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{1,3-Bis[3-(2-pyridyl)-1*H*-pyrazol-1-yl]-propan-2-ol}silver(I) perchlorate

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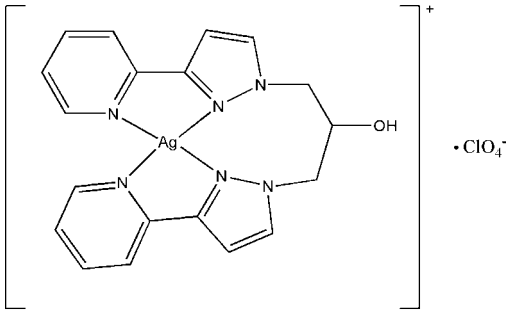
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; disorder in main residue; R factor = 0.038; wR factor = 0.088; data-to-parameter ratio = 14.1.

In the title compound, $[Ag(C_{19}H_{18}N_6O)]ClO_4$, the cation and anion both lie on crystallographic twofold rotation axes. The hydroxyl group of the cation is disordered across the twofold rotation axis. The Ag^I centre is four-coordinated by four N atoms from the 1,3-bis[3-(2-pyridyl)-1*H*-pyrazole]propan-2-ol ligand in a distorted tetrahedral coordination environment. O—H···O and weak C—H···O hydrogen-bonding interactions link adjacent mononuclear Ag^I units and perchlorate ions, forming a chain.

Related literature

For general background, see: Bell *et al.* (2003); Paul *et al.* (2004); Ruben *et al.* (2004); Steel (2005); Zhang *et al.* (2005). For hydrogen-bonding, see: Barberà *et al.* (2002); Desiraju & Steiner (1999).



Experimental

Crystal data

$[Ag(C_{19}H_{18}N_6O)]ClO_4$
M_r = 553.71
Monoclinic, $C2/c$
a = 8.687 (3) Å
b = 21.863 (9) Å
c = 10.904 (4) Å
β = 95.482 (7)°
V = 2061.4 (14) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.15 mm^{-1}
T = 293 (2) K
0.20 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.802, T_{max} = 0.819
5960 measured reflections
2135 independent reflections
1497 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.088
S = 1.00
2135 reflections
151 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ag1—N2	2.260 (3)	Ag1—N1	2.391 (3)
N2—Ag1—N2^{i}	134.08 (15)	N2—Ag1—N1^{i}	72.03 (10)
N2—Ag1—N1	148.68 (10)	N1—Ag1—N1^{i}	91.21 (14)

Symmetry code: (i) $-x+1, y, -z+\frac{3}{2}$.

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···O3^{ii}	0.82	2.21	3.017 (7)	170
C8—H8A···O2^{iii}	0.93	2.60	3.315 (5)	134

Symmetry codes: (ii) $x+\frac{1}{2}, -y+\frac{3}{2}, z-\frac{1}{2}$; (iii) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{3}{2}$.

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* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2407).

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supplementary materials

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{1,3-Bis[3-(2-pyridyl)-1*H*-pyrazol-1-yl]propan-2-ol}silver(I) perchlorate

C.-S. Liu and X.-S. Shi

Comment

In recent years, 3-(2-pyridyl)pyrazole-based ligands have found a wide range of application in the area of coordination chemistry, because they can act as bridging or chelate ligands and exhibit a series of intriguing structures and potential applications as functional materials (Ruben *et al.*, 2004; Steel *et al.*, 2005). Nowadays, much attention has been focused on the synthetic approach and the structural control of coordination architectures (Bell *et al.*, 2003; Paul *et al.*, 2004). We report here the structure of a mononuclear silver complex, {1,3-bis[3-(2-pyridyl)pyrazole]propan-2-ol}silver(I) perchlorate.

