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Bis[μ-(*E*)-*N*-(pyridin-3-ylmethylidene)-hydroxyamine]-$\kappa^2N^1:N^3;\kappa^2N^3:N^1$-bis-{[(*E*)-*N*-(pyridin-3-ylmethylidene-κN)-hydroxyamine]silver(I)} dinitrate

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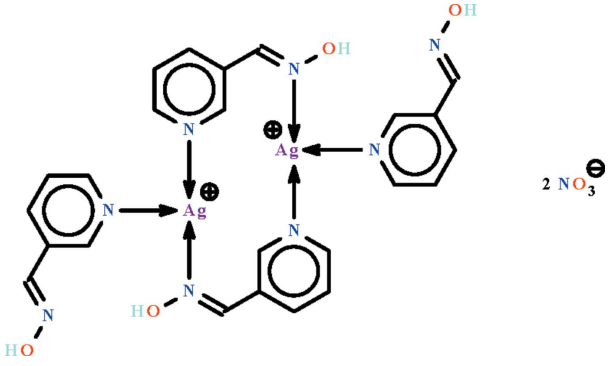
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.040; wR factor = 0.107; data-to-parameter ratio = 15.9.

In the centrosymmetric dinuclear title Ag^{I} compound, $[Ag_2(C_6H_6N_2O)_4](NO_3)_2$, the aromatic amine-coordinated Ag^{I} atom is further bridged by two hydroxylamine molecules that use aromatic and oxime N atoms for bridging, and it exists in a distorted trigonal-planar geometry. In the crystal, the nitrate anions link to the dinuclear compound molecules *via* O—H···O hydrogen bonds, generating a chain running along the *a*-axis direction.

Related literature

For bis(nicotinylaldehyde oxime)silver perchlorate, see: Xu *et al.* (2012) and for (nitrato)(picolinaldehyde oxime)silver, see: Abu-Youssef *et al.* (2010).



Experimental

Crystal data

$[Ag_2(C_6H_6N_2O)_4](NO_3)_2$ M_r = 828.27
Triclinic, $P\overline{1}$
a = 7.2913 (10) Å
b = 8.3395 (10) Å
c = 13.1415 (17) Å
α = 92.934 (4)°
β = 95.008 (4)°
γ = 111.360 (3)°
V = 738.38 (16) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 1.40 mm^{-1}
T = 293 K
0.24 × 0.21 × 0.17 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.730, T_{max} = 0.797
7293 measured reflections
3341 independent reflections
2896 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.107
S = 1.11
3341 reflections
210 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.71 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O3^{i}	0.84	2.39	3.189 (6)	158
O1—H1···O4^{i}	0.84	2.20	2.925 (5)	145
O2—H2···O4^{ii}	0.84	1.92	2.745 (4)	169

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

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalClear* (Rigaku/MSC, 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); 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: XU5646).

References

Abu-Youssef, M. A., Soliman, S. V., Langer, V., Gohar, Y. M., Hasanen, A. A., Makhyoun, M. A., Zaky, A. H. & Öhrström, L. R. (2010). *Inorg. Chem.* **49**, 9788–9797.
Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Rigaku (1998). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2002). *CrystalClear*. Rigaku/MSC Inc., The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
Xu, J., Gao, S., Ng, S. W. & Tiekink, E. R. T. (2012). *Acta Cryst.* E**68**, m735–m736.

supporting information

Acta Cryst. (2012). E**68**, m1547 [doi:10.1107/S1600536812045898]

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S1. Comment

A previous study on the adducts of silver salts with nicotinylaldehyde oxime reported the salt, $[Ag(C_6H_6N_2O)_2]$ ClO_4; the metal atom shows linear coordination (Xu *et al.*, 2012). The aromatic amine-coordinated Ag^I atom in dinuclear $[Ag(C_6H_6N_2O)_2]_2$ $2NO_3$ is bridged by another hydroxylamine molecule that uses its aromatic and oxime N atoms for bridging, and it exists in a trigonal planar geometry (Scheme I, Fig. 1). The hydroxyl OH groups forms H atoms to the nitrate O atoms of adjacent molecules to generate a chain running along the shortest axis of the orthorhombic unit cell (Table 1).

