

## Bis(1,10-phenanthroline-5,6-dione- $\kappa^2N,N'$ )silver(I) perchlorate

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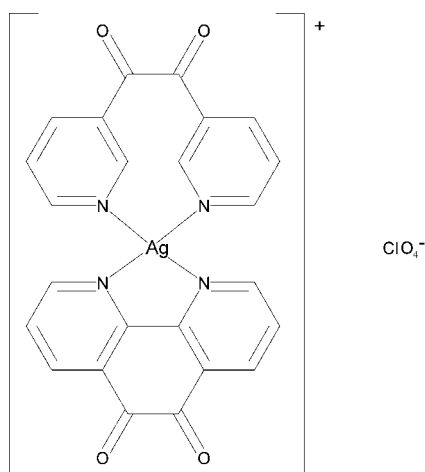
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Key indicators: single-crystal X-ray study;  $T = 93$  K; mean  $\sigma(C-C) = 0.007$  Å;  $R$  factor = 0.066;  $wR$  factor = 0.184; data-to-parameter ratio = 17.5.

At 93 K, in the structure of a new polymorph of  $[AgL_2]^+$  ( $ClO_4^-$  ( $L = 1,10$ -phenanthroline-5,6-dione) or  $[Ag(C_{12}H_6N_2O_2)_2]ClO_4$ , there is a complete formula unit in the asymmetric unit. The dihedral angle between the two phendione ligands is  $36.7(2)^\circ$ . The geometry about the Ag atom is distorted tetrahedral. There are short contacts between the perchlorate anion and the phendione ligands [ $O \cdots C = 2.845(5)$  Å], as well as unusual and different  $O-C-C-O$  torsion angles for the two phendione ligands [ $-21.9(7)$  and  $-5.0(7)^\circ$ ], which reflects the fact that in this polymorph there is no crystallographically imposed symmetry in the cation.

### Related literature

For related literature, see: Allen (2002); Galet *et al.* (2005); Leschke *et al.* (2002); McCann *et al.* (2004); Pallenberg *et al.* (1997); Paramonov *et al.* (2003); Titze *et al.* (1997); Wen *et al.* (2006).



### Experimental

#### Crystal data

$[Ag(C_{12}H_6N_2O_2)_2]ClO_4$	$\gamma = 97.378(4)^\circ$
$M_r = 627.70$	$V = 1097.8(5)$ Å <sup>3</sup>
Triclinic, $P\bar{1}$	$Z = 2$
$a = 8.493(2)$ Å	Mo $K\alpha$ radiation
$b = 11.260(3)$ Å	$\mu = 1.10$ mm <sup>-1</sup>
$c = 13.190(4)$ Å	$T = 93(2)$ K
$\alpha = 112.525(3)^\circ$	$0.38 \times 0.32 \times 0.12$ mm
$\beta = 103.682(4)^\circ$	

#### Data collection

Bruker APEX II CCD area-detector diffractometer	11431 measured reflections
Absorption correction: multi-scan (SADABS; Bruker, 2000)	6019 independent reflections
$T_{min} = 0.679$ , $T_{max} = 0.879$	4791 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.067$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.066$	343 parameters
$wR(F^2) = 0.184$	H-atom parameters constrained
$S = 1.07$	$\Delta\rho_{max} = 2.36$ e Å <sup>-3</sup>
6019 reflections	$\Delta\rho_{min} = -1.63$ e Å <sup>-3</sup>

Table 1

Selected geometric parameters (Å, °).

Ag—N2A	2.261(3)	Ag—N2B	2.349(3)
Ag—N1B	2.331(4)	Ag—N1A	2.453(4)
N2A—Ag—N1B	129.69(12)	N2A—Ag—N1A	70.77(11)
N2A—Ag—N2B	158.05(13)	N1B—Ag—N1A	144.28(12)
N1B—Ag—N2B	71.16(12)	N2B—Ag—N1A	95.79(12)

Table 2

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
C8A—H8AA $\cdots$ O1B <sup>i</sup>	0.93	2.54	3.170(5)	125
C1B—H1BA $\cdots$ O14 <sup>ii</sup>	0.93	2.50	3.380(5)	157
C8B—H8BA $\cdots$ O11 <sup>i</sup>	0.93	2.58	3.155(6)	121
C1A—H1AA $\cdots$ O2B <sup>iii</sup>	0.93	2.47	3.159(5)	131
C6B—H6BA $\cdots$ O2A <sup>iv</sup>	0.93	2.47	3.203(5)	136
C6A—H6AA $\cdots$ O13 <sup>v</sup>	0.93	2.45	3.235(6)	142
C8B—H8BA $\cdots$ O1A <sup>vi</sup>	0.93	2.48	3.158(6)	130
C2A—H2AA $\cdots$ O12 <sup>vii</sup>	0.93	2.55	3.268(6)	134
C3A—H3AA $\cdots$ O11 <sup>vii</sup>	0.93	2.57	3.464(7)	162

Symmetry codes: (i)  $-x, -y + 1, -z + 1$ ; (ii)  $-x - 1, -y + 1, -z + 1$ ; (iii)  $-x + 1, -y + 2, -z + 1$ ; (iv)  $x + 1, y + 1, z + 1$ ; (v)  $x, y - 1, z - 1$ ; (vi)  $-x + 1, -y + 1, -z$ ; (vii)  $x + 1, y, z - 1$ .

