μ-Biphenyl-4,4′-dicarboxylato-bis{[2-(piperidin-1-yl)ethanamine]silver(I)} dihydrate

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The title complex, $[Ag_2(C_{14}H_8O_4)(C_7H_{16}N_2)_2] \cdot 2H_2O$, consists of a biphenyl-4,4′-dicarboxylate-bridged centrosymmetric dinuclear silver(I) complex and two uncoordinated water molecules. The Ag atom is three-coordinated by two N atoms of 2-(piperidin-1-yl)ethanamine and one O atom of biphenyl-4,4′-dicarboxylate, forming a distorted T-shaped coordination environment, the distortion being caused by the strain created by the five-membered chelate Ag—N—C—C—N ring. In the crystal structure, molecules are linked through intermolecular N—H···O and O—H···O hydrogen bonds, forming layers parallel to the bc plane.

Related literature

For related literature, see: Kristiansson (2001); Melcer et al. (2001); Nomiya et al. (2000); Schultheiss et al. (2003); Smith et al. (1996); Wang & Mak (2003); Wei et al. (1998).
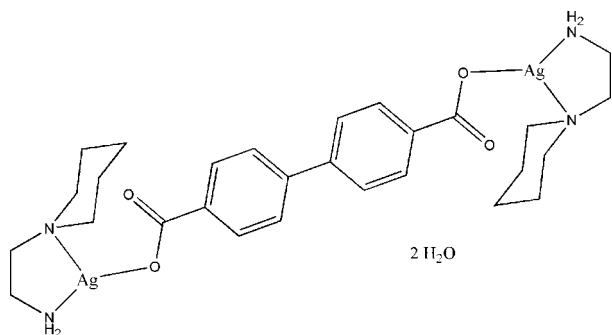
2 H₂O

Experimental

Crystal data

$[Ag_2(C_{14}H_8O_4)(C_7H_{16}N_2)_2] \cdot 2H_2O$
$M_r = 748.41$

Triclinic, $P\bar{1}$
$a = 7.2410$ (14) Å

$b = 8.3920$ (17) Å
$c = 13.154$ (3) Å
$\alpha = 82.95$ (3)°
$\beta = 76.61$ (3)°
$\gamma = 77.34$ (3)°
$V = 756.5$ (3) Å³

$Z = 1$
Mo $K\alpha$ radiation
$\mu = 1.34$ mm⁻¹
$T = 298$ (2) K
$0.40 \times 0.37 \times 0.32$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
$T_{min} = 0.616$, $T_{max} = 0.674$

6271 measured reflections
3279 independent reflections
2614 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.078$
$S = 1.06$
3279 reflections
187 parameters
3 restraints

H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max} = 0.44$ e Å⁻³
$\Delta\rho_{min} = -0.39$ e Å⁻³

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O2ⁱ	0.90	2.24	3.134 (4)	175
N2—H2B···O2ⁱⁱ	0.90	2.13	2.994 (4)	161
O3—H3A···O2	0.84 (3)	2.38 (2)	3.174 (4)	156 (4)
O3—H3B···O2ⁱⁱ	0.85 (3)	2.02 (3)	2.867 (4)	175 (4)

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

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 1998); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 1998); software used to prepare material for publication: SHELXTL.

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doi:10.1107/S160053680706103X

supplementary materials

μ-Biphenyl-4,4'-dicarboxylato-bis{[2-(piperidin-1-yl)ethanamine]silver(I)} dihydrate

C.-L. Yuan

Comment

The structural characterization of silver(I) complexes with carboxylate anions as counter-ions or ligands has attracted much interest in the last few years (Smith *et al.*, 1996; Wei *et al.*, 1998; Nomiya *et al.*, 2000; Kristiansson, 2001). As a further investigation of such silver(I) complexes, in this paper, a new silver(I) complex is reported.

