Orthorhombic
*Pna*2$_1$
$a = 7.675\,(1)$ Å
$b = 25.400\,(7)$ Å
$c = 7.270\,(2)$ Å
$V = 1417.3\,(5)$ Å^3
$Z = 4$
$D_x = 2.220$ Mg m^{-3}
D_m not measured

Cell parameters from 25 reflections
$\theta = 10.0$–$15.0°$
$\mu = 10.064$ mm^{-1}
$T = 295.2$ K
Platelet
0.30 × 0.30 × 0.25 mm
Pale yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan (North, Phillips & Mathews, 1968)
$T_{\min} = 0.052$, $T_{\max} = 0.081$
2811 measured reflections
2406 independent reflections

1803 reflections with $I > 3\sigma(I)$
$R_{\rm int} = 0.030$
$\theta_{\max} = 29.96°$
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 35$
$l = -10 \rightarrow 10$
3 standard reflections every 200 reflections
intensity decay: none

Refinement

Refinement on F
$R = 0.029$
$wR = 0.033$
$S = 1.282$
1803 reflections
172 parameters
H atoms not refined
$w = 1/\sigma^2(F_o) = 1/[\sigma^2{}_c(F_o) + p^2/4F_o^2]$, where $p = 0.010$
$(\Delta/\sigma)_{\max} = 0.003$

$\Delta\rho_{\max} = 0.90$ e Å^{-3}
$\Delta\rho_{\min} = -1.00$ e Å^{-3}
Extinction correction: Zachariasen (1967) type 2, Gaussian isotropic
Extinction coefficient: 11.342×10^{-5}
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

Pt—Cl	2.294 (3)	O(1)—C(1)	1.34 (1)
Pt—N(1)	2.027 (7)	O(1)—C(6)	1.38 (1)
Pt—N(2)	2.042 (7)	N(1)—C(1)	1.30 (1)
Pt—N(3)	2.007 (8)	N(1)—C(7)	1.45 (1)
Cl—Pt—N(1)	92.3 (2)	N(2)—Pt—N(3)	88.5 (6)
Cl—Pt—N(2)	177.5 (5)	C(1)—O(1)—C(6)	106.2 (7)
Cl—Pt—N(3)	89.0 (2)	Pt—N(1)—C(1)	131.7 (7)
N(1)—Pt—N(2)	90.3 (6)	Pt—N(1)—C(7)	122.6 (6)
N(1)—Pt—N(3)	178.3 (3)	C(1)—N(1)—C(7)	105.7 (8)

The H atoms were either located from the Fourier difference electron-density maps or calculated, and included in the structure model. The largest hole in the final difference map was 4.11 Å from the N(3) atom and the largest peak was 0.90 Å from the Pt atom.

Data collection: *CAD-4-PC Software* (Enraf–Nonius, 1991). Cell refinement: *CAD-4-PC Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1985, 1992). Program(s) used to solve structure: *SIR*92 (Altomare, Cascarano, Giacovazzo & Guagliardi, 1993). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: TA1154). Services for accessing these data are described at the back of the journal.

References

Altomare, A., Cascarano, M., Giacovazzo, C. & Guagliardi, A. (1993). *J. Appl. Cryst.* **26**, 343–350.

Enraf–Nonius (1991). *CAD-4-PC Software*. Version 1.1. Enraf–Nonius, Delft, The Netherlands.

Gomez, M., Muir, M., Muir, J. & Cox, O. (1988). *Acta Cryst.* **C44**, 1554–1557.

Hollis, L., Amundsen, A. & Stern, E. (1981). *J. Med. Chem.* **32**, 128–136.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5183. Oak Ridge National Laboratory, Tennessee, USA.

Massacessi, M., Pinna, R. & Ponticelli, G. (1981). *Spectrochim. Acta*, **37**A, 441–444.

Molecular Structure Corporation (1985, 1992). *TEXSAN. Crystal Structure Analysis Package*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Muir, M., Cox, O., Bernard, L. & Muir, J. (1992). *Acta Cryst.* **C48**, 583–585.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.

Zachariasen, W. H. (1967). *Acta Cryst.* **23**, 558–564.

