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catena-Poly[[silver(I)-μ-{bis[4-(2-pyridylmethyleneamino)phenyl] ether}] trifluoromethanesulfonate 0.4-hydrate]: a zigzag coordination polymer

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The title compound, $[Ag(C_{24}H_{18}N_4O)]CF_3SO_3\cdot 0.4H_2O$, can be described as a zigzag polymer. The Ag atom is coordinated to the N atoms of two pyridylimine units resulting in a distorted tetrahedral coordination geometry. Both the Ag atom and the central O atom of the ligand are situated on mutually perpendicular crystallographic twofold axes.

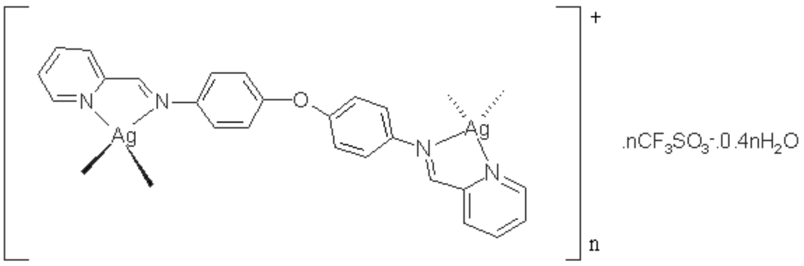
Key indicators

Single-crystal X-ray study
T = 153 K
Mean σ(C–C) = 0.009 Å
H-atom completeness 96%
Disorder in solvent or counterion
R factor = 0.042
wR factor = 0.095
Data-to-parameter ratio = 12.2

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

Comment

The reaction of the ligand bis[4-(2-pyridilmethyleneamino)-phenyl] ether, L1, with $Ag(CF_3SO_3)$ yielded an ionic complex, AgL1, (I), consisting of a one-dimensional zigzag coordination polymer, Fig. 1. Similar single-stranded silver polymers have been reported previously (Bowyer *et al.*, 1998; Carlucci *et al.*, 1998; Suzuki *et al.*, 1995; Withersby *et al.*, 1997). Using L1 and $AgNO_3$ very small poor crystals of a similar zigzag polymer have also been prepared (Tesouro Vallina & Stoeckli-Evans, 1999*a*). Using BF_4^- as counter-ion Cheng *et al.* (2000) have recently published the structure of a silver(I) double-stranded helix, previously postulated by Hannon *et al.* (1999) and Yoshida *et al.* (2000), for a similar ligand where the central ether linkage is replaced by a CH_2 group. In AgL1, the $Ag—N_{py}$ and $Ag—N_{im}$ bond distances, 2.283 (4) and 2.343 (4) Å, respectively, are similar to those observed in the above-mentioned complexes. The chelate bite angle of 72.69 (16)° is also within the expected range. The ligand is twisted about the central O atom with the two benzene rings being inclined by 51.09 (2)°. The two halves of the ligand (related by a twofold axis) are fairly planar which contrasts with the structure of the ligand itself, where one half of the ligand is much less planar than the other (Tesouro Vallina & Stoeckli-Evans, 2001). The best plane through atoms N1/C1–C6/N2/C7–C12 is planar to within 0.055 (5) Å and the Ag atom is displaced from this plane by 0.227 (5) Å. In the crystal, the chains stack up the c axis with a π–π overlap of symmetry-related aromatic rings; the shortest intermolecular C···C distance is *ca* 3.52 (2) Å, Fig. 2.



(I)

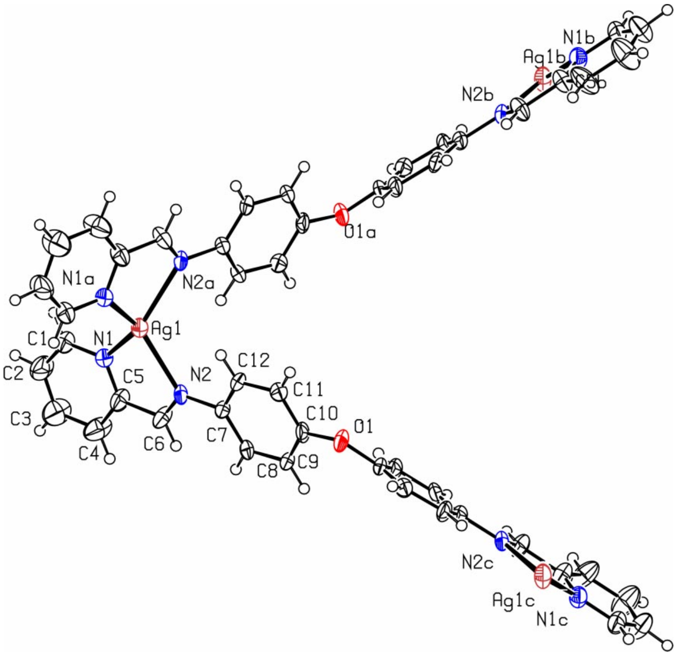


Figure 1
View of the title compound showing the numbering scheme and displacement ellipsoids at 50% probability level. The disordered $CF_3SO_3^-$ anion and partially occupied water molecule have been omitted for clarity.