Data collection: APEX2 (Bruker, 2006); cell refinement: SAINT (Bruker, 2002); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1990); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 2000); software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CS2045).

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**supplementary materials**

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## Bis(1,10-phenanthroline-5,6-dione- $\kappa^2N,N'$ )silver(I) perchlorate

J. Onuegbu, R. J. Butcher, C. Hosten, U. C. Udeochu and O. Bakare

### Comment

Phendione (1,10-phenanthroline-5,6-dione) is an excellent ligand that incorporates two functional groups with different coordination properties. Since the stereochemical behavior of  $\text{Cu}^+$  and  $\text{Ag}^+$  are similar it is interesting to compare the behavior of these cations with phendione. Isomorphous and isostructural  $\text{Cu}^+$  and  $\text{Ag}^+$  derivatives  $\{[M(L)_2](\text{ClO}_4)_2\}$ , where  $M = \text{Cu}^+$  and  $\text{Ag}^+$  have recently been determined (Galet *et al.*, 2005; McCann *et al.*, 2004) as well as a  $\text{Ag}^+$  complex, with trifluoromethanesulfonate instead of perchlorate as counterion (Wen *et al.*, 2006). In addition a polymeric  $\text{Ag}^+$  phendione complex where the ligand coordinates to Ag through N and O donors has been reported (Wen *et al.*, 2006). In this paper we report the synthesis and characterization of the title compound,  $[\text{AgL}_2]^+(\text{ClO}_4)^-(\text{I})$ .

The structure of the title compound, shown in Figure 1, is made up of an  $[\text{Ag}(L)_2]^+$  cation and a perchlorate anion. Each silver atom is coordinated to the two nitrogen atoms of both phendione ligands. In contrast to the previous structure determination of this complex where there is crystallographically imposed symmetry on both the anion and cation in the present case there is no such symmetry. Table 1 gives a listing of selected bond lengths and bond angles. The C=O bond lengths in the phendione ligands (1.220 (5) Å and 1.207 (5) Å) are comparable to those values found in other such complexes (Allen, 2002). The metrical parameters for the phendione ligand is in the normal ranges observed for complexes where only the N atoms are coordinated to a metal (Allen, 2002). The Ag—N bond lengths (2.261 (3), 2.331 (4) Å, 2.349 (3) Å, and 2.453 (4) Å) are similar to those found in related phenanthroline derivatives of silver (McCann *et al.*, 2004; Wen *et al.*, 2006; Leschke *et al.*, 2002; Paramonov *et al.*, 2003; Pallenberg *et al.*, 1997; Titze *et al.*, 1997). In I, silver is in a distorted tetrahedral environment. This is best illustrated by the dihedral angle between the planes of the coordinated ligands which would be 90° for tetrahedral and 0° for planar. In this case the angle is 36.7 (2)° which is intermediate between these extremes.

There are weak C—H...O hydrogen bonds between the hydrogen atoms on C1A, C1B, C2A, C6A, C6B, C8A, and C8B and either perchlorate O atoms or phendione O atoms from an adjoining cation. In addition, there are short contacts between the perchlorate anion and the phendione ligands (O14...C4B 2.845 (5) Å) as well as unusual and different torsion angles for O1—C4—C5—O2 for the two phendione ligands (−21.9 (7)° and −5.0 (7)°) which reflects the fact that in this polymorph there is no crystallographically imposed symmetry on the cation.

### Experimental

A flask containing 1,10-phenanthroline hydrate (1.00 g, 5.04 mmol) and potassium bromide (5.95 g, 50.0 mmol) was placed in an ice bath. Concentrated sulfuric acid (20 cm<sup>3</sup>) was added in small portions, followed by drop wise addition of concentrated nitric acid (10 cm<sup>3</sup>). The resulting solution was heated for 2 h at 80–85° C and cooled to room temperature. The solution was then poured into 400 cm<sup>3</sup> of water and neutralized with sodium bicarbonate, after which the phendione was extracted with dichloromethane, and recrystallized using a methanol-water mixture.

## supplementary materials

The title compound was synthesized in an atmosphere saturated with N<sub>2</sub>. To a solution of AgClO<sub>4</sub> in 15 ml of CH<sub>3</sub>CN, was added drop-wise a solution (15 ml) of CH<sub>3</sub>CN containing 0.26 g of phendione. The final yellowish solution was filtered and allowed to slowly evaporate for about a week yielding reddish brown prismatic crystals of the title compound suitable for X-ray studies.

### Refinement

H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with C—H distances of 0.93 Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

### Figures

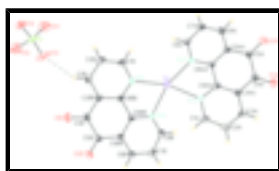


Fig. 1. View of the title compound (I), [Ag(L)<sub>2</sub>]<sup>+</sup>(ClO<sub>4</sub>)<sup>-</sup>, showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level.

