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catena-Poly[[silver(I)- μ -N-(3-pyridyl-methyl)pyridine-4-carboxamide] nitrate monohydrate]

Yu-Tao Ma and Qi-Hua Zhao*

School of Chemical Science and Technology, Key Laboratory of Medicinal Chemistry for Natural Resources, Ministry of Education, Yunnan University, Kunming 650091, People's Republic of China

Correspondence e-mail: qhzhao@ynu.edu.cn

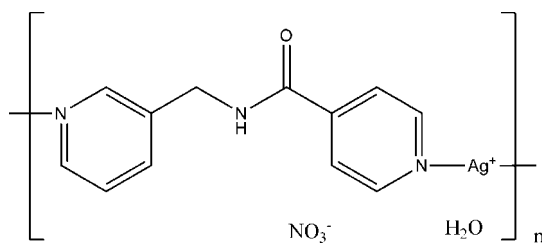
Received 13 November 2007; accepted 30 November 2007

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.042; wR factor = 0.101; data-to-parameter ratio = 16.2.

In the title compound, $\{[\text{Ag}(\text{C}_{12}\text{H}_{11}\text{N}_3\text{O})]\text{NO}_3 \cdot \text{H}_2\text{O}\}_n$, the Ag atom is coordinated by two N atoms from the heterocyclic ligand, giving a linear polycationic chain. Two long $\text{Ag} \cdots \text{O}_{\text{nitrate}}$ interactions [2.667 (3) and 2.840 (3) Å] result in a three-dimensional network. The water molecule consolidates the network structure by forming hydrogen bonds, one to the polycationic chain and one to the nitrate anion.

Related literature

For related literature, see: Cordes & Hanton (2007); Kumar *et al.* (2006); Tong *et al.* (2002).



Experimental

Crystal data

 $[\text{Ag}(\text{C}_{12}\text{H}_{11}\text{N}_3\text{O})]\text{NO}_3 \cdot \text{H}_2\text{O}$ $M_r = 401.13$ Monoclinic, $P2_1/c$ $a = 12.177$ (2) Å $b = 13.022$ (3) Å $c = 8.9109$ (18) Å $\beta = 94.21$ (3)° $V = 1409.2$ (5) Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 1.46$ mm⁻¹ $T = 293$ (2) K $0.6 \times 0.4 \times 0.2$ mm

Data collection

Rigaku Mercury CCD

diffractometer

Absorption correction: multi-scan

(CrystalClear; Rigaku/MS, 2003) $T_{\text{min}} = 0.503$, $T_{\text{max}} = 0.742$

14304 measured reflections

3230 independent reflections

2399 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.065$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.042$ $wR(F^2) = 0.101$ $S = 1.06$

3230 reflections

199 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.35$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.43$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{N4}-\text{H4A} \cdots \text{O1W}^i$	0.86	2.04	2.837 (4)	154
$\text{O1W}-\text{H1WA} \cdots \text{O1}$	0.85	1.94	2.790 (4)	174
$\text{O1W}-\text{H1WB} \cdots \text{O3}^{ii}$	0.85	2.04	2.886 (4)	171

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

Data collection: *CrystalClear* (Rigaku/MS, 2003); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1999); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2069).

References

- Cordes, D. B. & Hanton, L. R. (2007). *Inorg. Chem.* **46**, 1634–1644.
 Kumar, D. K., Das, A. & Dastidar, P. (2006). *Cryst. Growth Des.* **6**, 1903–1909.
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supplementary materials

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***catena*-Poly[[silver(I)- μ -*N*-(3-pyridylmethyl)pyridine-4-carboxamide] nitrate monohydrate]**

Y.-T. Ma and Q.-H. Zhao

Comment

The reactions of silver(I) salts with flexible pyridyl type ligands have received considerable attention (Cordes *et al.*, 2007; Kumar *et al.*, 2006; Tong *et al.*, 2002). Here, we report a new silver(I) complex (Fig. 1), which was prepared by the reaction of *N*-(3-pyridinylmethyl)-4-pyridine-carboxamide acting as a bidentate bridge ligand with AgNO₃. In the cation, the Ag(I) atom is in a linear coordination environment and the Ag1—N1A and Ag1—N3 bond length are 2.152 (3) and 2.157 (3) Å, respectively. The N3—Ag1—N1ⁱ (*i* = -1 + *x*, 0.5 - *y*, 1/2 + *z*) bond angle is 172.55 (15) °, indicating that the N—Ag—N skeleton that gives rise to a chain structure is distorted by the presence of two Ag···O_{nitrate} interactions. If these are regarded as formal bonds, the compound may be described as a three dimensional network structure (Fig. 2).