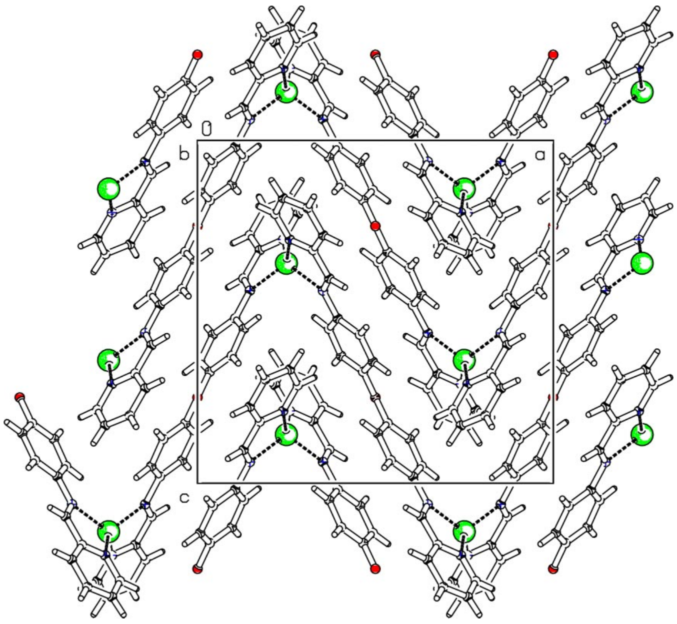


Figure 2
Crystal packing of (I) showing the π–π interactions. The H atoms and the disordered $CF_3SO_3^-$ anion and partially occupied water molecule have been omitted for clarity.

61 Despite the polymeric nature of the title compound, (I), in the solid state, positive ESI–MS (electrospray ionization mass spectroscopy) in acetonitrile indicates that the most abundant ion in solution is $[Ag_2L_2]^{2+}$ at *m*/*z* 486, which corresponds to a binuclear complex, probably the double helix.

Experimental

The synthesis of bis[4-(2-pyridilmethyleneamino)phenyl]ether, L1, has been reported elsewhere (Cheng *et al.*, 2000; Tesouro Vallina & Stoeckli-Evans, 1999*b*). A methanolic solution of $AgCF_3SO_3$ (1 equivalent, 0.1 mmol, 0.0257 g per 15 ml) was added slowly to a methanolic solution of L1 (1 equivalent, 0.1 mmol, 0.0378 g per 10 ml) under N_2 and protected from the light. The colour of the mixture changed to deep yellow and a precipitate appeared. The mixture was heated to *ca* 313 K with stirring for 2 h, cooled to room temperature and filtered. The yellow solid obtained was dissolved in acetonitrile and left undisturbed in the dark for *ca* two weeks, whereupon yellow–green crystals were formed. IR (KBr pellet, cm^{-1}): 3468, 3067 (C—H), 1626, 1592, 1561, 1496, 1441, 1420 (C═C, C═N), 1251 (Ph—O), 1056, 1029, 1008, 837, 775 (CF_3). Analysis for $[AgL][CF_3SO_3]\cdot 2H_2O$ (623.41 g mol^{-1}), calculated: C 48.17, H 3.71, N 8.98%; found: C 48.43, H 3.36, N 8.20%. MS (ESI) *m*/*z*: 1121, $[Ag_2L_2CF_3SO_3]^+$; 865, $[AgL_2]^+$; 742, $[Ag_2LCF_3SO_3]^+$ and 486, $[Ag_2L_2]^{2+}$. UV/Vis (λ_{max}/nm, ethanol solution): 330, 390. ^1H NMR (DMSO-d^6): 7.09 (4H, *d*, Ph), 7.50 (4H, *d*, Ph), 7.70 (2H, *m*, py), 8.13 (4H, *dd*, py), 8.78 (2H, *d*, py), 8.89 (2H, *s*, C═N).

Crystal data

$[Ag(C_{24}H_{18}N_4O)]CF_3SO_3\cdot 0.4H_2O$
$M_r = 642.57$
Orthorhombic, *Pcca*
$a = 15.3582$ (15) Å
$b = 11.6957$ (12) Å
$c = 14.8253$ (12) Å
$V = 2663.0$ (4) $Å^3$
$Z = 4$
$D_x = 1.603$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 5109 reflections
$\theta = 2.6$–25.9°
$\mu = 0.90$ mm^{-1}
$T = 153$ (2) K
Block, pale yellow
0.25 × 0.10 × 0.05 mm

Data collection

Stoe IPDS diffractometer
φ oscillation scans
19 496 measured reflections
2588 independent reflections
1105 reflections with $I > 2\sigma(I)$

$R_{int} = 0.180$
$\theta_{max} = 25.9°$
$h = -18 \rightarrow 18$
$k = -14 \rightarrow 14$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.095$
$S = 0.74$
2588 reflections
212 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.04P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.59$ e $Å^{-3}$
$\Delta\rho_{min} = -0.68$ e $Å^{-3}$
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0014 (3)

Table 1
Selected geometric parameters (Å, °).

Ag1—N1	2.283 (4)	Ag1—N2	2.343 (4)
N1—Ag1—N1^i	126.9 (2)	N1—Ag1—N2	72.69 (16)
N1—Ag1—N2^i	138.59 (17)	N2^i—Ag1—N2	119.2 (2)

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

The $CF_3SO_3^-$ anion was disordered about a center of symmetry. The H atoms were included in calculated positions and treated as riding atoms using *SHELXL* default parameters. The H atoms of the partially occupied water molecule could not be located. The R_{int} value of 0.18 is due to the poor quality of the crystal, which did not diffract significantly above 40° in 2θ, and the low $I/\sigma(I)$ ratio obtained. The ratio of parameters to observed data is at the lower end

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of the scale, *ca* 5, but the s.u.'s. are reasonable. This is also responsable for the GOF value which is slightly less than 0.8.

Data collection: *EXPOSE* (Stoe & Cie, 1997); cell refinement: *CELL* (Stoe & Cie, 1997); data reduction: *INTEGRATE* (Stoe & Cie, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON*99 (Spek, 1990); software used to prepare material for publication: *SHELXL*97.

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