With picolinylaldehyde oxime in place of nicotinylaldehyde oxime, the synthesis yields the monomeric adduct in which the metal atom is chelated by the ligand (Abu-Youssef *et al.*, 2010).

S2. Experimental

Nicotinyladehyde oxime was synthesized from the reaction of nicotinylaldehyde and hydroxylamine. Silver nitrate (1 mmol) dissolved in water (5 ml) was added to nicotinylaldehyde oxime (1 mmol) dissolved in ethanol (5 ml). The solution was filtered and set aside, away from light, for the growth of colorless crystals.

S3. Refinement

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

The (-1 3 3), (5 - 10 1), (6 - 9 5) and (0 9 1) reflections were omitted.

The final difference Fourier map had a peak at 0.88 Å from Ag1.

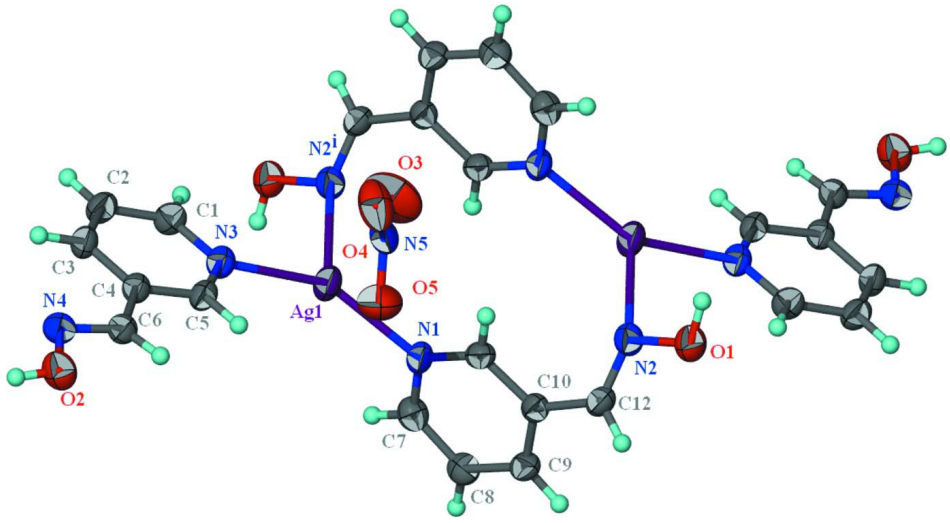

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $[Ag(C_6H_6N_2O)_2]_2\ 2NO_3$ at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

