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

Grant A. Broker and Edward R. T. Tiekink*

Department of Chemistry, University of Texas at San Antonio, One UTSA Circle, San Antonio, TX 78249-0698, USA
Correspondence e-mail: edward.tiekink@utsa.edu

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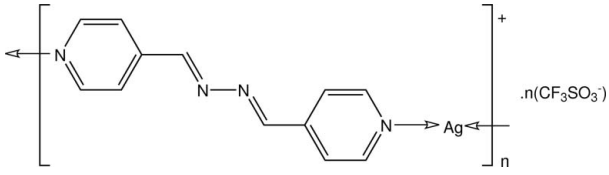
Key indicators: single-crystal X-ray study; T = 98 K; mean σ(C–C) = 0.004 Å; R factor = 0.025; wR factor = 0.068; data-to-parameter ratio = 11.9.

In the polymeric title compound, $\{[Ag(C_{12}H_{10}N_4)](CF_3SO_3)\}_n$, the Ag atom lies within an almost linear N_2 geometry and the topology of the polymer is linear. Weak Ag···O interactions lead to supramolecular chains that pack into layers, with connections between layers being of types Ag···Ag and Ag···O.

Related literature

For related polymeric silver salts containing the 4-pyridinealdazine ligand, see: Kennedy *et al.* (2005); Broker & Tiekink (2007*c*); Shi *et al.* (2002); Patra & Goldberg (2003). For related literature, see: Broker & Tiekink (2007*a*,*b*).



Experimental

Crystal data

$[Ag(C_{12}H_{10}N_4)](CF_3SO_3)$
M_r = 467.18
Triclinic, $P\overline{1}$
a = 8.9608 (18) Å
b = 8.9816 (18) Å
c = 10.804 (2) Å
α = 75.99 (3)°
β = 71.09 (3)°
γ = 83.30 (3)°
V = 797.4 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.45 mm^{-1}
T = 98 (2) K
0.45 × 0.36 × 0.21 mm

Data collection

Rigaku AFC12κ/SATURN724 diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.845, T_{max} = 1.000
(expected range = 0.623–0.738)
7718 measured reflections
2695 independent reflections
2654 reflections with $I > 2\sigma(I)$
R_{int} = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.068
S = 1.07
2695 reflections
226 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.55 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ag—N1	2.144 (2)	Ag—N4^{i}	2.150 (2)
N1—Ag—N4^{i}	172.36 (8)		

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

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O1	0.95	2.47	3.360 (4)	155
C10—H10···O3^{ii}	0.95	2.49	3.274 (3)	139
C11—H11···O2^{iii}	0.95	2.54	3.235 (4)	130

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

Data collection: *CrystalClear* (Rigaku/MSC, 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: HB2524).

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supplementary materials

Acta Cryst. (2007). E**63**, m2442 [**doi:10.1107/S1600536807041888**]

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

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

Comment

The polymeric title compound, $[Ag(C_{12}H_{10}N_4)]_n.n(CF_3SO_3)$, or $[Ag(4—PA)(CF_3SO_3)]_n$ (I), was investigated as a part of an on-going study of the structural chemistry of the Ag salts of isomeric n-pyridinealdazine molecules, n = 2, 3 and 4 (Broker & Tiekink, 2007*a*,b,c). The 4-pyridinealdazine (4-PA) ligand in (I) is bidentate bridging which results in the formation of polymeric chains and a near linear AgN_2 geometry for the silver atom (Fig. 1 & Table 1).

The linear chains are connected into supramolecular double chains (Fig. 2) *via* a combination of C—H···O interactions (Table 2) and a weak Ag···O3^{i} (i = 1 − *x*, 1 − *y*, 1 − *z*) contact of 2.759 (3) Å. The chains stack side by side to form layers in the *ac* plane and these layers are connected to adjacent layers on either side *via* Ag···Ag^{ii} (ii = 1 − *x*, 1 − *y*, −*z*) agentophilic interactions [3.2738 (13) Å] as well as Ag···O2^{iii} (iii = *x*, *y*, −1 + *z*) contacts of 2.854 (3) Å.

