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Poly[[bis(acetonitrile-κN)bis[μ_2-2,2′-(methylenedithio)bis(1,3,4-thiadiazole)-$\kappa^2N^4:N^{4'}$]copper(II)] bis(perchlorate) acetonitrile solvate]

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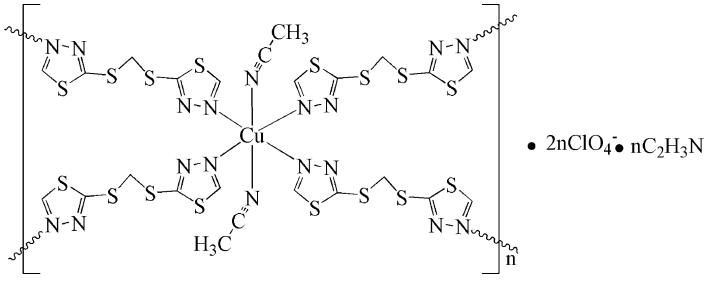
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.015 Å; disorder in solvent or counterion; R factor = 0.057; wR factor = 0.181; data-to-parameter ratio = 15.2.

In the title compound, $\{[Cu(C_5H_4N_4S_4)_2(C_2H_3N)_2](ClO_4)_2\cdot C_2H_3N\}_n$, the Cu^{II} atom occupies a crystallographic inversion centre and is six-coordinated by six N atoms of four symmetry-related 2,2′-(methylenedithio)bis(1,3,4-thiadiazole) (L) ligands and two acetonitrile molecules in a slightly distorted octahedral geometry. The ligand L adopts an N:N'-bidentate bridging mode in a *trans* configuration, bridging the Cu atoms *via* translation symmetry, forming a two-dimensional layer-like structure. The perchlorate ions serve as acceptors for intermolecular C—H···O hydrogen bonds, which link the layers into a three-dimensional network. The ClO_4^- anion is disordered with an occupation ratio of 0.658:0.342.

Related literature

For literature on Cu—N bonds, see: Huang *et al.* (2009); Qin *et al.* (2009); Wang *et al.* (2008).



Experimental

Crystal data

$[Cu(C_5H_4N_4S_4)_2(C_2H_3N)_2](ClO_4)_2\cdot C_2H_3N$
M_r = 1764.65
Monoclinic, $C2/c$
a = 19.3144 (18) Å
b = 9.9450 (9) Å
c = 18.8722 (18) Å
β = 98.876 (1)°
V = 3581.6 (6) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.28 mm^{-1}
T = 294 K
0.43 × 0.32 × 0.30 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.608, T_{max} = 0.702
12066 measured reflections
3285 independent reflections
2568 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.181
S = 1.07
3285 reflections
216 parameters
304 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.86 e Å^{-3}
$\Delta\rho_{min}$ = −0.83 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1···O3^{i}	0.93	2.35	2.955 (8)	123
C3—H3A···O1^{ii}	0.97	2.41	3.277 (9)	149
C5—H5···O1^{iii}	0.93	2.45	3.169 (9)	135

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x+\frac{1}{2}, -y+\frac{3}{2}, -z+1$; (iii) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; 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*.

The authors thank Luoyang Normal University for supporting this work.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2101).

References

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Wang, J. G., Qin, J. H., Hu, P. Z. & Zhao, B. T. (2008). *Z. Kristallogr. New Cryst. Struct.* **223**, 225–227.

supporting information

Acta Cryst. (2009). E**65**, m415 [doi:10.1107/S1600536809008708]

Poly[[bis(acetonitrile-κN)bis[μ_2-2,2′-(methylenedithio)bis(1,3,4-thiadiazole)-$\kappa^2N^4:N^{4'}$]copper(II)] bis(perchlorate) acetonitrile solvate]

