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Bis(μ-bis{[4-(2-pyridyl)pyrimidin-2-yl]-sulfanyl}methane)disilver(I) bis(perchlorate)

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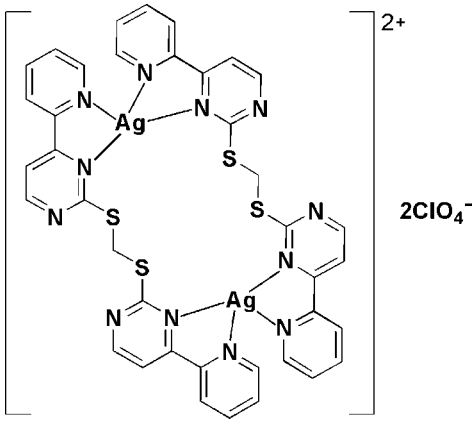
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.009 Å; disorder in solvent or counterion; R factor = 0.046; wR factor = 0.172; data-to-parameter ratio = 16.0.

In the macrocyclic centrosymmetric dinuclear complex, $[Ag_2(C_{19}H_{14}N_6S_2)_2](ClO_4)_2$, the Ag^I atom, bis{[4-(2-pyridyl)pyrimidin-2-yl]sulfanyl}methane (2-bppt) ligand and perchlorate anion each lie on a twofold rotation axis. The 2-bppt ligand chelates two four-coordinated Ag^I atoms through its two bipyridine-like arms. The O atoms of the perchlorate anion are disordered each over two positions of equal occupancy. Adjacent complex molecules are linked by π–π interactions between the pyridine and pyrimidine rings [centroid–centroid distance = 3.663 (8) Å].

Related literature

For Ag(I) coordination polymers, see: Chen *et al.* (2006). For the coordination chemistry of 4-(pyridin-*n*-yl)pyrimidin-2-thiol (*n* = 2, 3, 4) and their derivatives, see: Dong *et al.* (2009); Huang *et al.* (2007); Zhu *et al.* (2010).



Experimental

Crystal data

$[Ag_2(C_{19}H_{14}N_6S_2)_2](ClO_4)_2$
M_r = 1195.64
Orthorhombic, *Fddd*
a = 10.4382 (16) Å
b = 27.896 (4) Å
c = 30.089 (5) Å
V = 8761 (2) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 1.27 mm^{-1}
T = 298 K
0.15 × 0.12 × 0.10 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.832, T_{max} = 0.880
14503 measured reflections
2705 independent reflections
1640 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.172
S = 1.05
2705 reflections
169 parameters
24 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.64 e Å^{-3}

Table 1
Selected bond lengths (Å).

Ag1—N1	2.277 (4)	Ag1—N2	2.398 (3)

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT-Plus* (Bruker, 2007); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2383).

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supporting information

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Bis(*μ*-bis{[4-(2-pyridyl)pyrimidin-2-yl]sulfanyl}methane)disilver(I) bis-(perchlorate)

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S1. Comment

The supramolecular chemistry of Ag(I) coordination polymers is being a dynamic and thriving research field, which has attracted considerable interest (Chen *et. al*, 2006). For a long time, we have focused on the coordination chemistry of 4-(pyridin-*n*-yl)pyrimidin-2-thiol (*n* = 2, 3, 4) and their derivatives (Dong *et al.*, 2009; Huang *et al.*, 2007; Zhu *et al.*, 2010). Herein, we report a macrocyclic Ag(I) complex with bis[4-(2-pyridyl)pyrimidin-2-ylthio]methane (2-bppt) ligand.

The title compound shows a discrete macrocylic dinuclear structure, with perchlorate anions uncoordinated (Fig. 1). Each Ag^{I} ion is chelated by two sets of N,*N*-chelating donors from two 2-bppt ligands. The Ag—N bond distances are 2.277 (4) and 2.398 (3) Å (Table 1), while the N—Ag—N angles are in the range of 70.96 (13) to 158.7 (2)°. The Ag—Ag separation across the macrocycle is 8.167 (1) Å.