In the title compound, the cation and anion both lie on crystallographic twofold rotation axes. In the cation, the twofold axis passes through atoms Ag1 and C1, and as a result the hydroxyl group is disordered. The Ag^{I} center is four-coordinated by four N donors from a 1,3-bis[3-(2-pyridyl)pyrazole]propan-2-ol ligand (Table 1). The coordination geometry around the Ag^{I} center can be described as a distorted tetrahedron (Fig. 1).

The Ag^{I} mononuclear units are linked to the perchlorate ions through O—H···O hydrogen bonds (Table 2) and weak C—H···O interactions (Desiraju *et al.*, 1999; Barberà *et al.*, 2002) leading to the formation of a one-dimensional chain (Fig. 2).

Experimental

The ligand 1,3-bis[3-(2-pyridyl)-1*H*-pyrazole]propan-2-ol (*L*) was synthesized according to the method reported in the literature (Zhang *et al.*, 2005). A solution of $AgClO_4$ (22 mg, 0.1 mmol) in ethanol (10 ml) was added to a solution of *L* (35 mg, 0.1 mmol) in acetonitrile (20 ml) in a 50 ml beaker and the resulted solution was kept at room temperature in the dark. Single crystals of (**I**) suitable for X-ray analysis were obtained after 10 d (yield: 45%). Analysis calculated for ($C_{19}H_{18}AgClN_6O_5$): C 41.18, H 3.25, N 15.17%; found: C 41.36, H 3.64, N 14.91%.

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) and O—H = 0.82 Å, and with U_{iso}(H) = 1.2U_{eq}(C) and 1.5U_{eq}(O).

Figures

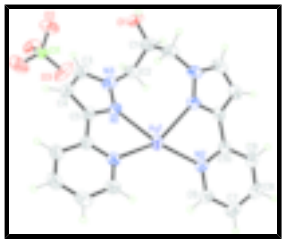

Fig. 1. The molecular structure of the title compound. Displacement ellipsoids are drawn at the 30% probability level. Unlabelled atoms in the cation are related to labelled atoms by (1 − *x*, *y*, 3/2 − *z*). Unlabelled atoms in the anion are related to labelled atoms by (1 − *x*, *y*, 5/2 − *z*). For clarity only one disorder component is shown.

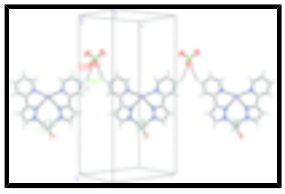

Fig. 2. Part of the crystal packing in the title compound, showing a C—H···O hydrogen-bonded (dashed) chain. The atom labelled with the suffix B is generated by the symmetry operation (1/2 − *x*, *y* − 1/2, 3/2 − *z*). For clarity only one disorder component is shown.

{1,3-Bis[3-(2-pyridyl)-1*H*-pyrazol-1-yl]propan-2-ol}silver(I) perchlorate

Crystal data

$[Ag(C_{19}H_{18}N_6O)]ClO_4$	F_{000} = 1112
M_r = 553.71	D_x = 1.784 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 946 reflections
a = 8.687 (3) Å	θ = 2.6–22.8º
b = 21.863 (9) Å	μ = 1.15 mm^{-1}
c = 10.904 (4) Å	*T* = 293 (2) K
β = 95.482 (7)º	Block, colourless
V = 2061.4 (14) Å^3	0.20 × 0.20 × 0.18 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	2135 independent reflections
Radiation source: fine-focus sealed tube	1497 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.031
T = 293(2) K	θ_{max} = 26.5º
φ and ω scans	θ_{min} = 2.5º
Absorption correction: multi-scan (SADABS; Bruker, 1998)	*h* = −6→10
T_{min} = 0.802, T_{max} = 0.819	*k* = −27→24
5960 measured reflections	*l* = −13→13