Experimental

An aqueous solution (5 ml) of silver nitrate (1.0 mmol) was layered carefully over a methanol (5 ml) solution of *N*-(4-pyridylmethyl)-4-pyridinecarboxamide (1.0 mmol) in a tube, which was covered and kept away from light. Colorless crystals were obtained after two weeks. These were washed with methanol and collected in 50% yield. CHN elemental analysis: found C 35.86, H 3.55, N 13.79%; calc. for C₁₂H₁₃AgN₄O₅: C 35.93, H 3.27, N 13.96%.

Refinement

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances of 0.93–0.97 Å, N—H distances of 0.86 Å and OW1—H distances of 0.85 Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C, N or O})$.

Figures

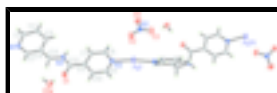


Fig. 1. The structure of the title complex, showing 30% probability displacement ellipsoids and the atom-numbering scheme. Symmetry-generated atoms in the plot are related by (-1 + *x*, 0.5 - *y*, 1/2 + *z*).

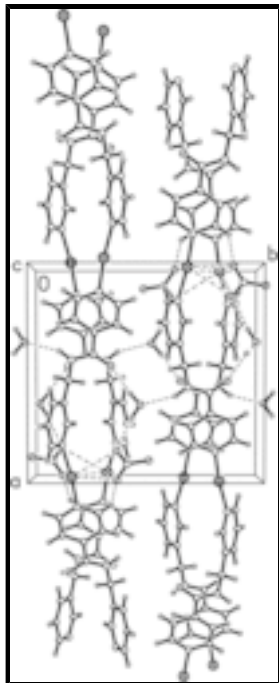


Fig. 2. Crystal packing viewed down the *c* axis.

catena-Poly[[silver(I)-μ-N-(3-pyridylmethyl)pyridine-4-carboxamide] nitrate monohydrate]

Crystal data

[Ag(C₁₂H₁₁N₃O)]NO₃·H₂O

M_r = 401.13

Monoclinic, *P*2₁/*c*

Hall symbol: -*P* 2ybc

a = 12.177 (2) Å

b = 13.022 (3) Å

c = 8.9109 (18) Å

β = 94.21 (3)°

V = 1409.2 (5) Å³

Z = 4

*F*₀₀₀ = 800

D_x = 1.891 Mg m⁻³

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 5866 reflections

θ = 3.2–27.5°

μ = 1.46 mm⁻¹

T = 293 (2) K

Block, colorless

0.6 × 0.4 × 0.2 mm

Data collection

Rigaku Mercury CCD
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

T = 293(2) K

ω scans

Absorption correction: multi-scan
(CrystalClear; Rigaku/MSO, 2003)

T_{min} = 0.503, *T_{max}* = 0.742

3230 independent reflections

2399 reflections with *I* > 2σ(*I*)

