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Bis[2-(thiophen-2-yl)quinoxaline-κN^4]silver(I) perchlorate

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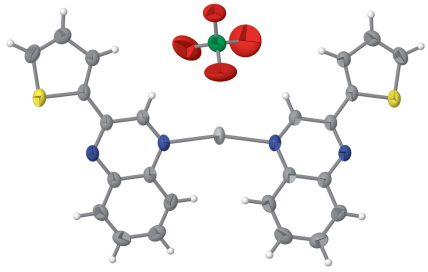
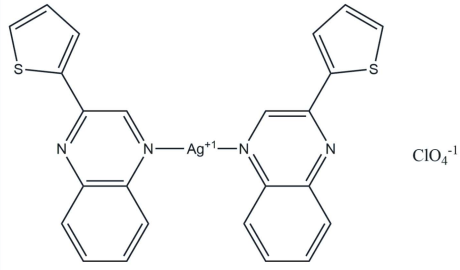
Structural data: full structural data are available from iucrdata.iucr.org

The crystal of the title salt, $[Ag(C_{12}H_8N_2S)_2]ClO_4$, has *C*2/*c* symmetry whereby the silver(I) atom sits on a twofold rotation axis, as does the perchlorate anion, which is disordered about this axis. The thienylquinoxaline ligand is nearly planar with the thienyl ring making a dihedral angle of 10.88 (8)° with respect to the quinoxaline moiety.

3D view



Chemical scheme



Structure description

The silver(I) metal center sits on a twofold symmetry axis (Fig. 1). As a result of the position of the twofold axis, the two thienylquinoxaline ligands, which are bonding *via* their quinoxaline N atoms, adopt a configuration whereby both of the quinoxaline units are pointing to the same side of the molecule, as opposed to the tetrafluoridoborate complex with the same cation, which crystallizes with the two ligands pointing in opposite directions (Crundwell, 2013). The thienylquinoxaline ligand is nearly planar, with the thienyl ring making a dihedral angle of 10.88 (8)° with respect to the quinoxaline moiety. This is similar to the nearly planar ligand configuration in the tetrafluoridoborate salt (Crundwell, 2013).

Synthesis and crystallization

Crystals were grown by combining warmed methanolic solutions of $AgClO_4$ and 2-thienylquinoxaline in a 1:2 molar ratio. The combined solution was pipetted into test tubes, which were then placed into amber vials and loosely sealed until small colorless crystals were observed. Crystals were harvested and used immediately since the silver salts deteriorate in light over days. When measuring of melting points was attempted, the crystals decomposed.

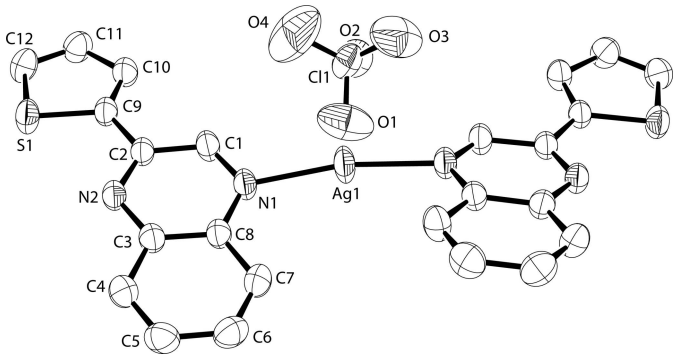


Figure 1
A view of the title compound (Farrugia, 2012). Displacement ellipsoids are drawn at the 50% probability level.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. The perchlorate disorder was treated by suppressing the generation of additional solvent atoms due to the anion's position on the symmetry axis. The perchlorate bond distances and oxygen-to-oxygen distances were restrained to 1.41 (1) and 2.30 (2) Å, respectively.

Funding information

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Table 1
Experimental details.