Refinement

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

Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.038$	H-atom parameters constrained
$wR(F^2) = 0.088$	$w = 1/[\sigma^2(F_o^2) + (0.0393P)^2 + 1.8827P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{max} = 0.001$
2135 reflections	$\Delta\rho_{max} = 0.53$ e Å^{-3}
151 parameters	$\Delta\rho_{min} = -0.46$ 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 $F^2 > 2$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}	Occ. (<1)
Ag1	0.5000	0.510403 (19)	0.7500	0.0676 (2)	
Cl1	0.5000	0.76495 (5)	1.2500	0.0524 (3)	
N1	0.5693 (3)	0.58693 (12)	0.9020 (2)	0.0499 (7)	
N2	0.3199 (3)	0.47008 (13)	0.6097 (3)	0.0523 (7)	
N3	0.5527 (3)	0.64717 (13)	0.9227 (3)	0.0550 (7)	
C1	0.5000	0.7204 (3)	0.7500	0.0764 (18)	
H1B	0.4098	0.7391	0.7082	0.092*	0.50
C2	0.4355 (4)	0.68233 (16)	0.8498 (4)	0.0610 (10)	
H2A	0.3847	0.7093	0.9040	0.073*	
H2B	0.3581	0.6545	0.8117	0.073*	
C3	0.6549 (5)	0.66635 (19)	1.0135 (4)	0.0652 (11)	
H3A	0.6642	0.7061	1.0438	0.078*	
C4	0.7424 (5)	0.61826 (18)	1.0539 (3)	0.0626 (10)	
H4A	0.8231	0.6179	1.1164	0.075*	
C5	0.6859 (4)	0.56931 (16)	0.9822 (3)	0.0494 (8)	
C6	0.2745 (5)	0.41184 (17)	0.6109 (3)	0.0629 (10)	
H6A	0.3154	0.3869	0.6750	0.075*	
C7	0.1708 (5)	0.38710 (19)	0.5223 (4)	0.0716 (11)	
H7A	0.1399	0.3465	0.5271	0.086*	
C8	0.1142 (5)	0.4230 (2)	0.4277 (4)	0.0732 (12)	
H8A	0.0438	0.4073	0.3662	0.088*	
C9	0.1609 (4)	0.48229 (19)	0.4231 (4)	0.0648 (10)	

H9A	0.1238	0.5071	0.3576	0.078*	
C10	0.2638 (4)	0.50556 (15)	0.5161 (3)	0.0474 (8)	
O1	0.5842 (6)	0.7655 (2)	0.7817 (5)	0.0669 (14)	0.50
H1A	0.6650	0.7632	0.7489	0.100*	0.50
O2	0.5854 (3)	0.80126 (13)	1.1757 (3)	0.0911 (10)	
O3	0.3944 (4)	0.72815 (17)	1.1794 (4)	0.1176 (13)	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ag1	0.0799 (3)	0.0611 (3)	0.0565 (3)	0.000	−0.0203 (2)	0.000
Cl1	0.0450 (7)	0.0449 (7)	0.0679 (8)	0.000	0.0082 (6)	0.000
N1	0.0484 (17)	0.0463 (16)	0.0551 (17)	0.0007 (12)	0.0061 (14)	−0.0066 (13)
N2	0.0617 (19)	0.0466 (17)	0.0474 (17)	−0.0030 (13)	−0.0007 (14)	−0.0032 (13)
N3	0.0512 (18)	0.0479 (17)	0.068 (2)	−0.0006 (13)	0.0166 (15)	−0.0087 (14)
C1	0.079 (4)	0.051 (3)	0.105 (5)	0.000	0.040 (4)	0.000
C2	0.056 (2)	0.048 (2)	0.082 (3)	0.0067 (17)	0.023 (2)	−0.0028 (19)
C3	0.070 (3)	0.061 (2)	0.067 (3)	−0.015 (2)	0.019 (2)	−0.027 (2)
C4	0.064 (2)	0.072 (3)	0.052 (2)	−0.010 (2)	0.0030 (18)	−0.0135 (19)
C5	0.047 (2)	0.059 (2)	0.0423 (19)	−0.0041 (16)	0.0093 (16)	−0.0089 (16)
C6	0.078 (3)	0.051 (2)	0.059 (2)	−0.0030 (19)	0.006 (2)	−0.0029 (18)
C7	0.077 (3)	0.061 (3)	0.078 (3)	−0.013 (2)	0.013 (2)	−0.020 (2)
C8	0.068 (3)	0.079 (3)	0.069 (3)	−0.007 (2)	−0.007 (2)	−0.027 (2)
C9	0.062 (2)	0.079 (3)	0.051 (2)	0.006 (2)	−0.0058 (18)	−0.008 (2)
C10	0.0418 (18)	0.058 (2)	0.0427 (18)	0.0064 (15)	0.0079 (14)	−0.0030 (16)
O1	0.067 (3)	0.052 (3)	0.082 (4)	−0.020 (2)	0.009 (3)	−0.020 (3)
O2	0.086 (2)	0.080 (2)	0.113 (3)	0.0011 (16)	0.0363 (18)	0.0338 (18)
O3	0.077 (2)	0.118 (3)	0.156 (3)	−0.0239 (19)	0.006 (2)	−0.072 (3)