S2. Experimental

A CH_3CN solution of $AgClO_4$ (0.1 mmol) was layered above a CH_2Cl_2 solution of 2-bppt (0.1 mmol). Colorless crystals were obtained after one week. The crystals were collected and dried under vacuum (yield: 46%).

S3. Refinement

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93 and 0.97 Å and with U_{iso}(H) = 1.2U_{eq}(C).

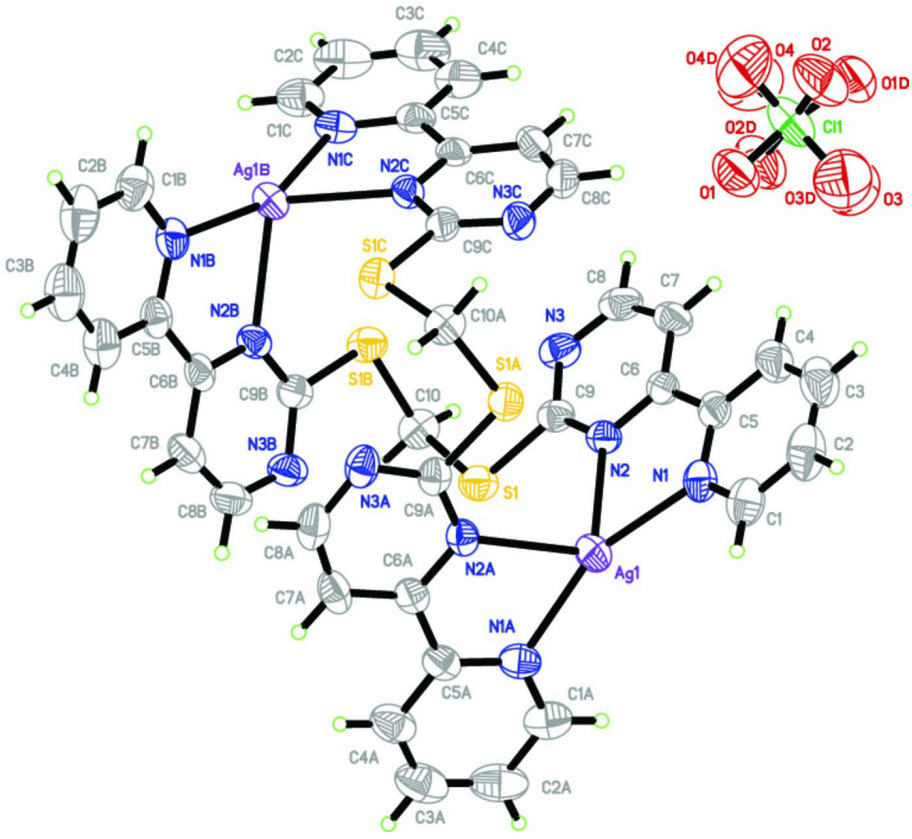

Figure 1

Molecular structure of the title compound, with the 30% probability displacement ellipsoids. [Symmetry codes: (A) 7/4 - *x*, 3/4 - *y*, *z*; (B) *x*, 3/4 - *y*, -1/4 - *z*; (C) 7/4 - *x*, *y*, -1/4 - *z*; (D) 9/4 - *x*, 1/4 - *y*, *z*.]

Bis(*μ*-bis{[4-(2-pyridyl)pyrimidin-2-yl]sulfanyl}methane)disilver(I) bis(perchlorate)

