Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis[μ-1-(2-pyridylmethyl)-1*H*-benzotriazole]disilver(I) bis(perchlorate)

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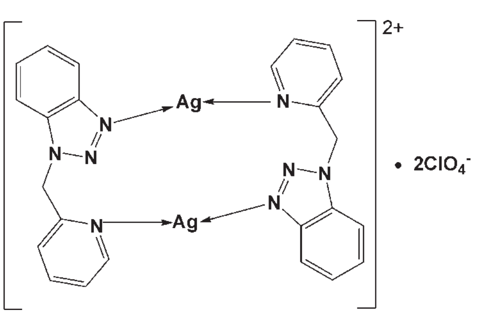
Received 14 October 2009; accepted 22 October 2009

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.012 Å; R factor = 0.043; wR factor = 0.137; data-to-parameter ratio = 12.5.

In the title centrosymmetric binuclear Ag^I complex, $[Ag_2(C_{12}H_{10}N_4)_2](ClO_4)_2$, each Ag^I center is two-coordinated by one pyridine and one benzotriazole *N*-donor atom of two inversion-related 1-(2-pyridylmethyl)-1*H*-benzotriazole (*L*) ligands. This forms a unique box-like cyclic dimer with an intramolecular Ag$\cdots$Ag separation of 4.479 (2) Å. Intermolecular C—H$\cdots$O hydrogen-bonding interactions, involving uncoordinated ClO_4^- ions, link the binuclear units, forming a two-dimensional network parallel to $(10\overline{2})$.

Related literature

Bis-heterocyclic chelating or bridging ligands have been used extensively to construct functional coordination complexes that contain different hetero-aromatic ring systems, see: Constable (1989); Constable & Steel (1989); Steel (2005). For related structures, see: Hu *et al.* (2008); Huang *et al.* (2008); Liu *et al.* (2006, 2007); Liu, Sun *et al.* (2008); Liu, Zhou *et al.* (2008); Richardson & Steel (2003). For the synthesis of ligand *L*, see: Liu, Sun *et al.* (2008).



Experimental

Crystal data

$[Ag_2(C_{12}H_{10}N_4)_2](ClO_4)_2$
M_r = 835.12
Monoclinic, $P2_1/c$
a = 9.4273 (4) Å
b = 16.0863 (7) Å
c = 11.9152 (7) Å
β = 128.448 (3)°
V = 1415.15 (13) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.64 mm^{-1}
T = 293 K
0.24 × 0.23 × 0.03 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.695, T_{max} = 0.960
9388 measured reflections
2484 independent reflections
1658 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.137
S = 1.08
2484 reflections
199 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.62 e Å^{-3}
$\Delta\rho_{min}$ = −0.53 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ag1—N2^i	2.159 (5)	Ag1—N1	2.201 (5)
N2^i—Ag1—N1	155.9 (2)		

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

Table 2
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C1—H11$\cdots$O3^i	0.97	2.31	3.264 (15)	168
C1—H12$\cdots$O2^{ii}	0.97	2.58	3.415 (11)	144
C3—H3$\cdots$O1^{ii}	0.93	2.60	3.512 (10)	168
C11—H1$\cdots$O1^{ii}	0.93	2.56	3.481 (11)	170

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

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

This work was supported by the start-up fund for PhDs in Natural Scientific Research of Zhengzhou University of Light Industry (No. 2007BSJJ001 to CSL).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZQ2014).

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supplementary materials

Acta Cryst. (2009). E**65**, m1453-m1454 [**doi:10.1107/S1600536809043669**]

Bis[μ-1-(2-pyridylmethyl)-1*H*-benzotriazole]disilver(I) bis(perchlorate)

