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catena-Poly[[silver(I)-μ-1,4-di-3-pyridyl-2,3-diazabuta-1,3-diene] methanesulfonate]

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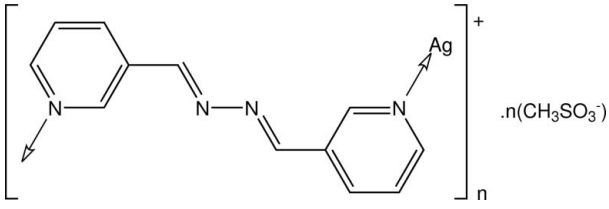
Key indicators: single-crystal X-ray study; T = 98 K; mean σ(C–C) = 0.007 Å; R factor = 0.044; wR factor = 0.115; data-to-parameter ratio = 14.0.

In the polymeric title compound, $\{[Ag(C_{12}H_{10}N_4)](CH_3SO_3)\}_n$, the Ag atom exists in an almost linear NAgN geometry and the topology of the supramolecular chain is a zigzag. Adjacent chains are linked *via* weak argentophilic Ag···Ag interactions [3.1402 (8) Å] to form double chains and the anions are associated with this *via* Ag···O contacts [2.508 (3) Å]. Layers are formed through C—H···O interactions and the layers stack *via* π–π interactions [centroid–centroid separation = 3.751 (3) Å].

Related literature

For related polymeric silver salts containing the 3-pyridinealdazine ligand, see: Kennedy *et al.* (2005). For related literature, see: Broker & Tiekink (2007*a*,*b*).



Experimental

Crystal data

$[Ag(C_{12}H_{10}N_4)](CH_3SO_3)$
M_r = 413.21
Triclinic, $P\overline{1}$
a = 7.7818 (13) Å
b = 9.6799 (16) Å
c = 9.9742 (15) Å
α = 106.50 (1)°
β = 91.12 (1)°
γ = 106.50 (1)°
V = 686.63 (19) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.64 mm^{-1}
T = 98 (2) K
0.15 × 0.05 × 0.03 mm

Data collection

Rigaku AFC12κ Saturn724 diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.641, T_{max} = 1.000
(expected range = 0.610–0.952)
4287 measured reflections
2793 independent reflections
2686 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.115
S = 1.09
2793 reflections
199 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.33 e Å^{-3}
$\Delta\rho_{min}$ = −1.74 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ag—N1	2.247 (4)	Ag···Ag^{ii}	3.1402 (8)
Ag—N4^{i}	2.227 (4)		
N4^{i}—Ag—N1	169.11 (14)		

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

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

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12···O2^{iii}	0.95	2.56	3.283 (6)	133
C2—H2···O3^{iv}	0.95	2.47	3.268 (7)	142
C13—H13*A*···N3^{v}	0.98	2.56	3.500 (7)	160

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

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2526).

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supplementary materials

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catena-Poly[[silver(I)-μ-1,4-di-3-pyridyl-2,3-diazabuta-1,3-diene] methanesulfonate]

G. A. Broker and E. R. T. Tiekink

Comment

The title compound, $[Ag(C_{12}H_{10}N_4)]_n.n(CH_3SO_3\}$, (I), was investigated in connection with on-going studies of Ag salts of isomeric n-pyridinealdazine, n = 2, 3 or 4, molecules (Broker & Tiekink, 2007*a*, b). In (I), (Fig. 1 & Table 1), the Ag atom exists in a linear N_2 geometry and the topology of the chain is zigzag owing to the relative disposition of the N-donor atoms. Similar polymeric chains are found for the perchlorate and tetrafluoroborate salts, each as acetonitrile solvates (Kennedy *et al.*, 2005).

In (I), the chains are connected into double chains *via* weak argentophilic interactions [Ag···Ag = 3.1402 (8) Å] and the methanesulfonate anions are linked to this *via* Ag···O interactions [2.508 (3) Å]. The presence of C—H···O interactions consolidate the double-chains into layers and additional stabilization is afforded by C—H···N contacts between a methyl-H and an azo-N atom (Fig. 2 & Table 2). Successive layers are connected primarily by π···π contacts so that the distance between the centroids of the N1/C1—C5 and $(N4/C8—C12)^{i}$ rings is 3.751 (3) Å (i = 1 − *x*, 1 − *y*, −*z*).

Experimental

$Ag(CF_3SO_3)$ (Aldrich, 0.05 g, 0.25 mmol) was dissolved in CH_3CN (20 ml) and layered on top of a CH_2Cl_2 solution (20 ml) containing 0.05 g (0.25 mmol) of 3-pyridinealdazine (Aldrich). After three days, yellow rods and blocks of (I) were observed at the interface between the two layers; m.p. 555–557 K.