Geometric parameters (Å, °)

Ag1—N2	2.260 (3)	C2—H2A	0.97
Ag1—N2^{i}	2.260 (3)	C2—H2B	0.97
Ag1—N1	2.391 (3)	C3—C4	1.346 (5)
Ag1—N1^{i}	2.391 (3)	C3—H3A	0.93
Cl1—O3	1.395 (3)	C4—C5	1.387 (5)
Cl1—O3^{ii}	1.395 (3)	C4—H4A	0.93
Cl1—O2^{ii}	1.397 (3)	C5—C10^{i}	1.460 (5)
Cl1—O2	1.397 (3)	C6—C7	1.368 (5)
N1—C5	1.330 (4)	C6—H6A	0.93
N1—N3	1.346 (4)	C7—C8	1.351 (6)
N2—C6	1.333 (4)	C7—H7A	0.93
N2—C10	1.337 (4)	C8—C9	1.361 (6)
N3—C3	1.333 (5)	C8—H8A	0.93
N3—C2	1.451 (5)	C9—C10	1.383 (5)
C1—O1	1.257 (6)	C9—H9A	0.93
C1—O1^{i}	1.257 (6)	C10—C5^{i}	1.460 (5)

C1—C2^{i}	1.519 (5)	O1—O1^{i}	1.557 (10)
C1—C2	1.519 (5)	O1—H1A	0.82
C1—H1B	0.96		
N2—Ag1—N2^{i}	134.08 (15)	C1—C2—H2A	108.9
N2—Ag1—N1	148.68 (10)	N3—C2—H2B	108.9
N2^{i}—Ag1—N1	72.03 (10)	C1—C2—H2B	108.9
N2—Ag1—N1^{i}	72.03 (10)	H2A—C2—H2B	107.7
N2^{i}—Ag1—N1^{i}	148.68 (10)	N3—C3—C4	108.2 (3)
N1—Ag1—N1^{i}	91.21 (14)	N3—C3—H3A	125.9
O3—Cl1—O3^{ii}	109.6 (4)	C4—C3—H3A	125.9
O3—Cl1—O2^{ii}	106.87 (19)	C3—C4—C5	105.1 (4)
O3^{ii}—Cl1—O2^{ii}	111.4 (2)	C3—C4—H4A	127.5
O3—Cl1—O2	111.4 (2)	C5—C4—H4A	127.5
O3^{ii}—Cl1—O2	106.87 (19)	N1—C5—C4	110.7 (3)
O2^{ii}—Cl1—O2	110.8 (3)	N1—C5—C10^{i}	119.5 (3)
C5—N1—N3	105.1 (3)	C4—C5—C10^{i}	129.8 (3)
C5—N1—Ag1	112.2 (2)	N2—C6—C7	123.1 (4)
N3—N1—Ag1	140.9 (2)	N2—C6—H6A	118.4
C6—N2—C10	118.4 (3)	C7—C6—H6A	118.4
C6—N2—Ag1	123.5 (2)	C8—C7—C6	118.4 (4)
C10—N2—Ag1	118.0 (2)	C8—C7—H7A	120.8
C3—N3—N1	111.0 (3)	C6—C7—H7A	120.8
C3—N3—C2	128.6 (3)	C7—C8—C9	119.6 (4)
N1—N3—C2	120.5 (3)	C7—C8—H8A	120.2