Acta Cryst. (1998). **C54**, 16–18

Bis[μ-methylenebis(diphenylphosphine)-*P*:*P*′]disilver(I) Diperchlorate Bis(dichloromethane) Solvate

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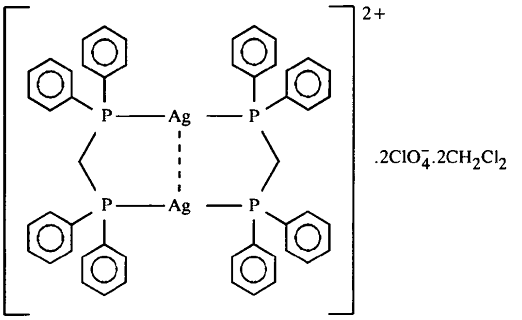
Abstract

The title compound, [Ag$_2$(C$_{25}$H$_{22}$P$_2$)$_2$](ClO$_4$)$_2$.2CH$_2$Cl$_2$, contains eight-membered rings with crystallographic inversion symmetry, with linear coordination at silver [P—Ag—P 170.72 (4)°] and a short intra-annular Ag···Ag contact of 2.9532 (7) Å.

Comment

The use of dppm [bis(diphenylphosphino)methane] as a ligand in coinage metal chemistry often leads to complexes with central eight-membered rings of the form [(dppm)$_2M_2$]; particularly for copper and silver, additional interactions from the metal to the counterion may be observed, *e.g.* in $[Cu_2(\mu\text{-dppm})_2(O_2CPh)_2]$ (Manotti-Lanfredi *et al.*, 1993), $[Ag(OAc)(dppm)]_2$ (Neo *et al.*, 1995) and $[Au_2(\mu\text{-dppm})_2Cl_2]$ (Schmidbaur *et al*, 1977). Short transannular $M\cdots M$ contacts are a common feature of such systems; Ag···Ag contacts (in the range 2.80–3.30 Å) have been reviewed by Jansen (1987), and Au···Au contacts (with similar distances) by Jones (1986), Schmidbaur (1990, 1995) and Pathaneni & Desiraju (1993).

We have now determined the structure of such a silver-based system, (1), with perchlorate as anion. The eight-membered ring displays crystallographic inversion symmetry with a pseudo-chair conformation; the C atoms of the methylene bridges lie 0.736 (4) Å out of the plane of the four P atoms. This conformation is often (but not exclusively) observed in gold or silver systems, *e.g.* in $[Au_2(dppm)_2](NO_3)_2$ (Wang, Khan & Fackler, 1989), $[Au_2(dppm)_2](BF_4)_2$ (Porter *et al.*, 1989) and $[Ag_2(dppm)_2(NO_3)_2]$ (Tiekink, 1990), and in those described by Schmidbaur *et al.* (1977) and Neo *et al.* (1995).



(1)

An exception is furnished by a second form of $[Ag_2(dppm)_2(NO_3)_2]$ (Ho & Bau, 1983), which has a pseudo-boat conformation. This form is more frequently observed for copper systems {*e.g.* in $[Cu_2(\mu\text{-dppm})_2(O_2CPh)(\mu\text{-}O_2CPh)].H_2O$ (Manotti-Lanfredi *et al.*, 1993) and $[Cu_2(\mu\text{-dppm})_2(\mu\text{-mpyO})]BF_4$ (mpyO is 6-methylpyridin-2-olate) (Diez *et al.*, 1990)} than the pseudo-boat conformation {$[Cu_2(\mu\text{-dppm})_2(O_2CPh)_2]$ (Manotti-Lanfredi *et al.*, 1993)}.

The intramolecular Ag···Ag contact in (1) is 2.9532 (7) Å, which is markedly shorter than in the other silver systems {*cf.* 3.194 (2) Å in $[Ag(OAc)(dppm)]_2$ (Neo *et al.*, 1995), 3.109 (1) and 3.085 (1) Å in $[Ag(dppm)(NO_3)]_2$ (Tiekink, 1990; Ho & Bau, 1983)}, but very similar to the value of 2.953 (1) Å in the ylid complex $[Ag_2\{\mu\text{-}\{CH(COOEt)\}_2PPh_2\}_2]$ (Vicente *et al.*, 1989). As would be expected, these values are affected by the coordination number at silver. Whereas the structures with longer Ag···Ag contacts involve anions coordinated to silver [Ag—O 2.416 (5)–2.689 (6) Å], the perchlorate ion in (1) is involved in two much longer Ag···O contacts, of 2.959 (3) Å to O1 and 3.514 (2) Å to O1^{i}, and the coordination number at silver is thus effectively 2, with linear coordination [P—Ag—P^{i} 170.72 (4)°; symmetry code: (i) $1-x$, $1-y$, $1-z$]. The ylid structure displays no extra-annular contacts (Vicente *et al.*, 1989).