C.-S. Liu, X.-G. Xiao and M. Hu

Comment

Numerous related bis-heterocyclic chelating or bridging ligands have been synthesized and used extensively to construct functional coordination complexes that contain different hetero-aromatic ring systems, for example, pyridine, pyrazine, quinoline, quinoxaline, pyrazole, imidazole, thiazoles and their benzo analogues (Constable, 1989; Constable & Steel, 1989; Steel, 2005). The structures of five N-containing bis-heterocyclic ligands bearing 1-substituted benzotriazole subunits, such as 1-(2-pyridylmethyl)-1*H*-benzotriazole and its Ru^{II}, Cu^{II}, Pd^{II}, Ag^{I}, Zn^{II} and Hg^{II} complexes, have been well documented previously (Huang *et al.* 2008; Liu, Zhou *et al.* 2008; Richardson & Steel, 2003). In our previous work, to further investigate the influences of the N-donor spatial position of pendant pyridyl group in structurally related benzotriazol-1-yl-based pyridyl ligands on the structures of their coordination complexes, two new N-containing heterocyclic ligands 1-(4-pyridylmethyl)-1*H*-benzotriazole (4-pbt) and 2-(3-pyridylmethyl)-2*H*-benzotriazole (3-pbt) were designed and prepared. Their reaction with $AgNO_3$ offered an one-dimensional double helical coordination polymer $\{[Ag(4\text{-pbt})](NO_3)\}_\infty$ and a centrosymmetric binuclear complex $[Ag_2(3\text{-pbt})_2 (NO_3)_2]$, respectively (Hu *et al.* 2008; Liu, Sun *et al.* 2008). To further investigate the influence of different counter-anions on the self-assembly process of coordination complexes, we chose to use *L* to construct new functional Ag^{I} complexes through its reaction with $AgClO_4$. Here we report the crystal structure of $[Ag(L)]_2(ClO_4)_2$.

The structure of the title compound (I) consists of a centrosymmetric binuclear $[Ag(L)]_2^{2+}$ unit and two uncoordinated ClO_4^- ions. The binuclear $[Ag(L)]_2^{2+}$ cation (Fig. 1) comprises two *L* ligands and two Ag^{I} centers. The intramolecular non-bonding Ag···Ag separation is 4.479 (2) Å. There is only one crystallographic independent Ag^{I} center, which is two coordinated by two N-atom donors, one N donor being from benzotriazole ring of one *L* ligand and the other being from pyridyl ring of another *L* ligand. In this case the 14-membered dimetallocyclic ring is far from planar as a result of the presence of the tetrahedral methylene group of the *L* ligand. All the Ag—N bond distances are in the normal range found for similar complexes (Liu *et al.*, 2006; Liu *et al.*, 2007).

In the crystal structure adjacent discrete binuclear $[Ag(L)]_2^{2+}$ units are assembled into one-dimensional chains by intermolecular C—H···O hydrogen-bonding interactions between the *L* ligands and the uncoordinated ClO_4^- (Table 2). The net result is a two-dimensional network running parallel to the $(10\overline{2})$ plane (Fig. 2). In addition, the crystal structure of (I) also contains intermolecular face-to-face π···π stacking interactions between the pyridyl ring involving N1/C2/C3/C4/C5/C6 (centroid *Cg*1) and N1A/C2A/C3A/C4A/C5A/C6A (centroid *Cg*2) of distinct *L* ligands [the centroid–centroid separation being 3.685 (1) Å, symmetry code A: $-x$, $-y + 1$, $-z + 1$], that interlink the two-dimensional sheets to form a three-dimensional framework.

Experimental

1-(2-Pyridylmethyl)-1*H*-benzotriazole (*L*) was synthesized according to the literature procedure of Liu, Sun *et al.* (2008). Complex (I) was prepared by adding a solution of *L* (0.05 mmol) in CH_3OH (10 ml) on top of an aqueous solution (15 ml) of $AgClO_4$ (0.1 mmol) in a test tube. Yellow single crystals suitable for X-ray structural analysis appeared at the tube wall after *ca* one month at room temperature (yield ~30% based on *L*). Elemental analysis calculated for ($C_{24}H_{20}Ag_2Cl_2N_8O_8$): H 2.41, C 34.52, N 13.42%; found: H 2.30, C 34.67, N 13.36%.

Refinement

H atoms were included in calculated positions and treated in the subsequent refinement as riding atoms, with C—H = 0.93 (aromatic) or 0.97 Å (methylene), with U_{iso}(H) = 1.2 U_{eq}(C).