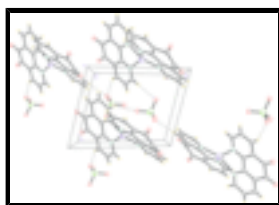


Fig. 2. The molecular packing of (I) viewed approximately along the *c* axis. Dotted lines indicate the hydrogen bonding interactions.

### Bis(1,10-phenanthroline-5,6-dione-κ<sup>2</sup>N,N')silver(I) perchlorate

#### Crystal data

[Ag(C<sub>12</sub>H<sub>6</sub>N<sub>2</sub>O<sub>2</sub>)<sub>2</sub>]ClO<sub>4</sub>

*M<sub>r</sub>* = 627.70

Triclinic, *P* $\bar{1}$

*a* = 8.493 (2) Å

*b* = 11.260 (3) Å

*c* = 13.190 (4) Å

$\alpha$  = 112.525 (3)°

$\beta$  = 103.682 (4)°

$\gamma$  = 97.378 (4)°

*V* = 1097.8 (5) Å<sup>3</sup>

*Z* = 2

*F*<sub>000</sub> = 624

*D<sub>x</sub>* = 1.899 Mg m<sup>-3</sup>

Mo *K*α radiation

$\lambda$  = 0.71073 Å

Cell parameters from 6560 reflections

$\theta$  = 2.6–29.9°

$\mu$  = 1.10 mm<sup>-1</sup>

*T* = 93 (2) K

Prism, red-brown

0.38 × 0.32 × 0.12 mm

#### Data collection

Bruker APEX II CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

6019 independent reflections

4791 reflections with  $I > 2\sigma(I)$

*R*<sub>int</sub> = 0.067

$T = 273(2)$  K  $\theta_{\max} = 30.4^\circ$   
 $\varphi$  and  $\omega$  scans  $\theta_{\min} = 2.0^\circ$   
 Absorption correction: multi-scan (SADABS; Bruker, 2000)  $h = -10 \rightarrow 12$   
 $T_{\min} = 0.679$ ,  $T_{\max} = 0.879$   $k = -15 \rightarrow 15$   
 11431 measured reflections  $l = -18 \rightarrow 17$

### Refinement

Refinement on  $F^2$  Secondary atom site location: difference Fourier map  
 Least-squares matrix: full Hydrogen site location: inferred from neighbouring sites  
 $R[F^2 > 2\sigma(F^2)] = 0.066$  H-atom parameters constrained  
 $wR(F^2) = 0.184$   $w = 1/[\sigma^2(F_o^2) + (0.107P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $S = 1.07$   $(\Delta/\sigma)_{\max} = 0.001$   
 6019 reflections  $\Delta\rho_{\max} = 2.36 \text{ e } \text{\AA}^{-3}$   
 343 parameters  $\Delta\rho_{\min} = -1.63 \text{ e } \text{\AA}^{-3}$   
 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.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Ag	0.13352 (4)	0.50994 (3)	0.25677 (2)	0.02407 (14)
Cl	-0.44552 (14)	0.71466 (11)	0.77901 (11)	0.0285 (3)
O1A	0.2299 (5)	0.2746 (3)	-0.2772 (3)	0.0303 (7)
C9BB	0.1294 (5)	0.7156 (4)	0.5018 (3)	0.0169 (7)
N2B	0.3438 (5)	0.6887 (3)	0.4071 (3)	0.0193 (7)
N1B	0.0294 (4)	0.6065 (3)	0.4095 (3)	0.0197 (7)
C9AA	0.1678 (5)	0.4269 (4)	0.0013 (3)	0.0160 (7)
O14	-0.6087 (4)	0.7408 (3)	0.7568 (3)	0.0302 (7)
O2B	0.4469 (5)	1.0657 (3)	0.7624 (3)	0.0358 (8)
N2A	0.0182 (4)	0.3212 (3)	0.0921 (3)	0.0174 (6)
O1B	0.1547 (5)	0.9601 (3)	0.7828 (3)	0.0308 (7)
C8A	-0.0858 (5)	0.2175 (4)	0.0876 (4)	0.0215 (8)
H8AA	-0.1122	0.2253	0.1541	0.026*
C1B	-0.1214 (5)	0.5583 (4)	0.4135 (4)	0.0232 (8)
H1BA	-0.1916	0.4830	0.3500	0.028*
C5AA	-0.0150 (5)	0.1964 (4)	-0.1072 (3)	0.0176 (7)
C8BB	0.2980 (5)	0.7637 (4)	0.4973 (3)	0.0169 (7)