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Hall symbol: -P 1
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$\beta = 95.008$ (4)°
$\gamma = 111.360$ (3)°
$V = 738.38$ (16) Å^3
$Z = 1$
$F(000) = 412$
$D_x = 1.863$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6130 reflections
$\theta = 3.0$–27.4°
$\mu = 1.40$ mm^{-1}
$T = 293$ K
Prism, colorless
0.24 × 0.21 × 0.17 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scan
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.730$, $T_{max} = 0.797$
7293 measured reflections
3341 independent reflections
2896 reflections with $I > 2\sigma(I)$
$R_{int} = 0.035$
$\theta_{max} = 27.4°$, $\theta_{min} = 3.0°$
$h = -9\rightarrow9$
$k = -10\rightarrow10$
$l = -17\rightarrow16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.107$
$S = 1.11$
3341 reflections
210 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map	$w = 1/[\sigma^2(F_o^2) + (0.056P)^2 + 0.3361P]$ where $P = (F_o^2 + 2F_c^2)/3$
Hydrogen site location: inferred from neighbouring sites	$(\Delta/\sigma)_{max} = 0.001$
H-atom parameters constrained	$\Delta\rho_{max} = 1.71$ e Å^{-3}
	$\Delta\rho_{min} = -0.34$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
Ag1	0.38614 (4)	0.61092 (3)	0.314850 (18)	0.04700 (13)
O1	1.0366 (4)	0.7195 (3)	0.7651 (2)	0.0501 (6)
H1	1.0758	0.6378	0.7541	0.075*
O2	0.9485 (4)	1.1845 (3)	−0.0856 (2)	0.0522 (6)
H2	0.9451	1.2272	−0.1419	0.078*
O3	0.7359 (7)	0.5156 (5)	0.3151 (4)	0.1102 (16)
O4	1.0106 (5)	0.6444 (5)	0.2602 (3)	0.0751 (10)
O5	0.8581 (6)	0.7867 (4)	0.3354 (3)	0.0792 (10)
N1	0.4261 (4)	0.7161 (4)	0.4767 (2)	0.0393 (6)
N2	0.8633 (4)	0.6911 (4)	0.7003 (2)	0.0426 (6)
N3	0.4227 (4)	0.6552 (4)	0.1507 (2)	0.0393 (6)
N4	0.7795 (4)	1.0353 (4)	−0.0856 (2)	0.0411 (6)
N5	0.8690 (4)	0.6504 (4)	0.3059 (2)	0.0436 (7)
C1	0.2918 (5)	0.5528 (4)	0.0748 (3)	0.0420 (7)
H1A	0.1875	0.4574	0.0915	0.050*
C2	0.3047 (5)	0.5827 (4)	−0.0277 (3)	0.0438 (7)
H2A	0.2104	0.5087	−0.0783	0.053*
C3	0.4576 (5)	0.7221 (4)	−0.0537 (3)	0.0410 (7)
H3	0.4689	0.7437	−0.1220	0.049*
C4	0.5962 (5)	0.8315 (4)	0.0240 (2)	0.0354 (6)
C5	0.5728 (5)	0.7911 (4)	0.1242 (2)	0.0366 (6)
H5	0.6663	0.8621	0.1762	0.044*
C6	0.7622 (5)	0.9865 (4)	0.0046 (3)	0.0375 (7)
H6	0.8559	1.0499	0.0586	0.045*
C7	0.3097 (5)	0.7957 (4)	0.5116 (3)	0.0430 (7)
H7	0.1944	0.7853	0.4708	0.052*
C8	0.3541 (5)	0.8913 (4)	0.6046 (3)	0.0429 (7)
H8	0.2721	0.9467	0.6256	0.051*
C9	0.5228 (5)	0.9041 (4)	0.6669 (3)	0.0407 (7)
H9	0.5570	0.9704	0.7296	0.049*
C10	0.6402 (5)	0.8177 (4)	0.6350 (2)	0.0361 (6)
C11	0.5871 (5)	0.7270 (4)	0.5384 (2)	0.0369 (6)
H11	0.6675	0.6713	0.5156	0.044*
C12	0.8170 (5)	0.8228 (4)	0.6996 (2)	0.0396 (7)
H12	0.8952	0.9234	0.7401	0.048*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ag1	0.05390 (19)	0.0543 (2)	0.03057 (17)	0.01848 (14)	0.00080 (12)	0.00222 (11)

