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Acetonitrile(6,6′-dimethyl-2,2′-dipyridyl)silver(I) trifluoromethanesulfonate

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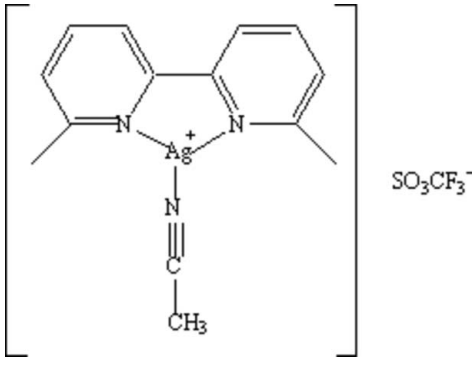
Key indicators: single-crystal X-ray study; T = 110 K; mean σ(C–C) = 0.003 Å; R factor = 0.025; wR factor = 0.062; data-to-parameter ratio = 15.0.

In the title compound, $[Ag(CH_3CN)(C_{12}H_{12}N_2)]CF_3SO_3$, the Ag atom is three-coordinated by one 6,6′-dimethyl-2,2′-dipyridyl ligand and one acetonitrile molecule, with trifluoromethanesulfonate acting as a noncoordinating counter-ion. The two pyridyl rings of the bipyridine ligand are nearly coplanar with a twist angle of 5.35 (5)° between the two mean planes. The commonly seen π-stacking interaction between the aromatic rings in bipyridine complexes, which reinforces further silver–silver interactions, is not observed in the crystal structure of the sterically hindered compound.

Related literature

For a general background on silver complexes of 2,2′-bipyridine derivatives, see: Swarnabala & Rajasekharan (1989); Sbrana (1990); Xu *et al.* (2001); Effendy *et al.* (2007); Di Nicola *et al.* (2007).



Experimental

Crystal data

$[Ag(C_2H_3N)(C_{12}H_{12}N_2)]CF_3O_3S$
M_r = 482.23
Orthorhombic, *Pbcn*
a = 21.9546 (11) Å
b = 6.9806 (4) Å
c = 22.6474 (11) Å
V = 3470.9 (3) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 1.33 mm^{-1}
T = 110 (2) K
0.40 × 0.26 × 0.20 mm

Data collection

Bruker X8 APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.66, T_{max} = 0.77
28162 measured reflections
3564 independent reflections
3067 reflections with $I > 2\sigma(I)$
R_{int} = 0.047

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.062
S = 1.04
3564 reflections
238 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.51 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ag1—N3	2.096 (2)	Ag1—N2	2.270 (2)
Ag1—N1	2.2684 (18)		
N3—Ag1—N1	138.71 (7)	N1—Ag1—N2	73.67 (7)
N3—Ag1—N2	147.05 (7)		

Data collection: *APEX2* (Bruker, 2003); cell refinement: *APEX2* and *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2000); software used to prepare material for publication: *SHELXTL*.

The Bruker X8 APEX diffractometer was purchased with funds received from the National Science Foundation Major Research Instrumentation Program grant CHE-0321214. KK thanks the Robert A. Welch Foundation for support (AA-1508).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2157).

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supplementary materials

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Acetonitrile(6,6'-dimethyl-2,2'-dipyridyl)silver(I) trifluoromethanesulfonate

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Comment

Silver(I) complexes of 2,2'-bipyridine and its bis-methylated derivatives have long been known (Swarnabala & Rajasekharan, 1989; Sbrana, 1990). These ligands normally coordinate in a chelating fashion and have been studied and used in a variety of approaches dealing with structural coordination chemistry in functionalized silver systems (Xu *et al.*, 2001; Effendy *et al.*, 2007). The bis-methylated 2,2'-bipyridine family of ligands has been extensively used as metal chelating group due to their redox stability and ease of functionalization (Di Nicola *et al.*, 2007). As neutral ligands, bipyridines form charged complexes with metal cations. In this study we present another coordination structure with 6,6'-dimethyl-2,2'-dipyridyl, coordinated to silver trifluoromethanesulfonate in a chelating fashion. A 1:1 metal to ligand ratio is observed in the crystal structure.

The title compound consists of one 6,6'-dimethyl-2,2'-dipyridyl ligand bound to the silver center in a chelating fashion, and one acetonitrile molecule which completes the coordination sphere of the metal atom. The angles around the silver center describe a distorted trigonal planar geometry, with the smallest angle being the N1—Ag1—N2 of 73.67 (7)°. The Ag—N distances fall in the range of reported values.