Crystal data	
Chemical formula	$[Ag(C_{12}H_8N_2S)_2]ClO_4$
M_r	631.85
Crystal system, space group	Monoclinic, *C*2/*c*
Temperature (K)	293
a, *b*, *c* (Å)	29.8739 (8), 10.6344 (4), 7.6425 (4)
β (°)	99.930 (4)
V (Å^3)	2391.58 (17)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	1.17
Crystal size (mm)	0.31 × 0.30 × 0.22
Data collection	
Diffractometer	Xcalibur CCD, Sapphire3
Absorption correction	Multi-scan (*CrysAlis PRO*; Agilent, 2014)
T_{min}, T_{max}	0.770, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	13270, 3957, 2441
R_{int}	0.027
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.753
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.030, 0.072, 0.82
No. of reflections	3957
No. of parameters	187
No. of restraints	10
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.36, −0.29

Computer programs: *CrysAlis CCD* (Oxford Diffraction, 2009), *CrysAlis RED* (Oxford Diffraction, 2009), *SHELXS97* (Sheldrick, 2008), *SHELXL* (Sheldrick, 2015), and *OLEX2* (Dolomanov *et al.*, 2009).

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full crystallographic data

IUCrData (2023). **8**, x230246 [https://doi.org/10.1107/S2414314623002468]

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Crystal data

$[Ag(C_{12}H_8N_2S)_2]ClO_4$
M_r = 631.85
Monoclinic, $C2/c$
a = 29.8739 (8) Å
b = 10.6344 (4) Å
c = 7.6425 (4) Å
β = 99.930 (4)°
V = 2391.58 (17) Å^3
Z = 4
$F(000)$ = 1264
D_x = 1.755 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5344 reflections
θ = 4.1–32.4°
μ = 1.17 mm^{-1}
T = 293 K
Block, colorless
0.31 × 0.30 × 0.22 mm

Data collection

Xcalibur CCD, Sapphire3 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (CrysAlisPRO; Agilent, 2014)
T_{min} = 0.770, T_{max} = 1.000
13270 measured reflections
3957 independent reflections
2441 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 32.4°, θ_{min} = 4.2°
h = −44→44
k = −15→15
l = −11→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.072
S = 0.82
3957 reflections
187 parameters
10 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0428P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Special details

Experimental. Hydrogen atoms were included in calculated positions with a C-H distance of 0.93 Å and were included in the refinement in riding motion approximation with $U_{iso} = 1.2U_{eq}$ of the carrier atom. Anion disorder treated with PART -1 and DFIX restraints.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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. Hydrogen atoms were placed in calculated positions with a C—H distance of 0.93 Å and were included in the refinement in a riding model approximation with $U_{iso} = 1.2\ U_{eq}$(C). Difference maps and oblong thermal parameters indicated that the perchlorate anion was disordered about a twofold axis.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Ag1	1.0000	0.15837 (2)	0.2500	0.05278 (10)	
S1	0.774221 (15)	0.02088 (5)	−0.17950 (8)	0.04958 (14)	
N1	0.92717 (5)	0.18494 (14)	0.1583 (2)	0.0404 (4)	
N2	0.83748 (4)	0.22291 (14)	−0.0167 (2)	0.0382 (4)	
C1	0.90225 (6)	0.09241 (18)	0.0828 (3)	0.0413 (4)	
H1	0.9148	0.0123	0.0854	0.050*	
C2	0.85637 (5)	0.11036 (17)	−0.0035 (3)	0.0353 (4)	
C3	0.86277 (6)	0.32029 (16)	0.0635 (3)	0.0372 (4)	
C4	0.84337 (7)	0.44149 (18)	0.0619 (3)	0.0458 (5)	
H4	0.8137	0.4548	0.0043	0.055*	
C5	0.86803 (8)	0.53904 (19)	0.1447 (3)	0.0503 (5)	
H5	0.8550	0.6186	0.1424	0.060*	
C6	0.91271 (8)	0.52096 (19)	0.2333 (3)	0.0514 (5)	
H6	0.9292	0.5887	0.2881	0.062*	
C7	0.93217 (6)	0.40506 (19)	0.2397 (3)	0.0472 (5)	
H7	0.9617	0.3935	0.3000	0.057*	
C8	0.90762 (5)	0.30265 (17)	0.1553 (3)	0.0366 (4)	
C9	0.82923 (6)	0.00112 (16)	−0.0714 (3)	0.0373 (4)	
C10	0.83990 (6)	−0.12423 (17)	−0.0520 (3)	0.0436 (5)	
H10	0.8681	−0.1542	0.0031	0.052*	
C11	0.80301 (7)	−0.2030 (2)	−0.1258 (3)	0.0509 (5)	
H11	0.8043	−0.2904	−0.1234	0.061*	
C12	0.76607 (7)	−0.1374 (2)	−0.1993 (3)	0.0514 (5)	
H12	0.7391	−0.1742	−0.2547	0.062*	
Cl1	0.9978 (2)	−0.17969 (10)	0.2283 (8)	0.0577 (9)	0.50
O1	1.00756 (19)	−0.0707 (4)	0.1315 (7)	0.1067 (16)	0.50
O2	1.00003 (13)	−0.2842 (3)	0.1151 (5)	0.0727 (10)	0.50
O3	0.95369 (19)	−0.1661 (5)	0.2619 (11)	0.124 (2)	0.50
O4	1.0300 (3)	−0.1931 (7)	0.3832 (9)	0.163 (3)	0.50

