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# Bis[(*E*)-*N*-(pyridin-3-ylmethylidene)-hydroxylamine- $\kappa$ N<sup>1</sup>]*silver*(I) perchlorate

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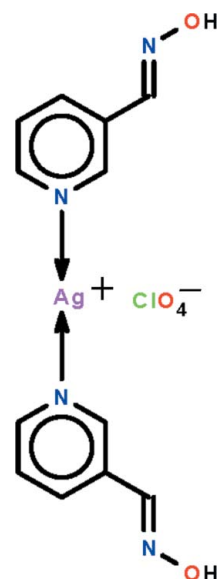
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Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.008$  Å; disorder in solvent or counterion;  $R$  factor = 0.048;  $wR$  factor = 0.147; data-to-parameter ratio = 13.9.

Each of the ions in the title salt,  $[\text{Ag}(\text{C}_6\text{H}_6\text{N}_2\text{O})_2]\text{ClO}_4$ , is completed by the application of crystallographic twofold symmetry. The  $\text{Ag}^{\text{I}}$  atom is coordinated by two pyridine N atoms in an almost linear fashion [ $\text{N}-\text{Ag}-\text{N} = 170.0$  ( $2^\circ$ )], with the T-shaped coordination geometry being completed by a weakly associated perchlorate-O atom. Supramolecular zigzag chains along  $[100]$  mediated by  $\text{O}-\text{H}\cdots\text{N}$  hydrogen bonds [as parts of  $R_2^2(6)$  loops] feature in the crystal packing. The perchlorate O atoms are disordered over two sets of sites in a statistical ratio.

## Related literature

For structural diversity in the structures of silver salts, see: Kundu *et al.* (2010). For related structures, see: Abu-Youssef *et al.* (2010); Xu *et al.* (2012).



## Experimental

### Crystal data

 $[\text{Ag}(\text{C}_6\text{H}_6\text{N}_2\text{O})_2]\text{ClO}_4$ 
 $M_r = 451.58$ 

 Monoclinic,  $C2/c$ 
 $a = 15.382$  (5) Å

 $b = 8.234$  (3) Å

 $c = 13.320$  (4) Å

 $\beta = 111.531$  ( $15^\circ$ )

 $V = 1569.3$  (8) Å<sup>3</sup>
 $Z = 4$ 

 Mo  $K\alpha$  radiation

 $\mu = 1.49$  mm<sup>-1</sup>
 $T = 293$  K

 $0.18 \times 0.16 \times 0.14$  mm

### Data collection

Rigaku R-Axis RAPID IP diffractometer

 Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)

 $T_{\text{min}} = 0.449$ ,  $T_{\text{max}} = 1.000$ 

7501 measured reflections

1795 independent reflections

 1176 reflections with  $I > 2\sigma(I)$ 
 $R_{\text{int}} = 0.052$ 

### Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.048$ 
 $wR(F^2) = 0.147$ 
 $S = 1.06$ 

1795 reflections

129 parameters

34 restraints

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.74$  e Å<sup>-3</sup>
 $\Delta\rho_{\text{min}} = -0.49$  e Å<sup>-3</sup>
**Table 1**

Selected bond lengths (Å).

Ag—N1	2.138 (5)	Ag—O2	2.843 (9)
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**Table 2**

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
O1—H1 $o$ ··N2 <sup>i</sup>	0.84	2.07	2.821 (7)	148

 Symmetry code: (i)  $-x + 1, -y + 1, -z + 1$ .

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalClear* (Rigaku/MSK, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008);

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program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6761).