Similar polymeric structures to those in (I) are found in the following salts: perchlorate, tetrafluoroborate (as acetonitrile solvates, Kennedy *et al.*, 2005), hexafluoroantimonate (as the acetonitrile water solvate, Kennedy *et al.*, 2005), methanesulfonate (Broker & Tiekink, 2007*c*), and nitrate, for which two polymorphs have been reported (Shi *et al.*, 2002; Patra & Goldberg, 2003; Kennedy *et al.*, 2005). In all but the nitrate polymorphs, the chains are essentially flat. Only in (I) and the methanesulfonate salt (Broker & Tiekink, 2007*c*) are Ag···Ag interactions found.

Experimental

$Ag(CF_3SO_3)$ (Aldrich, 0.05 g, 0.19 mmol) was dissolved in CH_3CN (20 ml) and layered on top of a CH_2Cl_2 solution (20 ml) containing 0.04 g (0.19 mmol) of 4-pyridinealdazine (Aldrich). After three days, yellow blocks of (I) were observed at the interface between the two layers; m.p. 522–524 K.

Refinement

All the H atoms were included in the riding-model approximation, with C—H = 0.95 Å, and with U_{iso}(H) = 1.2U_{eq}(C).

Figures

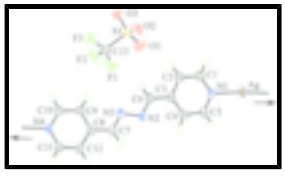

Fig. 1. The asymmetric unit of (I) showing displacement ellipsoids at the 70% probability level (arbitrary spheres for the H atoms).

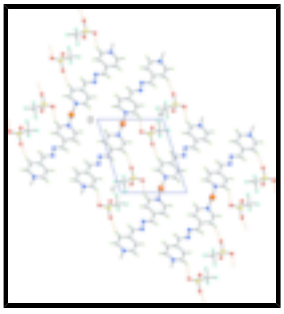

Fig. 2. View of the layers in (I) down the *b* axis highlighting the Ag···O interactions (orange dashed lines) leading to supramolecular double chains. Colour code: orange (silver), yellow (sulfur), red (oxygen), blue (nitrogen), grey (carbon) and green (hydrogen).

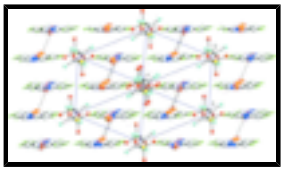

Fig. 3. View of the stacking of layers in (I) highlighting the aregentophilic interactions (black dotted lines). Colour code as for Fig. 2.

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

Crystal data

$[Ag(C_{12}H_{10}N_4)](CF_3SO_3)$	$Z = 2$
$M_r = 467.18$	$F_{000} = 460$
Triclinic, $P\overline{1}$	$D_x = 1.946$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71070$ Å
$a = 8.9608$ (18) Å	Cell parameters from 1270 reflections
$b = 8.9816$ (18) Å	$\theta = 3.3$–$30.2°$
$c = 10.804$ (2) Å	$\mu = 1.45$ mm^{-1}
$\alpha = 75.99$ (3)°	$T = 98$ (2) K
$\beta = 71.09$ (3)°	Block, yellow
$\gamma = 83.30$ (3)°	0.45 × 0.36 × 0.21 mm
$V = 797.4$ (3) Å^3	

Data collection

Rigaku AFC12κ/SATURN724 diffractometer	2695 independent reflections
Radiation source: fine-focus sealed tube	2654 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.036$
$T = 98(2)$ K	$\theta_{max} = 25.0°$
ω scans	$\theta_{min} = 3.1°$
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	$h = -9 \rightarrow 10$
$T_{min} = 0.845$, $T_{max} = 1.000$	$k = -10 \rightarrow 10$
7718 measured reflections	$l = -11 \rightarrow 12$

Refinement

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

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.025$

$wR(F^2) = 0.068$

$S = 1.07$

2695 reflections

226 parameters

Primary atom site location: structure-invariant direct methods

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0382P)^2 + 0.6435P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.47$ e Å^{-3}