Experimental

The title compound was obtained by mixing AgOtf (0.082 g, 0.3 mmol) and 6,6'-dimethyl-2,2'-dipyridyl (0.055 g, 0.3 mmol) in 20 ml of acetonitrile. The mixture was stirred for 10 min and the solvent removed *via* vacuum. Diffraction-quality crystals were obtained by slow diffusion of hexanes into a concentrated THF solution of the title compound in the presence of air.

Refinement

All hydrogen atoms were included in calculated positions (C—H = 0.930–0.970 Å); isotropic displacement parameters were fixed [U_{iso}(H) = 1.2U_{iso}(C)].

Figures

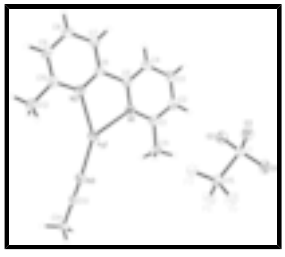


Fig. 1. A view of the molecular structure of the title compound; displacement ellipsoids are drawn at the 50% probability level

Acetonitrile(6,6'-dimethyl-2,2'-dipyridyl)silver(I) trifluoromethanesulfonate

Crystal data

$[Ag(C_2H_3N)(C_{12}H_{12}N_2)]CF_3O_3S$	F_{000} = 1920
M_r = 482.23	D_x = 1.846 Mg m^{-3}
Orthorhombic, *Pbcn*	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2n 2ab	θ = 3.1–26.4º
a = 21.9546 (11) Å	μ = 1.33 mm^{-1}
b = 6.9806 (4) Å	*T* = 110 (2) K
c = 22.6474 (11) Å	Block, colorless
V = 3470.9 (3) Å^3	0.40 × 0.26 × 0.20 mm
Z = 8	

Data collection

Bruker X8 APEX CCD area-detector diffractometer	3564 independent reflections
Radiation source: fine-focus sealed tube	3067 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.047
T = 110(2) K	θ_{max} = 26.4º
φ and ω scans	θ_{min} = 3.1º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	*h* = −27→27
T_{min} = 0.66, T_{max} = 0.77	*k* = −8→8
28162 measured reflections	*l* = −27→28

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.025	H-atom parameters constrained
$wR(F^2)$ = 0.062	$w = 1/[\sigma^2(F_o^2) + (0.0238P)^2 + 3.3879P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{max}$ = 0.001
3564 reflections	$\Delta\rho_{max}$ = 0.42 e Å^{-3}
238 parameters	$\Delta\rho_{min}$ = −0.51 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 $F^2 > 2\text{sigma}(F^2)$ 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	0.142814 (8)	0.60567 (3)	0.381071 (8)	0.02563 (7)
S1	0.08722 (3)	0.19499 (9)	0.10596 (2)	0.02357 (14)
F1	0.00400 (7)	−0.0158 (2)	0.15879 (7)	0.0360 (4)
F2	−0.00743 (7)	0.2881 (2)	0.17062 (7)	0.0367 (4)
F3	0.06284 (7)	0.1382 (2)	0.21841 (6)	0.0330 (4)
O1	0.04780 (8)	0.1974 (3)	0.05512 (7)	0.0336 (4)
O2	0.11393 (9)	0.3763 (3)	0.12188 (8)	0.0316 (4)
O3	0.12824 (8)	0.0343 (3)	0.10858 (8)	0.0292 (4)
N1	0.19042 (8)	0.6386 (3)	0.29276 (8)	0.0187 (4)
N2	0.24428 (9)	0.5966 (3)	0.40019 (8)	0.0197 (4)
N3	0.05438 (9)	0.6198 (3)	0.41563 (9)	0.0254 (5)
C1	0.16074 (11)	0.6647 (3)	0.24150 (10)	0.0216 (5)
C2	0.19238 (11)	0.7016 (3)	0.18942 (10)	0.0249 (5)
H2	0.1710	0.7199	0.1534	0.030*
C3	0.25485 (12)	0.7114 (4)	0.19066 (10)	0.0271 (5)
H3	0.2770	0.7368	0.1555	0.032*
C4	0.28528 (11)	0.6837 (3)	0.24355 (10)	0.0240 (5)
H4	0.3285	0.6897	0.2451	0.029*
C5	0.25181 (10)	0.6471 (3)	0.29427 (10)	0.0193 (5)
C6	0.28131 (10)	0.6156 (3)	0.35301 (10)	0.0190 (5)
C7	0.34430 (11)	0.6063 (4)	0.35921 (11)	0.0254 (5)
H7	0.3701	0.6213	0.3258	0.031*
C8	0.36892 (12)	0.5748 (4)	0.41459 (12)	0.0291 (6)
H8	0.4118	0.5668	0.4196	0.035*
C9	0.33067 (12)	0.5552 (3)	0.46256 (11)	0.0259 (5)
H9	0.3470	0.5335	0.5008	0.031*
C10	0.26820 (11)	0.5676 (3)	0.45434 (10)	0.0222 (5)
C11	0.09262 (11)	0.6522 (4)	0.24297 (11)	0.0277 (6)
H11A	0.0766	0.6616	0.2027	0.042*
H11B	0.0763	0.7573	0.2669	0.042*
H11C	0.0804	0.5294	0.2603	0.042*
C12	0.22439 (12)	0.5521 (4)	0.50488 (10)	0.0274 (6)
H12A	0.2014	0.6718	0.5085	0.041*
H12B	0.2470	0.5284	0.5415	0.041*
H12C	0.1962	0.4458	0.4977	0.041*
C13	0.00776 (11)	0.6529 (3)	0.43470 (10)	0.0202 (5)
C14	−0.05121 (10)	0.6967 (4)	0.45997 (10)	0.0245 (5)
H14A	−0.0459	0.7430	0.5005	0.037*

