa, M. P., GimeNo. J., Tiripicchio. A. & Camellini, M. T. (1987). *J. Chem. Soc. Dalton Trans.* pp. 1275–1278.
Franzoni, D., Pelizzi, G., Predieri, G., Tarasconi, P., Vitali, F. & Pelizzi, C. (1989). *J. Chem. Soc. Dalton Trans.* pp. 247–252.
Sheldrick, G. M. (1996). *SADABS.* University of Göttingen, Germany.
Siemens (1994). *SHELXTL.* Version 5. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Siemens (1996). *SMART* and *SAINT.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Zhang, P., Zhang, Y., Zheng, L. S., Yang, H. H. & Zhang, Q. E. (1990). *Xiamen Dax. Xuebao Zir. Kex.* **29**, 549–552. (In Chinese.)