$\Delta\rho_{min} = -0.55$ e Å^{-3}

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	0.36832 (2)	0.36786 (2)	0.060832 (18)	0.02608 (10)
S1	0.26232 (8)	0.68538 (8)	0.79915 (7)	0.02863 (17)
F1	−0.0358 (2)	0.7776 (2)	0.8486 (2)	0.0571 (5)
F2	0.1221 (3)	0.9605 (2)	0.7756 (2)	0.0600 (6)
F3	0.0766 (3)	0.8337 (2)	0.9775 (2)	0.0568 (6)
O1	0.2826 (3)	0.7020 (3)	0.6605 (2)	0.0583 (7)
O2	0.2017 (3)	0.5404 (2)	0.8843 (3)	0.0469 (6)
O3	0.3899 (3)	0.7370 (3)	0.8300 (2)	0.0436 (5)
N1	0.2426 (3)	0.4890 (2)	0.2149 (2)	0.0215 (4)
N2	−0.1451 (3)	0.8152 (2)	0.5065 (2)	0.0247 (5)
N3	−0.1980 (3)	0.8996 (3)	0.6078 (2)	0.0273 (5)
N4	−0.5349 (3)	1.2325 (2)	0.9117 (2)	0.0219 (4)
C1	0.3015 (3)	0.5068 (3)	0.3104 (3)	0.0234 (5)
H1	0.4007	0.4581	0.3132	0.028*
C2	0.2238 (3)	0.5927 (3)	0.4042 (3)	0.0223 (5)
H2	0.2690	0.6028	0.4700	0.027*
C3	0.0771 (3)	0.6650 (3)	0.4012 (2)	0.0215 (5)
C4	0.0148 (3)	0.6433 (3)	0.3041 (3)	0.0236 (5)
H4	−0.0855	0.6882	0.3005	0.028*
C5	0.1001 (3)	0.5563 (3)	0.2138 (3)	0.0251 (5)
H5	0.0568	0.5430	0.1479	0.030*
C6	−0.0050 (3)	0.7582 (3)	0.4990 (3)	0.0231 (5)

H6	0.0462	0.7768	0.5580	0.028*
C7	−0.3364 (3)	0.9599 (3)	0.6199 (3)	0.0277 (6)
H7	−0.3941	0.9449	0.5644	0.033*
C8	−0.4057 (3)	1.0528 (3)	0.7210 (3)	0.0237 (5)
C9	−0.3297 (3)	1.0618 (3)	0.8132 (3)	0.0225 (5)
H9	−0.2320	1.0071	0.8118	0.027*
C10	−0.3976 (3)	1.1503 (3)	0.9058 (2)	0.0216 (5)
H10	−0.3455	1.1540	0.9689	0.026*
C11	−0.6086 (3)	1.2238 (3)	0.8233 (3)	0.0289 (6)
H11	−0.7058	1.2803	0.8266	0.035*
C12	−0.5486 (3)	1.1358 (3)	0.7276 (3)	0.0299 (6)
H12	−0.6042	1.1321	0.6671	0.036*
C13	0.0992 (3)	0.8217 (3)	0.8526 (3)	0.0298 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ag	0.02502 (14)	0.02762 (14)	0.02719 (15)	0.00420 (9)	−0.00387 (9)	−0.01708 (9)
S1	0.0247 (3)	0.0372 (4)	0.0324 (4)	0.0105 (3)	−0.0154 (3)	−0.0196 (3)
F1	0.0286 (10)	0.0531 (12)	0.0942 (16)	0.0092 (8)	−0.0254 (10)	−0.0210 (11)
F2	0.0581 (13)	0.0333 (10)	0.0737 (14)	0.0066 (9)	−0.0168 (11)	0.0070 (9)
F3	0.0611 (14)	0.0662 (13)	0.0445 (11)	0.0323 (11)	−0.0170 (10)	−0.0297 (10)
O1	0.0528 (15)	0.094 (2)	0.0380 (13)	0.0212 (14)	−0.0215 (11)	−0.0332 (13)
O2	0.0479 (14)	0.0324 (11)	0.0642 (15)	0.0117 (10)	−0.0236 (12)	−0.0152 (10)
O3	0.0281 (11)	0.0606 (14)	0.0547 (14)	0.0096 (10)	−0.0201 (10)	−0.0310 (11)
N1	0.0208 (11)	0.0224 (10)	0.0211 (11)	0.0022 (8)	−0.0043 (8)	−0.0087 (8)
N2	0.0292 (12)	0.0236 (11)	0.0215 (11)	0.0036 (9)	−0.0039 (9)	−0.0126 (9)
N3	0.0296 (13)	0.0289 (11)	0.0254 (12)	0.0069 (9)	−0.0061 (9)	−0.0166 (9)
N4	0.0208 (11)	0.0218 (10)	0.0234 (11)	0.0020 (8)	−0.0044 (9)	−0.0098 (8)
C1	0.0213 (13)	0.0236 (12)	0.0236 (13)	0.0038 (10)	−0.0054 (10)	−0.0062 (10)
C2	0.0233 (13)	0.0233 (12)	0.0200 (13)	0.0002 (10)	−0.0065 (10)	−0.0048 (10)
C3	0.0239 (13)	0.0198 (12)	0.0187 (12)	−0.0001 (10)	−0.0021 (10)	−0.0067 (10)
C4	0.0177 (12)	0.0290 (13)	0.0244 (13)	0.0051 (10)	−0.0060 (10)	−0.0098 (10)
C5	0.0252 (13)	0.0286 (13)	0.0226 (13)	0.0023 (10)	−0.0077 (11)	−0.0086 (10)
C6	0.0246 (13)	0.0232 (12)	0.0225 (13)	0.0001 (10)	−0.0054 (10)	−0.0099 (10)
C7	0.0281 (15)	0.0302 (13)	0.0287 (14)	0.0063 (11)	−0.0105 (11)	−0.0147 (11)
C8	0.0235 (13)	0.0217 (12)	0.0272 (14)	0.0039 (10)	−0.0063 (11)	−0.0124 (10)
C9	0.0223 (13)	0.0227 (12)	0.0230 (13)	0.0037 (10)	−0.0069 (10)	−0.0080 (10)
C10	0.0213 (12)	0.0232 (12)	0.0213 (12)	0.0016 (10)	−0.0062 (10)	−0.0084 (10)
C11	0.0207 (13)	0.0335 (14)	0.0383 (16)	0.0088 (11)	−0.0110 (12)	−0.0206 (12)
C12	0.0267 (14)	0.0375 (15)	0.0364 (15)	0.0097 (12)	−0.0166 (12)	−0.0240 (12)
C13	0.0247 (14)	0.0297 (14)	0.0369 (16)	0.0058 (11)	−0.0138 (12)	−0.0077 (11)