O1—C1—O1^{i}	76.5 (6)	C9—C8—H8A	120.2
O1—C1—C2^{i}	112.5 (3)	C8—C9—C10	119.8 (4)
O1^{i}—C1—C2^{i}	118.5 (3)	C8—C9—H9A	120.1
O1—C1—C2	118.5 (3)	C10—C9—H9A	120.1
O1^{i}—C1—C2	112.5 (3)	N2—C10—C9	120.6 (3)
C2^{i}—C1—C2	113.6 (5)	N2—C10—C5^{i}	117.2 (3)
O1—C1—H1B	102.8	C9—C10—C5^{i}	122.1 (3)
C2^{i}—C1—H1B	103.5	C1—O1—O1^{i}	51.7 (3)
C2—C1—H1B	103.5	C1—O1—H1A	109.3
N3—C2—C1	113.2 (3)	O1^{i}—O1—H1A	127.8
N3—C2—H2A	108.9		
N2—Ag1—N1—C5	157.0 (2)	C2—N3—C3—C4	−179.3 (3)
N2^{i}—Ag1—N1—C5	6.1 (2)	N3—C3—C4—C5	−0.2 (4)
N1^{i}—Ag1—N1—C5	−147.0 (3)	N3—N1—C5—C4	0.3 (4)
N2—Ag1—N1—N3	−41.1 (4)	Ag1—N1—C5—C4	168.6 (2)
N2^{i}—Ag1—N1—N3	168.0 (3)	N3—N1—C5—C10^{i}	−179.4 (3)
N1^{i}—Ag1—N1—N3	14.9 (3)	Ag1—N1—C5—C10^{i}	−11.1 (4)
N2^{i}—Ag1—N2—C6	15.6 (3)	C3—C4—C5—N1	−0.1 (4)
N1—Ag1—N2—C6	−124.4 (3)	C3—C4—C5—C10^{i}	179.6 (3)

N1^{i}—Ag1—N2—C6	175.0 (3)	C10—N2—C6—C7	−1.5 (6)
N2^{i}—Ag1—N2—C10	−159.8 (3)	Ag1—N2—C6—C7	−176.8 (3)
N1—Ag1—N2—C10	60.2 (3)	N2—C6—C7—C8	1.5 (6)
N1^{i}—Ag1—N2—C10	−0.4 (2)	C6—C7—C8—C9	−0.2 (6)
C5—N1—N3—C3	−0.5 (4)	C7—C8—C9—C10	−1.0 (6)
Ag1—N1—N3—C3	−163.1 (3)	C6—N2—C10—C9	0.1 (5)
C5—N1—N3—C2	179.3 (3)	Ag1—N2—C10—C9	175.8 (3)
Ag1—N1—N3—C2	16.7 (5)	C6—N2—C10—C5^{i}	179.4 (3)
C3—N3—C2—C1	77.9 (5)	Ag1—N2—C10—C5^{i}	−5.0 (4)
N1—N3—C2—C1	−101.9 (4)	C8—C9—C10—N2	1.1 (5)
O1—C1—C2—N3	−69.5 (5)	C8—C9—C10—C5^{i}	−178.1 (3)
O1^{i}—C1—C2—N3	−155.9 (4)	C2^{i}—C1—O1—O1^{i}	115.6 (4)
C2^{i}—C1—C2—N3	65.9 (2)	C2—C1—O1—O1^{i}	−108.5 (4)
N1—N3—C3—C4	0.5 (4)		

