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(2,2′-Bipyridine)saccharinatosilver(I)

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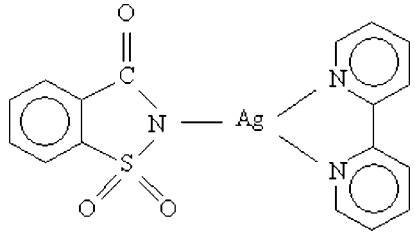
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.003 Å; R factor = 0.025; wR factor = 0.056; data-to-parameter ratio = 16.4.

The title complex, $[Ag(C_7H_4NO_3S)(C_{10}H_8N_2)]$, has a mononuclear structure in which the Ag^I ion is coordinated by two N atoms from 2,2′-bipyridine and one N atom from saccharinate, forming a distorted T-shaped (or trigonal-planar) AgN_3 arrangement. The bite angle of bpy is 71.69 (6)°, which contributes significantly to the distortion of the coordination geometry. Molecules are connected by C—H···O and π(bpy)–π(bpy) interactions with a centroid–centroid separation of 3.6741 (13) Å.

Related literature

For related literature, see: Weber *et al.* (1993); Yilmaz *et al.* (2004); Liu *et al.* (2006); Baran & Yilmaz (2006).



Experimental

Crystal data

$[Ag(C_7H_4NO_3S)(C_{10}H_8N_2)]$
M_r = 446.23
Monoclinic, $P2_1/n$
a = 10.7803 (2) Å
b = 9.76830 (10) Å
c = 15.6176 (2) Å
β = 100.1975 (8)°
V = 1618.64 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.40 mm^{-1}
T = 120 (2) K
0.40 × 0.04 × 0.02 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2003)
T_{min} = 0.605, T_{max} = 0.973
25291 measured reflections
3713 independent reflections
3194 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.056
S = 1.08
3713 reflections
227 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ag1—N1	2.1241 (18)	Ag1—N3	2.3488 (18)
Ag1—N2	2.2559 (18)		
N1—Ag1—N2	156.02 (7)	N2—Ag1—N3	71.69 (6)
N1—Ag1—N3	131.25 (7)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···O2^{i}	0.95	2.51	3.458 (3)	173
C16—H16···O1^{ii}	0.95	2.49	3.235 (3)	136

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

Data collection: *COLLECT* (Enraf–Nonius, 1999); cell refinement: *COLLECT*; data reduction: *COLLECT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2607).

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supplementary materials

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(2,2'-Bipyridine)saccharinatosilver(I)

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Comment

Saccharin is a well known artificial sweetener and it readily coordinates metal ions in the deprotonated form, saccharinate (sac) (Baran & Yilmaz 2006). The first silver(I) complexes of sac were reported by Weber *et al.* (1993) and Yilmaz *et al.* (2004). In this paper, the structure of the title compound, (I) (Fig. 1), containing a 2,2'-bipyridine (bpy) molecule and a sac anion is described.

In (I), $Ag(I)^{I}$ ion is coordinated by two N atoms from 2,2'-bipyridine and one N atom from saccharinate, forming a highly distorted T-shaped (or trigonal planar) coordination geometry (Table 1). The small bite angle of bpy [71.69 (6)°] is responsible for the distortion of the coordination geometry similar to that of a silver(I) complex of bpy (Liu *et al.*, 2006). Both ligands are essentially planar and the dihedral angle between the two pyridine rings of the bipy ligand is only 2.48 (11)°, while the dihedral angle between the sac benzene ring and sac 5-membered ring is 0.50 (8)°. The sac ion makes a dihedral angle of 7.20 (8)° with the complete bipy molecule.

The closest $Ag\cdots Ag^{i}$ contact is 4.0838 (3)Å (i = 1 − *x*, 2 − *y*, −*z*). There are two intramolecular CH···O hydrogen bonds involving the sac and bpy ring H atoms and sulfonly and carbony O atoms (Table 2). Furthermore, the molecules are further linked by π(bpy)···π(bpy) interactions with a $Cg—Cg^{i}$ separation of 3.6741 (13)Å (i = 1 − *x*, 1 − *y*, −*z*).