## supplementary materials

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C6B	0.5636 (5)	0.9226 (4)	0.5775 (3)	0.0215 (8)
H6BA	0.6364	1.0019	0.6341	0.026*
C3AA	0.2244 (5)	0.4194 (4)	-0.0913 (3)	0.0179 (7)
C7B	0.6096 (5)	0.8460 (4)	0.4850 (4)	0.0230 (8)
H7BA	0.7138	0.8712	0.4781	0.028*
O2A	-0.0645 (5)	0.1084 (4)	-0.3101 (3)	0.0352 (8)
C4B	0.1929 (6)	0.9039 (4)	0.6977 (4)	0.0223 (8)
C3B	-0.0743 (6)	0.7283 (5)	0.6016 (4)	0.0248 (9)
H3BA	-0.1084	0.7688	0.6659	0.030*
C6A	-0.1213 (6)	0.0888 (4)	-0.1098 (4)	0.0239 (8)
H6AA	-0.1679	0.0112	-0.1777	0.029*
N1A	0.2144 (4)	0.5387 (3)	0.0994 (3)	0.0199 (7)
C8AA	0.0549 (5)	0.3108 (4)	-0.0038 (3)	0.0166 (7)
C5A	0.0205 (6)	0.1890 (4)	-0.2137 (4)	0.0209 (8)
C4A	0.1674 (6)	0.2938 (4)	-0.1997 (4)	0.0226 (8)
C8B	0.4943 (6)	0.7290 (4)	0.4016 (4)	0.0229 (8)
H8BA	0.5245	0.6765	0.3388	0.027*
C5BB	0.4073 (5)	0.8811 (4)	0.5863 (3)	0.0174 (7)
C3BB	0.0804 (5)	0.7798 (4)	0.5979 (3)	0.0196 (8)
C2A	0.3813 (6)	0.6438 (4)	0.0180 (4)	0.0243 (9)
H2AA	0.4543	0.7186	0.0267	0.029*
C2B	-0.1769 (6)	0.6150 (4)	0.5072 (4)	0.0271 (9)
H2BA	-0.2814	0.5778	0.5069	0.033*
C3A	0.3321 (5)	0.5288 (4)	-0.0830 (4)	0.0232 (8)
H3AA	0.3704	0.5246	-0.1444	0.028*
O13	-0.3380 (5)	0.7847 (4)	0.7458 (4)	0.0480 (11)
C7A	-0.1556 (6)	0.0998 (4)	-0.0107 (4)	0.0247 (9)
H7AA	-0.2245	0.0293	-0.0097	0.030*
C5B	0.3604 (6)	0.9603 (4)	0.6878 (4)	0.0220 (8)
C1A	0.3188 (5)	0.6445 (4)	0.1060 (4)	0.0201 (8)
H1AA	0.3505	0.7224	0.1737	0.024*
O12	-0.3734 (6)	0.7661 (5)	0.9063 (4)	0.0550 (12)
O11	-0.4590 (6)	0.5775 (4)	0.7348 (5)	0.0566 (13)

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Ag	0.0258 (2)	0.01959 (18)	0.01995 (18)	0.00118 (13)	0.00769 (12)	0.00246 (13)
Cl	0.0157 (5)	0.0261 (5)	0.0515 (7)	0.0045 (4)	0.0112 (4)	0.0243 (5)
O1A	0.0285 (18)	0.0360 (18)	0.0269 (15)	0.0071 (15)	0.0155 (13)	0.0100 (14)
C9BB	0.0136 (18)	0.0170 (16)	0.0230 (17)	0.0055 (14)	0.0091 (13)	0.0090 (14)
N2B	0.0152 (17)	0.0213 (15)	0.0199 (15)	0.0058 (13)	0.0082 (12)	0.0053 (13)
N1B	0.0159 (17)	0.0212 (16)	0.0218 (15)	0.0051 (13)	0.0059 (12)	0.0089 (13)
C9AA	0.0108 (17)	0.0171 (16)	0.0227 (17)	0.0063 (13)	0.0061 (13)	0.0097 (14)
O14	0.0171 (16)	0.0337 (17)	0.0419 (18)	0.0067 (14)	0.0090 (13)	0.0183 (15)
O2B	0.044 (2)	0.0232 (15)	0.0261 (16)	-0.0054 (15)	0.0121 (14)	0.0001 (13)
N2A	0.0143 (16)	0.0183 (15)	0.0204 (15)	0.0036 (13)	0.0079 (12)	0.0078 (13)
O1B	0.041 (2)	0.0267 (16)	0.0269 (16)	0.0122 (15)	0.0188 (14)	0.0082 (13)