Refinement

All the H atoms were included in the riding-model approximation, with C—H = 0.95–0.98Å and $U_{iso}(H) = 1.2$ or $1.5U_{eq}(C)$. The maximum and minimum difference peaks are located 0.87 and 0.88 Å, respectively, from Ag.

Figures

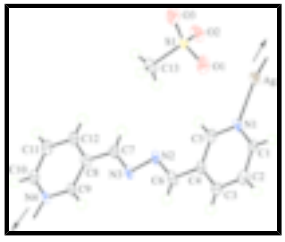

Fig. 1. The asymmetric unit of (I) showing displacement ellipsoids at the 70% probability level (arbitrary spheres for the H atoms).

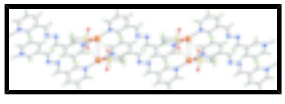

Fig. 2. View of the supramolecular double chains in (I) highlighting the Ag···Ag and Ag···O interactions (black dashed lines). Colour code: orange (silver), yellow (sulfur), red (oxygen), blue (nitrogen), grey (carbon) and green (hydrogen).

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Crystal data

$[Ag(C_{12}H_{10}N_4)](CH_3SO_3)$	$Z = 2$
$M_r = 413.21$	$F_{000} = 412$
Triclinic, $P\overline{1}$	$D_x = 1.994$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71070$ Å
$a = 7.7818$ (13) Å	Cell parameters from 6258 reflections
$b = 9.6799$ (16) Å	$\theta = 2.8$–29.7º
$c = 9.9742$ (15) Å	$\mu = 1.64$ mm^{-1}
$\alpha = 106.50$ (1)º	$T = 98$ (2) K
$\beta = 91.12$ (1)º	Rod, yellow
$\gamma = 106.50$ (1)º	0.15 × 0.05 × 0.03 mm
$V = 686.63$ (19) Å^3	

Data collection

Rigaku AFC12κ Saturn724 diffractometer	2793 independent reflections
Radiation source: fine-focus sealed tube	2686 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.030$
$T = 98(2)$ K	$\theta_{max} = 26.5$º
ω scans	$\theta_{min} = 2.6$º
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	$h = -9 \rightarrow 8$
$T_{min} = 0.641$, $T_{max} = 1.000$	$k = -10 \rightarrow 12$
4287 measured reflections	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.044$	H-atom parameters constrained
$wR(F^2) = 0.115$	$w = 1/[\sigma^2(F_o^2) + (0.0581P)^2 + 3.8177P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.09$	$(\Delta/\sigma)_{max} < 0.001$
2793 reflections	$\Delta\rho_{max} = 1.33$ e Å^{-3}
199 parameters	$\Delta\rho_{min} = -1.74$ 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}
Ag	−0.17114 (4)	0.45886 (4)	−0.42132 (3)	0.01878 (14)
S1	0.24710 (15)	0.79114 (12)	−0.26031 (11)	0.0167 (2)
O1	0.1198 (5)	0.6446 (4)	−0.2912 (4)	0.0296 (8)
O2	0.1607 (5)	0.9035 (4)	−0.2162 (4)	0.0279 (8)
O3	0.3649 (5)	0.8196 (5)	−0.3689 (4)	0.0334 (9)
N1	−0.1379 (5)	0.2995 (4)	−0.3042 (4)	0.0153 (7)
N2	0.2373 (5)	0.4241 (4)	0.0284 (4)	0.0167 (7)
N3	0.3507 (5)	0.4542 (4)	0.1490 (4)	0.0159 (7)
N4	0.7448 (5)	0.6106 (4)	0.4777 (4)	0.0153 (7)
C1	−0.2417 (6)	0.1603 (5)	−0.3322 (4)	0.0148 (8)
H1	−0.3284	0.1221	−0.4125	0.018*
C2	−0.2351 (6)	0.0603 (5)	−0.2512 (5)	0.0182 (9)
H2	−0.3170	−0.0391	−0.2792	0.022*
C3	−0.1162 (6)	0.1037 (5)	−0.1371 (5)	0.0169 (8)
H3	−0.1094	0.0398	−0.0817	0.020*
C4	−0.0069 (6)	0.2470 (5)	−0.1082 (4)	0.0141 (8)
C5	−0.0209 (6)	0.3421 (5)	−0.1940 (4)	0.0138 (8)
H5	0.0603	0.4418	−0.1685	0.017*
C6	0.1217 (6)	0.2981 (5)	0.0121 (5)	0.0181 (9)
H6	0.1204	0.2411	0.0756	0.022*
C7	0.4537 (6)	0.5846 (5)	0.1682 (4)	0.0161 (8)
H7	0.4445	0.6407	0.1056	0.019*
C8	0.5824 (6)	0.6447 (5)	0.2847 (4)	0.0139 (8)
C9	0.6240 (6)	0.5568 (5)	0.3700 (4)	0.0145 (8)
H9	0.5579	0.4525	0.3449	0.017*
C10	0.8239 (6)	0.7563 (5)	0.5078 (5)	0.0184 (9)
H10	0.9100	0.8032	0.5886	0.022*
C11	0.7911 (6)	0.8506 (5)	0.4285 (5)	0.0190 (9)
H11	0.8563	0.9552	0.4575	0.023*
C12	0.6709 (6)	0.7940 (5)	0.3151 (5)	0.0167 (8)
H12	0.6489	0.8537	0.2597	0.020*
C13	0.3873 (7)	0.7910 (5)	−0.1196 (5)	0.0212 (9)
H13A	0.4457	0.7119	−0.1524	0.032*

