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Bis(2-aminopyridine-κN^1)silver(I) perchlorate

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.011 Å
Disorder in solvent or counterion
R factor = 0.055
wR factor = 0.174
Data-to-parameter ratio = 11.1

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

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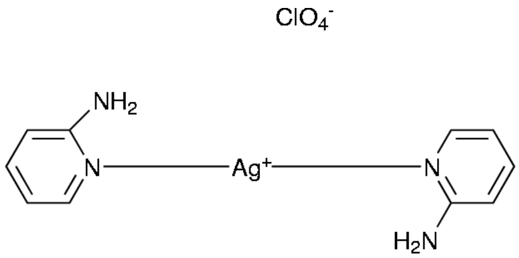
The title compound, $[Ag(C_5H_6N_2)_2]ClO_4$, is a mononuclear silver(I) complex. The Ag atom, lying on a twofold rotation axis, is bicoordinated in a distorted linear configuration by two N atoms from two symmetry-related 2-aminopyridine ligands. The perchlorate ion is disordered across a crystallographic mirror plane. The cations and anions are arranged as alternating layers along the *a* axis and are interlinked by weak N—H···O interactions in addition to the electrostatic interactions.

Comment

Metal complexes of pyridine and its derivatives constitute a hot topic in coordination chemistry. Indeed, numerous monomers, dimers and polymers have been prepared and structurally characterized. Recently, we reported a few transition metal complexes with pyridine derivatives (Yang *et al.*, 2004; Zhu *et al.*, 2001; Zhu, Qu *et al.*, 2003; Zhu, Shao *et al.*, 2003; Zhu, Usman *et al.*, 2003; Zhu, Zeng *et al.*, 2003; Zhu, Zhang *et al.*, 2003). In continuation of our investigation, some more silver(I) complexes with pyridine derivatives were prepared. We report here the structure of the silver(I) perchlorate complex with 2-aminopyridine, (I).



(I)

The title compound is a discrete mononuclear silver(I) complex which consists of a complex cation and a perchlorate anion (Fig. 1). In the cation, the Ag^I atom, which lies on a twofold rotation axis, is two-coordinated by two N atoms from two 2-aminopyridine ligands. The Ag^I atom adopts a slightly distorted linear coordination geometry, with an N—Ag—N angle of 178.2 (2)°. The coordination mode of silver(I) in (I) is different from that in a silver(I) benzoate complex with 2-aminopyridine (Zhu, Usman *et al.*, 2003). In the latter complex, the Ag^I atom is three-coordinated by two pyridine ligands and one carboxylate anion. The difference is mainly due to the poor coordination ability of the perchlorate anion in (I). The Ag—N bond length of 2.126 (5) Å is much shorter than those [2.205 (4) and 2.230 (3) Å] in the silver(I) benzoate

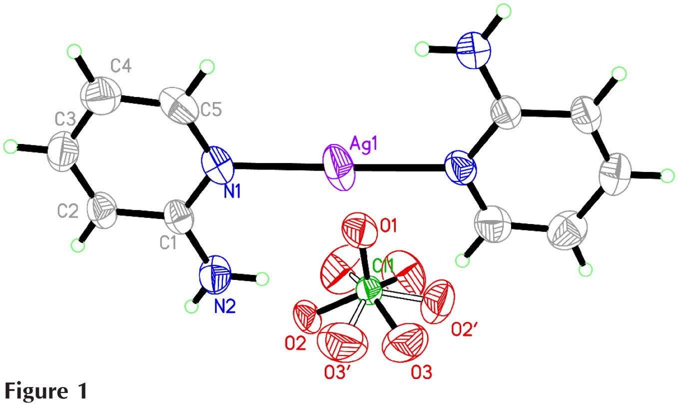


Figure 1
The structure of the title compound (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme. Both disorder components are shown.