C8A	0.018 (2)	0.0217 (18)	0.0253 (19)	0.0010 (15)	0.0075 (14)	0.0110 (16)
C1B	0.0146 (19)	0.027 (2)	0.0265 (19)	0.0001 (16)	0.0046 (15)	0.0126 (17)
C5AA	0.0125 (18)	0.0186 (16)	0.0216 (17)	0.0048 (14)	0.0054 (13)	0.0084 (14)
C8BB	0.0135 (18)	0.0170 (16)	0.0200 (17)	0.0054 (14)	0.0059 (13)	0.0071 (14)
C6B	0.018 (2)	0.0215 (18)	0.0217 (18)	-0.0018 (15)	0.0029 (14)	0.0101 (15)
C3AA	0.0131 (18)	0.0191 (17)	0.0221 (17)	0.0052 (14)	0.0071 (13)	0.0081 (15)
C7B	0.0141 (19)	0.027 (2)	0.032 (2)	0.0036 (16)	0.0082 (15)	0.0167 (18)
O2A	0.0292 (19)	0.0374 (18)	0.0263 (16)	-0.0060 (15)	0.0049 (13)	0.0072 (14)
C4B	0.030 (2)	0.0157 (16)	0.0217 (18)	0.0058 (16)	0.0103 (15)	0.0074 (15)
C3B	0.022 (2)	0.034 (2)	0.029 (2)	0.0143 (18)	0.0161 (16)	0.0173 (18)
C6A	0.021 (2)	0.0190 (17)	0.028 (2)	0.0041 (16)	0.0067 (16)	0.0072 (16)
N1A	0.0147 (16)	0.0157 (14)	0.0283 (17)	0.0022 (13)	0.0067 (13)	0.0088 (13)
C8AA	0.0115 (17)	0.0159 (16)	0.0230 (17)	0.0058 (14)	0.0053 (13)	0.0082 (14)
C5A	0.020 (2)	0.0210 (17)	0.0225 (18)	0.0056 (16)	0.0096 (14)	0.0079 (15)
C4A	0.019 (2)	0.0229 (18)	0.0253 (19)	0.0061 (16)	0.0080 (15)	0.0092 (16)
C8B	0.019 (2)	0.0259 (19)	0.0256 (19)	0.0088 (17)	0.0120 (15)	0.0093 (17)
C5BB	0.0178 (19)	0.0158 (16)	0.0190 (16)	0.0041 (14)	0.0067 (13)	0.0072 (14)
C3BB	0.017 (2)	0.0208 (17)	0.0221 (18)	0.0069 (15)	0.0078 (14)	0.0089 (15)
C2A	0.019 (2)	0.0214 (18)	0.032 (2)	0.0022 (16)	0.0058 (16)	0.0133 (17)
C2B	0.014 (2)	0.034 (2)	0.038 (2)	0.0055 (18)	0.0094 (16)	0.020 (2)
C3A	0.016 (2)	0.028 (2)	0.031 (2)	0.0050 (16)	0.0104 (15)	0.0172 (18)
O13	0.027 (2)	0.047 (2)	0.090 (4)	0.0095 (18)	0.030 (2)	0.043 (2)
C7A	0.023 (2)	0.0181 (18)	0.032 (2)	0.0001 (16)	0.0096 (16)	0.0109 (17)
C5B	0.026 (2)	0.0156 (16)	0.0233 (19)	0.0044 (16)	0.0083 (15)	0.0067 (15)
C1A	0.0139 (18)	0.0171 (17)	0.030 (2)	0.0028 (14)	0.0060 (14)	0.0114 (15)
O12	0.043 (3)	0.073 (3)	0.043 (2)	0.004 (2)	0.0069 (18)	0.026 (2)
O11	0.047 (3)	0.0230 (18)	0.099 (4)	0.0105 (18)	0.026 (3)	0.023 (2)

*Geometric parameters (Å, °)*

Ag—N2A	2.261 (3)	C5AA—C5A	1.478 (6)
Ag—N1B	2.331 (4)	C8BB—C5BB	1.403 (5)
Ag—N2B	2.349 (3)	C6B—C7B	1.375 (6)
Ag—N1A	2.453 (4)	C6B—C5BB	1.397 (6)
Cl—O13	1.385 (4)	C6B—H6BA	0.9300
Cl—O11	1.404 (4)	C3AA—C3A	1.388 (5)
Cl—O14	1.439 (4)	C3AA—C4A	1.492 (6)
Cl—O12	1.485 (5)	C7B—C8B	1.398 (6)
O1A—C4A	1.220 (5)	C7B—H7BA	0.9300
C9BB—N1B	1.345 (5)	O2A—C5A	1.215 (5)
C9BB—C3BB	1.389 (6)	C4B—C3BB	1.500 (6)
C9BB—C8BB	1.487 (6)	C4B—C5B	1.537 (7)
N2B—C8B	1.329 (6)	C3B—C3BB	1.386 (6)
N2B—C8BB	1.346 (5)	C3B—C2B	1.387 (6)
N1B—C1B	1.347 (6)	C3B—H3BA	0.9300
C9AA—N1A	1.341 (5)	C6A—C7A	1.369 (6)
C9AA—C3AA	1.392 (5)	C6A—H6AA	0.9300
C9AA—C8AA	1.488 (5)	N1A—C1A	1.351 (5)
O2B—C5B	1.207 (5)	C5A—C4A	1.527 (6)