H13B	0.3147	0.7713	−0.0441	0.032*
H13C	0.4795	0.8896	−0.0841	0.032*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ag	0.0180 (2)	0.0230 (2)	0.0156 (2)	0.00637 (14)	−0.00283 (13)	0.00638 (14)
S1	0.0159 (5)	0.0166 (5)	0.0142 (5)	0.0019 (4)	−0.0044 (4)	0.0028 (4)
O1	0.0236 (18)	0.0202 (17)	0.037 (2)	0.0029 (14)	−0.0091 (15)	0.0003 (15)
O2	0.032 (2)	0.0171 (16)	0.0312 (19)	0.0081 (14)	−0.0112 (15)	0.0028 (14)
O3	0.0238 (19)	0.051 (2)	0.0164 (17)	−0.0002 (17)	−0.0040 (14)	0.0090 (16)
N1	0.0149 (17)	0.0212 (18)	0.0095 (16)	0.0083 (14)	−0.0035 (13)	0.0015 (14)
N2	0.0123 (17)	0.0208 (18)	0.0114 (17)	0.0025 (14)	−0.0059 (13)	−0.0007 (14)
N3	0.0126 (17)	0.0221 (19)	0.0083 (16)	0.0043 (15)	−0.0057 (13)	−0.0013 (14)
N4	0.0144 (17)	0.0176 (17)	0.0132 (17)	0.0060 (14)	−0.0035 (13)	0.0029 (14)
C1	0.0118 (19)	0.0142 (19)	0.0130 (19)	0.0001 (15)	−0.0016 (15)	−0.0002 (15)
C2	0.018 (2)	0.0135 (19)	0.016 (2)	0.0016 (16)	−0.0020 (16)	−0.0033 (16)
C3	0.018 (2)	0.015 (2)	0.014 (2)	0.0010 (16)	−0.0016 (16)	0.0027 (16)
C4	0.0111 (19)	0.017 (2)	0.0113 (18)	0.0037 (16)	−0.0026 (15)	−0.0005 (15)
C5	0.0115 (19)	0.0123 (18)	0.0127 (19)	0.0036 (15)	−0.0013 (15)	−0.0039 (15)
C6	0.019 (2)	0.022 (2)	0.0111 (19)	0.0080 (18)	−0.0049 (16)	−0.0005 (16)
C7	0.013 (2)	0.020 (2)	0.0126 (19)	0.0042 (17)	−0.0022 (16)	0.0028 (16)
C8	0.016 (2)	0.0132 (19)	0.0104 (18)	0.0056 (16)	0.0000 (15)	0.0000 (15)
C9	0.0125 (19)	0.018 (2)	0.0112 (18)	0.0057 (16)	−0.0047 (15)	0.0003 (15)
C10	0.015 (2)	0.021 (2)	0.015 (2)	0.0039 (17)	−0.0036 (16)	−0.0008 (16)
C11	0.016 (2)	0.018 (2)	0.017 (2)	0.0035 (17)	−0.0036 (17)	−0.0013 (17)
C12	0.015 (2)	0.016 (2)	0.016 (2)	0.0050 (16)	−0.0023 (16)	0.0009 (16)
C13	0.025 (2)	0.026 (2)	0.012 (2)	0.0108 (19)	−0.0032 (17)	0.0025 (17)

Geometric parameters (Å, °)