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S1. Comment

The asymmetric unit of the title compound consists of half a Cu^{II} atom, two independent 2,2′-(methylenedithio)bis(1,3,4-thiadiazole) (*L*) ligands, one coordinated acetonitrile molecule, one uncoordinated acetonitrile molecule, and one perchlorate ion (Fig. 1). The Cu^{II} atom is coordinated by six N atoms, from four *L* ligands and two acetonitrile molecules, in a slightly distorted octahedral geometry. All six Cu—N bond distances are within the range expected for such coordination bonds (Huang *et al.*, 2009; Wang *et al.*, 2008; Qin *et al.*, 2009). The ligand *L* adopts a *N:N*′-bidentate bridging mode in *trans* configuration, so bridging the copper atoms *via* translation symmetry to form a two-dimensional layer-like structure, with a bridging Cu···Cu distance of 10.6661 (8) Å (Fig. 2). The centroid-centroid separation and dihedral angle of the thiadiazole rings are 6.3928 (5) Å and 81.86 (13)°, respectively.

In the crystal structure the region between the layes is taken up by perchlorate ions and uncoordinated acetonitrile molecules. The perchlorate ions serve as acceptors for C—H···O hydrogen-bonds, which link the chains into a three-dimensional network (Table 1 and Fig. 3).

S2. Experimental

The reaction of 2,2′-(methylenedithio)bis(1,3,4-thiadiazole) (0.2 mmol) with $Cu(ClO_4)_2$ (0.1 mmol) in an acetonitrile solution (20 ml) afforded a light blue solid after a few minutes. It was filtered off, washed with acetone, and dried in air. Single crystals, suitable for X-ray analysis, were obtained by slow diffusion of Et_2O into an acetonitrile solution of the solid.

S3. Refinement

All H-atoms were positioned geometrically and treated as riding: C—H = 0.93 - 09.7 Å with U_{iso}(H) = 1.2 or 1.5U_{eq}(parent C-atom).

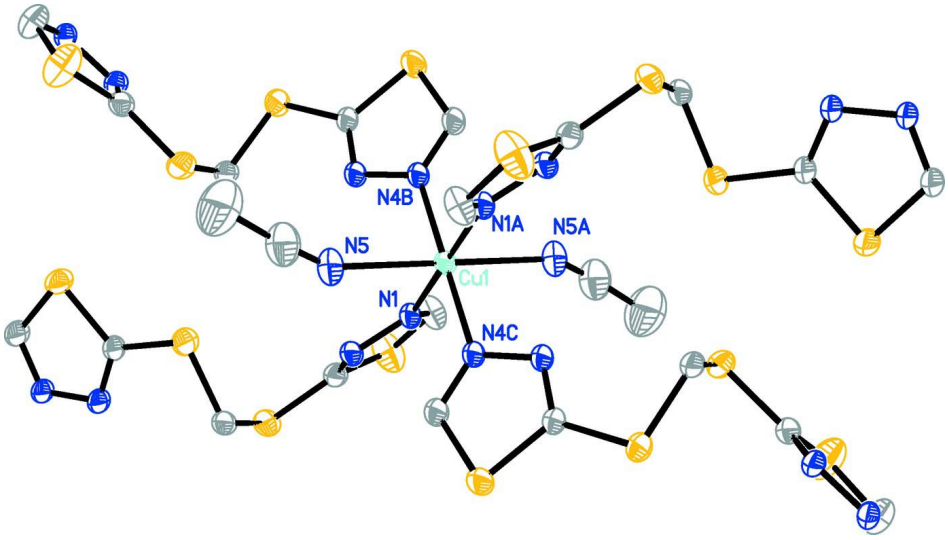

Figure 1

A view of the molecular structure of the cation in the title compound. Displacement ellipsoids are drawn at the 30% probability level. The H atoms, perchlorate ion, and uncoordinated acetonitrile molecules were omitted for clarity [symmetry operations: A: 1/2-x, 3/2-y, -z; B: 1/2-x, -1/2+y, 1/2-z; C: x, 2-y, -1/2+z].

