Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

Bis[μ-1-(4-pyridylmethyl)-1*H*-imidazole]-disilver(I) dinitrate

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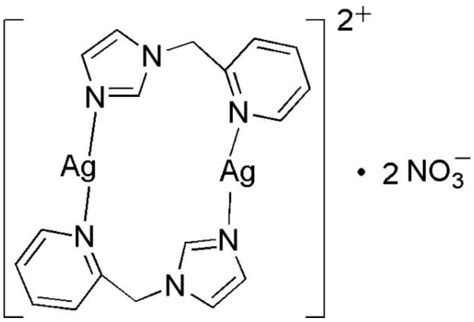
Received 12 March 2007; accepted 12 August 2007

Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.006 Å; R factor = 0.029; wR factor = 0.084; data-to-parameter ratio = 13.1.

The title compound, $[Ag_2(C_9H_9N_3)_2](NO_3)_2$, is a 14-membered metallamacrocycle formed by two Ag atoms bridging two *N*-(2-pyridylmethyl)imidazole (pymim) molecules. The asymmetric unit consists of one-half of the cation and one nitrate anion. The metallamacrocycle complex lies on an inversion centre. The nitrate anions are in close contact with the Ag centres of two neighbouring cations, which link the metallamacrocyclic units into a double-chain structure along the *a* axis.

Related literature

For related literature, see: Bondi (1964); Chiu *et al.* (2005); Lin *et al.* (2004); Peng *et al.* (2006); Telfer *et al.* (2006); Yue *et al.* (2005); Zhang *et al.* (2005); Zheng *et al.* (2003).



Experimental

Crystal data

$[Ag_2(C_9H_9N_3)_2](NO_3)_2$
M_r = 658.14
Monoclinic, $P2_1/n$
a = 5.4170 (9) Å
b = 13.020 (2) Å
c = 15.648 (3) Å
β = 92.915 (2)°
V = 1102.2 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.83 mm^{-1}
T = 291 (2) K
0.33 × 0.28 × 0.19 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
T_{min} = 0.587, T_{max} = 0.720
6892 measured reflections
2024 independent reflections
1809 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.084
S = 1.05
2024 reflections
154 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.85 e Å^{-3}
$\Delta\rho_{min}$ = −0.50 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ag1—N3^i	2.190 (3)	Ag1—N1	2.244 (3)
N3^i—Ag1—N1	162.80 (10)	C7—N3—Ag1^i	130.5 (2)
C5—N1—Ag1	126.3 (2)	C8—N3—Ag1^i	123.9 (2)
C1—N1—Ag1	115.1 (2)	N2—C6—C5	114.0 (3)

Symmetry code: (i) $-x+2, -y+1, -z+1$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The authors thank the Natural Science Foundation of Henan Province (No. 051105300) and the Creative Talented Persons Fund of Henan Province (Teach high [2005]-126) for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ER2033).

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doi:10.1107/S1600536807040032

supplementary materials

Acta Cryst. (2007). E**63**, m2371 [**doi:10.1107/S1600536807040032**]

Bis[μ-1-(4-pyridylmethyl)-1*H*-imidazole]disilver(I) dinitrate

J.-P. Xiao, Q.-X. Zhou and J.-H. Tu

Comment

The synthesis and properties of self-assembled hybrid organic–inorganic macrocyclic or polymeric compounds involving suitably designed ligands and transition-metal ions have drawn an ever-increasing level of attention (Yue *et al.*, 2005; Zheng *et al.*, 2003). There has been much progress recently in the study of crystal engineering of supramolecular architectures using N-donor ligands (Telfer *et al.*, 2006; Peng *et al.*, 2006). The cation of the title compound, $Ag_2(pymim)_2(NO_3)_2$ is a fourteen-membered metallomacrocycle formed by the two Ag atoms bridging two pymim molecules. The asymmetric unit consists of one-half of the molecular cation and one nitrate anion. The full cation and the other nitrate anion are generated by the symmetry operation of a crystallographic inversion centre. As shown Fig. 1 and Fig. 2, The ligands are arranged in a head-to-tail fashion. The macrocycle lie on an inversion centre and the silver atoms are coordinated by a pyridine and a imidazole moiety. The Ag···Ag distance of 5.040 (6) Å is much longer than the sum of van der Waals radii of two Ag atoms (3.40 Å, Bondi, 1964) and can be regarded as noninteracting. Each pyridine ring is highly twisted from its *trans* imidazole ring with an interplanar angle of 74.55 (11)°. The overall geometry of the metallomacrocycle is slightly twisted with the N1—Ag1···Ag1A—N3 dihedral angle of 1.37 (3)°. The O atoms of nitrate anions are in close contact with the silver centers of two neighboring cations having nonbonding distances of 2.715 (3)–2.918 (3) Å, which are shorter than the sum of van der Waals radii for the Ag and O atoms (3.24 Å, Bondi, 1964) and consistent with those reported for other silver nitrate complexes in the literature (Yue *et al.*, 2005; Zhang *et al.*, 2005; Lin *et al.*, 2004). These bridging nitrate interactions pull the silver centers in the metallomacrocyclic units away from each other resulting in very long Ag···Ag distance and a one-dimensional double-chain structure along the *a* axis.