Ag—N1	2.247 (4)	C3—C4	1.350 (6)
Ag—O1	2.508 (4)	C3—H3	0.9500
Ag—N4^{i}	2.227 (4)	C4—C6	1.421 (6)
Ag—Ag^{ii}	3.1402 (8)	C4—C5	1.447 (6)
S1—O2	1.409 (4)	C5—H5	0.9500
S1—O1	1.425 (4)	C6—H6	0.9500
S1—O3	1.468 (4)	C7—C8	1.391 (6)
S1—C13	1.761 (4)	C7—H7	0.9500
N1—C5	1.303 (5)	C8—C12	1.353 (6)
N1—C1	1.306 (6)	C8—C9	1.452 (6)
N2—C6	1.258 (6)	C9—H9	0.9500
N2—N3	1.388 (5)	C10—C11	1.435 (6)
N3—C7	1.247 (6)	C10—H10	0.9500
N4—C9	1.294 (6)	C11—C12	1.331 (6)
N4—C10	1.310 (6)	C11—H11	0.9500
N4—Ag^{iii}	2.227 (4)	C12—H12	0.9500
C1—C2	1.437 (6)	C13—H13A	0.9800

C1—H1	0.9500	C13—H13B	0.9800
C2—C3	1.339 (6)	C13—H13C	0.9800
C2—H2	0.9500		
$N4^{i}$—Ag—N1	169.11 (14)	C6—C4—C5	122.4 (4)
$N4^{i}$—Ag—O1	97.46 (13)	N1—C5—C4	124.0 (4)
N1—Ag—O1	88.96 (13)	N1—C5—H5	118.0
$N4^{i}$—Ag—Ag^{ii}	82.12 (10)	C4—C5—H5	118.0
N1—Ag—Ag^{ii}	108.76 (10)	N2—C6—C4	115.2 (4)
O1—Ag—Ag^{ii}	58.95 (10)	N2—C6—H6	122.4
O2—S1—O1	110.4 (2)	C4—C6—H6	122.4
O2—S1—O3	111.1 (3)	N3—C7—C8	117.4 (4)
O1—S1—O3	116.9 (2)	N3—C7—H7	121.3
O2—S1—C13	110.8 (2)	C8—C7—H7	121.3
O1—S1—C13	100.8 (2)	C12—C8—C7	115.5 (4)
O3—S1—C13	106.1 (2)	C12—C8—C9	120.8 (4)
S1—O1—Ag	148.7 (2)	C7—C8—C9	123.8 (4)
C5—N1—C1	113.6 (4)	N4—C9—C8	124.5 (4)
C5—N1—Ag	122.4 (3)	N4—C9—H9	117.7
C1—N1—Ag	123.8 (3)	C8—C9—H9	117.7
C6—N2—N3	107.0 (4)	N4—C10—C11	125.3 (4)
C7—N3—N2	105.2 (4)	N4—C10—H10	117.4
C9—N4—C10	113.5 (4)	C11—C10—H10	117.4
C9—N4—Ag^{iii}	121.2 (3)	C12—C11—C10	121.1 (4)
C10—N4—Ag^{iii}	124.8 (3)	C12—C11—H11	119.4
N1—C1—C2	125.2 (4)	C10—C11—H11	119.4
N1—C1—H1	117.4	C11—C12—C8	114.8 (4)
C2—C1—H1	117.4	C11—C12—H12	122.6
C3—C2—C1	121.6 (4)	C8—C12—H12	122.6
C3—C2—H2	119.2	S1—C13—H13A	109.5
C1—C2—H2	119.2	S1—C13—H13B	109.5
C2—C3—C4	113.7 (4)	H13A—C13—H13B	109.5
C2—C3—H3	123.2	S1—C13—H13C	109.5
C4—C3—H3	123.2	H13A—C13—H13C	109.5
C3—C4—C6	115.7 (4)	H13B—C13—H13C	109.5
C3—C4—C5	122.0 (4)		
O2—S1—O1—Ag	52.9 (5)	Ag—N1—C5—C4	174.0 (3)
O3—S1—O1—Ag	−75.4 (5)	C3—C4—C5—N1	0.3 (7)
C13—S1—O1—Ag	170.1 (5)	C6—C4—C5—N1	−179.4 (4)
$N4^{i}$—Ag—O1—S1	−3.4 (5)	N3—N2—C6—C4	−179.5 (4)
N1—Ag—O1—S1	−174.6 (5)	C3—C4—C6—N2	171.5 (4)
Ag^{ii}—Ag—O1—S1	72.7 (5)	C5—C4—C6—N2	−8.8 (6)
$N4^{i}$—Ag—N1—C5	−113.0 (7)	N2—N3—C7—C8	179.4 (4)
O1—Ag—N1—C5	13.4 (3)	N3—C7—C8—C12	−169.5 (4)
Ag^{ii}—Ag—N1—C5	69.9 (3)	N3—C7—C8—C9	11.0 (7)
$N4^{i}$—Ag—N1—C1	61.4 (8)	C10—N4—C9—C8	2.6 (6)
O1—Ag—N1—C1	−172.2 (4)	Ag^{iii}—N4—C9—C8	−169.7 (3)

