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[*N*,*N*′-Bis(3-phenylprop-2-enylidene)ethylenediamine-κ^2*N*,*N*′]bis(triphenylphosphine-κ*P*)silver(I) nitrate 1.36-acetonitrile 1.64-methanol 0.53-diethyl ether solvate

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In the title compound $[Ag(C_{20}H_{20}N_2)(C_{18}H_{15}P)_2]NO_3\cdot 1.36NCCH_3\cdot 1.64CH_3OH\cdot 0.53H_3CH_2COCH_2CH_3$, the AgN_2P_2 coordination polyhedron can be described as a distorted tetrahedron. The packing of the cations leaves quite large voids in which there are disordered anions and solvent molecules.

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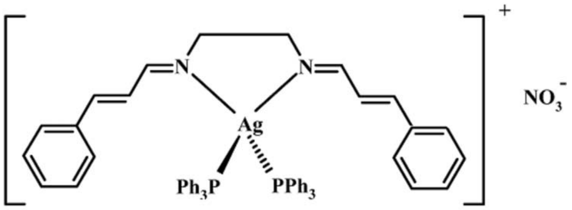
Key indicators

Single-crystal X-ray study
T = 140 K
Mean σ(C–C) = 0.004 Å
H-atom completeness 76%
Disorder in main residue
R factor = 0.055
wR factor = 0.137
Data-to-parameter ratio = 19.4

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

Comment

Silver(I) triphenylphosphine complexes have been increasingly studied over the past decade because of their interesting structural, antimicrobial and anticancer properties (Hannon *et al.*, 1999; Schmittel *et al.*, 2000; Berners-Price *et al.*, 1987). The number of ligands bound to silver(I) is greatly influenced by the chemical nature of the ligand *L* (Amirnasr *et al.*, 2005; Liu *et al.*, 2005; Amirnasr *et al.*, 2006; Khalaji *et al.*, 2006, 2007). In this context, we decided to examine the nature of the complex formed with a conjugated Schiff base ligand (ca_2en) and triphenylphosphine (PPh_3), and (I) has been synthesized and structurally investigated.



$+1.36\ NCCH_3\cdot 1.64\ CH_3OH\cdot 0.53\ H_3CH_2COCH_2CH_3$

(I)

Compound (I) is a monomeric complex (Fig. 1) in which the ca_2en ligand is bidentate and the triphenylphosphine molecules are monodentate. Selected geometric parameters are listed in Table 1. The complex exists as discrete $[Ag(ca_2en)(PPh_3)_2]^+$ cations and disordered NO_3^- anions in the solid and is completed with a number of disordered solvent molecules lying in the voids, with partial occupancy: 1.36 molecules of acetonitrile, 1.64 of methanol and 0.53 of diethyl ether. The coordination environment around the metal ion in this complex is pseudo-tetrahedral with large angular distortion arising from the small intraligand N1—Ag—N2 chelate angle. This angle, fixed by the bite size of the ligand, is similar to the one found for ethylenediamine chelate compounds (75.8°; Khalaji *et al.*, 2007). The P—Ag—N angles deviate somewhat from those of a regular tetrahedron. However, P1—Ag—P2 is larger than expected owing to the steric effects of the bulky PPh_3 ligands. This angle is comparable to that observed in

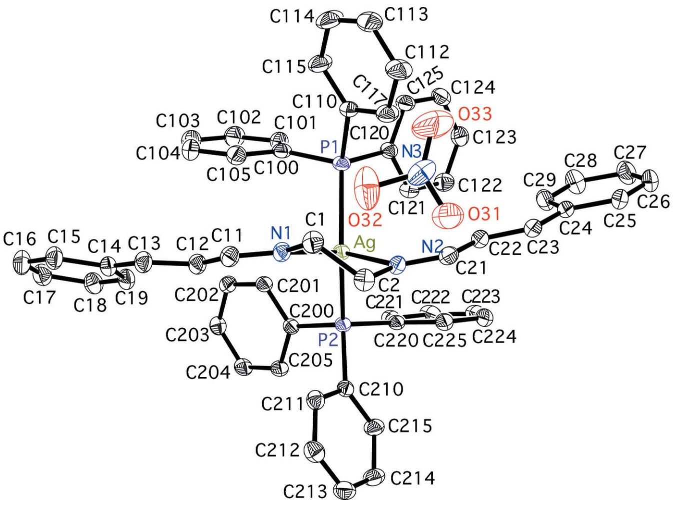


Figure 1
The structure of the $[Ag(ca_2en)(PPh_3)_2]^+$ cation showing displacement ellipsoids drawn at the 50% probability level. The disordered nitrate counter-ion and disordered solvate molecules have been omitted for clarity. H atoms are not shown.

