Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis[2-(benzylamino)pyridine-κN]silver(I) perchlorate

Chong-Qing Wan, Qing-Shan Li,* Hai-Bin Song, Feng-Bo Xu and Zheng-Zhi Zhang

State Key Laboratory and Institute of Elemento-Organic Chemistry, Nankai University, Tianjin, Weijin Road No. 94, Tianjin, People's Republic of China

Correspondence e-mail: qli@nankai.edu.cn

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.017 Å
Disorder in solvent or counterion
R factor = 0.067
wR factor = 0.200
Data-to-parameter ratio = 14.2

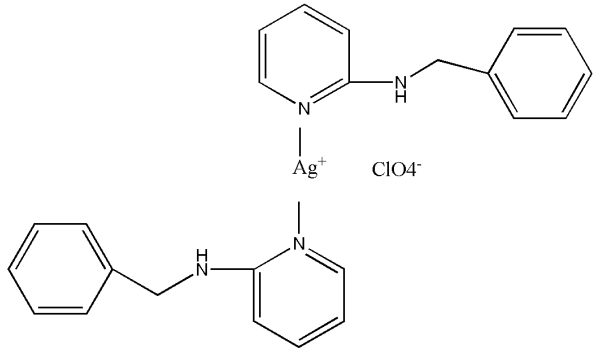
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 25 October 2004
Accepted 23 November 2004
Online 30 November 2004

The title compound, $[Ag(C_{12}H_{12}N_2)_2]ClO_4$, is a mononuclear silver(I) complex. The Ag^I atom is coordinated by two pyridine N atoms from two benzylaminopyridine ligands. In the crystal structure, the molecules are linked by N—H···O intermolecular hydrogen bonds, forming chains along the c axis.

Comment

Pyridine and its derivatives are versatile precursors of multidentate ligands. The synthesis, crystal structures and properties of these compounds have been reported (Faar *et al.*, 1983; Zhang & Cheng, 1996). A major interest arises from the rigid *ortho* bridge structure, forming dinuclear metallic compounds (Barder *et al.*, 1983), and the various amino derivatives, affording a series of multidentate ligands (Cui *et al.*, 2001; Li *et al.*, 2003; Kamar *et al.*, 2002). We report here the crystal structure of the silver(I) complex, (I), with the 2-benzylaminopyridine ligand.



(I)

The structure of the cation of (I), a mononuclear silver(I) complex, is shown in Fig. 1. The Ag^I atom is in a linear coordination environment and is coordinated by two pyridine N atoms from two benzylaminopyridine ligands. The average

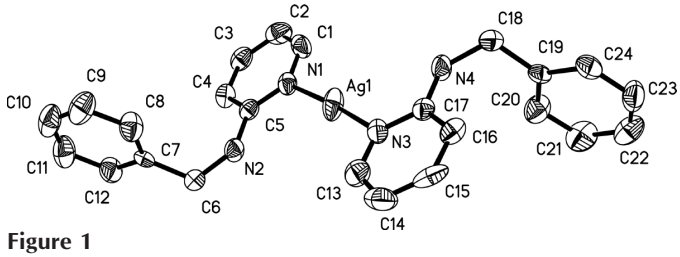


Figure 1
The structure of the title compound, showing 40% probability displacement ellipsoids and the atom-numbering scheme. The perchlorate anion and H atoms have been omitted for clarity.

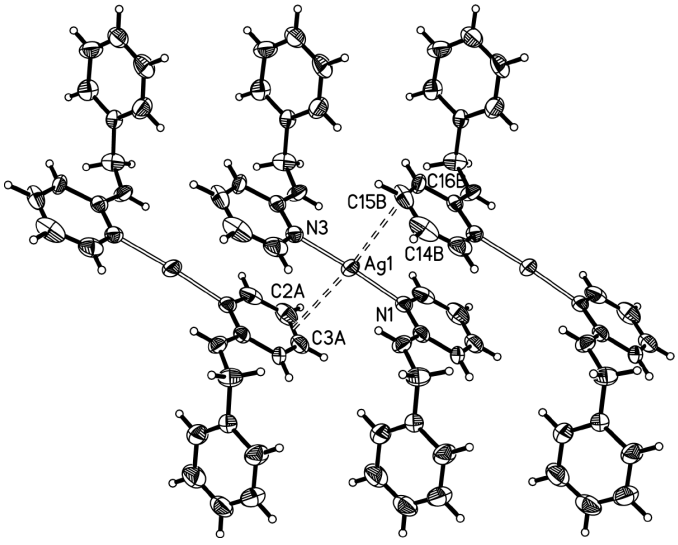


Figure 2
Ring–metal interactions in the title compound. [Symmetry codes: (*A*) *x*, 1 + *y*, *z*; (*B*) *x*, *y* − 1, *z*].

Ag—N bond length is 2.153 (8) Å and the N—Ag—N angle is 175.1 (4)°. There is η^2-bonding between Ag^+ and C2*A*/C3*A* and η^3-bonding between Ag^+ and C14*B*/C15*B*/C16*B*, as shown in Fig. 2 [symmetry codes: (*A*) *x*, 1 + *y*, *z*; (*B*) *x*, *y* − 1, *z*]. The Ag1···*Cg*1(*x*, 1 + *y*, *z*) and Ag1···*Cg*2(*x*, *y* − 1, *z*) distances are 3.441 and 3.408 Å, respectively, where *Cg*1 and *Cg*2 are the centroids of the N1-pyridine and N3-pyridine rings.