Experimental

$Na(sac)\cdot 2H_2O$ (0.24 g, 1 mmol) was added to a 320 ml solution of $AgNO_3$ (0.17 g, 1 mmol) dissolved in a mixture of water, methanol and dichloromethane (1:1:1, *v*/*v*) with stirring. The solution immediately became milky and the addition of bpy (0.16 g, 1 mmol) to the milky suspension resulted in a clear solution. The resulting solution was stirred for 30 min at room temperature and was allowed to stand in darkness at room temperature. Colorless crystals of the title compound were obtained after a week.

Refinement

All hydrogen atoms were placed in idealized locations and refined by riding, with C—H = 0.95 Å and $U_{iso}(H) = 1.2\,x\,U_{eq}(C)$.

Figures

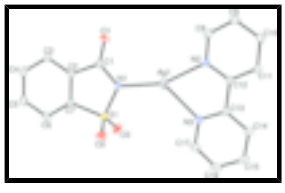


Fig. 1. The molecular structure of (I) showing 40% displacement ellipsoids (arbitrary spheres for the H atoms). C—H hydrogen atoms were removed for clarity.

(2,2'-Bipyridine)saccharinatosilver(I)

Crystal data

$[Ag(C_7H_4NO_3S)(C_{10}H_8N_2)]$	F_{000} = 888
M_r = 446.23	D_x = 1.831 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 3928 reflections
a = 10.7803 (2) Å	θ = 2.9–27.5º
b = 9.76830 (10) Å	μ = 1.40 mm^{-1}
c = 15.6176 (2) Å	T = 120 (2) K
β = 100.1975 (8)º	Needle, colourless
V = 1618.64 (4) Å^3	0.40 × 0.04 × 0.02 mm
Z = 4	

Data collection

Nonius KappaCCD diffractometer	3713 independent reflections
Radiation source: fine-focus sealed tube	3194 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.038
T = 120(2) K	θ_{max} = 27.5º
ω and φ scans	θ_{min} = 3.3º
Absorption correction: multi-scan (SADABS; Bruker, 2003)	h = −14→13
T_{min} = 0.605, T_{max} = 0.973	k = −12→12
25291 measured reflections	l = −20→20

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.025	$w = 1/[\sigma^2(F_o^2) + (0.0156P)^2 + 1.822P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.056	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.08	$\Delta\rho_{max}$ = 0.43 e Å^{-3}
3713 reflections	$\Delta\rho_{min}$ = −0.46 e Å^{-3}
227 parameters	Extinction correction: SHELXL, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.00128 (18)
Secondary atom site location: difference Fourier map	