Figures

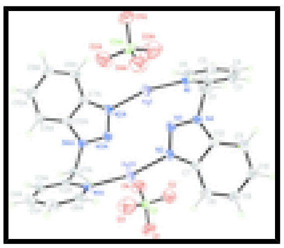

Fig. 1. The molecular structure of complex (I). Displacement ellipsoids are drawn at the 30% probability level. Atoms labelled with the suffix A are generated by the symmetry operation ($-x + 1$, $-y + 1$, $-z + 1$).

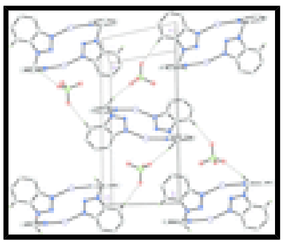

Fig. 2. A view of the two-dimensional network of complex (I), running parallel to the (10$\bar{2}$) plane, formed by the intermolecular C—H···O (fine dashed lines) interactions. For clarity, only H atoms involved in the interactions are shown.

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Crystal data

$[Ag_2(C_{12}H_{10}N_4)_2](ClO_4)_2$	F_{000} = 824
M_r = 835.12	D_x = 1.960 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 3182 reflections
a = 9.4273 (4) Å	θ = 3.0–26.3º
b = 16.0863 (7) Å	μ = 1.64 mm^{-1}
c = 11.9152 (7) Å	*T* = 293 K
β = 128.448 (3)º	Block, yellow
V = 1415.15 (13) Å^3	0.24 × 0.23 × 0.03 mm
Z = 2	

Data collection

Bruker SMART CCD area-detector diffractometer	2484 independent reflections
Radiation source: fine-focus sealed tube	1658 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.037$
$T = 293$ K	$\theta_{max} = 25.0°$
φ and ω scans	$\theta_{min} = 3.0°$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -11 \rightarrow 11$
$T_{min} = 0.695$, $T_{max} = 0.960$	$k = -15 \rightarrow 19$
9388 measured reflections	$l = -13 \rightarrow 14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.043$	H-atom parameters constrained
$wR(F^2) = 0.137$	$w = 1/[\sigma^2(F_o^2) + (0.0614P)^2 + 2.0392P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.08$	$(\Delta/\sigma)_{max} = 0.001$
2484 reflections	$\Delta\rho_{max} = 0.62$ e Å^{-3}
199 parameters	$\Delta\rho_{min} = -0.53$ 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.

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

	x	y	z	U_{iso}*/U_{eq}
Ag1	0.42617 (7)	0.60201 (4)	0.58311 (7)	0.0599 (3)
N1	0.1418 (7)	0.6101 (3)	0.4978 (6)	0.0368 (13)
N2	0.3065 (6)	0.4519 (4)	0.2962 (5)	0.0385 (14)
N3	0.2868 (7)	0.5274 (4)	0.3285 (6)	0.0407 (14)
N4	0.1091 (6)	0.5452 (3)	0.2391 (5)	0.0347 (13)

