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A bent two-coordinate silver(I) complex with 2,2′,3,3′-tetrahydro-4,4′-dithia-1,1′-binaphylidene

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Key indicators

Single-crystal X-ray study
T = 200 K
Mean σ(C–C) = 0.005 Å
R factor = 0.034
wR factor = 0.079
Data-to-parameter ratio = 13.9

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

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The Ag^{I} complex of 2,2′,3,3′-tetrahydro-4,4′-dithia-1,1′-binaphylidene (ttp) has been synthesized and its molecular structure determined as its PF_6^- salt, *i.e.* (2,2′,3,3′-tetrahydro-4,4′-dithia-1,1′-binaphylidene)silver(I) hexafluorophosphate, $[Ag(C_{18}H_{16}S_2)]PF_6$. The Ag^{I} ion has a distorted linear geometry comprised of the two S atoms of ttp, with an Ag—S distance of 2.4889 (8) Å and an S—Ag—S angle of 157.48 (4)°; the cation has twofold symmetry. This coordination mode results in the formation of a nine-membered ring.

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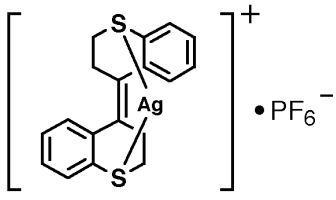
Comment

The systematic study of cyclic sulfur ligands and their coordination complexes is of current interest in the fields of molecular organic conductors and inorganic chemistry (Blake, Brooks *et al.*, 2000; Blake, Champness *et al.*, 2000; Blake *et al.*, 1997; Desper & Gellman, 1994; Misaki *et al.*, 1993). According to this research, metal complexes of the cyclic sulfur ligands 1,4-dithiane, TTF (tetrathiafulvalene) and dmit (4,5-ethylenedithio-1,3-dithiole-2-thione) show different functional frameworks and exhibit metallic conductivity or even superconductivity, as there is a rich diversity of packing arrangments for these complexes compared with the organic molecules themselves (Munakata, Kuroda-Sowa *et al.*, 1995; Munakata, Wu *et al.*, 1995; Gan *et al.*, 1994; Williams *et al.*, 1991).



(I)

As part of our continuing interest in the complexation properties of cyclic sulfur mixed-donor ligands with silver ions (Dai, Munakata *et al.*, 1997; Dai, Kuroda-Sowa *et al.*, 1997), the ligand 2,2′,3,3′-tetrahydro-4,4′-dithia-1,1′-binaphylidene (ttp) has been synthesized and its silver(I) complex, $[Ag(ttp)]^+$, as the PF_6^- salt, prepared and the crystal structure determined.

The Ag^{I} and P1 atoms are each located on a crystallographic twofold axis. The Ag^{I} atom is coordinated by two S atoms of the chelating ligand (Fig. 1) so that the Ag—S distance is 2.4889 (8) Å and the S—Ag—S^{i} bond angle is 157.48 (4)° [symmetry code: (i) $\frac{3}{2} - x, \frac{1}{2} - y, z$]. The bidentate

coordination mode of the ligand results in the formatation of a nine-membered ring.

The Ag—S bond lengths reported for the silver(I) complexes of 1,4-dithiane (Blake, Brooks *et al.*, 2000) and 4,5-ethylenedithio-1,3-dithiole-2-thione (Dai, Munakata *et al.*, 1997; Dai, Kuroda-Sowa *et al.*, 1997), are in the ranges 2.5027 (8)–2.4999 (11) Å and 2.596 (1)–2.841 (1) Å, respectively, and these are longer than that found in the present complex.

Experimental

To tetrahydrofuran (10 ml) were added $AgPF_6$ (25.3 mg, 10 mmol) and 2,2′,3,3′-tetrahydro-4,4′-dithia-1,1′-binaphylidene (29.8 mg, 10 mmol). The mixture was stirred and filtered. A portion of the colorless filtrate was transferred to a 7 mm glass tube under argon and layered with *n*-pentane. The glass tube was sealed under argon and wrapped with aluminium foil. Colorless needles of $[Ag(ttp)]PF_6$ were obtained from the solution after standing for 2 d at room temperature in the dark. Found: C 38.54, H 2.81%; calculated for $C_{18}H_{16}AgF_6PS_2$: C 39.36, H 2.94%. IR data as KBr disks (cm^{-1}): 2970.74 (*w*), 2926.37 (*w*), 1466.08 (*s*), 794.77 (*w*), 761.97 (*s*), 736.90 (*w*).

Crystal data

$[Ag(C_{18}H_{16}S_2)]PF_6$
M_r = 549.28
Orthorhombic, *Pccn*
a = 7.109 (3) Å
b = 15.014 (2) Å
c = 17.139 (2) Å
V = 1829.4 (8) Å^3
Z = 4
D_x = 1.994 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 28404 reflections
θ = 6.3–27.6°
μ = 1.48 mm^{-1}
T = 200 K
Block, colorless
0.20 × 0.20 × 0.10 mm

Data collection

Quantum CCD/Rigaku AFC-8 diffractometer
ω scans
Absorption correction: multi-scan (*REQAB*; Jacobson, 1998)
T_{min} = 0.694, T_{max} = 0.863
28 404 measured reflections
2082 independent reflections
1777 reflections with $I > 2\sigma(I)$
R_{int} = 0.039
θ_{max} = 27.6°
$h = -8 \rightarrow 9$
$k = -18 \rightarrow 19$
$l = -22 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.079
S = 1.13
1777 reflections
128 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0266P)^2 + 4.3325P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.88 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ag1—S1	2.4889 (8)	S1—C9	1.773 (3)
S1—C1	1.823 (3)		
S1—Ag1—S1^{i}	157.48 (4)	Ag1—S1—C9	99.04 (9)
Ag1—S1—C1	99.59 (10)	C1—S1—C9	94.1 (1)

Symmetry code: (i) $\frac{3}{2} - x, \frac{1}{2} - y, z$.

All H atoms were placed in idealized positions and included as riding atoms with fixed isotropic displacement parameters.

Data collection: *D*TREK* (Rigaku Corporation, 1991); cell refinement: *D*TREK*; data reduction: *TEXSAN* (Molecular Structure Corporation & Rigaku Corporation, 1999); program(s) used to solve structure: *MULTAN*88 (Debaerdemaeker *et al.*, 1988); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *TEXSAN*.

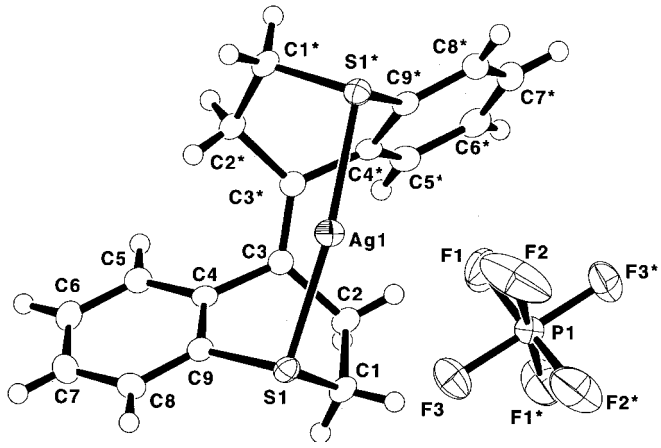


Figure 1
The structure of (I) showing 50% probability displacement ellipsoids (*ORTEP*II; Johnson, 1976).

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