

Bis(4-aminopyridine)silver(I) trifluoroacetate

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Key indicators

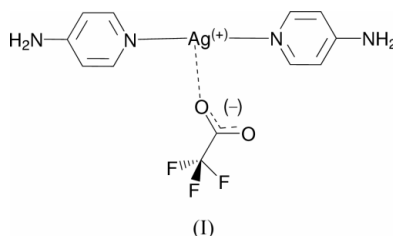
Single-crystal X-ray study
T = 298 K
Mean $\sigma(\text{C}-\text{C}) = 0.008 \text{ \AA}$
R factor = 0.044
wR factor = 0.133
Data-to-parameter ratio = 13.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the title mononuclear complex, $[\text{Ag}(\text{C}_5\text{H}_6\text{N}_2)_2](\text{CF}_3\text{O}_2)$, the Ag atom is coordinated in an almost linear configuration by the pyridine N atoms of two 4-aminopyridine ligands. An O atom from the trifluoroacetate anion weakly ligates to the Ag atom to give a T-shaped environment. In the crystal structure, the two moieties are interconnected by N—H...O hydrogen bonds and weak Ag...F interactions to form layers.

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Comment

The coinage metals, especially silver, have been the subject of investigation for preparing novel complexes for decades. Interest in this area grew out of the diverse structural motifs displayed by these superficially similar monovalent cations. Recently, we reported several 2-aminopyrimidine complexes (Zhu, Usman *et al.*, 2003; Zhu, Wang *et al.*, 2003) with Ag^{I} atoms, which exhibit interesting bioactivities (to be reported elsewhere). We report here the crystal structure of a new 4-aminopyridinesilver(I) trifluoroacetate complex, (I).



As shown in Fig. 1, atom Ag1 is coordinated by the pyridine N atoms of two independent 4-aminopyridine ligands. One O atom from the trifluoroacetate anion weakly ligates to the Ag atom [$\text{Ag1}-\text{O1} = 2.843(4) \text{ \AA}$]. The Ag—N distances [$\text{Ag1}-\text{N1} = 2.111(4) \text{ \AA}$ and $\text{Ag1}-\text{N3} = 2.119(4) \text{ \AA}$] are within acceptable values, but are shorter than those in the silver(I) complexes of 2-aminopyridines [$2.230(3)$ and $2.205(4) \text{ \AA}$ (Zhu, Usman *et al.*, 2003), and $2.137(2) \text{ \AA}$ (Zhu, Wang *et al.*, 2003)]. The bond angles [$\text{N1}-\text{Ag1}-\text{N3} = 166.5(15)^\circ$, $\text{N1}-\text{Ag1}-\text{O1} = 94.8(2)^\circ$ and $\text{N3}-\text{Ag1}-\text{O1} = 96.2(2)^\circ$] indicate a distorted T-shaped coordination environment of atom Ag1. To reduce steric effects, the two pyridine rings connected by Ag1 are nearly perpendicular to one another, with a dihedral angle of $78.4(2)^\circ$. All other bond lengths and angles are in the normal ranges.

In the crystal structure of (I) (Fig. 2), discrete cations are interconnected by N—H...O hydrogen bonds to form a one-dimensional chain (see Table 1 for details). These chains are further linked by three short Ag...F contacts [$\text{Ag1}\cdots\text{F1} = 3.414(4) \text{ \AA}$, $\text{Ag1}\cdots\text{F1}^i = 3.376(4) \text{ \AA}$ and $\text{Ag1}\cdots\text{F2}^i = 3.593(4) \text{ \AA}$; symmetry code: (i) $2 - x, 2 - y, 1 - z$], forming a layer-like structure (Fig. 3).

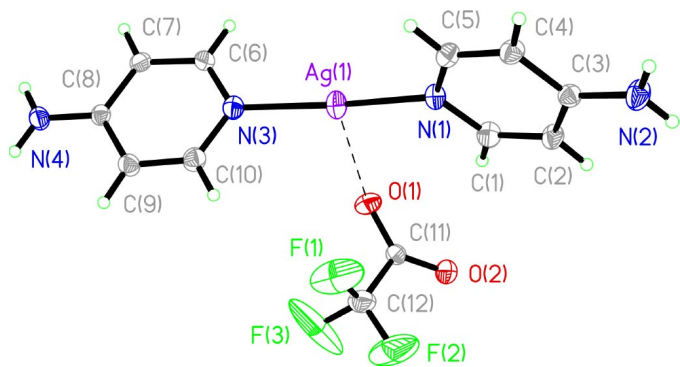


Figure 1
The structure of the title compound, (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

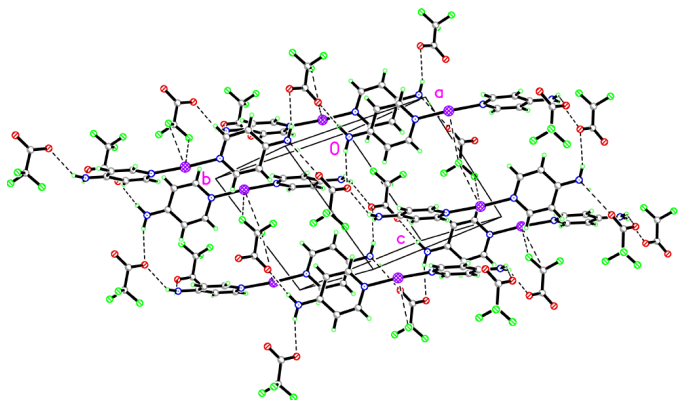


Figure 2
The crystal packing of (I), showing the hydrogen bonds as dashed lines.