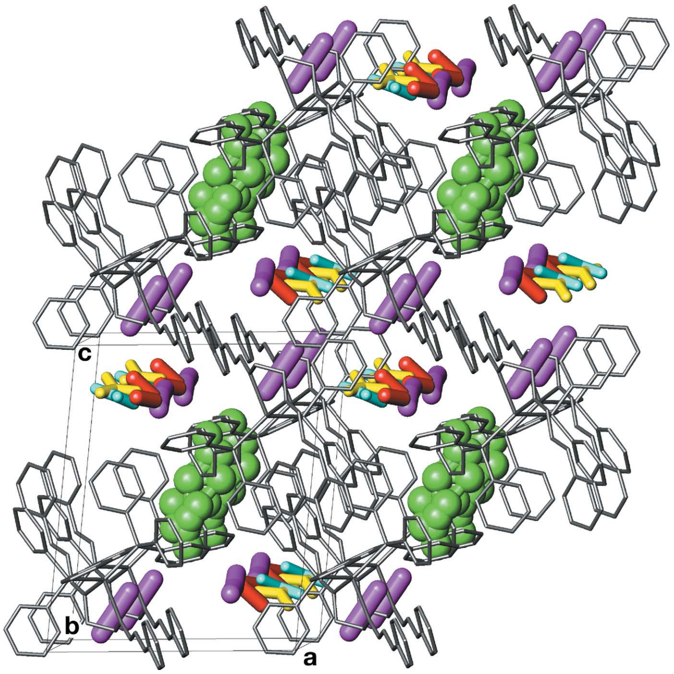


Figure 2
The packing of (I) viewed approximately along the *b* axis. No H atoms are shown. (Acetonitrile molecules are mauve, methanol red, diethyl ether green and nitrate anions turquoise or yellow.)

$[Ag\{H_2B(pz)_2\}(PPh_3)_2]$ [122.51 (3)°; Effendy *et al.*, 2000]. The average Ag—N [2.370 (4) Å] and Ag—P distances [2.45 (1) Å] agree well with those found for analogous silver(I) complexes (Effendy *et al.*, 2000; Richmond *et al.*, 1988), Ag—N = 2.349 Å for $[Ag\{N,N'\text{-bis(orthobromobenzaldehyde)-1,2-diaminoethane}\}_2]^+$ (Richmond *et al.*, 1988), and Ag—P = 2.448 Å for $[Ag(tetz)(PPh_3)_2]_n$ (Nomiya *et al.*, 2000). Despite the fact that the donor N atoms are sp^2 hybridized, the chelate ring is significantly puckered in this complex and some strain in the chelate ring is suggested by the deviation from 120° angles about the nitrogen atoms. Atom C1 is 0.423 (4) Å below the MN_2 plane defined by N1—Ag—N2 and atom C2 is 0.332 (4) Å above this plane. The dihedral angle between the AgN_2 and AgP_2 planes is 83.16 (5) °. The crystal packing of (I) is shown in Fig. 2.

Experimental

To a stirred solution of 170 mg (1 mmol) $AgNO_3$ in 2 ml acetonitrile was added dropwise a solution of 524 mg (2 mmol) of PPh_3 in 5 ml acetonitrile at room temperature, and the mixture was stirred for 15 min. The volume of the solution was then reduced under vacuum to about 3 ml, a solution of 288 mg (1 mmol) of ca_2en in 3 ml methanol was added and the reaction mixture was stirred for an additional 20 min. Colourless crystals of (I) (809 mg, 72% yield) suitable for X-ray crystallography were obtained by the diffusion of diethyl ether vapour into the solution at 273 K. The crystals were collected by filtration and dried under vacuum. Found: C 66.16, H 5.85, N 5.17°; $C_{62.5}H_{65.9}N_{4.3}AgO_{5.1}P_2$ requires: C 66.59, H 5.77, N 5.34%.

Crystal data

$[Ag(C_{20}H_{20}N_2)(C_{18}H_{15}P)_2]NO_3\cdot{}$
$1.36C_2H_3N\cdot1.64CH_4O\cdot{}$
$0.53C_4H_{10}O$
$M_r = 1130.66$
Triclinic, $P\overline{1}$
$a = 13.5727\ (9)$ Å
$b = 14.9626\ (10)$ Å
$c = 15.7828\ (11)$ Å
$\alpha = 80.578\ (5)°$
$\beta = 82.201\ (5)°$
$\gamma = 64.867\ (4)°$
$V = 2854.7\ (4)$ Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 0.46$ mm^{-1}
$T = 140\ (1)$ K
0.71 × 0.32 × 0.23 mm