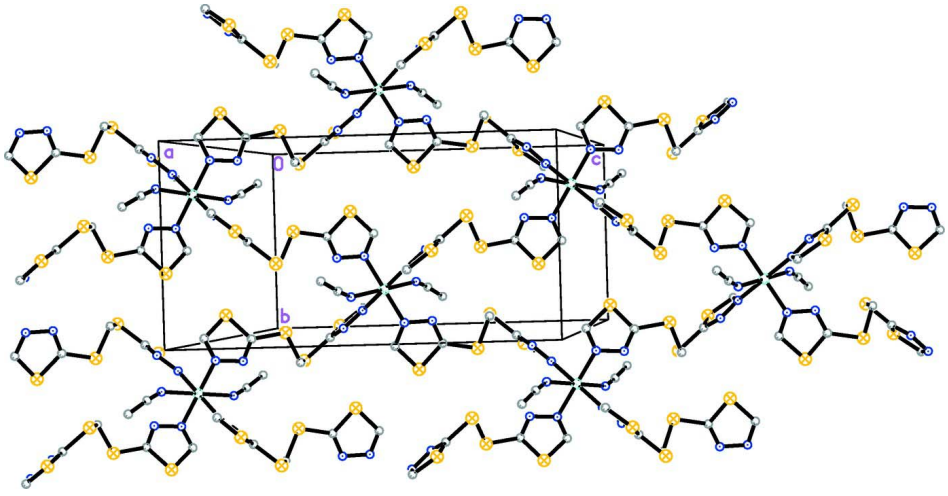

Figure 2

A view of the two-dimensional network in the title compound.

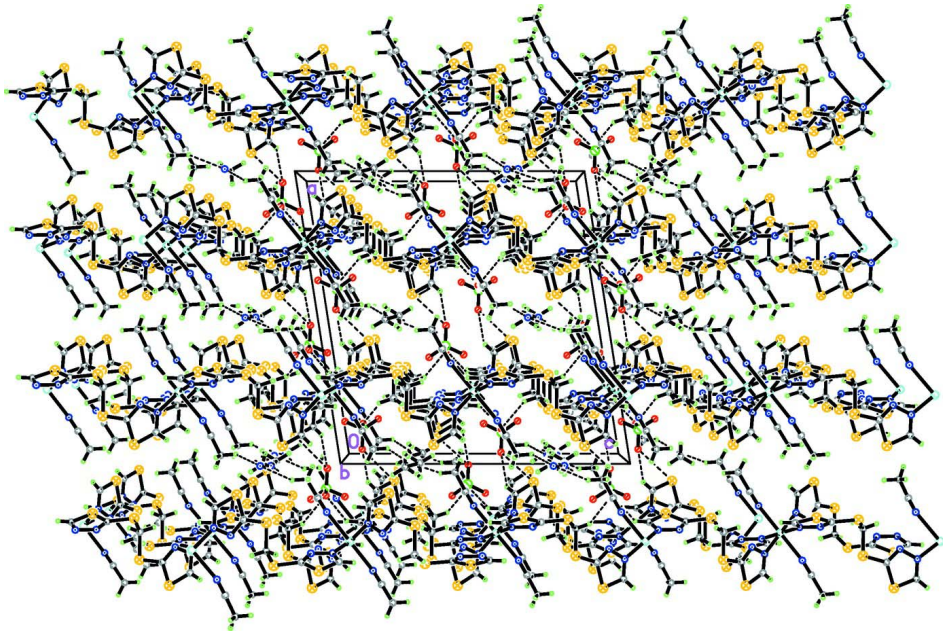

Figure 3

A view down the *b* axis of the crystal packing of the title compound.

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Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
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b = 9.9450 (9) Å
c = 18.8722 (18) Å
β = 98.876 (1)°
V = 3581.6 (6) Å^3
Z = 2
F(000) = 1780
D_x = 1.636 Mg m^{-3}
Mo *K*α radiation, λ = 0.71073 Å
Cell parameters from 5001 reflections
θ = 2.3–28.0°
μ = 1.28 mm^{-1}
T = 294 K
Block, blue
0.43 × 0.32 × 0.30 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.608, T_{max} = 0.702
12066 measured reflections
3285 independent reflections
2568 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 25.5°, θ_{min} = 2.6°
h = −23→23
k = −12→12
l = −22→22