Experimental

Silver trifluoromethanesulfonate and 4-aminopyridine were available commercially and were used without further purification. $\text{CF}_3\text{SO}_3\text{Ag}$ (1.0 mmol, 257 mg) and 4-aminopyridine (1 mmol, 94 mg) were dissolved in an aqueous solution of ammonia (10 ml). The mixture was stirred for ca 10 min to obtain a clear solution. After allowing the resulting solution to stand in air for 2 d, large colorless crystals were formed on slow evaporation of the solvent. The crystals were isolated, washed with water three times, and dried in a vacuum desiccator using CaCl_2 (yield: 69%).

Crystal data

$[\text{Ag}(\text{C}_5\text{H}_6\text{N}_2)_2](\text{CF}_3\text{O}_2)$
 $M_r = 409.13$
 Triclinic, $P\bar{1}$
 $a = 9.492$ (7) Å
 $b = 9.803$ (7) Å
 $c = 10.002$ (7) Å
 $\alpha = 115.206$ (9)°
 $\beta = 108.078$ (8)°
 $\gamma = 92.793$ (10)°
 $V = 782.6$ (10) Å³

$Z = 2$
 $D_x = 1.736$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 2092 reflections
 $\theta = 2.3$ – 25.1 °
 $\mu = 1.33$ mm⁻¹
 $T = 298$ (2) K
 Prism, colorless
 $0.32 \times 0.27 \times 0.15$ mm

Data collection

Siemens SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.676$, $T_{\max} = 0.826$
 3984 measured reflections

2720 independent reflections
 2200 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.015$
 $\theta_{\text{max}} = 25.0$ °
 $h = -11 \rightarrow 11$
 $k = -7 \rightarrow 11$
 $l = -11 \rightarrow 11$

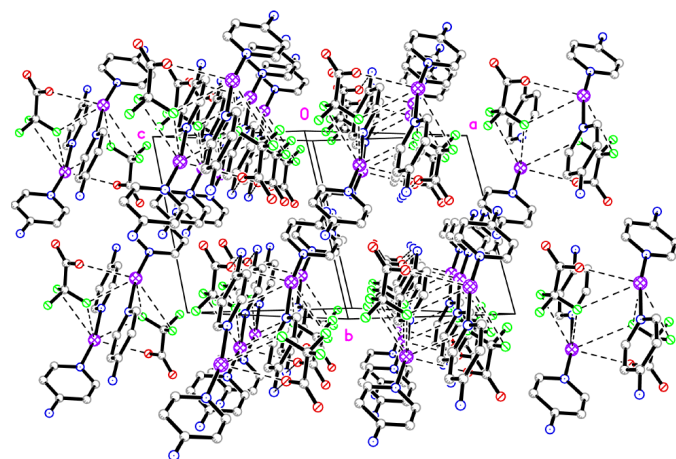


Figure 3
The crystal packing of (I), showing the $\text{Ag} \cdots \text{O}$ and $\text{Ag} \cdots \text{F}$ interactions as dashed lines.

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.133$
 $S = 1.06$
 2720 reflections
 199 parameters
 H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0848P)^2 + 0.1491P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.94 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.67 \text{ e \AA}^{-3}$

Table 1

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{N2}-\text{H2A} \cdots \text{O2}^i$	0.86	2.25	2.969 (6)	142
$\text{N2}-\text{H2B} \cdots \text{O2}^{ii}$	0.86	2.37	3.126 (7)	146
$\text{N4}-\text{H4A} \cdots \text{O1}^{iii}$	0.86	2.05	2.887 (6)	164
$\text{N4}-\text{H4B} \cdots \text{O2}^{iv}$	0.86	2.28	3.108 (6)	162

Symmetry codes: (i) $2-x, 1-y, 1-z$; (ii) $1+x, y, z$; (iii) $1-x, 2-y, -z$; (iv) $x, 1+y, z$.

All the H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with $\text{N}-\text{H}$ distances of 0.86 Å, $\text{C}-\text{H}$ distances of 0.97 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}$ of the parent atom. The F-atom displacement parameters were quite large, presumably because these atoms are slightly disordered; however, no attempt was made to model this.

Data collection: SMART (Siemens, 1996); cell refinement: SMART; data reduction: SAINT (Siemens, 1996) and SHELXTL (Sheldrick, 1997); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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