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 $F^2 > 2\text{sigma}(F^2)$ 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	0.501633 (17)	0.793746 (18)	0.021427 (11)	0.02327 (7)
C1	0.4298 (2)	1.0121 (2)	0.15235 (13)	0.0158 (4)
C2	0.33090 (19)	1.1138 (2)	0.16617 (13)	0.0145 (4)
C3	0.3360 (2)	1.2084 (2)	0.23278 (14)	0.0185 (4)
H3	0.4093	1.2169	0.2763	0.022*
C4	0.2318 (2)	1.2901 (2)	0.23427 (14)	0.0229 (5)
H4	0.2339	1.3561	0.2792	0.028*
C5	0.1232 (2)	1.2772 (2)	0.17078 (15)	0.0221 (5)
H5	0.0526	1.3341	0.1735	0.026*
C6	0.1172 (2)	1.1826 (2)	0.10380 (14)	0.0190 (5)
H6	0.0439	1.1729	0.0605	0.023*
C7	0.2232 (2)	1.1032 (2)	0.10334 (13)	0.0144 (4)
N1	0.39147 (17)	0.93554 (19)	0.07890 (11)	0.0182 (4)
O1	0.53026 (15)	0.99921 (17)	0.20193 (10)	0.0240 (4)
O2	0.16452 (15)	0.85874 (16)	0.03892 (11)	0.0271 (4)
O3	0.24637 (18)	1.02164 (18)	−0.05562 (10)	0.0295 (4)
S1	0.24856 (5)	0.97131 (6)	0.03116 (3)	0.01733 (12)
C8	0.7656 (2)	0.6447 (2)	0.08002 (15)	0.0244 (5)
H8	0.7734	0.7113	0.1250	0.029*
C9	0.8607 (2)	0.5498 (2)	0.08214 (15)	0.0237 (5)
H9	0.9324	0.5509	0.1273	0.028*
C10	0.8489 (2)	0.4530 (2)	0.01676 (15)	0.0224 (5)
H10	0.9124	0.3857	0.0165	0.027*
C11	0.7438 (2)	0.4551 (2)	−0.04827 (14)	0.0182 (5)
H11	0.7345	0.3894	−0.0938	0.022*
C12	0.6517 (2)	0.5545 (2)	−0.04641 (13)	0.0147 (4)
C13	0.5363 (2)	0.5625 (2)	−0.11505 (13)	0.0158 (4)
C14	0.5170 (2)	0.4753 (3)	−0.18644 (15)	0.0248 (5)
H14	0.5774	0.4070	−0.1927	0.030*
C15	0.4087 (2)	0.4890 (3)	−0.24846 (15)	0.0283 (6)
H15	0.3943	0.4307	−0.2979	0.034*
C16	0.3221 (2)	0.5881 (3)	−0.23762 (15)	0.0268 (5)
H16	0.2464	0.5986	−0.2787	0.032*

C17	0.3481 (2)	0.6718 (3)	−0.16543 (16)	0.0265 (5)
H17	0.2888	0.7408	−0.1581	0.032*
N2	0.66271 (17)	0.64772 (19)	0.01771 (11)	0.0184 (4)
N3	0.45297 (17)	0.6604 (2)	−0.10520 (12)	0.0197 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ag1	0.02769 (11)	0.01844 (10)	0.02661 (11)	0.00675 (7)	0.01280 (7)	−0.00129 (7)