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## supplementary materials

*Acta Cryst.* (2012). E68, m735–m736 [doi:10.1107/S1600536812019290]

**Bis[(*E*)-*N*-(pyridin-3-ylmethylidene)hydroxylamine- $\kappa$ N<sup>1</sup>]silver(I) perchlorate**

Jing Xu, Shan Gao, Seik Weng Ng and Edward R. T. Tiekink

**Comment**

The structural diversity of nitrogen adducts of silver salts are well known with distinct coordination geometries and supramolecular patterns being observed even when only a simple change, for example, in counter-ion, is made (Kundu *et al.*, 2010). The structural chemistry of pyridine-2-carboxaldoxime (LH) complexes with silver are relatively unexplored with only the Ag(LH)NO<sub>3</sub> salt (Abu-Youssef *et al.*, 2010) and the salt co-crystal [C<sub>12</sub>H<sub>11</sub>AgN<sub>4</sub>O<sub>2</sub>]<sup>+</sup>[ClO<sub>4</sub>]<sup>-</sup>[C<sub>12</sub>H<sub>12</sub>AgN<sub>4</sub>O<sub>2</sub>] (Xu *et al.*, 2012) having been reported previously. Herein, the crystal structure determination of the title salt, [Ag(C<sub>6</sub>H<sub>6</sub>N<sub>2</sub>O)<sub>2</sub>]ClO<sub>4</sub>, (I), is described.

In (I), Fig. 1, each of the ions has crystallographic twofold symmetry. The Ag<sup>+</sup> atom is coordinated by two N atoms in an almost linear array, Table 1. One of the perchlorate-O atoms is weakly associated with the silver atom so that the coordination geometry is T-shaped. The pyridine-2-carboxaldoxime ligand is planar as seen in the values of the C3—C4—C6—N2 and O1—N2—C6—C4 torsion angles of -2.3 (9) and -178.7 (5)°, respectively. The conformation about the imine bond [N2=C6 = 1.255 (7) Å] is *E*, and the nitrogen atoms are *anti*.

In the crystal packing, the oxime residues self-associate *via* O—H $\cdots$ N hydrogen bonds and six-membered { $\cdots$ HON}<sub>2</sub> synthons, Table 2. The result is the formation of a supramolecular chain with a zigzag topology along the *a* axis, Fig. 2.

**Experimental**

Silver perchlorate (1 mmol) and nicotinaldehyde oxime (1 mmol) was dissolved in methanol solution (10 ml). The solution was filtered and set aside, away from light, for the growth of crystals. Colourless crystals deposited after several days.

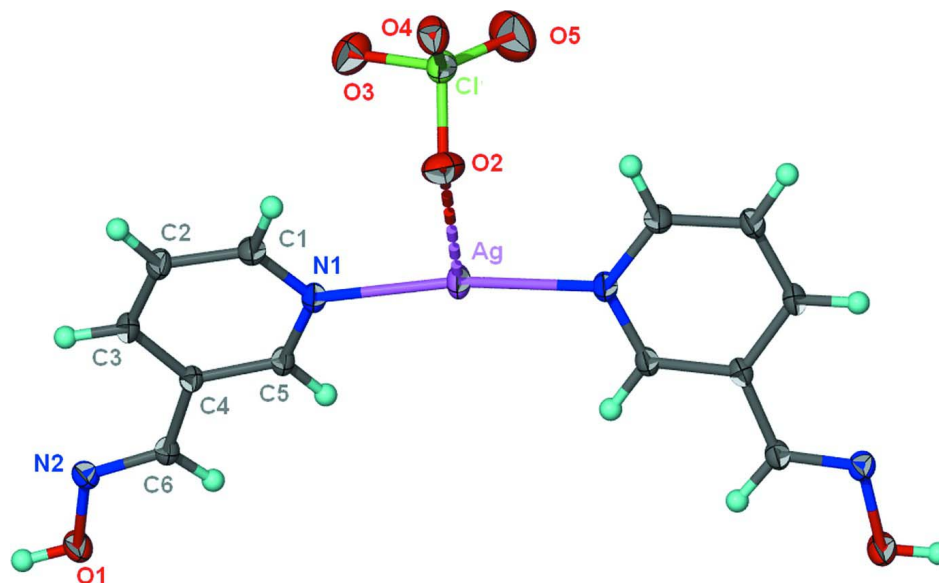
**Refinement**

Carbon- and oxygen-bound H-atoms were placed in calculated positions (C—H = 0.93 and O—H = 0.84 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2–1.5*U*<sub>eq</sub>(C, O).