C1	0.0453 (9)	0.6239 (4)	0.2566 (7)	0.0403 (17)
H11	0.1386	0.6658	0.2933	0.048*
H12	−0.0612	0.6425	0.1637	0.048*
C2	−0.0004 (9)	0.6159 (4)	0.3575 (7)	0.0325 (15)
C3	−0.1725 (9)	0.6158 (4)	0.3083 (8)	0.0435 (18)
H3	−0.2665	0.6190	0.2102	0.052*
C4	−0.2110 (10)	0.6110 (4)	0.4035 (9)	0.051 (2)
H4	−0.3297	0.6105	0.3711	0.062*
C5	−0.0673 (11)	0.6068 (4)	0.5464 (9)	0.052 (2)
H5	−0.0869	0.6050	0.6138	0.063*
C6	0.1070 (10)	0.6055 (4)	0.5901 (8)	0.0472 (18)
H6	0.2035	0.6012	0.6874	0.057*
C7	0.1415 (8)	0.4210 (4)	0.1852 (6)	0.0318 (15)
C8	0.0907 (9)	0.3446 (4)	0.1149 (7)	0.0400 (17)
H8	0.1757	0.3039	0.1390	0.048*
C9	−0.0903 (10)	0.3326 (5)	0.0089 (8)	0.0500 (19)
H9	−0.1290	0.2828	−0.0419	0.060*
C10	−0.2202 (10)	0.3928 (5)	−0.0261 (8)	0.052 (2)
H10	−0.3421	0.3811	−0.0980	0.062*
C11	−0.1737 (8)	0.4676 (5)	0.0417 (7)	0.0407 (17)
H1	−0.2595	0.5076	0.0183	0.049*
C12	0.0111 (8)	0.4803 (4)	0.1486 (6)	0.0310 (14)
Cl1	0.4844 (2)	0.21950 (12)	0.5792 (2)	0.0510 (5)
O1	0.4745 (9)	0.1317 (4)	0.5670 (7)	0.089 (2)
O2	0.3143 (9)	0.2532 (5)	0.4653 (7)	0.116 (3)
O3	0.6166 (13)	0.2454 (6)	0.5735 (14)	0.168 (5)
O4	0.5263 (10)	0.2432 (5)	0.7115 (7)	0.109 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ag1	0.0348 (3)	0.0672 (5)	0.0583 (4)	0.0086 (3)	0.0195 (3)	−0.0078 (3)
N1	0.041 (3)	0.034 (3)	0.038 (3)	0.002 (2)	0.026 (3)	−0.003 (3)
N2	0.033 (3)	0.047 (4)	0.032 (3)	0.007 (3)	0.019 (2)	0.002 (3)
N3	0.037 (3)	0.045 (4)	0.035 (3)	0.004 (3)	0.019 (3)	−0.002 (3)
N4	0.037 (3)	0.036 (3)	0.034 (3)	0.006 (2)	0.023 (2)	0.000 (3)
C1	0.059 (4)	0.026 (4)	0.040 (4)	0.006 (3)	0.033 (4)	0.004 (3)
C2	0.044 (4)	0.017 (3)	0.032 (3)	0.003 (3)	0.021 (3)	−0.003 (3)
C3	0.039 (4)	0.046 (5)	0.038 (4)	0.008 (3)	0.020 (3)	0.004 (3)
C4	0.050 (4)	0.049 (5)	0.064 (5)	0.010 (3)	0.040 (4)	0.008 (4)
C5	0.074 (6)	0.046 (5)	0.061 (5)	0.012 (4)	0.054 (5)	0.012 (4)
C6	0.057 (4)	0.036 (4)	0.043 (4)	0.008 (3)	0.028 (4)	0.001 (3)
C7	0.036 (3)	0.034 (4)	0.026 (3)	0.004 (3)	0.020 (3)	0.006 (3)
C8	0.055 (4)	0.032 (4)	0.043 (4)	0.005 (3)	0.036 (4)	0.001 (3)
C9	0.058 (5)	0.036 (4)	0.052 (5)	−0.011 (4)	0.032 (4)	−0.009 (4)
C10	0.043 (4)	0.051 (5)	0.050 (4)	−0.011 (4)	0.023 (4)	−0.001 (4)
C11	0.033 (3)	0.050 (5)	0.037 (4)	0.005 (3)	0.021 (3)	0.008 (3)
C12	0.039 (3)	0.028 (4)	0.032 (4)	0.000 (3)	0.025 (3)	0.002 (3)

Cl1	0.0455 (9)	0.0446 (11)	0.0560 (12)	−0.0073 (8)	0.0281 (9)	−0.0116 (9)
O1	0.094 (4)	0.051 (4)	0.090 (5)	−0.012 (3)	0.041 (4)	−0.011 (3)
O2	0.098 (5)	0.117 (6)	0.066 (5)	0.042 (5)	0.018 (4)	−0.015 (4)
O3	0.171 (8)	0.115 (7)	0.317 (14)	−0.064 (6)	0.201 (10)	−0.063 (8)
O4	0.114 (5)	0.116 (6)	0.060 (4)	0.019 (5)	0.037 (4)	−0.028 (4)

Geometric parameters (Å, °)