Crystal data

$[Ag_2(C_{19}H_{14}N_6S_2)_2](ClO_4)_2$
$M_r = 1195.64$
Orthorhombic, *Fddd*
Hall symbol: -F 2uv 2vw
$a = 10.4382\ (16)$ Å
$b = 27.896\ (4)$ Å
$c = 30.089\ (5)$ Å
$V = 8761\ (2)$ Å^3
$Z = 8$
$F(000) = 4768$
$D_x = 1.813$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2705 reflections
$\theta = 2.3–25.5°$
$\mu = 1.27$ mm^{-1}
$T = 298$ K
Block, colorless
0.15 × 0.12 × 0.10 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min} = 0.832$, $T_{max} = 0.880$
14503 measured reflections
2705 independent reflections
1640 reflections with $I > 2\sigma(I)$
$R_{int} = 0.035$
$\theta_{max} = 28.3°$, $\theta_{min} = 2.2°$
$h = -13\rightarrow12$
$k = -36\rightarrow35$
$l = -35\rightarrow40$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.172$
$S = 1.05$
2705 reflections
169 parameters
24 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
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.54$ e Å^{-3}
$\Delta\rho_{min} = -0.64$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Ag1	0.8750	0.3750	0.010711 (18)	0.0724 (3)	
S1	0.64901 (13)	0.37857 (5)	−0.07449 (5)	0.0703 (4)	
Cl1	1.1250	0.1250	−0.08929 (11)	0.1119 (9)	
C6	0.8563 (4)	0.27205 (15)	−0.03645 (15)	0.0602 (11)	
N2	0.7995 (3)	0.31493 (12)	−0.03991 (12)	0.0577 (9)	
N3	0.6715 (4)	0.28754 (15)	−0.10059 (13)	0.0708 (10)	
C9	0.7124 (4)	0.32014 (15)	−0.07181 (14)	0.0598 (10)	
C5	0.9565 (5)	0.26651 (16)	−0.00209 (16)	0.0652 (12)	
C10	0.5575 (6)	0.3750	−0.1250	0.0715 (18)	
H10A	0.5025	0.4030	−0.1267	0.086*	0.50
H10B	0.5025	0.3470	−0.1233	0.086*	0.50
N1	0.9792 (4)	0.30490 (14)	0.02472 (13)	0.0693 (10)	
C8	0.7272 (6)	0.24468 (18)	−0.09552 (17)	0.0804 (15)	
H8	0.7013	0.2199	−0.1141	0.096*	
C7	0.8198 (6)	0.23519 (16)	−0.06458 (18)	0.0761 (14)	
H7	0.8571	0.2050	−0.0625	0.091*	
C3	1.1171 (7)	0.2208 (3)	0.0336 (3)	0.107 (2)	
H3	1.1630	0.1924	0.0365	0.128*	
C1	1.0704 (5)	0.3010 (2)	0.05571 (19)	0.0873 (15)	
H1	1.0864	0.3268	0.0745	0.105*	
C2	1.1432 (5)	0.2585 (3)	0.0605 (3)	0.105 (2)	
H2	1.2078	0.2565	0.0817	0.125*	
C4	1.0244 (6)	0.2245 (2)	0.0026 (2)	0.0892 (16)	
H4	1.0062	0.1985	−0.0158	0.107*	
O1	1.1499 (10)	0.1740 (3)	−0.0861 (3)	0.120 (3)	0.50
O2	1.2437 (12)	0.1043 (4)	−0.0898 (4)	0.142 (4)	0.50
O3	1.0656 (13)	0.1194 (5)	−0.0423 (5)	0.166 (5)	0.50
O4	1.0504 (13)	0.1200 (6)	−0.1265 (5)	0.176 (5)	0.50

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ag1	0.0848 (4)	0.0529 (3)	0.0795 (4)	−0.0060 (2)	0.000	0.000
S1	0.0718 (8)	0.0712 (7)	0.0679 (8)	0.0074 (5)	−0.0057 (6)	−0.0091 (6)