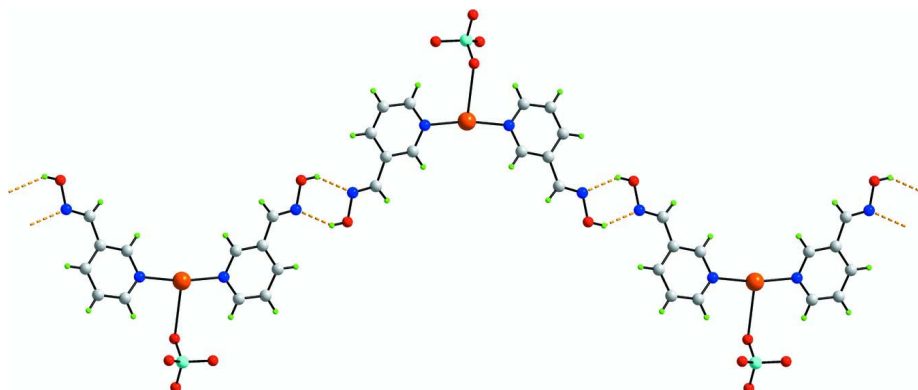
The oxygen atoms of the perchlorate ion are disordered about a twofold rotation axis, and four oxygen atoms were given 0.5 occupancies. The Cl—O distances were restrained to 1.41±0.01 Å and the O $\cdots$ O distances to 2.30±0.01 Å. The anisotropic displacement parameters of the oxygen atoms were restrained to be nearly isotropic.

**Computing details**

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO* (Rigaku, 1998); data reduction: *CrystalClear* (Rigaku/MSO, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *pubCIF* (Westrip, 2010).


**Figure 1**

The molecular structure of (I) showing displacement ellipsoids at the 20% probability level. Each ion has crystallographic twofold symmetry. Only one orientation for the disordered perchlorate anion is shown.


**Figure 2**

A view of the zigzag supramolecular chain along [100] in (I). The O—H...N hydrogen bonds are shown as orange dashed lines. Only one orientation for the disordered perchlorate anion is shown.

### Bis[(*E*)-*N*-(pyridin-3-ylmethylidene)hydroxylamine- $\kappa N^1$ ][silver(I) perchlorate

#### Crystal data

[Ag(C<sub>6</sub>H<sub>6</sub>N<sub>2</sub>O)<sub>2</sub>]ClO<sub>4</sub>

$M_r = 451.58$

Monoclinic, *C2/c*

Hall symbol: -C 2yc

$a = 15.382(5) \text{ \AA}$

$b = 8.234(3) \text{ \AA}$

$c = 13.320(4) \text{ \AA}$

$\beta = 111.531(15)^\circ$

$V = 1569.3(8) \text{ \AA}^3$

$Z = 4$

$F(000) = 896$

$D_x = 1.911 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 3613 reflections

$\theta = 3.0\text{--}27.5^\circ$

$\mu = 1.49 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Prism, colourless

$0.18 \times 0.16 \times 0.14 \text{ mm}$

Data collection

Rigaku R-AXIS RAPID IP diffractometer	7501 measured reflections
Radiation source: fine-focus sealed tube	1795 independent reflections
Graphite monochromator	1176 reflections with $I > 2\sigma(I)$
$\omega$ scan	$R_{\text{int}} = 0.052$
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	$\theta_{\text{max}} = 27.5^\circ$ , $\theta_{\text{min}} = 3.0^\circ$
$T_{\text{min}} = 0.449$ , $T_{\text{max}} = 1.000$	$h = -19 \rightarrow 19$
	$k = -10 \rightarrow 10$
	$l = -17 \rightarrow 17$

Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.048$	H-atom parameters constrained
$wR(F^2) = 0.147$	$w = 1/[\sigma^2(F_o^2) + (0.0776P)^2 + 1.2356P]$
$S = 1.06$	where $P = (F_o^2 + 2F_c^2)/3$
1795 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
129 parameters	$\Delta\rho_{\text{max}} = 0.74 \text{ e } \text{\AA}^{-3}$
34 restraints	$\Delta\rho_{\text{min}} = -0.49 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Ag	1.0000	0.04678 (8)	0.7500	0.0718 (3)	
Cl	1.0000	-0.4118 (3)	0.7500	0.0779 (6)	
O1	0.5863 (3)	0.6168 (5)	0.5648 (4)	0.0819 (13)	
H1o	0.5297	0.6370	0.5298	0.123*	
N1	0.8519 (3)	0.0695 (5)	0.6720 (4)	0.0573 (11)	
N2	0.6021 (3)	0.4515 (5)	0.5606 (4)	0.0603 (12)	
C1	0.7951 (4)	-0.0529 (6)	0.6259 (5)	0.0617 (14)	
H1	0.8207	-0.1559	0.6285	0.074*	
C2	0.7003 (4)	-0.0342 (7)	0.5744 (5)	0.0645 (14)	
H2	0.6630	-0.1234	0.5437	0.077*	
C3	0.6612 (4)	0.1150 (7)	0.5686 (4)	0.0587 (13)	
H3	0.5971	0.1289	0.5337	0.070*	
C4	0.7187 (3)	0.2477 (6)	0.6158 (4)	0.0495 (11)	
C5	0.8130 (3)	0.2174 (7)	0.6658 (4)	0.0544 (12)	
H5	0.8521	0.3044	0.6971	0.065*	
C6	0.6859 (4)	0.4136 (7)	0.6113 (4)	0.0571 (12)	
H6	0.7283	0.4943	0.6469	0.068*	
O2	1.0050 (8)	-0.2768 (10)	0.6778 (8)	0.105 (3)	0.50
O3	0.9040 (6)	-0.4057 (13)	0.7376 (10)	0.110 (4)	0.50
O4	1.0044 (7)	-0.5524 (9)	0.6836 (7)	0.089 (3)	0.50
O5	1.0649 (10)	-0.4065 (18)	0.8439 (7)	0.171 (6)	0.50

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Ag	0.0392 (4)	0.0813 (5)	0.0859 (5)	0.000	0.0121 (3)	0.000

C1	0.0624 (13)	0.0605 (12)	0.1049 (17)	0.000	0.0238 (13)	0.000
O1	0.064 (3)	0.054 (2)	0.106 (3)	0.005 (2)	0.005 (3)	0.007 (2)
N1	0.042 (2)	0.061 (3)	0.063 (3)	-0.0016 (19)	0.013 (2)	0.000 (2)
N2	0.054 (3)	0.054 (3)	0.066 (3)	0.005 (2)	0.013 (2)	0.006 (2)
C1	0.056 (3)	0.051 (3)	0.072 (3)	0.000 (2)	0.017 (3)	0.000 (3)
C2	0.048 (3)	0.058 (3)	0.075 (4)	-0.009 (3)	0.009 (3)	-0.006 (3)
C3	0.040 (3)	0.068 (3)	0.060 (3)	-0.003 (3)	0.009 (2)	0.002 (3)
C4	0.043 (2)	0.055 (3)	0.045 (2)	-0.006 (2)	0.010 (2)	0.002 (2)
C5	0.040 (3)	0.059 (3)	0.057 (3)	-0.005 (2)	0.010 (2)	-0.003 (2)
C6	0.045 (3)	0.057 (3)	0.062 (3)	-0.004 (2)	0.010 (2)	0.002 (2)
O2	0.123 (7)	0.082 (5)	0.125 (7)	-0.002 (5)	0.063 (6)	0.020 (5)
O3	0.098 (7)	0.104 (6)	0.148 (8)	-0.010 (6)	0.068 (7)	-0.018 (6)
O4	0.072 (5)	0.074 (5)	0.107 (6)	0.006 (4)	0.018 (5)	-0.014 (4)
O5	0.166 (11)	0.162 (9)	0.137 (9)	-0.001 (9)	-0.001 (8)	-0.008 (8)