Ag1—N2^{i}	2.159 (5)	C5—C6	1.381 (11)
Ag1—N1	2.201 (5)	C5—H5	0.9300
N1—C6	1.332 (9)	C6—H6	0.9300
N1—C2	1.347 (8)	C7—C12	1.394 (8)
N2—N3	1.322 (8)	C7—C8	1.394 (9)
N2—C7	1.362 (8)	C8—C9	1.365 (9)
N2—Ag1^{i}	2.159 (5)	C8—H8	0.9301
N3—N4	1.344 (7)	C9—C10	1.406 (10)
N4—C12	1.367 (8)	C9—H9	0.9300
N4—C1	1.470 (8)	C10—C11	1.361 (10)
C1—C2	1.514 (10)	C10—H10	0.9300
C1—H11	0.9698	C11—C12	1.393 (8)
C1—H12	0.9699	C11—H1	0.9300
C2—C3	1.339 (9)	Cl1—O3	1.355 (7)
C3—C4	1.392 (11)	Cl1—O2	1.414 (6)
C3—H3	0.9300	Cl1—O1	1.417 (6)
C4—C5	1.367 (11)	Cl1—O4	1.418 (7)
C4—H4	0.9299		
N2^{i}—Ag1—N1	155.9 (2)	C6—C5—H5	120.2
C6—N1—C2	117.5 (6)	N1—C6—C5	122.3 (7)
C6—N1—Ag1	118.1 (5)	N1—C6—H6	118.8
C2—N1—Ag1	124.3 (5)	C5—C6—H6	118.9
N3—N2—C7	109.6 (5)	N2—C7—C12	107.9 (6)
N3—N2—Ag1^{i}	119.5 (4)	N2—C7—C8	131.5 (6)
C7—N2—Ag1^{i}	131.0 (4)	C12—C7—C8	120.6 (6)
N2—N3—N4	107.3 (5)	C9—C8—C7	116.4 (6)
N3—N4—C12	111.1 (5)	C9—C8—H8	121.8
N3—N4—C1	119.3 (5)	C7—C8—H8	121.8
C12—N4—C1	129.4 (5)	C8—C9—C10	122.3 (7)
N4—C1—C2	112.5 (5)	C8—C9—H9	118.9
N4—C1—H11	109.0	C10—C9—H9	118.8
C2—C1—H11	109.1	C11—C10—C9	122.2 (6)
N4—C1—H12	109.2	C11—C10—H10	118.9
C2—C1—H12	109.1	C9—C10—H10	118.9
H11—C1—H12	107.8	C10—C11—C12	115.5 (6)
C3—C2—N1	123.0 (6)	C10—C11—H1	122.3
C3—C2—C1	121.2 (6)	C12—C11—H1	122.2
N1—C2—C1	115.9 (6)	N4—C12—C11	132.9 (6)
C2—C3—C4	120.1 (7)	N4—C12—C7	104.1 (5)
C2—C3—H3	119.9	C11—C12—C7	123.0 (6)

C4—C3—H3	119.9	O3—Cl1—O2	111.3 (7)
C5—C4—C3	117.3 (7)	O3—Cl1—O1	107.7 (5)
C5—C4—H4	121.4	O2—Cl1—O1	108.7 (4)
C3—C4—H4	121.2	O3—Cl1—O4	110.1 (6)
C4—C5—C6	119.7 (7)	O2—Cl1—O4	109.2 (4)
C4—C5—H5	120.1	O1—Cl1—O4	109.7 (5)

$N2^{i}$—Ag1—N1—C6	53.0 (7)	C4—C5—C6—N1	1.5 (11)
$N2^{i}$—Ag1—N1—C2	−124.0 (6)	N3—N2—C7—C12	−1.4 (7)
C7—N2—N3—N4	0.4 (7)	$Ag1^{i}$—N2—C7—C12	178.4 (4)
$Ag1^{i}$—N2—N3—N4	−179.4 (4)	N3—N2—C7—C8	−178.6 (7)
N2—N3—N4—C12	0.8 (7)	$Ag1^{i}$—N2—C7—C8	1.1 (11)
N2—N3—N4—C1	176.1 (5)	N2—C7—C8—C9	178.0 (7)
N3—N4—C1—C2	−89.7 (7)	C12—C7—C8—C9	1.1 (10)
C12—N4—C1—C2	84.6 (8)	C7—C8—C9—C10	−1.5 (11)
C6—N1—C2—C3	−1.1 (9)	C8—C9—C10—C11	1.2 (12)
Ag1—N1—C2—C3	175.9 (5)	C9—C10—C11—C12	−0.3 (11)
C6—N1—C2—C1	178.0 (5)	N3—N4—C12—C11	177.9 (7)
Ag1—N1—C2—C1	−4.9 (7)	C1—N4—C12—C11	3.3 (11)
N4—C1—C2—C3	−106.9 (7)	N3—N4—C12—C7	−1.6 (7)
N4—C1—C2—N1	74.0 (7)	C1—N4—C12—C7	−176.2 (6)
N1—C2—C3—C4	1.1 (10)	C10—C11—C12—N4	−179.6 (7)
C1—C2—C3—C4	−178.0 (6)	C10—C11—C12—C7	−0.2 (10)
C2—C3—C4—C5	0.2 (11)	N2—C7—C12—N4	1.8 (7)
C3—C4—C5—C6	−1.4 (11)	C8—C7—C12—N4	179.4 (6)
C2—N1—C6—C5	−0.2 (10)	N2—C7—C12—C11	−177.8 (6)
Ag1—N1—C6—C5	−177.4 (5)	C8—C7—C12—C11	−0.2 (10)