R_{int} = 0.065

θ_{max} = 27.5°

θ_{min} = 3.1°

h = -15→15

k = -16→16

14304 measured reflections

$l = -11 \rightarrow 11$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.042$	H-atom parameters constrained
$wR(F^2) = 0.101$	$w = 1/[\sigma^2(F_o^2) + (0.0433P)^2 + 0.2355P]$
$S = 1.06$	where $P = (F_o^2 + 2F_c^2)/3$
3230 reflections	$(\Delta/\sigma)_{\max} < 0.001$
199 parameters	$\Delta\rho_{\max} = 0.35 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\min} = -0.43 \text{ e } \text{\AA}^{-3}$
	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Ag1	-0.01568 (2)	0.32484 (2)	0.11987 (4)	0.04917 (14)
N3	0.1306 (2)	0.3030 (2)	0.0011 (3)	0.0377 (7)
C9	0.1867 (3)	0.3832 (3)	-0.0444 (4)	0.0453 (9)
H9A	0.1644	0.4486	-0.0176	0.054*
C7	0.2524 (3)	0.1954 (3)	-0.1219 (4)	0.0426 (9)
H7A	0.2731	0.1291	-0.1467	0.051*
C8	0.1637 (3)	0.2107 (3)	-0.0387 (4)	0.0448 (9)
H8A	0.1249	0.1537	-0.0087	0.054*
C11	0.3109 (3)	0.2785 (3)	-0.1690 (4)	0.0321 (7)
C10	0.2755 (3)	0.3746 (3)	-0.1283 (4)	0.0418 (9)
H10A	0.3120	0.4331	-0.1580	0.050*
C12	0.4107 (3)	0.2716 (3)	-0.2571 (4)	0.0348 (8)
N4	0.4370 (2)	0.1789 (2)	-0.3061 (3)	0.0396 (7)
H4A	0.3962	0.1273	-0.2872	0.047*
C13	0.5327 (3)	0.1630 (3)	-0.3904 (4)	0.0444 (9)
H13A	0.5431	0.2231	-0.4520	0.053*
H13B	0.5188	0.1051	-0.4576	0.053*

supplementary materials

C4	0.6375 (3)	0.1431 (3)	-0.2938 (4)	0.0335 (8)
C5	0.7368 (3)	0.1578 (3)	-0.3533 (4)	0.0358 (8)
H5A	0.7365	0.1849	-0.4499	0.043*
N1	0.8337 (2)	0.1358 (2)	-0.2817 (3)	0.0387 (7)
C1	0.8340 (3)	0.0972 (3)	-0.1428 (4)	0.0472 (9)
H1B	0.9007	0.0798	-0.0917	0.057*
C2	0.7385 (3)	0.0825 (3)	-0.0737 (4)	0.0485 (10)
H2A	0.7410	0.0577	0.0243	0.058*
C3	0.6391 (3)	0.1044 (3)	-0.1492 (4)	0.0413 (9)
H3A	0.5738	0.0933	-0.1038	0.050*
O1	0.4653 (2)	0.3480 (2)	-0.2803 (3)	0.0550 (8)
O2	-0.0055 (2)	0.1263 (3)	0.1929 (4)	0.0669 (8)
N2	-0.0802 (3)	0.0813 (3)	0.2490 (3)	0.0427 (7)
O3	-0.1439 (3)	0.1264 (3)	0.3279 (4)	0.0735 (9)
O1W	0.6452 (2)	0.4755 (2)	-0.2057 (4)	0.0624 (8)
H1WA	0.5932	0.4332	-0.2267	0.075*
H1WB	0.7033	0.4393	-0.1928	0.075*
O4	-0.0959 (3)	-0.0117 (2)	0.2228 (4)	0.0702 (9)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ag1	0.03123 (18)	0.0563 (2)	0.0621 (2)	0.00455 (13)	0.01775 (14)	-0.00438 (15)
N3	0.0300 (16)	0.0400 (18)	0.0435 (18)	0.0011 (13)	0.0066 (13)	-0.0052 (13)
C9	0.042 (2)	0.034 (2)	0.061 (3)	0.0054 (17)	0.0109 (19)	-0.0029 (18)
C7	0.041 (2)	0.033 (2)	0.057 (2)	-0.0023 (16)	0.0209 (18)	-0.0077 (16)
C8	0.039 (2)	0.043 (2)	0.055 (2)	-0.0071 (17)	0.0197 (18)	-0.0027 (18)
C11	0.0277 (17)	0.0366 (19)	0.0319 (18)	0.0012 (15)	0.0013 (14)	0.0008 (14)
C10	0.039 (2)	0.035 (2)	0.053 (2)	-0.0021 (17)	0.0105 (17)	0.0004 (17)
C12	0.0267 (18)	0.042 (2)	0.0363 (19)	-0.0045 (16)	0.0048 (14)	-0.0007 (16)
N4	0.0270 (15)	0.0483 (19)	0.0448 (18)	-0.0020 (13)	0.0127 (13)	-0.0045 (14)
C13	0.032 (2)	0.062 (3)	0.041 (2)	0.0028 (18)	0.0105 (16)	-0.0027 (17)
C4	0.0324 (19)	0.0360 (18)	0.0324 (18)	0.0020 (15)	0.0060 (15)	-0.0046 (14)
C5	0.0305 (18)	0.041 (2)	0.0373 (19)	0.0019 (15)	0.0103 (15)	0.0014 (15)
N1	0.0285 (16)	0.0443 (17)	0.0447 (18)	-0.0018 (14)	0.0108 (13)	0.0025 (14)
C1	0.036 (2)	0.056 (3)	0.050 (2)	0.0012 (18)	0.0028 (17)	0.0017 (19)
C2	0.045 (2)	0.066 (3)	0.036 (2)	0.002 (2)	0.0075 (17)	0.0058 (18)
C3	0.037 (2)	0.048 (2)	0.041 (2)	-0.0032 (17)	0.0150 (16)	-0.0004 (17)
O1	0.0404 (16)	0.0537 (17)	0.073 (2)	-0.0142 (13)	0.0212 (14)	-0.0028 (14)
O2	0.055 (2)	0.068 (2)	0.080 (2)	-0.0126 (16)	0.0235 (16)	0.0100 (18)
N2	0.0371 (18)	0.050 (2)	0.0413 (18)	-0.0045 (15)	0.0028 (14)	0.0039 (15)
O3	0.066 (2)	0.076 (2)	0.082 (2)	-0.0028 (18)	0.0310 (18)	-0.0178 (18)
O1W	0.0471 (17)	0.0461 (17)	0.094 (2)	-0.0057 (14)	0.0067 (15)	-0.0046 (15)
O4	0.068 (2)	0.0443 (18)	0.098 (3)	-0.0020 (16)	0.0053 (18)	0.0006 (16)