H14B	−0.0764	0.5809	0.4602	0.037*
H14C	−0.0712	0.7960	0.4363	0.037*
C15	0.03418 (11)	0.1500 (4)	0.16621 (10)	0.0256 (5)

Atomic displacement parameters (Å^2)

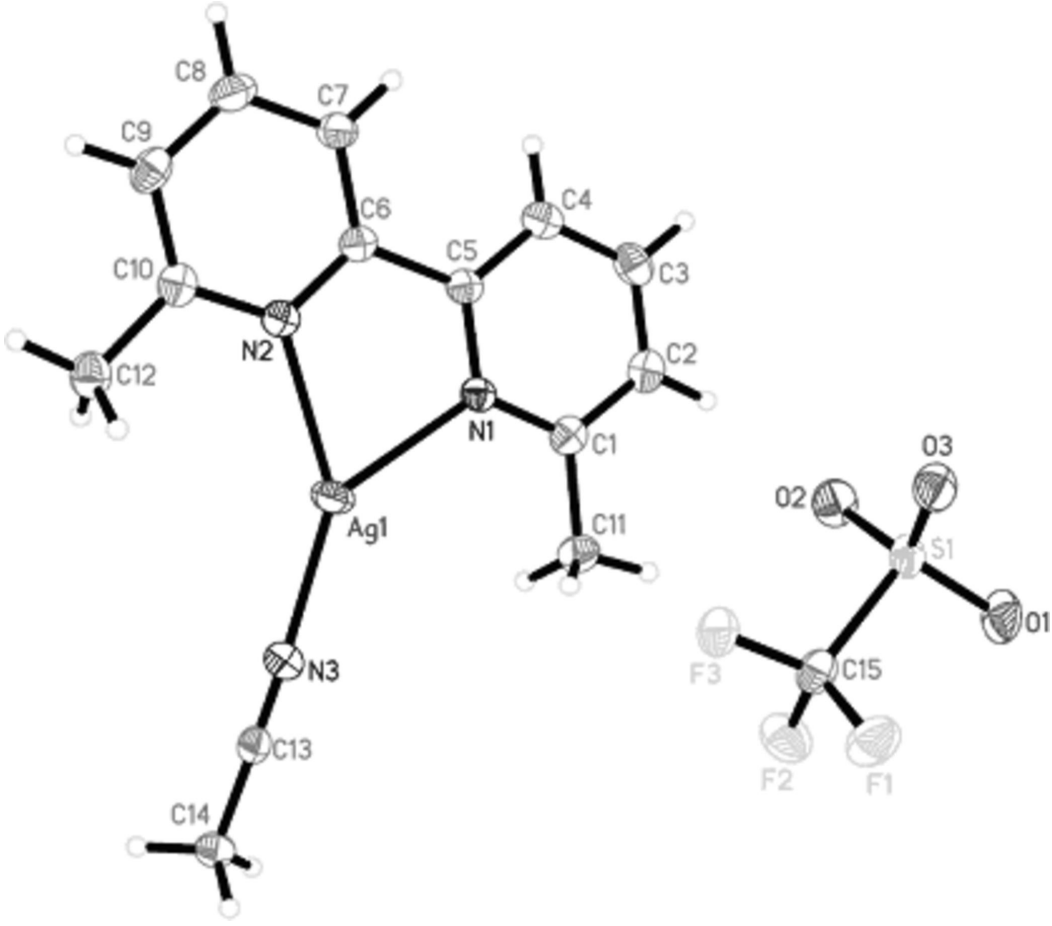
	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ag1	0.01857 (11)	0.02947 (12)	0.02884 (11)	−0.00015 (8)	0.00566 (7)	−0.00198 (8)
S1	0.0271 (3)	0.0254 (3)	0.0182 (3)	0.0020 (3)	−0.0020 (2)	−0.0009 (2)
F1	0.0345 (9)	0.0352 (9)	0.0384 (8)	−0.0113 (7)	−0.0048 (7)	0.0032 (7)
F2	0.0337 (9)	0.0407 (9)	0.0358 (8)	0.0115 (7)	0.0049 (7)	0.0019 (7)
F3	0.0329 (8)	0.0470 (10)	0.0190 (7)	−0.0028 (7)	−0.0040 (6)	0.0027 (6)
O1	0.0403 (11)	0.0416 (11)	0.0188 (8)	0.0067 (9)	−0.0077 (7)	0.0008 (8)
O2	0.0367 (11)	0.0258 (10)	0.0323 (10)	−0.0057 (8)	0.0026 (8)	−0.0009 (8)
O3	0.0301 (10)	0.0279 (10)	0.0295 (9)	0.0066 (8)	−0.0040 (7)	−0.0053 (8)
N1	0.0192 (10)	0.0158 (10)	0.0212 (9)	0.0009 (8)	0.0015 (7)	−0.0023 (8)
N2	0.0208 (10)	0.0153 (9)	0.0231 (9)	−0.0017 (8)	0.0010 (8)	−0.0020 (8)
N3	0.0229 (11)	0.0302 (12)	0.0232 (10)	−0.0004 (9)	0.0031 (8)	−0.0002 (9)
C1	0.0249 (12)	0.0141 (11)	0.0259 (12)	0.0018 (10)	−0.0020 (10)	−0.0018 (9)