## supplementary materials

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N2A—C8AA	1.339 (5)	C8B—H8BA	0.9300
N2A—C8A	1.344 (5)	C5BB—C5B	1.475 (6)
O1B—C4B	1.204 (5)	C2A—C3A	1.379 (6)
C8A—C7A	1.381 (6)	C2A—C1A	1.383 (6)
C8A—H8AA	0.9300	C2A—H2AA	0.9300
C1B—C2B	1.380 (6)	C2B—H2BA	0.9300
C1B—H1BA	0.9300	C3A—H3AA	0.9300
C5AA—C8AA	1.396 (5)	C7A—H7AA	0.9300
C5AA—C6A	1.399 (6)	C1A—H1AA	0.9300
N2A—Ag—N1B	129.69 (12)	O1B—C4B—C3BB	122.1 (4)
N2A—Ag—N2B	158.05 (13)	O1B—C4B—C5B	120.0 (4)
N1B—Ag—N2B	71.16 (12)	C3BB—C4B—C5B	117.9 (4)
N2A—Ag—N1A	70.77 (11)	C3BB—C3B—C2B	118.4 (4)
N1B—Ag—N1A	144.28 (12)	C3BB—C3B—H3BA	120.8
N2B—Ag—N1A	95.79 (12)	C2B—C3B—H3BA	120.8
O13—Cl—O11	115.7 (3)	C7A—C6A—C5AA	118.8 (4)
O13—Cl—O14	111.8 (2)	C7A—C6A—H6AA	120.6
O11—Cl—O14	110.3 (3)	C5AA—C6A—H6AA	120.6
O13—Cl—O12	105.8 (3)	C9AA—N1A—C1A	117.8 (4)
O11—Cl—O12	105.6 (3)	C9AA—N1A—Ag	113.5 (2)
O14—Cl—O12	106.9 (3)	C1A—N1A—Ag	127.8 (3)
N1B—C9BB—C3BB	122.2 (4)	N2A—C8AA—C5AA	121.3 (4)
N1B—C9BB—C8BB	116.7 (4)	N2A—C8AA—C9AA	118.2 (3)
C3BB—C9BB—C8BB	121.1 (4)	C5AA—C8AA—C9AA	120.4 (4)
C8B—N2B—C8BB	119.0 (4)	O2A—C5A—C5AA	123.5 (4)
C8B—N2B—Ag	124.3 (3)	O2A—C5A—C4A	119.2 (4)
C8BB—N2B—Ag	116.1 (3)	C5AA—C5A—C4A	117.1 (3)
C9BB—N1B—C1B	117.7 (4)	O1A—C4A—C3AA	122.7 (4)
C9BB—N1B—Ag	117.6 (3)	O1A—C4A—C5A	119.9 (4)
C1B—N1B—Ag	124.6 (3)	C3AA—C4A—C5A	117.3 (4)
N1A—C9AA—C3AA	121.4 (3)	N2B—C8B—C7B	123.5 (4)
N1A—C9AA—C8AA	117.1 (4)	N2B—C8B—H8BA	118.2
C3AA—C9AA—C8AA	121.5 (4)	C7B—C8B—H8BA	118.2
C8AA—N2A—C8A	118.5 (3)	C6B—C5BB—C8BB	118.6 (4)
C8AA—N2A—Ag	119.7 (2)	C6B—C5BB—C5B	119.9 (4)
C8A—N2A—Ag	121.8 (3)	C8BB—C5BB—C5B	121.5 (4)
N2A—C8A—C7A	123.3 (4)	C3B—C3BB—C9BB	119.5 (4)
N2A—C8A—H8AA	118.4	C3B—C3BB—C4B	119.7 (4)
C7A—C8A—H8AA	118.4	C9BB—C3BB—C4B	120.8 (4)
N1B—C1B—C2B	123.2 (4)	C3A—C2A—C1A	118.0 (4)
N1B—C1B—H1BA	118.4	C3A—C2A—H2AA	121.0
C2B—C1B—H1BA	118.4	C1A—C2A—H2AA	121.0
C8AA—C5AA—C6A	119.3 (4)	C1B—C2B—C3B	118.9 (4)
C8AA—C5AA—C5A	120.6 (4)	C1B—C2B—H2BA	120.6
C6A—C5AA—C5A	120.1 (4)	C3B—C2B—H2BA	120.6
N2B—C8BB—C5BB	121.3 (4)	C2A—C3A—C3AA	118.8 (4)
N2B—C8BB—C9BB	117.8 (3)	C2A—C3A—H3AA	120.6
C5BB—C8BB—C9BB	120.8 (4)	C3AA—C3A—H3AA	120.6
C7B—C6B—C5BB	119.8 (4)	C6A—C7A—C8A	118.7 (4)