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.181$
$S = 1.07$
3285 reflections
216 parameters
304 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
$w = 1/[\sigma^2(F_o^2) + (0.1007P)^2 + 13.6762P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.86$ e Å^{-3}
$\Delta\rho_{min} = -0.83$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Cl1	0.40159 (11)	0.1887 (3)	0.92446 (12)	0.1040 (7)	0.658 (6)
O1	0.3842 (4)	0.3266 (6)	0.9249 (4)	0.135 (2)	0.658 (6)
O2	0.3626 (4)	0.1399 (7)	0.8620 (4)	0.146 (3)	0.658 (6)
O3	0.4718 (3)	0.1679 (9)	0.9382 (4)	0.139 (2)	0.658 (6)
O4	0.3720 (5)	0.1390 (8)	0.9838 (4)	0.150 (3)	0.658 (6)
Cl1′	0.40159 (11)	0.1887 (3)	0.92446 (12)	0.1040 (7)	0.342 (6)
O1′	0.3333 (3)	0.2118 (9)	0.9325 (5)	0.135 (2)	0.342 (6)
O2′	0.4400 (4)	0.0929 (7)	0.9670 (4)	0.146 (3)	0.342 (6)
O3′	0.4391 (4)	0.3116 (7)	0.9371 (5)	0.139 (2)	0.342 (6)
O4′	0.4083 (5)	0.1678 (8)	0.8518 (3)	0.150 (3)	0.342 (6)
Cu1	0.2500	0.7500	0.0000	0.0403 (3)	
S1	0.41916 (9)	0.9410 (2)	0.16187 (11)	0.0858 (6)	
S2	0.32021 (8)	1.10571 (14)	0.23555 (7)	0.0551 (4)	
S3	0.18788 (8)	0.94899 (14)	0.24653 (6)	0.0542 (4)	
S4	0.17640 (9)	0.85456 (14)	0.39769 (7)	0.0584 (4)	
N1	0.3156 (2)	0.8589 (4)	0.07448 (19)	0.0424 (9)	
N2	0.2872 (2)	0.9444 (4)	0.12019 (19)	0.0421 (9)	
N3	0.2193 (2)	1.0883 (4)	0.36936 (19)	0.0445 (9)	
N4	0.2208 (2)	1.0822 (4)	0.44266 (19)	0.0425 (9)	
N5	0.1534 (3)	0.7792 (6)	0.0641 (3)	0.0662 (13)	
C1	0.3821 (3)	0.8491 (7)	0.0899 (3)	0.0651 (16)	
H1	0.4084	0.7955	0.0637	0.078*	
C2	0.3356 (3)	0.9939 (5)	0.1685 (2)	0.0454 (11)	
C3	0.2265 (3)	1.1032 (5)	0.2201 (3)	0.0498 (12)	
H3A	0.2105	1.1181	0.1694	0.060*	
H3B	0.2095	1.1773	0.2461	0.060*	
C4	0.1974 (3)	0.9770 (5)	0.3392 (2)	0.0410 (10)	
C5	0.1996 (3)	0.9686 (5)	0.4642 (3)	0.0509 (13)	
H5	0.1973	0.9505	0.5121	0.061*	
C6	0.1050 (4)	0.7464 (9)	0.0852 (4)	0.088 (2)	
C7	0.0400 (6)	0.6925 (15)	0.1112 (7)	0.144 (4)	
H7A	−0.0006	0.7375	0.0865	0.216*	
H7B	0.0434	0.7083	0.1618	0.216*	
H7C	0.0361	0.5977	0.1019	0.216*	

N6	1.000 (2)	−0.171 (2)	0.2314 (17)	0.212 (10)	0.50
C8	0.9970 (14)	−0.059 (2)	0.2284 (11)	0.208 (11)	0.50
C9	0.993 (2)	0.093 (2)	0.2242 (18)	0.204 (11)	0.50
H9A	1.0400	0.1296	0.2300	0.306*	0.50
H9B	0.9696	0.1270	0.2617	0.306*	0.50
H9C	0.9682	0.1199	0.1785	0.306*	0.50