C2	0.0313 (14)	0.0201 (12)	0.0234 (11)	0.0012 (11)	−0.0031 (10)	0.0017 (10)
C3	0.0329 (14)	0.0238 (13)	0.0245 (12)	−0.0020 (11)	0.0069 (10)	0.0020 (10)
C4	0.0231 (12)	0.0189 (12)	0.0300 (12)	−0.0011 (10)	0.0035 (10)	−0.0012 (10)
C5	0.0197 (11)	0.0138 (11)	0.0245 (11)	0.0013 (9)	0.0005 (9)	−0.0038 (9)
C6	0.0196 (12)	0.0127 (11)	0.0247 (11)	−0.0005 (9)	−0.0007 (9)	−0.0033 (9)
C7	0.0197 (12)	0.0287 (14)	0.0278 (12)	−0.0006 (10)	0.0012 (10)	−0.0056 (11)
C8	0.0207 (13)	0.0304 (15)	0.0363 (14)	0.0027 (11)	−0.0051 (11)	−0.0076 (11)
C9	0.0293 (14)	0.0206 (12)	0.0279 (12)	−0.0003 (10)	−0.0078 (10)	−0.0032 (10)
C10	0.0277 (13)	0.0144 (11)	0.0245 (12)	0.0008 (10)	−0.0013 (10)	−0.0021 (9)
C11	0.0223 (13)	0.0309 (14)	0.0299 (13)	0.0032 (11)	−0.0028 (10)	0.0000 (11)
C12	0.0320 (14)	0.0248 (13)	0.0254 (12)	−0.0026 (11)	−0.0009 (10)	−0.0008 (10)
C13	0.0226 (13)	0.0204 (12)	0.0177 (10)	−0.0012 (10)	−0.0024 (9)	0.0008 (9)
C14	0.0189 (12)	0.0278 (13)	0.0267 (12)	0.0031 (10)	0.0021 (9)	−0.0010 (11)
C15	0.0261 (13)	0.0278 (14)	0.0229 (12)	0.0013 (11)	−0.0066 (10)	0.0014 (10)

Geometric parameters (Å, °)