Geometric parameters (\AA , $^\circ$)

Ag1—N1 ⁱ	2.152 (3)	C13—H13A	0.9700
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Ag1—N3	2.157 (3)	C13—H13B	0.9700
N3—C8	1.324 (5)	C4—C5	1.369 (5)
N3—C9	1.328 (5)	C4—C3	1.382 (5)
C9—C10	1.363 (5)	C5—N1	1.331 (4)
C9—H9A	0.9300	C5—H5A	0.9300
C7—C8	1.369 (5)	N1—C1	1.336 (5)
C7—C11	1.378 (5)	N1—Ag1 ⁱⁱ	2.152 (3)
C7—H7A	0.9300	C1—C2	1.368 (5)
C8—H8A	0.9300	C1—H1B	0.9300
C11—C10	1.381 (5)	C2—C3	1.370 (5)
C11—C12	1.497 (5)	C2—H2A	0.9300
C10—H10A	0.9300	C3—H3A	0.9300
C12—O1	1.222 (4)	O2—N2	1.220 (4)
C12—N4	1.331 (4)	N2—O3	1.234 (4)
N4—C13	1.447 (5)	N2—O4	1.245 (4)
N4—H4A	0.8600	O1W—H1WA	0.8499
C13—C4	1.508 (5)	O1W—H1WB	0.8500
N1 ⁱ —Ag1—N3	172.19 (11)	C4—C13—H13A	108.7
C8—N3—C9	117.3 (3)	N4—C13—H13B	108.7
C8—N3—Ag1	122.0 (2)	C4—C13—H13B	108.7
C9—N3—Ag1	120.5 (2)	H13A—C13—H13B	107.6
N3—C9—C10	123.3 (3)	C5—C4—C3	117.3 (3)
N3—C9—H9A	118.3	C5—C4—C13	119.3 (3)
C10—C9—H9A	118.3	C3—C4—C13	123.3 (3)
C8—C7—C11	119.8 (3)	N1—C5—C4	124.2 (3)
C8—C7—H7A	120.1	N1—C5—H5A	117.9
C11—C7—H7A	120.1	C4—C5—H5A	117.9
N3—C8—C7	123.0 (3)	C5—N1—C1	117.8 (3)
N3—C8—H8A	118.5	C5—N1—Ag1 ⁱⁱ	120.4 (2)
C7—C8—H8A	118.5	C1—N1—Ag1 ⁱⁱ	121.6 (2)
C7—C11—C10	117.0 (3)	N1—C1—C2	121.7 (4)
C7—C11—C12	124.8 (3)	N1—C1—H1B	119.2
C10—C11—C12	118.3 (3)	C2—C1—H1B	119.2
C9—C10—C11	119.6 (4)	C1—C2—C3	120.0 (4)
C9—C10—H10A	120.2	C1—C2—H2A	120.0
C11—C10—H10A	120.2	C3—C2—H2A	120.0
O1—C12—N4	122.4 (3)	C2—C3—C4	119.0 (3)
O1—C12—C11	120.8 (3)	C2—C3—H3A	120.5
N4—C12—C11	116.8 (3)	C4—C3—H3A	120.5
C12—N4—C13	121.5 (3)	O2—N2—O3	121.6 (4)
C12—N4—H4A	119.2	O2—N2—O4	119.9 (4)
C13—N4—H4A	119.2	O3—N2—O4	118.4 (3)
N4—C13—C4	114.1 (3)	H1WA—O1W—H1WB	105.6
N4—C13—H13A	108.7		
C8—N3—C9—C10	-0.2 (6)	C11—C12—N4—C13	-179.1 (3)
Ag1—N3—C9—C10	-175.6 (3)	C12—N4—C13—C4	87.7 (4)
C9—N3—C8—C7	0.6 (6)	N4—C13—C4—C5	-160.2 (3)
Ag1—N3—C8—C7	175.9 (3)	N4—C13—C4—C3	23.9 (5)