Experimental

All reagents were of analytical grade and used without further purification. Pymim was prepared by the general procedure of Chiu *et al.* (2005). A solution of pymim (0.4 mmol, 64 mg) in MeOH (4 ml) was added to a stirring solution of $AgNO_3$ (0.4 mmol, 68 mg) in MeOH (8 ml). The white precipitate that formed immediately was collected, washed with MeOH and dried. Colorless single crystals were grown from diffusion of dithyl ether into a DMF solution containing the silver complexes. Yield: 35%. Analysis found: C 33.01, H 2.79, N 16.93%.; calculated for $Ag_2(C_9H_9N_3)_2(NO_3)_2$: C 32.85, H 2.76, N 17.03%.

Refinement

C-bound H atoms were positioned geometrically and treated as riding with C—H = 0.93–0.97 Å, and with $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

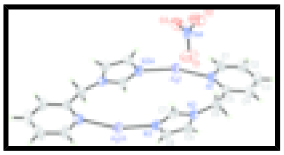

Fig. 1. The asymmetric unit of (I) and parts of adjacent units, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 40% probability level. [Symmetry code (A): $-x + 2, -y + 1, -z + 1$]

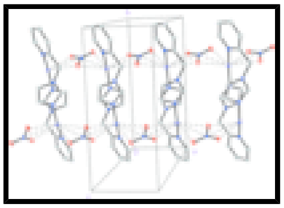

Fig. 2. Bridging nitrate interactions in (I) along the *a* axis. Hydrogen atoms are omitted for clarity.

Bis[μ-1-(4-pyridylmethyl)-1*H*-imidazole]disilver(I) dinitrate

Crystal data

$[Ag_2(C_9H_9N_3)_2](NO_3)_2$	$F_{000} = 648$
$M_r = 658.14$	$D_x = 1.983$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 3520 reflections
$a = 5.4170$ (9) Å	$\theta = 2.6$–27.7°
$b = 13.020$ (2) Å	$\mu = 1.83$ mm^{-1}
$c = 15.648$ (3) Å	$T = 291$ (2) K
$\beta = 92.915$ (2)°	Block, colourless
$V = 1102.2$ (3) Å^3	0.33 × 0.28 × 0.19 mm
$Z = 2$	

Data collection

Bruker SMART 1K CCD area-detector diffractometer	2024 independent reflections
Radiation source: fine-focus sealed tube	1809 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.022$
Detector resolution: 10 pixels mm^{-1}	$\theta_{max} = 25.5$°
$T = 291(2)$ K	$\theta_{min} = 2.6$°
φ and ω scans	$h = -5 \rightarrow 6$
Absorption correction: multi-scan (SADABS; Bruker, 2001)	$k = -15 \rightarrow 15$
$T_{min} = 0.587$, $T_{max} = 0.720$	$l = -18 \rightarrow 18$
6892 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map

Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.029$	H-atom parameters constrained
$wR(F^2) = 0.084$	$w = 1/[\sigma^2(F_o^2) + (0.0546P)^2 + 0.4881P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} = 0.001$
2024 reflections	$\Delta\rho_{max} = 0.85$ e Å^{-3}
154 parameters	$\Delta\rho_{min} = -0.50$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	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 $F^2 > \sigma(F^2)$ 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. Highest peak 0.85 at 0.2188 0.3146 0.5020 [0.90 A from AG1] Deepest hole −0.50 at 0.0726 0.3653 0.5062 [0.80 A from AG1]