C1	0.0157 (11)	0.0180 (11)	0.0142 (10)	−0.0019 (9)	0.0041 (8)	0.0016 (8)
C2	0.0166 (10)	0.0129 (10)	0.0144 (10)	0.0002 (8)	0.0039 (8)	0.0027 (8)
C3	0.0216 (11)	0.0183 (11)	0.0149 (10)	−0.0024 (9)	0.0016 (9)	−0.0011 (9)
C4	0.0347 (13)	0.0165 (11)	0.0189 (11)	0.0021 (10)	0.0082 (10)	−0.0039 (9)
C5	0.0256 (12)	0.0180 (12)	0.0240 (11)	0.0085 (10)	0.0084 (10)	0.0026 (9)
C6	0.0180 (11)	0.0178 (11)	0.0204 (11)	0.0027 (9)	0.0017 (9)	0.0025 (9)
C7	0.0183 (11)	0.0116 (10)	0.0135 (10)	−0.0004 (8)	0.0034 (8)	0.0004 (8)
N1	0.0172 (9)	0.0196 (10)	0.0173 (9)	0.0048 (8)	0.0018 (7)	−0.0030 (7)
O1	0.0152 (8)	0.0314 (10)	0.0235 (8)	0.0034 (7)	−0.0015 (7)	−0.0012 (7)
O2	0.0234 (9)	0.0167 (8)	0.0379 (10)	−0.0006 (7)	−0.0040 (7)	−0.0056 (7)
O3	0.0416 (11)	0.0304 (10)	0.0147 (8)	0.0090 (8)	0.0002 (7)	−0.0023 (7)
S1	0.0194 (3)	0.0152 (3)	0.0157 (2)	0.0030 (2)	−0.0016 (2)	−0.0033 (2)
C8	0.0290 (13)	0.0225 (12)	0.0195 (11)	0.0017 (10)	−0.0014 (10)	−0.0054 (9)
C9	0.0233 (12)	0.0214 (12)	0.0230 (12)	0.0017 (10)	−0.0057 (9)	0.0012 (10)
C10	0.0201 (12)	0.0173 (11)	0.0289 (12)	0.0058 (9)	0.0020 (10)	0.0021 (9)
C11	0.0202 (11)	0.0151 (11)	0.0195 (11)	0.0004 (9)	0.0037 (9)	−0.0024 (9)
C12	0.0158 (11)	0.0138 (10)	0.0152 (10)	−0.0015 (9)	0.0042 (8)	0.0014 (8)
C13	0.0167 (11)	0.0150 (10)	0.0163 (10)	−0.0027 (9)	0.0044 (8)	0.0019 (8)
C14	0.0239 (12)	0.0254 (13)	0.0238 (12)	0.0005 (10)	0.0004 (10)	−0.0054 (10)
C15	0.0291 (14)	0.0314 (14)	0.0221 (12)	−0.0062 (11)	−0.0020 (10)	−0.0048 (10)
C16	0.0186 (12)	0.0366 (15)	0.0228 (12)	−0.0070 (11)	−0.0028 (9)	0.0063 (10)
C17	0.0181 (12)	0.0334 (14)	0.0274 (12)	0.0049 (10)	0.0023 (10)	0.0046 (11)
N2	0.0204 (10)	0.0164 (9)	0.0177 (9)	0.0035 (8)	0.0017 (8)	−0.0021 (7)
N3	0.0166 (9)	0.0236 (10)	0.0184 (9)	0.0030 (8)	0.0018 (7)	0.0000 (8)