Geometric parameters (Å, °)

Ag—N1	2.144 (2)	C1—H1	0.9500
Ag—N4^{i}	2.150 (2)	C2—C3	1.403 (4)
Ag—Ag^{ii}	3.2738 (13)	C2—H2	0.9500

S1—O1	1.422 (2)	C3—C4	1.397 (4)
S1—O3	1.442 (2)	C3—C6	1.463 (3)
S1—O2	1.449 (3)	C4—C5	1.376 (4)
S1—C13	1.830 (3)	C4—H4	0.9500
F1—C13	1.333 (3)	C5—H5	0.9500
F2—C13	1.317 (3)	C6—H6	0.9500
F3—C13	1.328 (3)	C7—C8	1.469 (4)
N1—C5	1.350 (3)	C7—H7	0.9500
N1—C1	1.351 (3)	C8—C12	1.395 (4)
N2—C6	1.284 (4)	C8—C9	1.397 (4)
N2—N3	1.407 (3)	C9—C10	1.372 (4)
N3—C7	1.273 (4)	C9—H9	0.9500
N4—C11	1.346 (3)	C10—H10	0.9500
N4—C10	1.350 (3)	C11—C12	1.385 (4)
N4—Ag^{iii}	2.150 (2)	C11—H11	0.9500
C1—C2	1.379 (4)	C12—H12	0.9500
N1—Ag—N4^{i}	172.36 (8)	N1—C5—H5	118.6
N1—Ag—Ag^{ii}	89.29 (6)	C4—C5—H5	118.6
N4^{i}—Ag—Ag^{ii}	97.80 (6)	N2—C6—C3	121.0 (2)
O1—S1—O3	115.54 (16)	N2—C6—H6	119.5
O1—S1—O2	115.44 (17)	C3—C6—H6	119.5
O3—S1—O2	113.75 (15)	N3—C7—C8	119.1 (2)
O1—S1—C13	103.95 (14)	N3—C7—H7	120.4
O3—S1—C13	103.31 (13)	C8—C7—H7	120.4
O2—S1—C13	102.47 (14)	C12—C8—C9	117.9 (2)
C5—N1—C1	118.0 (2)	C12—C8—C7	121.2 (2)
C5—N1—Ag	118.75 (17)	C9—C8—C7	121.0 (2)
C1—N1—Ag	123.24 (17)	C10—C9—C8	119.4 (2)
C6—N2—N3	110.3 (2)	C10—C9—H9	120.3
C7—N3—N2	113.4 (2)	C8—C9—H9	120.3
C11—N4—C10	117.7 (2)	N4—C10—C9	123.1 (2)
C11—N4—Ag^{iii}	122.05 (17)	N4—C10—H10	118.4
C10—N4—Ag^{iii}	120.26 (17)	C9—C10—H10	118.4
N1—C1—C2	122.8 (2)	N4—C11—C12	122.7 (2)
N1—C1—H1	118.6	N4—C11—H11	118.6
C2—C1—H1	118.6	C12—C11—H11	118.6
C1—C2—C3	119.1 (2)	C11—C12—C8	119.3 (2)
C1—C2—H2	120.5	C11—C12—H12	120.4
C3—C2—H2	120.5	C8—C12—H12	120.4
C4—C3—C2	118.0 (2)	F2—C13—F3	107.5 (2)
C4—C3—C6	122.6 (2)	F2—C13—F1	106.6 (2)
C2—C3—C6	119.4 (2)	F3—C13—F1	107.1 (3)
C5—C4—C3	119.4 (2)	F2—C13—S1	112.5 (2)
C5—C4—H4	120.3	F3—C13—S1	111.70 (18)
C3—C4—H4	120.3	F1—C13—S1	111.1 (2)
N1—C5—C4	122.8 (2)		
Ag^{ii}—Ag—N1—C5	111.84 (19)	C12—C8—C9—C10	0.2 (4)