Cl1	0.1036 (17)	0.0619 (12)	0.170 (3)	0.0054 (11)	0.000	0.000
C6	0.068 (3)	0.051 (2)	0.062 (3)	−0.0062 (19)	0.026 (2)	0.0064 (19)
N2	0.059 (2)	0.0522 (18)	0.062 (2)	−0.0070 (15)	0.0100 (18)	−0.0039 (15)
N3	0.074 (2)	0.071 (3)	0.067 (2)	−0.022 (2)	0.007 (2)	−0.0131 (19)
C9	0.058 (2)	0.063 (2)	0.059 (2)	−0.0073 (19)	0.012 (2)	−0.006 (2)
C5	0.065 (3)	0.062 (3)	0.069 (3)	0.002 (2)	0.023 (2)	0.013 (2)
C10	0.050 (3)	0.089 (5)	0.076 (4)	0.000	0.000	0.000 (3)
N1	0.066 (2)	0.068 (2)	0.074 (2)	−0.0072 (19)	0.004 (2)	0.0169 (19)
C8	0.095 (4)	0.075 (3)	0.071 (3)	−0.026 (3)	0.020 (3)	−0.020 (3)
C7	0.091 (4)	0.050 (2)	0.087 (4)	−0.008 (2)	0.034 (3)	−0.007 (2)
C3	0.092 (5)	0.102 (5)	0.126 (6)	0.029 (4)	0.023 (4)	0.036 (5)
C1	0.074 (3)	0.101 (4)	0.087 (4)	−0.006 (3)	−0.007 (3)	0.023 (3)
C2	0.065 (4)	0.129 (6)	0.120 (5)	0.007 (3)	−0.003 (3)	0.050 (5)
C4	0.087 (4)	0.081 (3)	0.099 (4)	0.022 (3)	0.027 (3)	0.019 (3)
O1	0.152 (7)	0.076 (5)	0.132 (7)	0.008 (5)	−0.029 (5)	−0.009 (4)
O2	0.133 (7)	0.105 (6)	0.188 (8)	0.044 (6)	−0.017 (6)	−0.012 (6)
O3	0.176 (9)	0.166 (8)	0.155 (8)	−0.016 (7)	0.028 (7)	0.007 (7)
O4	0.167 (8)	0.198 (9)	0.162 (8)	−0.025 (8)	−0.076 (7)	−0.041 (7)

Geometric parameters (Å, º)

Ag1—N1	2.277 (4)	C5—C4	1.377 (6)
Ag1—N2	2.398 (3)	C10—S1^{i}	1.798 (4)
S1—C9	1.761 (4)	C10—H10A	0.9700
S1—C10	1.798 (4)	C10—H10B	0.9700
Cl1—O4	1.370 (11)	N1—C1	1.337 (6)
Cl1—O2	1.367 (11)	C8—C7	1.368 (8)
Cl1—O1	1.395 (9)	C8—H8	0.9300
Cl1—O3	1.552 (13)	C7—H7	0.9300
C6—N2	1.339 (5)	C3—C2	1.355 (11)
C6—C7	1.385 (7)	C3—C4	1.348 (9)
C6—C5	1.479 (7)	C3—H3	0.9300
N2—C9	1.330 (5)	C1—C2	1.416 (9)
N3—C9	1.326 (5)	C1—H1	0.9300
N3—C8	1.338 (6)	C2—H2	0.9300
C5—N1	1.361 (6)	C4—H4	0.9300
N1—Ag1—N1^{ii}	158.7 (2)	S1^{i}—C10—H10A	108.3
N1—Ag1—N2	71.00 (14)	S1—C10—H10B	108.3
N1^{ii}—Ag1—N2	124.11 (13)	S1^{i}—C10—H10B	108.3
N1—Ag1—N2^{ii}	124.11 (13)	H10A—C10—H10B	107.4
N1^{ii}—Ag1—N2^{ii}	71.00 (14)	C5—N1—C1	118.3 (5)
N2—Ag1—N2^{ii}	101.13 (16)	C5—N1—Ag1	118.9 (3)
C9—S1—C10	100.78 (17)	C1—N1—Ag1	122.6 (4)
O4—Cl1—O4^{iii}	70.5 (13)	N3—C8—C7	123.9 (4)
O4—Cl1—O2	117.6 (10)	N3—C8—H8	118.1
O4^{iii}—Cl1—O2	61.2 (7)	C7—C8—H8	118.1
O4—Cl1—O2^{iii}	61.2 (7)	C8—C7—C6	117.8 (5)