Geometric parameters (Å, °)

C1—O1	1.221 (3)	C9—H9	0.9500
C1—N1	1.370 (3)	C10—C11	1.381 (3)
C1—C2	1.500 (3)	C10—H10	0.9500
C2—C7	1.385 (3)	C11—C12	1.393 (3)
C2—C3	1.385 (3)	C11—H11	0.9500
C3—C4	1.382 (3)	C12—N2	1.343 (3)
C3—H3	0.9500	C12—C13	1.493 (3)
C4—C5	1.399 (3)	C13—N3	1.339 (3)
C4—H4	0.9500	C13—C14	1.389 (3)
C5—C6	1.389 (3)	C14—C15	1.385 (3)
C5—H5	0.9500	C14—H14	0.9500
C6—C7	1.383 (3)	C15—C16	1.375 (4)
C6—H6	0.9500	C15—H15	0.9500

C7—S1	1.764 (2)	C16—C17	1.381 (3)
N1—S1	1.6262 (18)	C16—H16	0.9500
O2—S1	1.4434 (17)	C17—N3	1.341 (3)
O3—S1	1.4379 (17)	C17—H17	0.9500
C8—N2	1.340 (3)	Ag1—N1	2.1241 (18)
C8—C9	1.378 (3)	Ag1—N2	2.2559 (18)
C8—H8	0.9500	Ag1—N3	2.3488 (18)
C9—C10	1.381 (3)		
O1—C1—N1	125.2 (2)	C10—C9—H9	120.9
O1—C1—C2	123.36 (19)	C9—C10—C11	119.3 (2)
N1—C1—C2	111.43 (18)	C9—C10—H10	120.3
C7—C2—C3	119.95 (19)	C11—C10—H10	120.3
C7—C2—C1	112.09 (18)	C10—C11—C12	119.3 (2)
C3—C2—C1	127.95 (19)	C10—C11—H11	120.3
C4—C3—C2	118.3 (2)	C12—C11—H11	120.3
C4—C3—H3	120.9	N2—C12—C11	121.25 (19)
C2—C3—H3	120.9	N2—C12—C13	116.89 (18)
C3—C4—C5	121.1 (2)	C11—C12—C13	121.86 (19)
C3—C4—H4	119.4	N3—C13—C14	121.4 (2)
C5—C4—H4	119.4	N3—C13—C12	116.45 (18)
C6—C5—C4	121.0 (2)	C14—C13—C12	122.1 (2)
C6—C5—H5	119.5	C15—C14—C13	119.4 (2)
C4—C5—H5	119.5	C15—C14—H14	120.3
C7—C6—C5	116.7 (2)	C13—C14—H14	120.3
C7—C6—H6	121.7	C16—C15—C14	119.2 (2)
C5—C6—H6	121.7	C16—C15—H15	120.4
C6—C7—C2	122.98 (19)	C14—C15—H15	120.4
C6—C7—S1	129.37 (16)	C15—C16—C17	118.2 (2)
C2—C7—S1	107.61 (15)	C15—C16—H16	120.9
C1—N1—S1	112.78 (15)	C17—C16—H16	120.9
C1—N1—Ag1	126.97 (14)	N3—C17—C16	123.2 (2)
S1—N1—Ag1	120.10 (10)	N3—C17—H17	118.4
O3—S1—O2	115.70 (11)	C16—C17—H17	118.4
O3—S1—N1	111.21 (10)	C8—N2—C12	118.60 (19)
O2—S1—N1	110.83 (10)	C8—N2—Ag1	122.51 (15)
O3—S1—C7	111.97 (10)	C12—N2—Ag1	118.75 (14)
O2—S1—C7	109.39 (10)	C13—N3—C17	118.6 (2)
N1—S1—C7	96.03 (10)	C13—N3—Ag1	115.99 (14)
N2—C8—C9	123.3 (2)	C17—N3—Ag1	125.41 (16)
N2—C8—H8	118.3	N1—Ag1—N2	156.02 (7)
C9—C8—H8	118.3	N1—Ag1—N3	131.25 (7)
C8—C9—C10	118.2 (2)	N2—Ag1—N3	71.69 (6)
C8—C9—H9	120.9		
O1—C1—C2—C7	−176.8 (2)	N2—C8—C9—C10	−0.1 (4)
N1—C1—C2—C7	2.0 (3)	C8—C9—C10—C11	0.5 (4)
O1—C1—C2—C3	1.4 (4)	C9—C10—C11—C12	−0.2 (3)
N1—C1—C2—C3	−179.7 (2)	C10—C11—C12—N2	−0.5 (3)
C7—C2—C3—C4	0.0 (3)	C10—C11—C12—C13	179.7 (2)