Ag^{ii}—Ag—N1—C1	−65.45 (19)	C7—C8—C9—C10	179.7 (2)
C6—N2—N3—C7	179.7 (2)	C11—N4—C10—C9	1.2 (4)
C5—N1—C1—C2	−1.3 (4)	Ag^{iii}—N4—C10—C9	179.98 (19)
Ag—N1—C1—C2	176.01 (19)	C8—C9—C10—N4	−1.0 (4)
N1—C1—C2—C3	0.1 (4)	C10—N4—C11—C12	−0.5 (4)
C1—C2—C3—C4	1.4 (4)	Ag^{iii}—N4—C11—C12	−179.3 (2)
C1—C2—C3—C6	−179.1 (2)	N4—C11—C12—C8	−0.3 (4)
C2—C3—C4—C5	−1.7 (4)	C9—C8—C12—C11	0.5 (4)
C6—C3—C4—C5	178.9 (2)	C7—C8—C12—C11	−179.1 (3)
C1—N1—C5—C4	1.0 (4)	O1—S1—C13—F2	−51.7 (3)
Ag—N1—C5—C4	−176.4 (2)	O3—S1—C13—F2	69.4 (2)
C3—C4—C5—N1	0.4 (4)	O2—S1—C13—F2	−172.2 (2)
N3—N2—C6—C3	180.0 (2)	O1—S1—C13—F3	−172.7 (2)
C4—C3—C6—N2	5.4 (4)	O3—S1—C13—F3	−51.7 (2)
C2—C3—C6—N2	−174.0 (2)	O2—S1—C13—F3	66.7 (2)
N2—N3—C7—C8	−179.3 (2)	O1—S1—C13—F1	67.8 (2)
N3—C7—C8—C12	172.3 (3)	O3—S1—C13—F1	−171.2 (2)
N3—C7—C8—C9	−7.2 (4)	O2—S1—C13—F1	−52.7 (2)

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O1	0.95	2.47	3.360 (4)	155
C10—H10···$O3^{iv}$	0.95	2.49	3.274 (3)	139
C11—H11···$O2^{v}$	0.95	2.54	3.235 (4)	130