*Geometric parameters (Å, °)*

Ag—N1 <sup>i</sup>	2.138 (5)	C2—H2	0.9300
Ag—N1	2.138 (5)	C3—C4	1.402 (8)
Ag—O2	2.843 (9)	C3—H3	0.9300
O1—N2	1.387 (7)	C4—C5	1.378 (7)
O1—H1 <sub>o</sub>	0.8400	C4—C6	1.449 (8)
N1—C1	1.327 (7)	C5—H5	0.9300
N1—C5	1.346 (7)	C6—H6	0.9300
N2—C6	1.255 (7)	Cl—O5	1.283 (7)
C1—C2	1.374 (9)	Cl—O3	1.426 (7)
C1—H1	0.9300	Cl—O4	1.474 (6)
C2—C3	1.357 (8)	Cl—O2	1.490 (6)
N1 <sup>i</sup> —Ag—N1	170.0 (2)	C5—C4—C3	117.1 (5)
N1 <sup>i</sup> —Ag—O2	95.1 (3)	C5—C4—C6	118.6 (5)
N1—Ag—O2	94.2 (3)	C3—C4—C6	124.3 (5)
N2—O1—H1 <sub>o</sub>	109.5	N1—C5—C4	124.0 (5)
C1—N1—C5	117.2 (5)	N1—C5—H5	118.0
C1—N1—Ag	124.1 (4)	C4—C5—H5	118.0
C5—N1—Ag	118.6 (3)	N2—C6—C4	122.0 (5)
C6—N2—O1	112.5 (5)	N2—C6—H6	119.0
N1—C1—C2	122.9 (5)	C4—C6—H6	119.0
N1—C1—H1	118.6	O5—Cl—O3	120.9 (7)
C2—C1—H1	118.6	O5—Cl—O4	114.9 (7)
C3—C2—C1	119.8 (5)	O3—Cl—O4	103.4 (5)
C3—C2—H2	120.1	O5—Cl—O2	113.4 (7)
C1—C2—H2	120.1	O3—Cl—O2	101.3 (5)
C2—C3—C4	119.1 (5)	O4—Cl—O2	100.0 (5)
C2—C3—H3	120.5	Cl—O2—Ag	117.8 (5)
C4—C3—H3	120.5		
N1 <sup>i</sup> —Ag—N1—C1	168.7 (5)	Ag—N1—C5—C4	178.7 (4)
O2—Ag—N1—C1	9.2 (5)	C3—C4—C5—N1	-0.4 (8)
N1 <sup>i</sup> —Ag—N1—C5	-9.2 (4)	C6—C4—C5—N1	-177.8 (5)

O2—Ag—N1—C5	-168.7 (4)	O1—N2—C6—C4	-178.7 (5)
C5—N1—C1—C2	-0.7 (9)	C5—C4—C6—N2	175.0 (5)
Ag—N1—C1—C2	-178.7 (5)	C3—C4—C6—N2	-2.3 (9)
N1—C1—C2—C3	0.5 (10)	O5—Cl—O2—Ag	-57.3 (9)
C1—C2—C3—C4	-0.2 (9)	O3—Cl—O2—Ag	73.9 (7)
C2—C3—C4—C5	0.1 (8)	O4—Cl—O2—Ag	179.9 (5)
C2—C3—C4—C6	177.4 (5)	N1—Ag—O2—Cl	-93.0 (6)
C1—N1—C5—C4	0.7 (8)		

Symmetry code: (i)  $-x+2, y, -z+3/2$ .

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O1—H1 <sup><i>o</i></sup> ...N2 <sup><i>ii</i></sup>	0.84	2.07	2.821 (7)	148

Symmetry code: (ii)  $-x+1, -y+1, -z+1$ .