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Ag1	1.08496 (5)	0.313742 (18)	0.533996 (15)	0.04668 (14)
O1	0.5896 (5)	0.2907 (3)	0.5539 (2)	0.0823 (10)
O2	0.3913 (6)	0.2095 (3)	0.6460 (2)	0.0767 (9)
O3	0.7815 (5)	0.2392 (2)	0.6697 (2)	0.0716 (8)
N1	1.0499 (6)	0.19166 (19)	0.43290 (18)	0.0445 (7)
N2	0.8319 (5)	0.37732 (19)	0.36086 (15)	0.0396 (6)
N3	0.9081 (5)	0.5376 (2)	0.40039 (16)	0.0442 (6)
N4	0.5879 (5)	0.2460 (2)	0.62429 (17)	0.0439 (6)
C1	1.1948 (8)	0.1093 (3)	0.4469 (3)	0.0606 (10)
H1	1.3174	0.1123	0.4906	0.073*
C2	1.1706 (9)	0.0201 (3)	0.3995 (3)	0.0713 (12)
H2	1.2752	−0.0354	0.4108	0.086*
C3	0.9894 (9)	0.0152 (3)	0.3354 (3)	0.0724 (12)
H3	0.9656	−0.0445	0.3034	0.087*
C4	0.8433 (8)	0.1000 (3)	0.3193 (2)	0.0622 (10)
H4	0.7216	0.0987	0.2752	0.075*
C5	0.8776 (7)	0.1871 (2)	0.3686 (2)	0.0415 (7)
C6	0.7082 (6)	0.2779 (3)	0.3527 (2)	0.0480 (8)
H6A	0.6313	0.2722	0.2956	0.058*
H6B	0.5780	0.2752	0.3930	0.058*
C7	0.7586 (6)	0.4584 (2)	0.40715 (19)	0.0438 (7)

H7	0.6199	0.4583	0.4398	0.053*
C8	1.0864 (7)	0.5054 (3)	0.34738 (19)	0.0454 (7)
H8	1.2188	0.5452	0.3313	0.054*
C9	1.0426 (7)	0.4081 (3)	0.3220 (2)	0.0459 (8)
H9	1.1357	0.3694	0.2854	0.055*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ag1	0.0523 (2)	0.04178 (19)	0.04488 (19)	0.00715 (10)	−0.00753 (13)	−0.01113 (9)
O1	0.057 (2)	0.136 (3)	0.0540 (16)	−0.0109 (18)	−0.0005 (13)	0.0275 (18)
O2	0.0523 (18)	0.098 (2)	0.079 (2)	−0.0163 (16)	−0.0053 (15)	0.0353 (17)
O3	0.0530 (17)	0.0734 (19)	0.0855 (19)	−0.0112 (14)	−0.0239 (15)	0.0270 (15)
N1	0.0514 (18)	0.0358 (15)	0.0454 (16)	0.0011 (11)	−0.0047 (13)	−0.0084 (10)
N2	0.0450 (16)	0.0357 (14)	0.0373 (13)	0.0038 (11)	−0.0044 (11)	−0.0037 (10)
N3	0.0555 (17)	0.0381 (14)	0.0382 (13)	0.0077 (12)	−0.0053 (12)	−0.0049 (10)
N4	0.0425 (16)	0.0399 (14)	0.0487 (15)	0.0027 (12)	−0.0018 (13)	0.0013 (12)
C1	0.069 (3)	0.042 (2)	0.070 (2)	0.0093 (17)	−0.0122 (19)	−0.0104 (16)
C2	0.090 (3)	0.0379 (19)	0.086 (3)	0.015 (2)	0.002 (3)	−0.0105 (19)
C3	0.100 (4)	0.039 (2)	0.077 (3)	−0.004 (2)	0.002 (3)	−0.0259 (19)
C4	0.074 (3)	0.056 (2)	0.055 (2)	−0.009 (2)	−0.0098 (19)	−0.0175 (17)
C5	0.047 (2)	0.0380 (17)	0.0395 (17)	−0.0067 (13)	0.0017 (14)	−0.0046 (12)
C6	0.047 (2)	0.0481 (18)	0.0484 (19)	−0.0061 (16)	−0.0068 (15)	−0.0028 (15)
C7	0.0440 (18)	0.0486 (18)	0.0383 (16)	0.0096 (15)	−0.0024 (13)	−0.0076 (13)
C8	0.0498 (19)	0.0407 (17)	0.0455 (17)	0.0032 (14)	0.0018 (15)	−0.0005 (14)
C9	0.054 (2)	0.0422 (17)	0.0422 (16)	0.0056 (15)	0.0062 (15)	−0.0046 (13)

Geometric parameters (Å, °)

Ag1—N3^{i}	2.190 (3)	C1—H1	0.9300
Ag1—N1	2.244 (3)	C2—C3	1.368 (6)
O1—N4	1.246 (4)	C2—H2	0.9300
O2—N4	1.230 (4)	C3—C4	1.375 (6)
O3—N4	1.240 (4)	C3—H3	0.9300
N1—C5	1.338 (5)	C4—C5	1.379 (5)
N1—C1	1.341 (4)	C4—H4	0.9300
N2—C7	1.351 (4)	C5—C6	1.509 (5)
N2—C9	1.380 (4)	C6—H6A	0.9700
N2—C6	1.460 (4)	C6—H6B	0.9700
N3—C7	1.319 (4)	C7—H7	0.9300
N3—C8	1.370 (4)	C8—C9	1.345 (5)
N3—Ag1^{i}	2.190 (3)	C8—H8	0.9300
C1—C2	1.380 (5)	C9—H9	0.9300
N3^{i}—Ag1—N1	162.80 (10)	C3—C4—C5	119.9 (4)
C5—N1—C1	117.9 (3)	C3—C4—H4	120.1
C5—N1—Ag1	126.3 (2)	C5—C4—H4	120.1
C1—N1—Ag1	115.1 (2)	N1—C5—C4	121.7 (3)
C7—N2—C9	106.3 (3)	N1—C5—C6	119.0 (3)