The two pyridine rings in the cation are coplanar [dihedral angle 3.0 (5)°]. The N1-pyridine ring and the N3-pyridine ring of the molecule translated by one unit along the *b* axis are stacked at a perpendicular distance of 3.385 Å [the centroid-centroid distance is 3.939 (6) Å and the dihedral angle between the planes is 3.0 (5)°], showing weak π–π stacking interactions. In the crystal structure, the molecules are linked by N—H···O intermolecular hydrogen bonds (Table 2), forming chains along the *c* axis.

Experimental

2-(Benzylamino)pyridine (*L*1) was prepared according to the literature method of Czuba & Kowalski (1980). 2-Aminopyridine (94 g, 1.0 mmol), benzyl alcohol (150 g, 1.39 mmol) and KOH (9 g, 0.16 mmol) were mixed and boiled. The water formed was slowly evaporated at 455 K. The reaction temperature was increased to 523 K and held for several minutes. The reaction mixture was then cooled to 373 K and poured into ice water (250 ml) with strong stirring. The solid formed was filtered and washed with water to give 180 g of *L*1 as white crystals (yield 98%; m.p. 368–369 K). *L*1 (0.184 g, 1.0 mmol) and $[Ag(CH_3CN)_4]ClO_4$ (0.186 g, 0.5 mmol) in CH_2Cl_2 (30 ml) were then stirred at room temperature for 0.5 h. The filtered solution was concentrated to about 5 ml and diethyl ether was allowed to diffuse into the solution, yielding 0.245 g of colourless crystals of the title compound (yield 85%). Analysis found: C 49.84, H 4.10, N 9.68%; calculated for $C_{24}H_{24}AgClN_4O_4$: C 50.06, H 4.20, N 9.73%. ^{1}H NMR: δ 7.99–8.02 (*d*, 2H), 7.40–7.66 (*t*, 2H), 7.22–7.26 (*m*, 10H), 6.56–6.59 (*t*, 2H), 6.43–6.47 (*d*, 2H), 4.42–4.45 (*d*, 4H).

Crystal data

$[Ag(C_{12}H_{12}N_2)_2]ClO_4$	D_x = 1.558 Mg m^{-3}
M_r = 575.79	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 784 reflections
a = 23.594 (7) Å	θ = 2.3–22.5°
b = 5.7205 (16) Å	μ = 0.97 mm^{-1}
c = 18.223 (5) Å	T = 293 (2) K
β = 93.308 (6)°	Thick plate, colourless
V = 2455.4 (12) Å^3	0.25 × 0.15 × 0.05 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	4327 independent reflections
φ and ω scans	1674 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	R_{int} = 0.096
T_{min} = 0.763, T_{max} = 0.953	θ_{max} = 25.0°
9722 measured reflections	$h = -28 \rightarrow 27$
	$k = -6 \rightarrow 6$
	$l = -11 \rightarrow 21$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.067	$w = 1/[\sigma^2(F_o^2) + (0.0844P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.200	$(\Delta/\sigma)_{max} < 0.001$
S = 0.96	$\Delta\rho_{max}$ = 0.73 e Å^{-3}
4327 reflections	$\Delta\rho_{min}$ = −0.69 e Å^{-3}
304 parameters	

Table 1
Selected geometric parameters (Å, °).

Ag1—N3	2.150 (8)	N1—C1	1.354 (14)
Ag1—N1	2.155 (8)	N2—C5	1.373 (12)
N1—C5	1.349 (11)	N2—C6	1.429 (11)
N3—Ag1—N1	175.1 (4)	C1—N1—Ag1	118.7 (8)
C5—N1—C1	116.6 (9)	C5—N2—C6	122.9 (9)
C5—N1—Ag1	124.5 (8)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2*A*···O3^{i}	0.86	2.07	2.770 (13)	138
N4—H4*A*···O4^{ii}	0.86	2.35	3.098 (15)	146

Symmetry codes: (i) *x*, *y*, *z* − 1; (ii) $x, \frac{1}{2} - y, z - \frac{1}{2}$.

The perchlorate counter-ion was found to be disordered over two orientations, with occupancies of 0.572 (13) and 0.478 (13); the disordered O atoms were refined isotropically. H atoms were placed in calculated positions, with C—H = 0.93 or 0.97 Å, and included in the final cycles of refinement using a riding model, with U_{iso}(H) = 1.2U_{eq}(parent atom). The low ratio of observed to unique reflections (39%) may be due to the poor diffraction quality of the crystal.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

This work was supported by the National Natural Science Foundation of China (No. 20102003).

References

Barder, T. J., Tetrik, S. M., Walton, R. A. Cotton, F. A. & Powell, G. L. (1983). *J. Am. Chem. Soc.* **105**, 4090–4091.

Bruker (1997). *SHELXTL.* Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.

Bruker (1998). *SMART* (Version 5.00) and *SAINT* (Version 5.00). Bruker AXS Inc., Madison, Wisconsin, USA.

Cui, D. J., Li, Q. S., Xu, F. B., Leng, X. B. & Zhang, Z. Z. (2001). *Organometallics*, **20**, 4126–4128.

Czuba, W. & Kowalski, P. (1980). *Pol. J. Chem.* **54**, 853–855.

Faar, J. P., Olmstead, M. M., Wood, F. E. & Balch, A. L. (1983). *J. Am. Chem. Soc.* **105**, 792–798.

Kamar, K. K., Saha, A., Eiras, A. C., Hung, C. H. & Goswami, S. (2002). *Inorg. Chem.* **41**, 4531–4538.

Li, Q. S., Xu, F. B., Cui, D. J., Yu, K., Zeng, X. S., Leng, X. B., Song, H. B. & Zhang, Z. Z. (2003). *J. Chem. Soc. Dalton Trans.* pp. 1551–1557.

Sheldrick, G. M. (1996). *SADABS.* University of Göttingen, Germany.

Zhang, Z. Z. & Cheng, H. (1996). *Coord. Chem. Rev.* **147**, 1–39.