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

Fig. 1

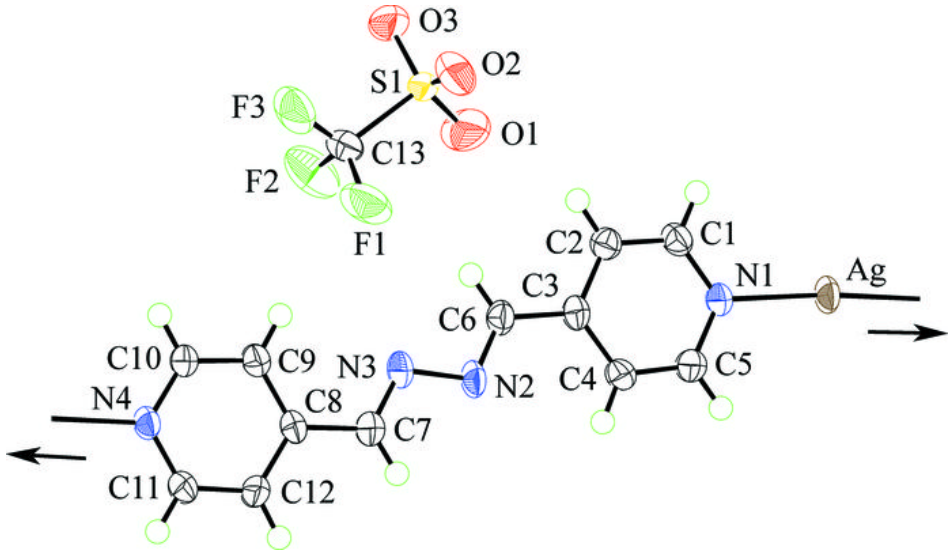
O3
O2
S1
O1
F3
C13
F2
F1
C2
C1
C3
N1
Ag
C6
C10
C9
N3
N2
C4
C5
N4
C8
C7
C11
C12

Fig. 2

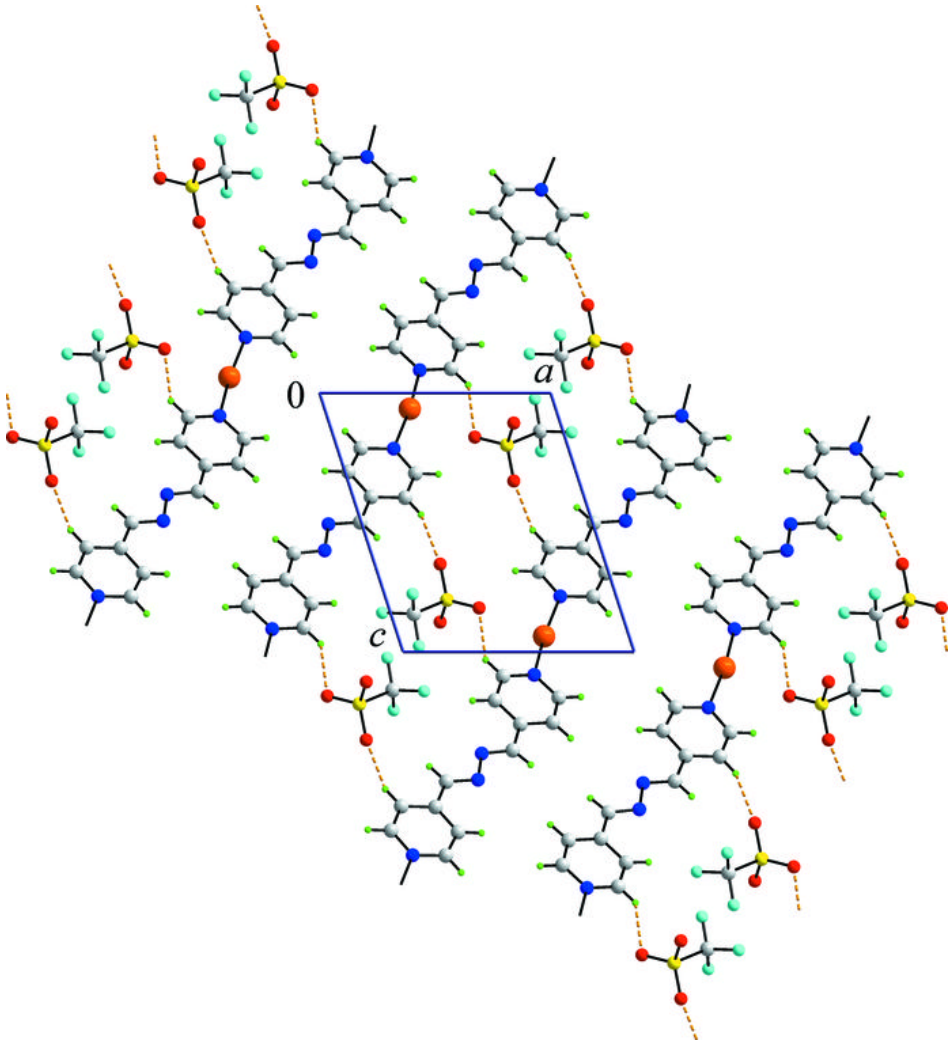

Fig. 3