The title complex consists of a biphenyl-4,4'-dicarboxylate bridged dinuclear silver(I) complex and two lattice water molecules. The Ag atom is three-coordinated by two N atoms of 2-piperidin-1-ylethylamine and one O atom of biphenyl-4,4'-dicarboxylate, forming a distorted T-shaped coordination environment (Fig. 1). The distortion of the T-shaped coordination is caused by the strain created by the five-membered chelate ring Ag1—N1—C9—C8—N2. All the coordination bond values (Table 1) are within normal ranges and comparable to the similar silver(I) complexes (Melcer *et al.*, 2001; Wang & Mak, 2003; Schultheiss *et al.*, 2003).

In the crystal, the lattice water molecules are linked to the silver(I) complex units through intermolecular hydrogen bonds O3–H3B···O2 and intramolecular hydrogen bonds O3–H3A···O2 (Table 2). The adjacent molecules are further linked through intermolecular hydrogen bonds N2–H2A···O2 and N2–H2B···O2, forming layers parallel to the *bc* plane (Fig. 2).

Experimental

Ag$_2$O (0.2 mmol, 46.3 mg) and biphenyl-4,4'-dicarboxylic acid (0.1 mmol, 24.2 mg) were dissolved in an ammonia solution (15 ml, 30%), and the mixture was stirred for 30 min at room temperature. To the above mixture was added with stirring a methanol solution (5 ml) of 2-piperidin-1-ylethylamine (0.2 mmol, 25.6 mg). The final mixture was further stirred for 30 min at room temperature. The resulting clear colorless solution was kept in dark for a week, yielding block-shaped colorless crystals. Analysis found: C 44.73, H 6.02, N 7.32%; calculated for C$_{28}$H$_{44}$Ag$_2$N$_4$O$_6$: C 44.94, H 5.93, N 7.49%. A broad absorption at 3412 cm^{-1} in the infrared spectrum is consistent with the presence of solvated water molecules.

Refinement

H3A and H3B were located from a difference Fourier map and refined isotropically, with O–H distances restrained to 0.85 (1) Å, H···H distance restrained to 1.37 (2) Å. Other H atoms were placed in idealized positions and constrained to ride on their parent atoms, with C–H distances in the range 0.93–0.97 Å, N–H distances of 0.90 Å, and with U_{iso}(H) = 1.2U_{eq}(C,N).

Figures
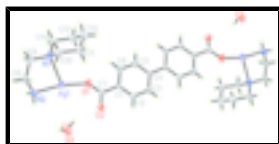
Fig. 1. The structure of the complex at the 30% probability level for non hydrogen atoms.
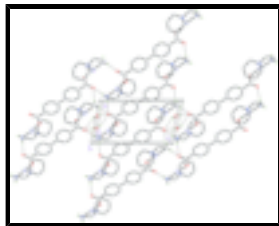
supplementary materials

 Fig. 2. Molecular packing of the complex.

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Crystal data

$[Ag_2(C_{14}H_8O_4)(C_7H_{16}N_2)_2] \cdot 2H_2O$	$Z = 1$
$M_r = 748.41$	$F_{000} = 382$
Triclinic, $P\bar{1}$	$D_x = 1.643$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 7.2410$ (14) Å	Cell parameters from 2907 reflections
$b = 8.3920$ (17) Å	$\theta = 2.4–27.3°$
$c = 13.154$ (3) Å	$\mu = 1.34$ mm^{-1}
$\alpha = 82.95$ (3)°	$T = 298$ (2) K
$\beta = 76.61$ (3)°	Block, colorless
$\gamma = 77.34$ (3)°	$0.40 \times 0.37 \times 0.32$ mm
$V = 756.5$ (3) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	3279 independent reflections
Radiation source: fine-focus sealed tube	2614 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.024$
$T = 298(2)$ K	$\theta_{max} = 27.5°$
ω scans	$\theta_{min} = 1.6°$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -9\rightarrow9$
$T_{min} = 0.616$, $T_{max} = 0.674$	$k = -10\rightarrow10$
6271 measured reflections	$l = -16\rightarrow16$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.036$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.078$	$w = 1/[\sigma^2(F_o^2) + (0.0338P)^2 + 0.0748P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} < 0.001$

3279 reflections

$\Delta\rho_{max} = 0.44$ e Å^{-3}

187 parameters

$\Delta\rho_{min} = -0.39$ e Å^{-3}

3 restraints

Extinction correction: none

Primary atom site location: structure-invariant direct methods

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.