supplementary materials

C11—C7—C8—N3	-0.5 (7)	C3—C4—C5—N1	1.0 (5)
C8—C7—C11—C10	-0.1 (6)	C13—C4—C5—N1	-175.1 (3)
C8—C7—C11—C12	178.4 (4)	C4—C5—N1—C1	-0.2 (5)
N3—C9—C10—C11	-0.3 (6)	C4—C5—N1—Ag1 ⁱⁱ	-174.5 (3)
C7—C11—C10—C9	0.4 (6)	C5—N1—C1—C2	-1.5 (6)
C12—C11—C10—C9	-178.1 (3)	Ag1 ⁱⁱ —N1—C1—C2	172.8 (3)
C7—C11—C12—O1	-171.7 (4)	N1—C1—C2—C3	2.2 (6)
C10—C11—C12—O1	6.7 (5)	C1—C2—C3—C4	-1.3 (6)
C7—C11—C12—N4	7.3 (6)	C5—C4—C3—C2	-0.3 (5)
C10—C11—C12—N4	-174.2 (3)	C13—C4—C3—C2	175.7 (4)
O1—C12—N4—C13	0.0 (6)		

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

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N4—H4A \cdots O1W ⁱⁱⁱ	0.86	2.04	2.837 (4)	154
O1W—H1WA \cdots O1	0.85	1.94	2.790 (4)	174
O1W—H1WB \cdots O3 ⁱⁱ	0.85	2.04	2.886 (4)	171

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

Fig. 1

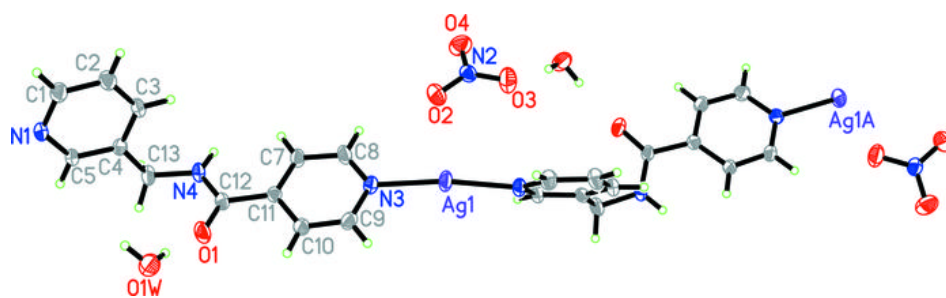


Fig. 2