Symmetry codes: (i) −*x*+1, *y*, −*z*+3/2; (ii) −*x*+1, *y*, −*z*+5/2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1A···O3^{iii}	0.82	2.21	3.017 (7)	170
C8—H8A···O2^{iv}	0.93	2.60	3.315 (5)	134

Symmetry codes: (iii) *x*+1/2, −*y*+3/2, *z*−1/2; (iv) −*x*+1/2, *y*−1/2, −*z*+3/2.

Fig. 1

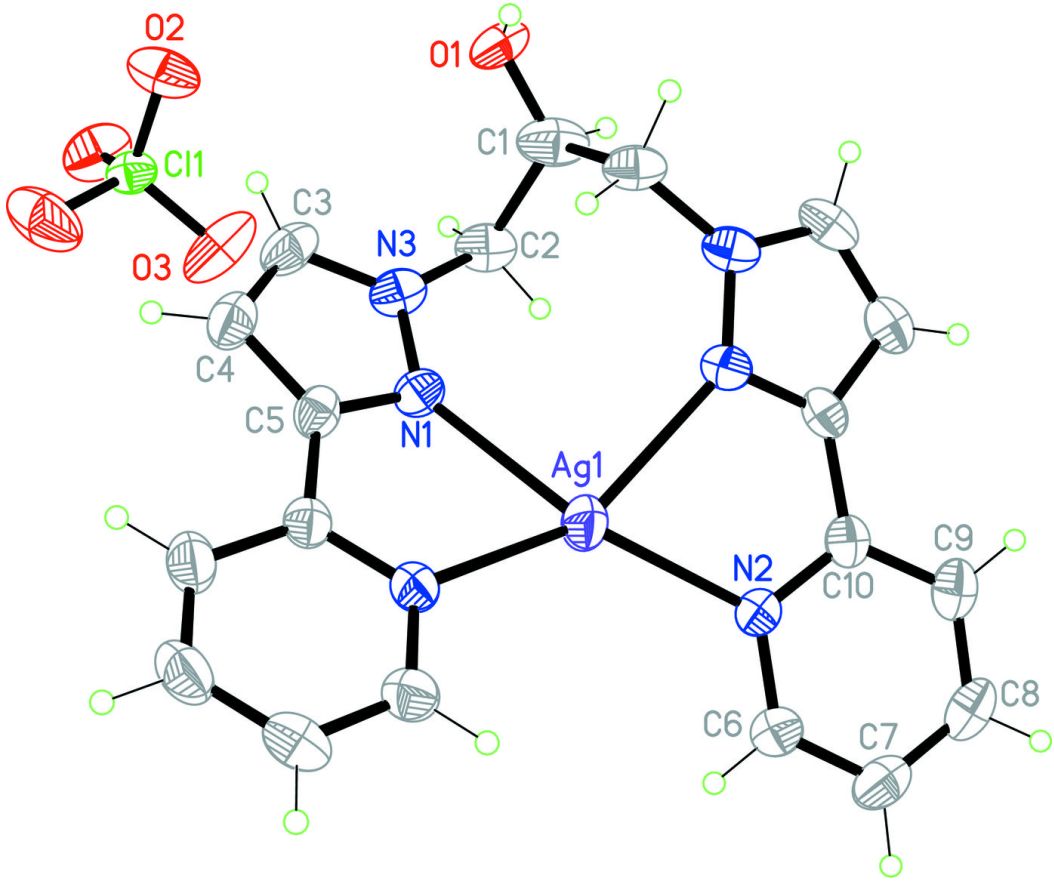

Fig. 2