C7—N2—C6	126.4 (3)	C4—C5—C6	119.2 (3)
C9—N2—C6	127.3 (3)	N2—C6—C5	114.0 (3)
C7—N3—C8	105.3 (3)	N2—C6—H6A	108.7
C7—N3—Ag1^{i}	130.5 (2)	C5—C6—H6A	108.7
C8—N3—Ag1^{i}	123.9 (2)	N2—C6—H6B	108.7
O2—N4—O3	122.2 (3)	C5—C6—H6B	108.7
O2—N4—O1	118.1 (3)	H6A—C6—H6B	107.6
O3—N4—O1	119.8 (3)	N3—C7—N2	111.7 (3)
N1—C1—C2	123.1 (4)	N3—C7—H7	124.1
N1—C1—H1	118.5	N2—C7—H7	124.1
C2—C1—H1	118.5	C9—C8—N3	110.4 (3)
C3—C2—C1	118.6 (4)	C9—C8—H8	124.8
C3—C2—H2	120.7	N3—C8—H8	124.8
C1—C2—H2	120.7	C8—C9—N2	106.3 (3)
C2—C3—C4	118.8 (3)	C8—C9—H9	126.8
C2—C3—H3	120.6	N2—C9—H9	126.8
C4—C3—H3	120.6		
N3^{i}—Ag1—N1—C5	−41.1 (5)	C7—N2—C6—C5	−130.2 (3)
N3^{i}—Ag1—N1—C1	149.0 (4)	C9—N2—C6—C5	51.1 (4)
C5—N1—C1—C2	−1.4 (6)	N1—C5—C6—N2	41.2 (4)
Ag1—N1—C1—C2	169.3 (4)	C4—C5—C6—N2	−141.9 (3)
N1—C1—C2—C3	−0.4 (7)	C8—N3—C7—N2	−0.3 (4)
C1—C2—C3—C4	1.8 (7)	Ag1^{i}—N3—C7—N2	−174.26 (19)
C2—C3—C4—C5	−1.4 (7)	C9—N2—C7—N3	−0.2 (4)
C1—N1—C5—C4	1.8 (5)	C6—N2—C7—N3	−179.1 (3)
Ag1—N1—C5—C4	−167.8 (3)	C7—N3—C8—C9	0.6 (4)
C1—N1—C5—C6	178.6 (3)	Ag1^{i}—N3—C8—C9	175.1 (2)
Ag1—N1—C5—C6	9.0 (4)	N3—C8—C9—N2	−0.7 (4)
C3—C4—C5—N1	−0.4 (6)	C7—N2—C9—C8	0.6 (3)
C3—C4—C5—C6	−177.2 (4)	C6—N2—C9—C8	179.4 (3)

Symmetry codes: (i) −*x*+2, −*y*+1, −*z*+1.

Fig. 1

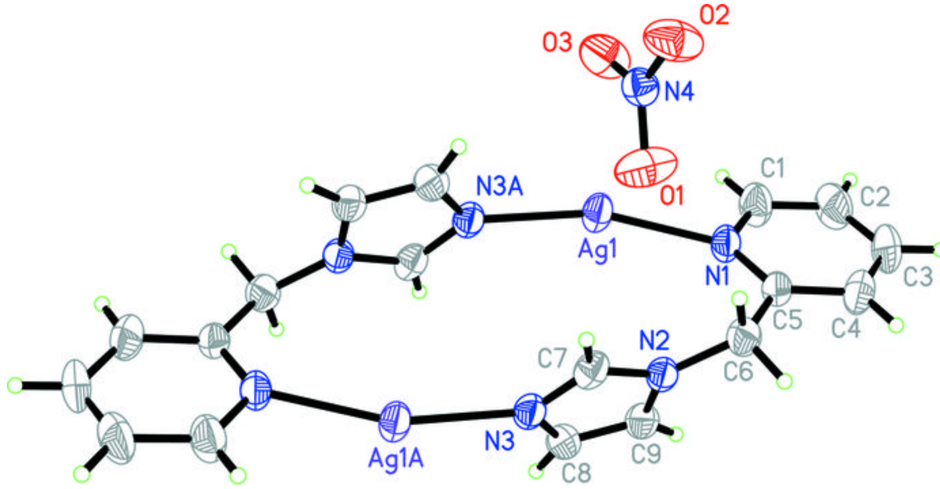

Fig. 2