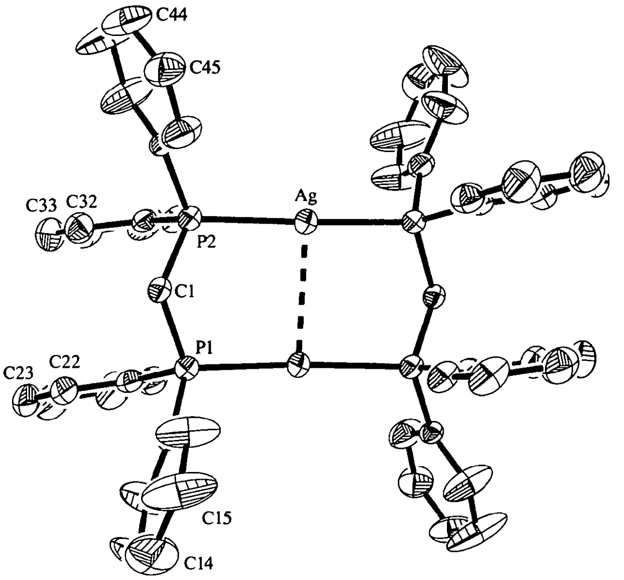


Fig. 1. The structure of the cation of the title compound in the crystal. Ellipsoids represent 50% probability levels. H atoms, anions and solvent have been omitted for clarity.

The P—Ag bond lengths in (1), 2.4017 (11) and 2.4060 (11) Å, are slightly shorter than those in the above-mentioned structures [2.417 (2)–2.473 (3) Å], which again may be related to the lack of further ligands at silver. This is in good agreement with the P—Ag bond length of 2.4106 (9) Å in $[(MePh_2P)_2AgClO_4]$ (Jones, 1995), where, in addition to the two Ag—P bonds, Ag shows a short contact of 3.485 (3) Å to one O atom of the ClO_4 anion.

The Ag atom in (1) is also involved in a weak contact to a solvent Cl atom [Ag···Cl1 3.551 (2) Å]. Furthermore, there are several C—H···O contacts with H···O 2.5–2.6 Å, which could be interpreted as hydrogen bonds; an example is C35—H35···O3^{i}, with C···O 3.230 (7), H···O 2.60 Å and C—H···O 124°.

Experimental

Crystals of compound (1) were obtained after unintentional selenium extrusion from a reaction mixture of dppmSe and $AgClO_4$ in acetone.

Crystal data

$[Ag_2(C_{25}H_{22}P_2)_2](ClO_4)_2.$-
$2CH_2Cl_2$
$M_r = 1353.22$
Monoclinic
$P2_1/n$

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 63 reflections
$\theta = 4.5$–$12.5°$

$a = 11.6819\,(14)$ Å
$b = 15.2304\,(16)$ Å
$c = 16.349\,(2)$ Å
$\beta = 107.548\,(10)°$
$V = 2773.5\,(6)$ Å^3
$Z = 2$
$D_x = 1.620$ Mg m^{-3}
D_m not measured

$\mu = 1.162$ mm^{-1}
$T = 173\,(2)$ K
Prism
$0.34 \times 0.32 \times 0.12$ mm
Colourless