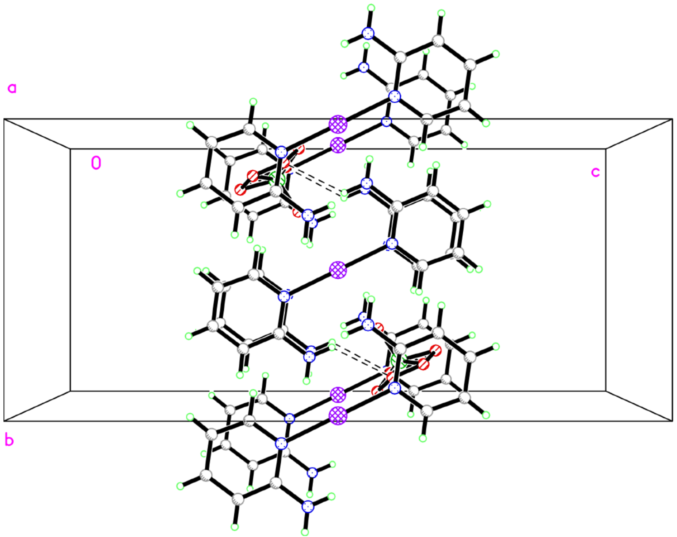


Figure 2
The crystal packing of (I), showing N—H···O hydrogen-bonding interactions as dashed lines.

complex with 2-aminopyridine (Zhu, Usman *et al.*, 2003), but is comparable to those in other silver(I) complexes with pyridine derivatives.

The perchlorate anion lies across a crystallographic mirror plane, and is disordered.

In the crystal structure, the cations and anions are arranged as alternating layers along the *a* axis, with weak N—H···O contacts between the adjacent layers (Fig. 2).

Experimental

Ag_2O (0.5 mmol, 116 mg) was dissolved in a 30% aqueous ammonia solution (10 ml) and stirred for *ca* 10 min to give a solution with a small amount of grey powder suspension. An acetonitrile solution (5 ml) of $NaClO_4$ (0.6 mmol, 74 mg) was added slowly to the above solution and filtered to remove some solid. After allowing the resulting solution to stand in air for 4 d, large colourless prisms were formed. The crystals were isolated, washed with water and acetonitrile in turn, and dried in a vacuum desiccator using $CaCl_2$ (yield 33%). Elemental analysis found: C 30.11, H 3.08, N 14.01%; calculated for $C_{10}H_{12}AgClN_4O_4$: C 30.36, H 3.06, N 14.16%.

Crystal data

$[Ag(C_5H_6N_2)_2]ClO_4$
$M_r = 395.56$
Orthorhombic, *Cmca*
$a = 12.720\ (4)$ Å
$b = 9.810\ (3)$ Å
$c = 21.710\ (7)$ Å
$V = 2709.0\ (15)$ Å^3
$Z = 8$
$D_x = 1.940$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 1237 reflections
$\theta = 2.5$–25°
$\mu = 1.70$ mm^{-1}
$T = 298\ (2)$ K
Prism, colourless
0.22 × 0.15 × 0.13 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.706$, $T_{max} = 0.809$
5599 measured reflections

1241 independent reflections
964 reflections with $I > 2\sigma(I)$
$R_{int} = 0.053$
$\theta_{max} = 25.0°$
$h = -9 \rightarrow 15$
$k = -11 \rightarrow 11$
$l = -25 \rightarrow 25$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.174$
$S = 1.10$
1241 reflections
112 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1098P)^2 + 2.44P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.13$ e Å^{-3}
$\Delta\rho_{min} = -0.75$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0065 (8)

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O3′	0.86	2.54	3.128 (16)	126
N2—H2A···O1	0.86	2.65	3.259 (8)	128
N2—H2B···O3′^{i}	0.86	2.31	3.130 (17)	159
N2—H2B···O2^{ii}	0.86	2.43	3.041 (9)	129

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

The perchlorate anion lies across a crystallographic mirror plane, with atoms Cl1, O1 and O2 lying on the mirror plane, and atom O3 located at a general position. Atoms O2 and O3 were found to be disordered over two positions each (O2/O2′ and O3/O3′) with equal site-occupancy factors. All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with N—H and C—H distances of 0.86 and 0.93 Å, respectively, and with U_{iso}(H) values set equal to $1.2U_{eq}$(C/N). The highest electron-density peak is located at (0.1373, 0, $\frac{1}{2}$).

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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