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H11···$O3^{i}$	0.97	2.31	3.264 (15)	168
C1—H12···$O2^{ii}$	0.97	2.58	3.415 (11)	144
C3—H3···$O1^{ii}$	0.93	2.60	3.512 (10)	168
C11—H1···$O1^{ii}$	0.93	2.56	3.481 (11)	170

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

Fig. 1

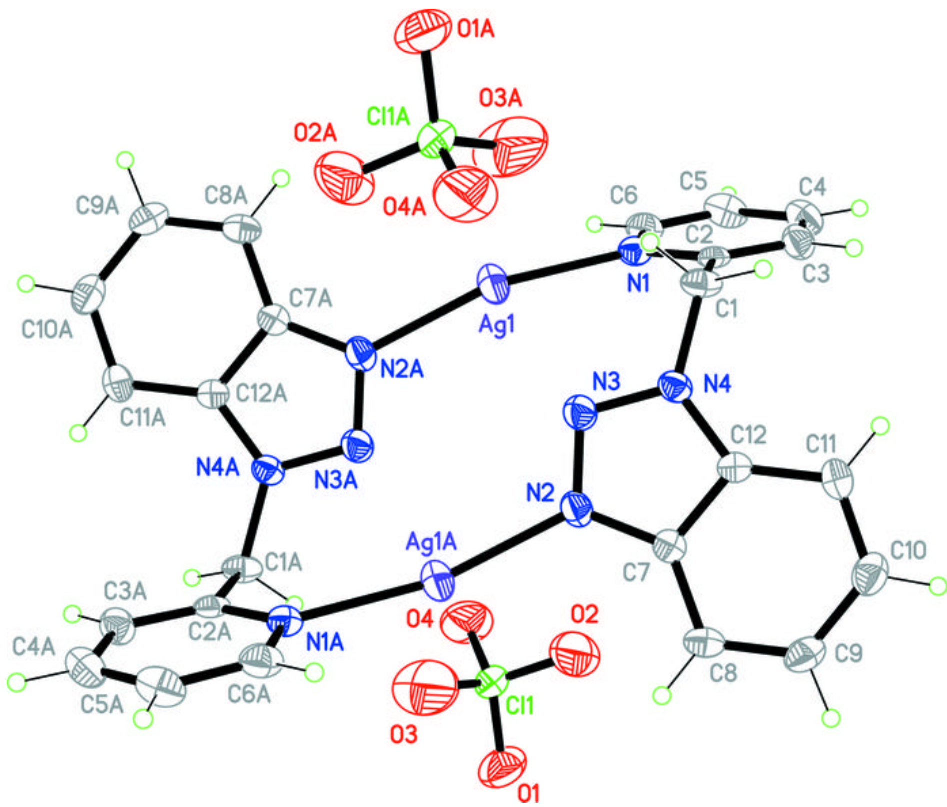
O1A
O3A
Cl1A
O2A
O4A
C9A
C8A
C6
C5
C4
C2
C3
N1
C1
C10A
C7A
Ag1
N2A
N3
N4
C12A
C11A
C12
C11
N4A
N3A
N2
C1A
Ag1A
C10
C7
C3A
C2A
N1A
O4
O2
C4A
C6A
C8
C9
C5A
Cl1
O3
O1

Fig. 2