O4^{iii}—Cl1—O2^{iii}	117.6 (10)	C8—C7—H7	121.1
O2—Cl1—O2^{iii}	178.7 (11)	C6—C7—H7	121.1
O4—Cl1—O1^{iii}	81.5 (8)	C2—C3—C4	119.8 (6)
O4^{iii}—Cl1—O1^{iii}	105.2 (8)	C2—C3—H3	120.1
O2—Cl1—O1^{iii}	75.8 (6)	C4—C3—H3	120.1
O2^{iii}—Cl1—O1^{iii}	104.3 (6)	N1—C1—C2	121.3 (6)
O4—Cl1—O1	105.2 (8)	N1—C1—H1	119.3
O4^{iii}—Cl1—O1	81.5 (8)	C2—C1—H1	119.3
O2—Cl1—O1	104.3 (6)	C3—C2—C1	118.9 (6)
O2^{iii}—Cl1—O1	75.8 (6)	C3—C2—H2	120.6
O1^{iii}—Cl1—O1	172.0 (9)	C1—C2—H2	120.6
O4—Cl1—O3^{iii}	168.9 (8)	C3—C4—C5	120.3 (6)
O4^{iii}—Cl1—O3^{iii}	120.5 (8)	C3—C4—H4	119.8
O2—Cl1—O3^{iii}	72.0 (7)	C5—C4—H4	119.8
O2^{iii}—Cl1—O3^{iii}	109.3 (9)	Cl1—O1—O2^{iii}	51.3 (4)
O1^{iii}—Cl1—O3^{iii}	96.3 (7)	Cl1—O1—O4^{iii}	48.6 (5)
O1—Cl1—O3^{iii}	76.3 (7)	O2^{iii}—O1—O4^{iii}	83.8 (7)
O4—Cl1—O3	120.5 (8)	Cl1—O1—O3^{iii}	55.7 (6)
O4^{iii}—Cl1—O3	168.9 (8)	O2^{iii}—O1—O3^{iii}	85.0 (7)
O2—Cl1—O3	109.3 (9)	O4^{iii}—O1—O3^{iii}	88.7 (8)
O2^{iii}—Cl1—O3	72.0 (7)	Cl1—O2—O4^{iii}	59.5 (7)
O1^{iii}—Cl1—O3	76.3 (7)	Cl1—O2—O1^{iii}	52.8 (5)
O1—Cl1—O3	96.3 (7)	O4^{iii}—O2—O1^{iii}	90.0 (9)
O3^{iii}—Cl1—O3	48.6 (10)	Cl1—O2—O3^{iii}	59.0 (6)
N2—C6—C7	119.6 (5)	O4^{iii}—O2—O3^{iii}	108.6 (10)
N2—C6—C5	117.4 (4)	O1^{iii}—O2—O3^{iii}	80.0 (8)
C7—C6—C5	123.0 (4)	O3^{iii}—O3—Cl1	65.7 (5)
C9—N2—C6	117.2 (4)	O3^{iii}—O3—O2^{iii}	104.2 (9)
C9—N2—Ag1	127.3 (3)	Cl1—O3—O2^{iii}	49.0 (5)
C6—N2—Ag1	115.4 (3)	O3^{iii}—O3—O1^{iii}	88.3 (12)
C9—N3—C8	113.5 (4)	Cl1—O3—O1^{iii}	48.0 (5)
N3—C9—N2	128.0 (4)	O2^{iii}—O3—O1^{iii}	75.8 (7)
N3—C9—S1	119.0 (4)	Cl1—O4—O2^{iii}	59.3 (7)
N2—C9—S1	113.0 (3)	Cl1—O4—O4^{iii}	54.8 (6)
N1—C5—C4	121.3 (5)	O2^{iii}—O4—O4^{iii}	103.6 (10)
N1—C5—C6	117.1 (4)	Cl1—O4—O1^{iii}	49.9 (6)
C4—C5—C6	121.6 (5)	O2^{iii}—O4—O1^{iii}	84.9 (9)
S1—C10—S1^{i}	115.8 (4)	O4^{iii}—O4—O1^{iii}	80.6 (11)
S1—C10—H10A	108.3		

Symmetry codes: (i) x, $-y+3/4$, $-z-1/4$; (ii) $-x+7/4$, $-y+3/4$, z; (iii) $-x+9/4$, $-y+1/4$, z.