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ag1	0.02387 (9)	0.05375 (14)	0.07518 (19)	0.000	−0.00703 (9)	0.000

S1	0.0328 (2)	0.0521 (3)	0.0581 (4)	−0.00168 (19)	−0.0080 (2)	0.0023 (2)
N1	0.0255 (6)	0.0442 (9)	0.0494 (10)	0.0015 (5)	0.0002 (6)	0.0004 (7)
N2	0.0285 (6)	0.0427 (8)	0.0410 (10)	0.0017 (6)	−0.0006 (6)	0.0004 (7)
C1	0.0281 (7)	0.0404 (10)	0.0531 (13)	0.0023 (7)	0.0007 (8)	0.0003 (9)
C2	0.0274 (7)	0.0420 (9)	0.0355 (10)	−0.0003 (7)	0.0023 (7)	0.0003 (8)
C3	0.0325 (8)	0.0417 (10)	0.0366 (11)	0.0030 (7)	0.0036 (7)	0.0020 (8)
C4	0.0430 (10)	0.0450 (10)	0.0468 (13)	0.0074 (8)	0.0007 (9)	0.0023 (9)
C5	0.0588 (12)	0.0395 (10)	0.0521 (14)	0.0082 (9)	0.0080 (10)	−0.0001 (9)
C6	0.0549 (12)	0.0460 (11)	0.0529 (14)	−0.0096 (9)	0.0077 (10)	−0.0094 (10)
C7	0.0353 (9)	0.0502 (11)	0.0535 (14)	−0.0054 (8)	0.0003 (9)	−0.0055 (10)
C8	0.0283 (7)	0.0421 (9)	0.0389 (11)	−0.0002 (6)	0.0043 (7)	0.0004 (8)
C9	0.0273 (7)	0.0459 (10)	0.0374 (11)	−0.0007 (7)	0.0016 (7)	0.0006 (8)
C10	0.0342 (8)	0.0433 (10)	0.0510 (13)	−0.0003 (7)	0.0012 (8)	−0.0034 (9)
C11	0.0516 (11)	0.0433 (10)	0.0545 (14)	−0.0052 (9)	−0.0002 (10)	−0.0024 (10)
C12	0.0429 (9)	0.0547 (12)	0.0527 (14)	−0.0124 (9)	−0.0030 (9)	−0.0010 (10)
Cl1	0.0621 (12)	0.0518 (5)	0.053 (3)	0.0063 (9)	−0.0079 (17)	−0.0050 (8)
O1	0.152 (4)	0.055 (2)	0.132 (4)	−0.002 (3)	0.078 (4)	−0.005 (2)
O2	0.088 (2)	0.0528 (19)	0.074 (3)	0.0035 (19)	0.006 (2)	−0.0154 (18)
O3	0.116 (4)	0.105 (4)	0.172 (7)	0.036 (3)	0.082 (4)	0.035 (4)
O4	0.206 (8)	0.177 (7)	0.081 (4)	0.003 (6)	−0.045 (5)	−0.009 (4)

Geometric parameters (Å, º)

Ag1—N1	2.1860 (13)	C5—C6	1.402 (3)
Ag1—N1^{i}	2.1861 (13)	C6—H6	0.9300
S1—C9	1.7202 (17)	C6—C7	1.360 (3)
S1—C12	1.704 (2)	C7—H7	0.9300
N1—C1	1.307 (2)	C7—C8	1.406 (3)
N1—C8	1.380 (2)	C9—C10	1.373 (3)
N2—C2	1.320 (2)	C10—H10	0.9300
N2—C3	1.364 (2)	C10—C11	1.421 (3)
C1—H1	0.9300	C11—H11	0.9300
C1—C2	1.427 (2)	C11—C12	1.344 (3)
C2—C9	1.460 (2)	C12—H12	0.9300
C3—C4	1.412 (2)	Cl1—O1	1.432 (5)
C3—C8	1.414 (2)	Cl1—O2	1.417 (5)
C4—H4	0.9300	Cl1—O3	1.392 (7)
C4—C5	1.363 (3)	Cl1—O4	1.399 (6)
C5—H5	0.9300		