Data collection

Stoe IPDS2 diffractometer
Absorption correction: integration
(*X-RED32*; Stoe & Cie, 2005)
$T_{min} = 0.804$, $T_{max} = 0.914$
27609 measured reflections
14231 independent reflections
12840 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.137$
$S = 1.12$
14231 reflections
735 parameters
17 restraints
H-atom parameters constrained
$\Delta\rho_{max} = 1.19$ e Å^{-3}
$\Delta\rho_{min} = -1.41$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ag—N1	2.3740 (17)	Ag—P1	2.4621 (6)
Ag—N2	2.366 (2)	Ag—P2	2.4393 (6)
C1—N1—C11	117.97 (19)	N1—Ag—P1	105.17 (5)
C1—N1—Ag	108.41 (13)	N1—Ag—P2	119.74 (5)
C2—N2—C21	118.9 (2)	N2—Ag—P1	113.12 (5)
C2—N2—Ag	109.84 (14)	N2—Ag—P2	112.10 (5)
N1—Ag—N2	74.05 (6)	P1—Ag—P2	122.70 (2)

H atoms on the aromatic rings were positioned geometrically (C—H = 0.93 Å), and refined as riding, with $U_{iso}(H) = 1.2U_{eq}(C)$. The counter-ion and the solvent molecules in the voids had to be refined with restraints [O—C = 1.48 (2) Å, C—C = 1.55 (2) Å, second neighbours 2.50 (2) Å, common isotropic *U*]. No H-atom positions

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could be found for them and these H atoms were, consequently, omitted. For the diethyl ether molecule, isotropic displacement parameters were used. In the treatment of the disorder, the site-occupancy factors were refined as follows: for the nitrate: major (N3) 0.609(3), minor (N3*A*) 0.391(3), sum = 1.00; for the acetonitrile: N72/C72/C73 0.362(4); for the methanol: O88/C88 0.638(4); for the diethyl ether 0.523(9). $\Delta\rho_{\rm max}$ is 0.73 Å from the Ag atom, and $\Delta\rho_{\rm min}$ is 0.71 Å from the same atom.

Data collection: *X-AREA* (Stoe & Cie , 2006); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2005); program(s) used to solve structure: *DIRDIF* (Beurskens *et al.*, 1996); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1996) and *ATOMS* (Dowty, 2006); software used to prepare material for publication: *SHELXTL*.

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References

Amirnasr, M., Khalaji, A. D., Falvello, L. R. & Soler, T. (2006). *Polyhedron*, **25**, 1967–1970.

Amirnasr, M., Welter, R. & Khalaji, A. D. (2005). *Acta Cryst.* E**61**, m1452–m1454.

Berners-Price, S. G., Johnson, R. K., Mirabelli, C. K., Faucette, L. F., McCabe, F. L. & Sadler, P. J. (1987). *Inorg. Chem.* **26**, 3383–3389.

Beurskens, P. T., Beurskens, G., Bosman, W. P., de Gelder, R., Garcia-Granda, S., Gould, R. O., Israël, R. & Smits, M. M. (1996). *The DIRDIF96 Program System*. Crystallography Laboratory, University of Nijmegen, The Netherlands.

Dowty, E. (2006). *ATOMS*. Version 6.3.1. Shape Software, 521 Hidden Valley Road, Kingsport, TN 37663, USA.

Effendy, Lobbia, G. G., Pellei, M., Pettinari, C., Santini, C., Skelton, B. W. & White, A. H. (2000). *J. Chem. Soc. Dalton Trans.* pp. 2123–2130.

Hannon, M. J., Painting, C. L. & Alkock, N. W. (1999). *Chem. Commun.* pp. 2023–2024.

Khalaji, A. D., Amirnasr, M. & Aoki, K. (2006). *Anal. Sci.* **22**, x87–x88.

Khalaji, A. D., Aoki, K. & Amirnasr, M. (2007). *J. Coord. Chem.* **60**, 201–206.

Liu, S.-Q., Konaka, H., Kuroda-Sowa, T., Ning, G.-L. & Munakata, M. (2005). *Acta Cryst.* C**61**, m194–m196.

Nomiya, K., Noguchi, R. & Oda, M. (2000). *Inorg. Chim. Acta*, **298**, 24–32.

Richmond, T. G., Kelson, E. P. & Arif, A. M. (1988). *J. Am. Chem. Soc.* **110**, 2334–2336.

Schmittel, M., Ganz, A., Fenske, D. & Herderich, M. (2000). *J. Chem. Soc. Dalton Trans.* pp. 353–360.

Sheldrick, G. M. (1996). *SHELXTL*. Version 5.05. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.

Stoe & Cie (2005). *X-RED*32. Version 1.31. Stoe & Cie GmbH, Darmstadt, Germany.

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