Data collection

Siemens *P*4 diffractometer
ω scans
Absorption correction: ψ scans (*XEMP*; Siemens 1994*a*)
$T_{\min} = 0.741$, $T_{\max} = 0.870$
5061 measured reflections
4869 independent reflections
3161 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.029$
$\theta_{\max} = 24.99°$
$h = -13 \rightarrow 13$
$k = -18 \rightarrow 1$
$l = -19 \rightarrow 0$
3 standard reflections every 247 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.073$
$S = 0.863$
4869 reflections
325 parameters
H atoms constrained
$w = 1/[\sigma^2(F_o^2) + (0.0314P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = -0.001$
$\Delta\rho_{\max} = 0.432$ e Å^{-3}
$\Delta\rho_{\min} = -0.393$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Ag—P1^i	2.4017 (11)	Ag···O1	2.959 (3)
Ag—P2	2.4060 (11)	P1—C1	1.839 (4)
Ag···Ag^i	2.9532 (7)	P2—C1	1.835 (4)
P1^i—Ag—P2	170.72 (4)	C11—P1—Ag^i	111.39 (13)
P1^i—Ag···Ag^i	92.62 (3)	C1—P1—Ag^i	112.34 (13)
P2—Ag···Ag^i	88.81 (3)	C31—P2—C41	106.68 (18)
P1^i—Ag···O1	108.07 (7)	C31—P2—C1	105.32 (19)
P2—Ag···O1	81.11 (7)	C41—P2—C1	105.03 (17)
Ag^i···Ag—O1	72.95 (7)	C31—P2—Ag	114.98 (13)
C21—P1—C11	105.39 (19)	C41—P2—Ag	112.33 (13)
C21—P1—C1	106.19 (18)	C1—P2—Ag	111.75 (13)
C11—P1—C1	105.20 (18)	P2—C1—P1	111.5 (2)
C21—P1—Ag^i	115.55 (14)		
C1—P2—P1^i—C1^i	51.7 (2)	Ag—P2—P1—Ag^i	−8.92 (4)

Symmetry code: (i) $1 - x, 1 - y, 1 - z$.

Data collection: *XSCANS* (Siemens, 1991). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *XP* (Siemens, 1994*b*). Software used to prepare material for publication: *SHELXL*93.

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References

Diez, J., Gamasa, P., Gimeno, J., Lanfranchi, M. & Tiripicchio, A. (1990). *J. Chem. Soc. Dalton Trans.* pp. 1027–1033, and references therein.
Ho, D. M. & Bau, R. (1983). *Inorg. Chem.* **22**, 4073–4079.
Jansen, M. (1987). *Angew. Chem.* **99**, 1136–1149.
Jones, P. G. (1986). *Gold Bull.* **19**, 46–57, and references therein.
Jones, P. G. (1995). *Z. Kristallogr.* **210**, 896.
Manotti-Lanfredi, A. M., Ugozzoli, F., Camus, A., Marsich, N. & Capelletti, R. (1993). *Inorg. Chim. Acta*, **206**, 173–185, and references therein.
Neo, S. P., Zhou, Z. Y., Mak, T. C. W. & Hor, T. S. A. (1995). *Inorg. Chem.* **34**, 520–523.
Pathaneni, S. S. & Desiraju, G. R. (1993). *J. Chem. Soc. Dalton Trans.* pp. 319–322.
Porter, L. C., Khan, M. N. I., King, C. & Fackler, J. P. Jr (1989). *Acta Cryst.* **C45**, 947–949.
Schmidbaur, H. (1990). *Gold Bull.* **23**, 11–21.
Schmidbaur, H. (1995). *Chem. Soc. Rev.* **24**, 391–400.
Schmidbaur, H., Wohlleben, A., Schubert, U., Frank, A. & Huttner, G. (1977). *Chem. Ber.* **110**, 2751–2757.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.
Siemens (1991). *XSCANS Users Manual.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Siemens (1994*a*). *XEMP. Empirical Absorption Correction Program.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Siemens (1994*b*). *XP. Molecular Graphics Program.* Version 5.03. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Tiekink, E. R. (1990). *Acta Cryst.* **C46**, 235–238.
Vicente, J., Chicote, M. T., Saura-Llamas, I. & Jones, P. G. (1989). *Organometallics*, **8**, 767–770.
Wang, J. C., Khan, M. N. I. & Fackler, J. P. Jr (1989). *Acta Cryst.* **C45**, 1482–1485.

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Tetrakis(acetonitrile-*N*)copper(I) Tetrafluoroborate

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Abstract

The title compound, $[Cu(H_3CCN)_4]BF_4$, is isomorphous with the analogous compounds $[M(H_3CCN)_4]ClO_4$ (M = Cu, Ag). It contains three independent formula units. A large spread of values of chemically equivalent dimensions is observed, notably in the cations, which display tetrahedral coordination at copper, with Cu—N