O1	0.0459 (13)	0.0570 (15)	0.0475 (15)	0.0220 (12)	−0.0050 (11)	0.0026 (11)
O2	0.0459 (13)	0.0490 (14)	0.0565 (16)	0.0103 (11)	0.0045 (12)	0.0153 (12)
O3	0.101 (3)	0.062 (2)	0.155 (5)	0.007 (2)	0.035 (3)	0.043 (3)
O4	0.0682 (19)	0.124 (3)	0.062 (2)	0.064 (2)	0.0219 (16)	0.034 (2)
O5	0.100 (3)	0.0675 (19)	0.079 (2)	0.0441 (19)	0.009 (2)	−0.0041 (16)
N1	0.0458 (14)	0.0412 (14)	0.0277 (13)	0.0131 (12)	0.0010 (11)	0.0040 (10)
N2	0.0430 (14)	0.0408 (14)	0.0401 (15)	0.0129 (12)	−0.0037 (12)	0.0043 (11)
N3	0.0400 (14)	0.0462 (15)	0.0333 (14)	0.0179 (12)	0.0039 (11)	0.0034 (11)
N4	0.0401 (14)	0.0401 (14)	0.0439 (16)	0.0154 (12)	0.0063 (12)	0.0039 (12)
N5	0.0483 (16)	0.0521 (17)	0.0383 (15)	0.0257 (15)	0.0081 (13)	0.0138 (13)
C1	0.0393 (16)	0.0407 (17)	0.0425 (18)	0.0106 (14)	0.0054 (14)	0.0021 (13)
C2	0.0442 (17)	0.0446 (17)	0.0386 (18)	0.0150 (15)	−0.0037 (14)	−0.0056 (14)
C3	0.0471 (18)	0.0475 (18)	0.0297 (15)	0.0197 (15)	0.0012 (14)	0.0024 (13)
C4	0.0349 (14)	0.0415 (16)	0.0328 (15)	0.0178 (13)	0.0035 (12)	0.0027 (12)
C5	0.0352 (15)	0.0437 (16)	0.0310 (15)	0.0163 (13)	−0.0002 (12)	−0.0023 (12)
C6	0.0367 (15)	0.0424 (16)	0.0373 (17)	0.0203 (14)	0.0013 (13)	0.0012 (13)
C7	0.0394 (16)	0.0468 (17)	0.0443 (19)	0.0166 (15)	0.0061 (14)	0.0098 (14)
C8	0.0470 (17)	0.0457 (18)	0.0413 (18)	0.0210 (15)	0.0138 (15)	0.0082 (14)
C9	0.0530 (19)	0.0390 (16)	0.0322 (16)	0.0188 (15)	0.0093 (14)	0.0022 (12)
C10	0.0424 (16)	0.0309 (14)	0.0319 (15)	0.0097 (13)	0.0039 (13)	0.0057 (11)
C11	0.0439 (16)	0.0315 (14)	0.0356 (16)	0.0141 (13)	0.0066 (13)	0.0021 (12)
C12	0.0455 (17)	0.0385 (16)	0.0315 (16)	0.0129 (14)	0.0015 (13)	−0.0022 (12)

Geometric parameters (Å, º)

Ag1—N1	2.212 (3)	C2—C3	1.369 (5)
Ag1—N3	2.229 (3)	C2—H2A	0.9300
Ag1—N2^{i}	2.498 (3)	C3—C4	1.396 (5)
O1—N2	1.397 (4)	C3—H3	0.9300
O1—H1	0.8400	C4—C5	1.385 (4)
O2—N4	1.396 (4)	C4—C6	1.466 (5)
O2—H2	0.8400	C5—H5	0.9300
O3—N5	1.208 (5)	C6—H6	0.9300
O4—N5	1.255 (4)	C7—C8	1.370 (5)
O5—N5	1.214 (4)	C7—H7	0.9300
N1—C11	1.339 (4)	C8—C9	1.383 (5)
N1—C7	1.349 (5)	C8—H8	0.9300
N2—C12	1.262 (4)	C9—C10	1.381 (5)
N2—Ag1^{i}	2.498 (3)	C9—H9	0.9300
N3—C1	1.337 (5)	C10—C11	1.392 (4)
N3—C5	1.344 (4)	C10—C12	1.466 (5)
N4—C6	1.274 (4)	C11—H11	0.9300
C1—C2	1.387 (5)	C12—H12	0.9300
C1—H1A	0.9300		
N1—Ag1—N3	149.49 (11)	C5—C4—C3	117.7 (3)
N1—Ag1—N2^{i}	107.79 (10)	C5—C4—C6	119.0 (3)
N3—Ag1—N2^{i}	101.48 (10)	C3—C4—C6	123.3 (3)