N1—Ag1—N1^{i}	165.14 (8)	C6—C7—H7	120.0
C12—S1—C9	91.83 (9)	C6—C7—C8	119.98 (18)
C1—N1—Ag1	120.27 (12)	C8—C7—H7	120.0
C1—N1—C8	117.94 (14)	N1—C8—C3	119.39 (15)
C8—N1—Ag1	121.17 (11)	N1—C8—C7	120.63 (15)
C2—N2—C3	117.24 (13)	C7—C8—C3	119.98 (17)
N1—C1—H1	118.9	C2—C9—S1	119.97 (13)
N1—C1—C2	122.13 (16)	C10—C9—S1	110.81 (13)

C2—C1—H1	118.9	C10—C9—C2	129.05 (16)
N2—C2—C1	121.37 (16)	C9—C10—H10	123.8
N2—C2—C9	119.37 (14)	C9—C10—C11	112.34 (17)
C1—C2—C9	119.20 (16)	C11—C10—H10	123.8
N2—C3—C4	119.64 (15)	C10—C11—H11	123.7
N2—C3—C8	121.78 (15)	C12—C11—C10	112.58 (18)
C4—C3—C8	118.53 (16)	C12—C11—H11	123.7
C3—C4—H4	119.9	S1—C12—H12	123.8
C5—C4—C3	120.16 (17)	C11—C12—S1	112.43 (15)
C5—C4—H4	119.9	C11—C12—H12	123.8
C4—C5—H5	119.6	O2—Cl1—O1	106.5 (5)
C4—C5—C6	120.87 (19)	O3—Cl1—O1	107.2 (4)
C6—C5—H5	119.6	O3—Cl1—O2	109.9 (5)
C5—C6—H6	119.8	O3—Cl1—O4	112.9 (6)
C7—C6—C5	120.47 (18)	O4—Cl1—O1	110.4 (6)
C7—C6—H6	119.8	O4—Cl1—O2	109.8 (4)

Ag1—N1—C1—C2	−170.09 (15)	C2—N2—C3—C8	0.8 (3)
Ag1—N1—C8—C3	167.57 (14)	C2—C9—C10—C11	175.2 (2)
Ag1—N1—C8—C7	−11.3 (3)	C3—N2—C2—C1	−3.4 (3)
S1—C9—C10—C11	0.0 (3)	C3—N2—C2—C9	173.90 (18)
N1^{i}—Ag1—N1—C1	162.84 (16)	C3—C4—C5—C6	−0.4 (3)
N1^{i}—Ag1—N1—C8	−7.97 (15)	C4—C3—C8—N1	−179.96 (19)
N1—C1—C2—N2	2.6 (3)	C4—C3—C8—C7	−1.1 (3)
N1—C1—C2—C9	−174.71 (19)	C4—C5—C6—C7	−0.6 (4)
N2—C2—C9—S1	3.9 (3)	C5—C6—C7—C8	0.8 (4)
N2—C2—C9—C10	−170.9 (2)	C6—C7—C8—N1	179.0 (2)
N2—C3—C4—C5	178.7 (2)	C6—C7—C8—C3	0.1 (3)
N2—C3—C8—N1	2.6 (3)	C8—N1—C1—C2	1.0 (3)
N2—C3—C8—C7	−178.51 (19)	C8—C3—C4—C5	1.2 (3)
C1—N1—C8—C3	−3.5 (3)	C9—S1—C12—C11	0.7 (2)
C1—N1—C8—C7	177.7 (2)	C9—C10—C11—C12	0.5 (3)
C1—C2—C9—S1	−178.79 (16)	C10—C11—C12—S1	−0.8 (3)
C1—C2—C9—C10	6.4 (4)	C12—S1—C9—C2	−176.09 (18)
C2—N2—C3—C4	−176.53 (19)	C12—S1—C9—C10	−0.42 (18)

Symmetry code: (i) −*x*+2, *y*, −*z*+1/2.