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0787 (12)	0.1298 (17)	0.0987 (14)	0.0298 (12)	−0.0016 (10)	−0.0166 (13)
O1	0.118 (4)	0.147 (4)	0.137 (4)	0.040 (4)	0.012 (4)	−0.018 (4)
O2	0.136 (5)	0.150 (4)	0.141 (4)	0.019 (4)	−0.016 (4)	−0.013 (4)
O3	0.105 (4)	0.156 (4)	0.156 (5)	0.017 (4)	0.018 (4)	−0.001 (4)
O4	0.141 (5)	0.173 (5)	0.138 (4)	0.009 (4)	0.036 (4)	0.000 (4)
Cl1′	0.0787 (12)	0.1298 (17)	0.0987 (14)	0.0298 (12)	−0.0016 (10)	−0.0166 (13)
O1′	0.118 (4)	0.147 (4)	0.137 (4)	0.040 (4)	0.012 (4)	−0.018 (4)
O2′	0.136 (5)	0.150 (4)	0.141 (4)	0.019 (4)	−0.016 (4)	−0.013 (4)
O3′	0.105 (4)	0.156 (4)	0.156 (5)	0.017 (4)	0.018 (4)	−0.001 (4)
O4′	0.141 (5)	0.173 (5)	0.138 (4)	0.009 (4)	0.036 (4)	0.000 (4)
Cu1	0.0650 (6)	0.0348 (4)	0.0214 (4)	0.0057 (3)	0.0075 (3)	−0.0001 (3)
S1	0.0569 (9)	0.1161 (16)	0.0808 (12)	0.0020 (9)	−0.0006 (8)	−0.0383 (11)
S2	0.0796 (9)	0.0467 (7)	0.0378 (7)	−0.0078 (6)	0.0053 (6)	−0.0117 (5)
S3	0.0840 (10)	0.0491 (7)	0.0300 (6)	−0.0130 (6)	0.0106 (6)	−0.0096 (5)
S4	0.0932 (11)	0.0443 (7)	0.0401 (7)	−0.0216 (7)	0.0176 (7)	−0.0046 (5)
N1	0.058 (3)	0.042 (2)	0.0291 (18)	0.0056 (18)	0.0118 (17)	0.0004 (16)
N2	0.058 (2)	0.039 (2)	0.0292 (18)	0.0026 (18)	0.0076 (17)	−0.0023 (15)
N3	0.067 (3)	0.041 (2)	0.0275 (18)	−0.0069 (19)	0.0127 (17)	−0.0023 (16)
N4	0.064 (3)	0.040 (2)	0.0253 (18)	−0.0051 (18)	0.0104 (17)	−0.0005 (15)
N5	0.072 (3)	0.082 (3)	0.049 (3)	0.001 (3)	0.022 (2)	−0.006 (2)
C1	0.061 (4)	0.078 (4)	0.057 (3)	0.007 (3)	0.011 (3)	−0.020 (3)
C2	0.063 (3)	0.040 (2)	0.033 (2)	−0.002 (2)	0.007 (2)	0.0015 (19)
C3	0.085 (4)	0.037 (2)	0.029 (2)	0.006 (2)	0.013 (2)	−0.0002 (19)
C4	0.054 (3)	0.041 (2)	0.029 (2)	−0.003 (2)	0.0103 (19)	−0.0025 (19)
C5	0.078 (4)	0.046 (3)	0.030 (2)	−0.011 (3)	0.013 (2)	−0.004 (2)
C6	0.085 (4)	0.112 (5)	0.066 (4)	−0.001 (4)	0.012 (3)	−0.008 (3)
C7	0.116 (6)	0.184 (8)	0.138 (7)	−0.028 (6)	0.037 (5)	0.012 (7)
N6	0.208 (12)	0.212 (11)	0.217 (14)	−0.002 (10)	0.033 (10)	0.002 (9)
C8	0.202 (12)	0.209 (12)	0.213 (14)	0.002 (9)	0.030 (9)	0.003 (9)
C9	0.197 (14)	0.205 (12)	0.211 (15)	0.003 (10)	0.036 (10)	0.002 (9)

