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(1,10-Phenanthroline-κ^2N,N')(triphenylphosphine-κP)silver(I) perchlorate acetonitrile hemisolvate

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The asymmetric unit of the title compound, $[Ag(C_{12}H_8N_2)(C_{18}H_{15}P)]ClO_4\cdot 0.5CH_3CN$, consists of two $[Ag(phen)(PPh_3)]^+$ complex cations (phen is 1,10-phenanthroline), two perchlorate anions and one acetonitrile solvent molecule. The Ag^I atoms assume a Y-shaped nearly planar geometry in which the metal ion is ligated by two phen N atoms and one P atom of a triphenylphosphine ligand. The two cations in the asymmetric unit are stacked in an inverse fashion through strong offset π–π stacking interactions with a face-to-face distance of 3.479 (2) Å, leading to accessible voids for acetonitrile solvent molecules. The disordered solvent molecule could not be satisfactorily modelled and, therefore, it was eliminated from the final refinement.

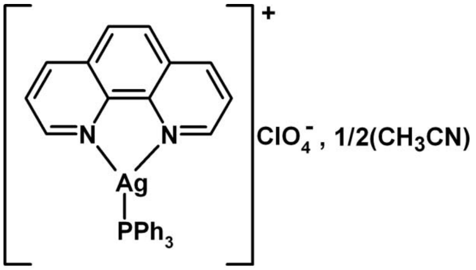
Key indicators

Single-crystal X-ray study
T = 180 K
Mean σ(C–C) = 0.007 Å
Some non-H atoms missing
R factor = 0.045
wR factor = 0.131
Data-to-parameter ratio = 20.1

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

Comment

Metal complexes with 1,10-phenantroline (phen) as a ligand have been increasingly studied over recent years (Bai *et al.*, 2005; Su & Xu, 2005; Pan & Xu, 2005; Hwang & Ha, 2006; Xu *et al.*, 2005; Zhang *et al.*, 2006; Du *et al.*, 2006; Liu *et al.*, 2006). We have examined the nature of the complex formed with 1,10-phenantroline (phen) and triphenylphosphine (PPh_3) ligand. The title compound $[Ag(phen)(PPh_3)]ClO_4\cdot 0.5CH_3CN$, (I), has been synthesized and structurally investigated.



(I)

The asymmetric unit of compound (I) contains two $[Ag(phen)(PPh_3)]^+$ cations, two ClO_4^- anions and one acetonitrile solvent molecule. Both Ag^I atoms assume a Y-shaped, roughly planar coordination in which the metal ions are ligated by two N atoms of a 1,10-phenantroline (phen) ligand and one P atom of a triphenylphosphine (PPh_3) ligand (Fig. 1). Ag1 and Ag2 lie out of the P1/N11/N12 and P2/N21/N22 planes by 0.196 (2) and 0.079 (2) Å, respectively. The two cations are stacked in an inverse fashion through strong offset π–π stacking interactions, with a face-to-face distance of 3.479 (2) Å. A similar situation was observed in the related [Ag(phen)(CN)] compound (Huang *et al.*, 2004). The N—

Ag—N and N—Ag—P bond angles as well as the Ag—N and Ag—P distances (Table 1) are similar to the corresponding bond angles and distances in the related complexes $[Ag(NO_3)(C_{10}H_8N_2)(C_{18}H_{15}P)]$ (Amirnasr *et al.*, 2005) and $[Ag_2(C_{10}H_8N_2)_2(C_{28}H_{28}P_2)](BF_4)_2$ (Zhang *et al.*, 2003).

Experimental

At room temperature a solution of PPh_3 (0.1 mmol) in acetonitrile (5 ml) was added dropwise to a stirred solution of $AgClO_4$ (0.1 mmol) in acetonitrile (2 ml), 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 phen (0.1 mmol) in methanol (3 ml) was added and the reaction mixture was stirred for another 20 min. Colourless crystals of (I) 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. Chemical analyses suggests the presence of half an acetonitrile solvent molecule per complex.

Crystal data

$[Ag(C_{12}H_8N_2)(C_{18}H_{15}P)]$-$ClO_4\cdot0.5C_2H_3N$
$M_r = 670.32$
Monoclinic, $P2_1/c$
$a = 16.6708\ (12)$ Å
$b = 22.9245\ (17)$ Å
$c = 14.7068\ (13)$ Å
$\beta = 94.063\ (6)^\circ$
$V = 5606.4\ (8)$ Å^3
$Z = 8$
$D_x = 1.588$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.91$ mm^{-1}
$T = 180\ (2)$ K
Lath, colourless
$0.49 \times 0.19 \times 0.09$ mm

Data collection

Oxford Diffraction Xcalibur diffractometer
ω and φ scans
Absorption correction: multi-scan (Blessing, 1995)
$T_{\min} = 0.798$, $T_{\max} = 0.943$
50182 measured reflections
13918 independent reflections
6516 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.061$
$\theta_{\max} = 28.3^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.131$
$S = 0.93$
13918 reflections
691 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0622P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.005$
$\Delta\rho_{\max} = 0.79$ e Å^{-3}
$\Delta\rho_{\min} = -1.18$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ag1—N11	2.264 (3)	Ag2—N21	2.281 (3)
Ag1—P1	2.3332 (11)	Ag2—N22	2.311 (3)
Ag1—N12	2.333 (3)	Ag2—P2	2.3524 (11)
N11—Ag1—P1	147.14 (9)	N21—Ag2—N22	73.11 (12)
N11—Ag1—N12	74.01 (12)	N21—Ag2—P2	145.71 (9)
P1—Ag1—N12	136.06 (9)	N22—Ag2—P2	140.72 (10)

All H atoms were positioned geometrically and treated as riding with C—H = 0.95 Å and $U_{\rm iso}(H) = 1.2U_{\rm eq}(C)$. Some residual electron density in the accesible voids of the structure was difficult to model. Therefore the SQUEEZE function of *PLATON* (Spek, 2003) was used to eliminate the contribution of the electron density in the solvent region from the intensity data, and the solvent-free model was employed for the final refinement. There are four cavities of *ca* 101 Å^3 per unit cell. *PLATON* estimated that each cavity contains 20 electrons, which may correspond to a solvent molecule of acetonitrile as suggested by chemical analyses. The deepest hole in the final Fourier map is 2.11 Å from atom H123.

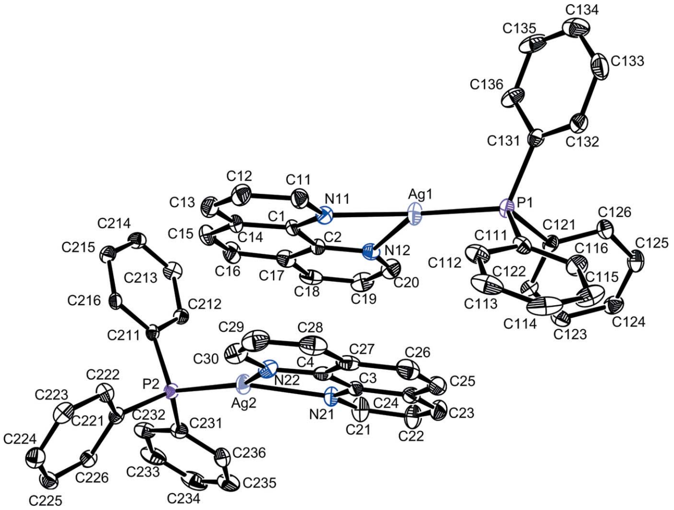


Figure 1
The structure of the two $[Ag(phen)(PPh_3)]^+$ cations with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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