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

	x	y	z	$U_{iso}*/U_{eq}$
Ag1	0.28889 (4)	0.34687 (3)	0.17052 (2)	0.05437 (12)
O1	0.4604 (3)	0.4290 (3)	0.26075 (17)	0.0610 (7)
O2	0.6870 (3)	0.5045 (3)	0.13123 (16)	0.0549 (6)
O3	0.3545 (6)	0.7009 (4)	0.0175 (3)	0.1041 (12)
N1	0.2171 (4)	0.0674 (3)	0.2252 (2)	0.0432 (6)
N2	0.1136 (4)	0.3156 (3)	0.0587 (2)	0.0477 (7)
H2A	−0.0067	0.3758	0.0766	0.057*
H2B	0.1673	0.3523	−0.0063	0.057*
C1	0.7356 (4)	0.4784 (3)	0.3058 (2)	0.0346 (6)
C2	0.8925 (4)	0.5553 (4)	0.2824 (2)	0.0394 (7)
H2	0.9298	0.6021	0.2149	0.047*
C3	0.9949 (4)	0.5637 (4)	0.3576 (2)	0.0411 (7)
H3	1.0996	0.6164	0.3396	0.049*
C4	0.9457 (4)	0.4958 (3)	0.4590 (2)	0.0358 (7)
C5	0.7885 (5)	0.4175 (5)	0.4813 (2)	0.0590 (10)
H5	0.7515	0.3695	0.5484	0.071*
C6	0.6863 (5)	0.4095 (5)	0.4064 (2)	0.0553 (9)
H6	0.5818	0.3564	0.4241	0.066*
C7	0.6205 (4)	0.4702 (4)	0.2251 (2)	0.0376 (7)
C8	0.1025 (6)	0.1431 (4)	0.0589 (3)	0.0595 (10)
H8A	0.2230	0.0846	0.0190	0.071*
H8B	−0.0011	0.1357	0.0254	0.071*
C9	0.0657 (5)	0.0650 (4)	0.1690 (3)	0.0591 (10)
H9A	−0.0572	0.1220	0.2075	0.071*
H9B	0.0552	−0.0477	0.1667	0.071*
C10	0.3912 (6)	−0.0542 (5)	0.1904 (3)	0.0592 (9)
H10A	0.4331	−0.0396	0.1148	0.071*