Ag^{ii}—Ag—N1—C1	−115.7 (3)	C12—C8—C9—N4	−0.3 (7)
C6—N2—N3—C7	−174.7 (4)	C7—C8—C9—N4	179.2 (4)
C5—N1—C1—C2	1.1 (6)	C9—N4—C10—C11	−2.9 (7)
Ag—N1—C1—C2	−173.7 (3)	Ag^{iii}—N4—C10—C11	169.1 (3)
N1—C1—C2—C3	−0.6 (7)	N4—C10—C11—C12	0.9 (8)
C1—C2—C3—C4	−0.2 (7)	C10—C11—C12—C8	1.5 (7)
C2—C3—C4—C6	−180.0 (4)	C7—C8—C12—C11	178.7 (4)
C2—C3—C4—C5	0.3 (6)	C9—C8—C12—C11	−1.8 (6)
C1—N1—C5—C4	−0.9 (6)		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12···$O2^{iv}$	0.95	2.56	3.283 (6)	133
C2—H2···$O3^{v}$	0.95	2.47	3.268 (7)	142
C13—H13A···$N3^{vi}$	0.98	2.56	3.500 (7)	160

Symmetry codes: (iv) −*x*+1, −*y*+2, −*z*; (v) *x*−1, *y*−1, *z*; (vi) −*x*+1, −*y*+1, −*z*.

Fig. 1

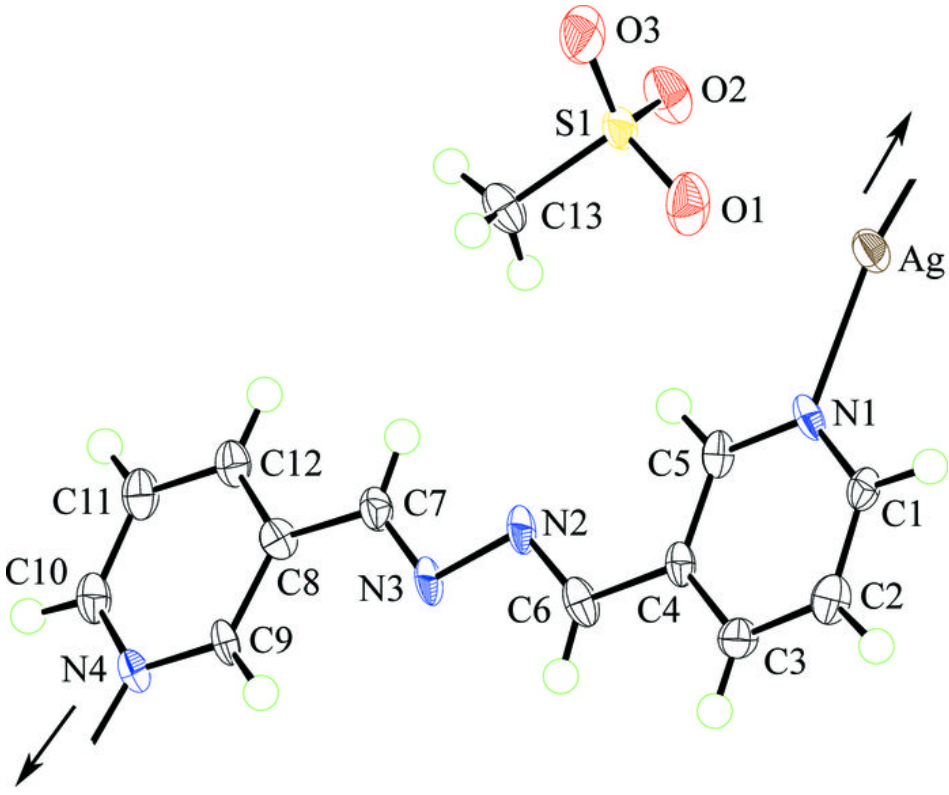

Fig. 2