C7B—C6B—H6BA	120.1	C6A—C7A—H7AA	120.6
C5BB—C6B—H6BA	120.1	C8A—C7A—H7AA	120.6
C3A—C3AA—C9AA	120.0 (4)	O2B—C5B—C5BB	123.2 (4)
C3A—C3AA—C4A	120.2 (4)	O2B—C5B—C4B	119.2 (4)
C9AA—C3AA—C4A	119.8 (4)	C5BB—C5B—C4B	117.5 (3)
C6B—C7B—C8B	117.7 (4)	N1A—C1A—C2A	123.9 (4)
C6B—C7B—H7BA	121.1	N1A—C1A—H1AA	118.0
C8B—C7B—H7BA	121.1	C2A—C1A—H1AA	118.0
N2A—Ag—N2B—C8B	19.0 (5)	C5A—C5AA—C8AA—N2A	177.0 (4)
N1B—Ag—N2B—C8B	-177.5 (4)	C6A—C5AA—C8AA—C9AA	-179.7 (4)
N1A—Ag—N2B—C8B	-31.7 (4)	C5A—C5AA—C8AA—C9AA	-0.6 (6)
N2A—Ag—N2B—C8BB	-170.1 (3)	N1A—C9AA—C8AA—N2A	-5.4 (5)
N1B—Ag—N2B—C8BB	-6.6 (3)	C3AA—C9AA—C8AA—N2A	173.7 (4)
N1A—Ag—N2B—C8BB	139.2 (3)	N1A—C9AA—C8AA—C5AA	172.3 (4)
C3BB—C9BB—N1B—C1B	-1.3 (6)	C3AA—C9AA—C8AA—C5AA	-8.5 (6)
C8BB—C9BB—N1B—C1B	177.5 (3)	C8AA—C5AA—C5A—O2A	-161.6 (4)
C3BB—C9BB—N1B—Ag	-179.1 (3)	C6A—C5AA—C5A—O2A	17.5 (7)
C8BB—C9BB—N1B—Ag	-0.3 (4)	C8AA—C5AA—C5A—C4A	15.2 (6)
N2A—Ag—N1B—C9BB	175.6 (3)	C6A—C5AA—C5A—C4A	-165.7 (4)
N2B—Ag—N1B—C9BB	3.5 (3)	C3A—C3AA—C4A—O1A	11.3 (6)
N1A—Ag—N1B—C9BB	-69.5 (4)	C9AA—C3AA—C4A—O1A	-169.4 (4)
N2A—Ag—N1B—C1B	-2.0 (4)	C3A—C3AA—C4A—C5A	-166.4 (4)
N2B—Ag—N1B—C1B	-174.1 (4)	C9AA—C3AA—C4A—C5A	12.9 (6)
N1A—Ag—N1B—C1B	112.9 (3)	O2A—C5A—C4A—O1A	-21.9 (7)
N1B—Ag—N2A—C8AA	150.5 (3)	C5AA—C5A—C4A—O1A	161.0 (4)
N2B—Ag—N2A—C8AA	-50.0 (5)	O2A—C5A—C4A—C3AA	155.9 (4)
N1A—Ag—N2A—C8AA	4.6 (3)	C5AA—C5A—C4A—C3AA	-21.2 (5)
N1B—Ag—N2A—C8A	-30.2 (4)	C8BB—N2B—C8B—C7B	0.2 (6)
N2B—Ag—N2A—C8A	129.3 (4)	Ag—N2B—C8B—C7B	170.8 (3)
N1A—Ag—N2A—C8A	-176.1 (3)	C6B—C7B—C8B—N2B	0.1 (7)
C8AA—N2A—C8A—C7A	0.7 (6)	C7B—C6B—C5BB—C8BB	-2.4 (6)
Ag—N2A—C8A—C7A	-178.5 (3)	C7B—C6B—C5BB—C5B	177.6 (4)
C9BB—N1B—C1B—C2B	-0.2 (6)	N2B—C8BB—C5BB—C6B	2.8 (6)
Ag—N1B—C1B—C2B	177.4 (3)	C9BB—C8BB—C5BB—C6B	-179.4 (4)
C8B—N2B—C8BB—C5BB	-1.7 (6)	N2B—C8BB—C5BB—C5B	-177.2 (4)
Ag—N2B—C8BB—C5BB	-173.1 (3)	C9BB—C8BB—C5BB—C5B	0.6 (6)
C8B—N2B—C8BB—C9BB	-179.5 (4)	C2B—C3B—C3BB—C9BB	-1.3 (6)
Ag—N2B—C8BB—C9BB	9.1 (4)	C2B—C3B—C3BB—C4B	178.7 (4)
N1B—C9BB—C8BB—N2B	-6.0 (5)	N1B—C9BB—C3BB—C3B	2.1 (6)
C3BB—C9BB—C8BB—N2B	172.9 (4)	C8BB—C9BB—C3BB—C3B	-176.7 (4)
N1B—C9BB—C8BB—C5BB	176.2 (3)	N1B—C9BB—C3BB—C4B	-177.9 (4)
C3BB—C9BB—C8BB—C5BB	-5.0 (6)	C8BB—C9BB—C3BB—C4B	3.3 (6)
N1A—C9AA—C3AA—C3A	0.3 (6)	O1B—C4B—C3BB—C3B	1.3 (7)
C8AA—C9AA—C3AA—C3A	-178.8 (4)	C5B—C4B—C3BB—C3B	-177.7 (4)
N1A—C9AA—C3AA—C4A	-179.0 (4)	O1B—C4B—C3BB—C9BB	-178.7 (4)
C8AA—C9AA—C3AA—C4A	1.9 (6)	C5B—C4B—C3BB—C9BB	2.3 (6)
C5BB—C6B—C7B—C8B	1.0 (6)	N1B—C1B—C2B—C3B	0.9 (7)
C8AA—C5AA—C6A—C7A	0.9 (6)	C3BB—C3B—C2B—C1B	-0.1 (6)
C5A—C5AA—C6A—C7A	-178.2 (4)	C1A—C2A—C3A—C3AA	-0.7 (6)