N2—O1—H1	109.5	N3—C5—C4	123.8 (3)
N4—O2—H2	109.5	N3—C5—H5	118.1
C11—N1—C7	117.6 (3)	C4—C5—H5	118.1
C11—N1—Ag1	119.9 (2)	N4—C6—C4	120.7 (3)
C7—N1—Ag1	121.6 (2)	N4—C6—H6	119.6
C12—N2—O1	112.4 (3)	C4—C6—H6	119.6
C12—N2—Ag1^{i}	123.3 (2)	N1—C7—C8	122.8 (3)
O1—N2—Ag1^{i}	115.10 (18)	N1—C7—H7	118.6
C1—N3—C5	117.2 (3)	C8—C7—H7	118.6
C1—N3—Ag1	121.6 (2)	C7—C8—C9	119.0 (3)
C5—N3—Ag1	121.1 (2)	C7—C8—H8	120.5
C6—N4—O2	110.6 (3)	C9—C8—H8	120.5
O3—N5—O5	120.1 (4)	C10—C9—C8	119.5 (3)
O3—N5—O4	117.9 (4)	C10—C9—H9	120.3
O5—N5—O4	121.9 (4)	C8—C9—H9	120.3
N3—C1—C2	122.8 (3)	C9—C10—C11	117.8 (3)
N3—C1—H1A	118.6	C9—C10—C12	121.5 (3)
C2—C1—H1A	118.6	C11—C10—C12	120.6 (3)
C3—C2—C1	119.4 (3)	N1—C11—C10	123.3 (3)
C3—C2—H2A	120.3	N1—C11—H11	118.4
C1—C2—H2A	120.3	C10—C11—H11	118.4
C2—C3—C4	119.0 (3)	N2—C12—C10	120.1 (3)
C2—C3—H3	120.5	N2—C12—H12	119.9
C4—C3—H3	120.5	C10—C12—H12	119.9

N3—Ag1—N1—C11	−93.9 (3)	O2—N4—C6—C4	−179.6 (3)
N2^{i}—Ag1—N1—C11	103.1 (2)	C5—C4—C6—N4	−174.4 (3)
N3—Ag1—N1—C7	75.2 (3)	C3—C4—C6—N4	4.9 (5)
N2^{i}—Ag1—N1—C7	−87.8 (3)	C11—N1—C7—C8	2.6 (5)
N1—Ag1—N3—C1	−147.5 (3)	Ag1—N1—C7—C8	−166.8 (2)
N2^{i}—Ag1—N3—C1	16.0 (3)	N1—C7—C8—C9	−1.5 (5)
N1—Ag1—N3—C5	29.4 (4)	C7—C8—C9—C10	−1.4 (5)
N2^{i}—Ag1—N3—C5	−167.1 (2)	C8—C9—C10—C11	2.9 (4)
C5—N3—C1—C2	−0.6 (5)	C8—C9—C10—C12	−178.1 (3)
Ag1—N3—C1—C2	176.4 (3)	C7—N1—C11—C10	−0.9 (5)
N3—C1—C2—C3	0.2 (5)	Ag1—N1—C11—C10	168.7 (2)
C1—C2—C3—C4	−0.4 (5)	C9—C10—C11—N1	−1.8 (5)
C2—C3—C4—C5	0.9 (5)	C12—C10—C11—N1	179.2 (3)
C2—C3—C4—C6	−178.4 (3)	O1—N2—C12—C10	179.4 (3)
C1—N3—C5—C4	1.2 (5)	Ag1^{i}—N2—C12—C10	−35.6 (4)
Ag1—N3—C5—C4	−175.8 (2)	C9—C10—C12—N2	144.0 (3)
C3—C4—C5—N3	−1.4 (5)	C11—C10—C12—N2	−37.1 (5)
C6—C4—C5—N3	178.0 (3)		

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O3^{ii}	0.84	2.39	3.189 (6)	158
O1—H1···O4^{ii}	0.84	2.20	2.925 (5)	145
O2—H2···O4^{iii}	0.84	1.92	2.745 (4)	169

Symmetry codes: (ii) −*x*+2, −*y*+1, −*z*+1; (iii) −*x*+2, −*y*+2, −*z*.