H10B	0.3630	−0.1633	0.2080	0.071*
C11	0.5512 (6)	−0.0368 (5)	0.2422 (3)	0.0723 (12)
H11A	0.6654	−0.1194	0.2192	0.087*
H11B	0.5845	0.0700	0.2209	0.087*
C12	0.4900 (7)	−0.0554 (6)	0.3597 (4)	0.0893 (15)
H12A	0.5885	−0.0313	0.3912	0.107*
H12B	0.4760	−0.1674	0.3821	0.107*
C13	0.2999 (7)	0.0599 (6)	0.3957 (3)	0.0850 (14)
H13A	0.2542	0.0372	0.4703	0.102*
H13B	0.3205	0.1716	0.3842	0.102*
C14	0.1473 (6)	0.0441 (5)	0.3391 (3)	0.0666 (11)
H14A	0.1148	−0.0635	0.3569	0.080*
H14B	0.0310	0.1254	0.3610	0.080*
H3A	0.413 (6)	0.643 (4)	0.062 (2)	0.080*
H3B	0.335 (6)	0.640 (4)	−0.024 (2)	0.080*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ag1	0.0592 (2)	0.06183 (18)	0.05765 (18)	−0.02704 (14)	−0.03305 (15)	0.00279 (12)
O1	0.0478 (15)	0.106 (2)	0.0438 (13)	−0.0359 (14)	−0.0163 (12)	−0.0092 (13)
O2	0.0590 (16)	0.0863 (17)	0.0314 (12)	−0.0338 (13)	−0.0173 (11)	0.0001 (11)
O3	0.129 (3)	0.093 (2)	0.107 (3)	0.009 (2)	−0.069 (2)	−0.040 (2)
N1	0.0429 (16)	0.0449 (14)	0.0444 (15)	−0.0102 (13)	−0.0132 (13)	−0.0027 (12)
N2	0.0488 (17)	0.0587 (17)	0.0404 (14)	−0.0154 (14)	−0.0180 (13)	0.0033 (12)
C1	0.0347 (17)	0.0418 (16)	0.0311 (14)	−0.0075 (13)	−0.0121 (13)	−0.0068 (12)
C2	0.0435 (19)	0.0506 (18)	0.0279 (14)	−0.0148 (15)	−0.0113 (13)	−0.0006 (13)
C3	0.0407 (19)	0.0540 (18)	0.0365 (16)	−0.0203 (15)	−0.0144 (14)	−0.0019 (14)
C4	0.0384 (18)	0.0431 (16)	0.0307 (15)	−0.0115 (14)	−0.0120 (13)	−0.0059 (12)
C5	0.067 (2)	0.093 (3)	0.0335 (16)	−0.049 (2)	−0.0215 (17)	0.0124 (17)
C6	0.055 (2)	0.085 (3)	0.0417 (18)	−0.042 (2)	−0.0203 (17)	0.0043 (17)
C7	0.0371 (18)	0.0427 (16)	0.0382 (16)	−0.0074 (14)	−0.0158 (14)	−0.0084 (13)
C8	0.068 (3)	0.062 (2)	0.063 (2)	−0.0134 (19)	−0.035 (2)	−0.0128 (18)
C9	0.055 (2)	0.051 (2)	0.082 (3)	−0.0179 (17)	−0.029 (2)	−0.0004 (18)
C10	0.060 (3)	0.059 (2)	0.056 (2)	−0.0015 (19)	−0.0151 (19)	−0.0090 (17)
C11	0.049 (2)	0.080 (3)	0.087 (3)	0.000 (2)	−0.029 (2)	0.006 (2)
C12	0.087 (4)	0.107 (4)	0.080 (3)	−0.017 (3)	−0.049 (3)	0.024 (3)
C13	0.102 (4)	0.113 (4)	0.044 (2)	−0.025 (3)	−0.024 (2)	0.004 (2)
C14	0.063 (3)	0.075 (3)	0.051 (2)	−0.011 (2)	−0.002 (2)	0.0107 (19)

Geometric parameters (Å, °)

Ag1—O1	2.164 (2)	C5—C6	1.378 (4)
Ag1—N2	2.225 (2)	C5—H5	0.9300
Ag1—N1	2.497 (3)	C6—H6	0.9300
O1—C7	1.252 (4)	C8—C9	1.504 (5)
O2—C7	1.240 (4)	C8—H8A	0.9700
O3—H3A	0.84 (3)	C8—H8B	0.9700
O3—H3B	0.85 (3)	C9—H9A	0.9700