Ag1—N3	2.096 (2)	C4—C5	1.387 (3)
Ag1—N1	2.2684 (18)	C4—H4	0.9500
Ag1—N2	2.270 (2)	C5—C6	1.496 (3)
S1—O3	1.4398 (19)	C6—C7	1.391 (3)
S1—O2	1.4405 (19)	C7—C8	1.383 (4)
S1—O1	1.4405 (17)	C7—H7	0.9500
S1—C15	1.821 (3)	C8—C9	1.380 (4)
F1—C15	1.344 (3)	C8—H8	0.9500
F2—C15	1.332 (3)	C9—C10	1.387 (3)
F3—C15	1.342 (3)	C9—H9	0.9500
N1—C1	1.344 (3)	C10—C12	1.499 (3)
N1—C5	1.349 (3)	C11—H11A	0.9800
N2—C6	1.349 (3)	C11—H11B	0.9800

N2—C10	1.349 (3)	C11—H11C	0.9800
N3—C13	1.135 (3)	C12—H12A	0.9800
C1—C2	1.393 (3)	C12—H12B	0.9800
C1—C11	1.499 (3)	C12—H12C	0.9800
C2—C3	1.373 (4)	C13—C14	1.448 (3)
C2—H2	0.9500	C14—H14A	0.9800
C3—C4	1.385 (3)	C14—H14B	0.9800
C3—H3	0.9500	C14—H14C	0.9800
N3—Ag1—N1	138.71 (7)	C6—C7—H7	120.4
N3—Ag1—N2	147.05 (7)	C9—C8—C7	119.5 (2)
N1—Ag1—N2	73.67 (7)	C9—C8—H8	120.3
O3—S1—O2	114.81 (11)	C7—C8—H8	120.3
O3—S1—O1	114.69 (11)	C8—C9—C10	119.3 (2)
O2—S1—O1	115.76 (11)	C8—C9—H9	120.3
O3—S1—C15	103.57 (11)	C10—C9—H9	120.3
O2—S1—C15	102.95 (11)	N2—C10—C9	121.1 (2)
O1—S1—C15	102.51 (11)	N2—C10—C12	117.1 (2)
C1—N1—C5	120.0 (2)	C9—C10—C12	121.8 (2)
C1—N1—Ag1	123.51 (15)	C1—C11—H11A	109.5
C5—N1—Ag1	116.25 (14)	C1—C11—H11B	109.5
C6—N2—C10	120.0 (2)	H11A—C11—H11B	109.5
C6—N2—Ag1	115.94 (15)	C1—C11—H11C	109.5
C10—N2—Ag1	124.02 (15)	H11A—C11—H11C	109.5
C13—N3—Ag1	170.9 (2)	H11B—C11—H11C	109.5
N1—C1—C2	121.0 (2)	C10—C12—H12A	109.5
N1—C1—C11	117.2 (2)	C10—C12—H12B	109.5
C2—C1—C11	121.8 (2)	H12A—C12—H12B	109.5
C3—C2—C1	119.3 (2)	C10—C12—H12C	109.5
C3—C2—H2	120.3	H12A—C12—H12C	109.5
C1—C2—H2	120.3	H12B—C12—H12C	109.5
C2—C3—C4	119.5 (2)	N3—C13—C14	178.9 (3)
C2—C3—H3	120.3	C13—C14—H14A	109.5
C4—C3—H3	120.3	C13—C14—H14B	109.5
C3—C4—C5	119.1 (2)	H14A—C14—H14B	109.5
C3—C4—H4	120.4	C13—C14—H14C	109.5
C5—C4—H4	120.4	H14A—C14—H14C	109.5
N1—C5—C4	121.1 (2)	H14B—C14—H14C	109.5
N1—C5—C6	116.65 (19)	F2—C15—F3	107.47 (19)
C4—C5—C6	122.3 (2)	F2—C15—F1	107.1 (2)
N2—C6—C7	120.9 (2)	F3—C15—F1	106.76 (19)
N2—C6—C5	117.3 (2)	F2—C15—S1	111.71 (17)
C7—C6—C5	121.8 (2)	F3—C15—S1	111.78 (17)
C8—C7—C6	119.2 (2)	F1—C15—S1	111.71 (17)
C8—C7—H7	120.4		

Fig. 1


C8
C7
C9
C6
C4
C10
C5
C3
N2
C12
C2
N1
C1
O3
O2
Ag1
C11
S1
O1
N3
F3
C15
C13
F2
F1
C14