## supplementary materials

C3AA—C9AA—N1A—C1A	-0.1 (6)	C9AA—C3AA—C3A—C2A	0.1 (6)
C8AA—C9AA—N1A—C1A	179.1 (3)	C4A—C3AA—C3A—C2A	179.4 (4)
C3AA—C9AA—N1A—Ag	-170.2 (3)	C5AA—C6A—C7A—C8A	1.0 (6)
C8AA—C9AA—N1A—Ag	9.0 (4)	N2A—C8A—C7A—C6A	-1.9 (7)
N2A—Ag—N1A—C9AA	-7.3 (3)	C6B—C5BB—C5B—O2B	4.6 (6)
N1B—Ag—N1A—C9AA	-139.6 (3)	C8BB—C5BB—C5B—O2B	-175.4 (4)
N2B—Ag—N1A—C9AA	154.9 (3)	C6B—C5BB—C5B—C4B	-175.1 (4)
N2A—Ag—N1A—C1A	-176.1 (4)	C8BB—C5BB—C5B—C4B	4.9 (6)
N1B—Ag—N1A—C1A	51.5 (4)	O1B—C4B—C5B—O2B	-5.0 (7)
N2B—Ag—N1A—C1A	-14.0 (3)	C3BB—C4B—C5B—O2B	173.9 (4)
C8A—N2A—C8AA—C5AA	1.3 (6)	O1B—C4B—C5B—C5BB	174.7 (4)
Ag—N2A—C8AA—C5AA	-179.4 (3)	C3BB—C4B—C5B—C5BB	-6.4 (6)
C8A—N2A—C8AA—C9AA	179.0 (3)	C9AA—N1A—C1A—C2A	-0.6 (6)
Ag—N2A—C8AA—C9AA	-1.7 (5)	Ag—N1A—C1A—C2A	167.9 (3)
C6A—C5AA—C8AA—N2A	-2.1 (6)	C3A—C2A—C1A—N1A	1.0 (6)

### 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>
C8A—H8AA...O1B <sup>i</sup>	0.93	2.54	3.170 (5)	125
C1B—H1BA...O14 <sup>ii</sup>	0.93	2.50	3.380 (5)	157
C8B—H8BA...O11 <sup>i</sup>	0.93	2.58	3.155 (6)	121
C1A—H1AA...O2B <sup>iii</sup>	0.93	2.47	3.159 (5)	131
C6B—H6BA...O2A <sup>iv</sup>	0.93	2.47	3.203 (5)	136
C6A—H6AA...O13 <sup>v</sup>	0.93	2.45	3.235 (6)	142
C8B—H8BA...O1A <sup>vi</sup>	0.93	2.48	3.158 (6)	130
C2A—H2AA...O12 <sup>vii</sup>	0.93	2.55	3.268 (6)	134
C3A—H3AA...O11 <sup>vii</sup>	0.93	2.57	3.464 (7)	162

Symmetry codes: (i)  $-x, -y+1, -z+1$ ; (ii)  $-x-1, -y+1, -z+1$ ; (iii)  $-x+1, -y+2, -z+1$ ; (iv)  $x+1, y+1, z+1$ ; (v)  $x, y-1, z-1$ ; (vi)  $-x+1, -y+1, -z$ ; (vii)  $x+1, y, z-1$ .

Fig. 1

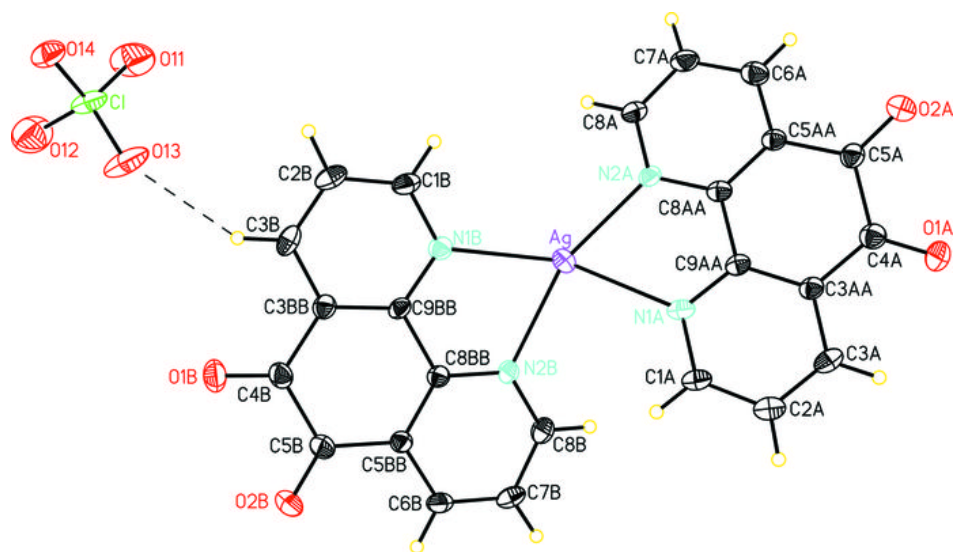


Fig. 2