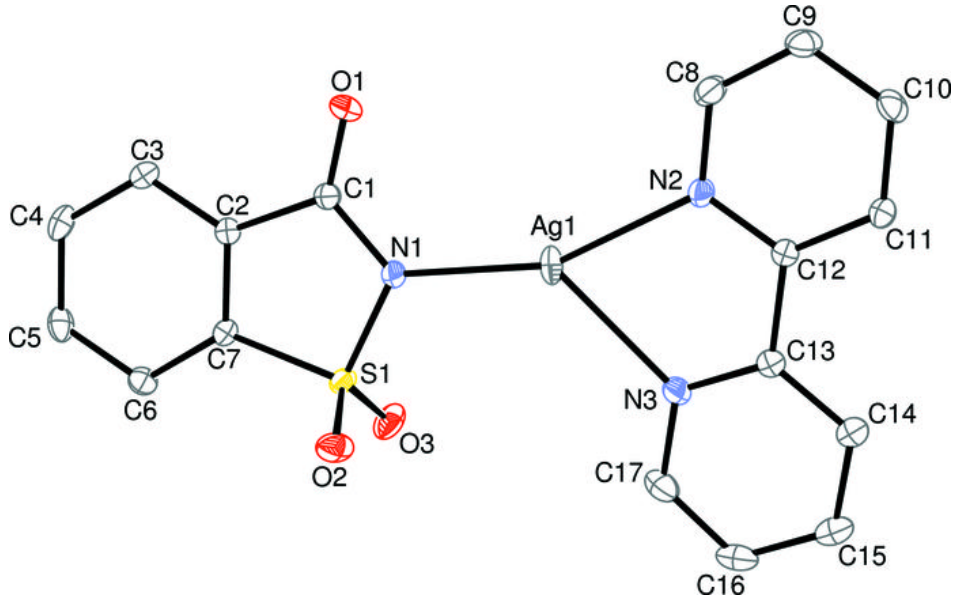
C1—C2—C3—C4	−178.1 (2)	N2—C12—C13—N3	−1.4 (3)
C2—C3—C4—C5	0.6 (3)	C11—C12—C13—N3	178.4 (2)
C3—C4—C5—C6	−0.5 (3)	N2—C12—C13—C14	177.3 (2)
C4—C5—C6—C7	−0.2 (3)	C11—C12—C13—C14	−2.9 (3)
C5—C6—C7—C2	0.8 (3)	N3—C13—C14—C15	−0.6 (4)
C5—C6—C7—S1	178.26 (17)	C12—C13—C14—C15	−179.2 (2)
C3—C2—C7—C6	−0.7 (3)	C13—C14—C15—C16	−0.5 (4)
C1—C2—C7—C6	177.67 (19)	C14—C15—C16—C17	1.0 (4)
C3—C2—C7—S1	−178.69 (17)	C15—C16—C17—N3	−0.6 (4)
C1—C2—C7—S1	−0.3 (2)	C9—C8—N2—C12	−0.5 (4)
O1—C1—N1—S1	175.89 (18)	C9—C8—N2—Ag1	175.15 (18)
C2—C1—N1—S1	−2.9 (2)	C11—C12—N2—C8	0.8 (3)
O1—C1—N1—Ag1	−8.7 (3)	C13—C12—N2—C8	−179.33 (19)
C2—C1—N1—Ag1	172.54 (14)	C11—C12—N2—Ag1	−175.03 (16)
N2—Ag1—N1—C1	21.5 (3)	C13—C12—N2—Ag1	4.8 (2)
N3—Ag1—N1—C1	−177.96 (16)	N1—Ag1—N2—C8	−15.4 (3)
N2—Ag1—N1—S1	−163.35 (13)	N3—Ag1—N2—C8	179.9 (2)
N3—Ag1—N1—S1	−2.81 (17)	N1—Ag1—N2—C12	160.26 (17)
C1—N1—S1—O3	118.83 (16)	N3—Ag1—N2—C12	−4.44 (15)
Ag1—N1—S1—O3	−56.97 (14)	C14—C13—N3—C17	1.0 (3)
C1—N1—S1—O2	−110.97 (16)	C12—C13—N3—C17	179.76 (19)
Ag1—N1—S1—O2	73.23 (14)	C14—C13—N3—Ag1	178.83 (17)
C1—N1—S1—C7	2.44 (17)	C12—C13—N3—Ag1	−2.5 (2)
Ag1—N1—S1—C7	−173.36 (11)	C16—C17—N3—C13	−0.5 (4)
C6—C7—S1—O3	65.3 (2)	C16—C17—N3—Ag1	−178.04 (18)
C2—C7—S1—O3	−116.97 (16)	N1—Ag1—N3—C13	−168.24 (14)
C6—C7—S1—O2	−64.4 (2)	N2—Ag1—N3—C13	3.56 (15)
C2—C7—S1—O2	113.41 (15)	N1—Ag1—N3—C17	9.4 (2)
C6—C7—S1—N1	−179.0 (2)	N2—Ag1—N3—C17	−178.8 (2)
C2—C7—S1—N1	−1.19 (16)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O2^{i}	0.95	2.51	3.458 (3)	173
C16—H16···O1^{ii}	0.95	2.49	3.235 (3)	136

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

Fig. 1


C9
C8
C10
O1
C3
C1
N2
C4
C2
Ag1
C11
N1
C12
C5
C13
C7
S1
N3
C14
C6
O3
O2
C17
C15
C16