N1—C10	1.457 (5)	C9—H9B	0.9700
N1—C9	1.462 (4)	C10—C11	1.514 (5)
N1—C14	1.469 (4)	C10—H10A	0.9700
N2—C8	1.468 (4)	C10—H10B	0.9700
N2—H2A	0.9000	C11—C12	1.505 (6)
N2—H2B	0.9000	C11—H11A	0.9700
C1—C6	1.376 (4)	C11—H11B	0.9700
C1—C2	1.383 (4)	C12—C13	1.510 (7)
C1—C7	1.510 (4)	C12—H12A	0.9700
C2—C3	1.384 (4)	C12—H12B	0.9700
C2—H2	0.9300	C13—C14	1.505 (6)
C3—C4	1.383 (4)	C13—H13A	0.9700
C3—H3	0.9300	C13—H13B	0.9700
C4—C5	1.393 (4)	C14—H14A	0.9700
C4—C4^i	1.490 (5)	C14—H14B	0.9700
O1—Ag1—N2	167.47 (10)	C9—C8—H8A	109.5
O1—Ag1—N1	115.68 (9)	N2—C8—H8B	109.5
N2—Ag1—N1	76.85 (9)	C9—C8—H8B	109.5
C7—O1—Ag1	125.81 (19)	H8A—C8—H8B	108.1
H3A—O3—H3B	110 (2)	N1—C9—C8	113.4 (3)
C10—N1—C9	111.7 (3)	N1—C9—H9A	108.9
C10—N1—C14	110.6 (3)	C8—C9—H9A	108.9
C9—N1—C14	111.3 (3)	N1—C9—H9B	108.9
C10—N1—Ag1	109.0 (2)	C8—C9—H9B	108.9
C9—N1—Ag1	102.13 (18)	H9A—C9—H9B	107.7
C14—N1—Ag1	111.9 (2)	N1—C10—C11	110.2 (3)
C8—N2—Ag1	111.07 (19)	N1—C10—H10A	109.6
C8—N2—H2A	109.4	C11—C10—H10A	109.6
Ag1—N2—H2A	109.4	N1—C10—H10B	109.6
C8—N2—H2B	109.4	C11—C10—H10B	109.6
Ag1—N2—H2B	109.4	H10A—C10—H10B	108.1
H2A—N2—H2B	108.0	C12—C11—C10	111.2 (4)
C6—C1—C2	117.6 (2)	C12—C11—H11A	109.4
C6—C1—C7	120.6 (3)	C10—C11—H11A	109.4
C2—C1—C7	121.8 (2)	C12—C11—H11B	109.4
C1—C2—C3	121.1 (3)	C10—C11—H11B	109.4
C1—C2—H2	119.5	H11A—C11—H11B	108.0
C3—C2—H2	119.5	C11—C12—C13	110.0 (3)
C4—C3—C2	121.8 (3)	C11—C12—H12A	109.7
C4—C3—H3	119.1	C13—C12—H12A	109.7
C2—C3—H3	119.1	C11—C12—H12B	109.7
C3—C4—C5	116.5 (2)	C13—C12—H12B	109.7
C3—C4—C4^i	122.4 (3)	H12A—C12—H12B	108.2
C5—C4—C4^i	121.1 (3)	C14—C13—C12	112.4 (4)
C6—C5—C4	121.7 (3)	C14—C13—H13A	109.1
C6—C5—H5	119.2	C12—C13—H13A	109.1
C4—C5—H5	119.2	C14—C13—H13B	109.1
C1—C6—C5	121.3 (3)	C12—C13—H13B	109.1

C1—C6—H6	119.3	H13A—C13—H13B	107.9
C5—C6—H6	119.3	N1—C14—C13	110.4 (3)
O2—C7—O1	125.1 (3)	N1—C14—H14A	109.6
O2—C7—C1	119.5 (3)	C13—C14—H14A	109.6
O1—C7—C1	115.3 (3)	N1—C14—H14B	109.6
N2—C8—C9	110.8 (3)	C13—C14—H14B	109.6
N2—C8—H8A	109.5	H14A—C14—H14B	108.1

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

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O2[ii]	0.90	2.24	3.134 (4)	175
N2—H2B···O2[iii]	0.90	2.13	2.994 (4)	161
O3—H3A···O2	0.84 (3)	2.38 (2)	3.174 (4)	156 (4)
O3—H3B···O2[iii]	0.85 (3)	2.02 (3)	2.867 (4)	175 (4)

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

Fig. 1

Fig. 2