Geometric parameters (Å, º)

Cl1—O3	1.356 (5)	N1—C1	1.277 (8)
Cl1—O2	1.385 (5)	N1—N2	1.382 (5)
Cl1—O1	1.411 (5)	N2—C2	1.298 (6)
Cl1—O4	1.422 (5)	N3—C4	1.286 (6)
Cl1′—O1′	1.371 (5)	N3—N4	1.380 (5)

Cl1′—O2′	1.385 (5)	N4—C5	1.288 (6)
Cl1′—O4′	1.413 (5)	N4—Cu1^{iv}	2.021 (4)
Cl1′—O3′	1.422 (5)	N5—C6	1.119 (8)
Cu1—N4^{i}	2.021 (4)	C1—H1	0.9300
Cu1—N4^{ii}	2.021 (4)	C3—H3A	0.9700
Cu1—N1^{iii}	2.050 (4)	C3—H3B	0.9700
Cu1—N1	2.050 (4)	C5—H5	0.9300
Cu1—N5^{iii}	2.393 (5)	C6—C7	1.515 (11)
Cu1—N5	2.393 (5)	C7—H7A	0.9600
S1—C1	1.700 (6)	C7—H7B	0.9600
S1—C2	1.720 (6)	C7—H7C	0.9600
S2—C2	1.744 (5)	N6—C8	1.114 (6)
S2—C3	1.789 (6)	C8—C9	1.523 (8)
S3—C4	1.752 (4)	C9—H9A	0.9600
S3—C3	1.809 (5)	C9—H9B	0.9600
S4—C5	1.699 (5)	C9—H9C	0.9600
S4—C4	1.734 (5)		

O3—Cl1—O2	120.3	N2—N1—Cu1	119.4 (3)
O3—Cl1—O1	112.3	C2—N2—N1	111.1 (4)
O2—Cl1—O1	104.3	C4—N3—N4	111.1 (4)
O3—Cl1—O4	107.9	C5—N4—N3	113.3 (4)
O2—Cl1—O4	108.5	C5—N4—Cu1^{iv}	129.1 (3)
O1—Cl1—O4	102.0	N3—N4—Cu1^{iv}	117.5 (3)
O1′—Cl1′—O2′	119.3	C6—N5—Cu1	154.6 (6)
O1′—Cl1′—O4′	111.4	N1—C1—S1	115.2 (4)
O2′—Cl1′—O4′	109.9	N1—C1—H1	122.4
O1′—Cl1′—O3′	108.2	S1—C1—H1	122.4
O2′—Cl1′—O3′	106.3	N2—C2—S1	114.5 (4)
O4′—Cl1′—O3′	99.7	N2—C2—S2	124.6 (4)
N4^{i}—Cu1—N4^{ii}	180.0	S1—C2—S2	121.0 (3)
N4^{i}—Cu1—N1^{iii}	91.30 (15)	S2—C3—S3	114.6 (3)
N4^{ii}—Cu1—N1^{iii}	88.70 (15)	S2—C3—H3A	108.6
N4^{i}—Cu1—N1	88.70 (15)	S3—C3—H3A	108.6
N4^{ii}—Cu1—N1	91.30 (15)	S2—C3—H3B	108.6
N1^{iii}—Cu1—N1	180.0	S3—C3—H3B	108.6
N4^{i}—Cu1—N5^{iii}	89.75 (18)	H3A—C3—H3B	107.6
N4^{ii}—Cu1—N5^{iii}	90.25 (18)	N3—C4—S4	114.6 (3)
N1^{iii}—Cu1—N5^{iii}	92.10 (17)	N3—C4—S3	123.8 (3)
N1—Cu1—N5^{iii}	87.90 (17)	S4—C4—S3	121.7 (3)
N4^{i}—Cu1—N5	90.25 (18)	N4—C5—S4	114.4 (4)
N4^{ii}—Cu1—N5	89.75 (18)	N4—C5—H5	122.8
N1^{iii}—Cu1—N5	87.90 (17)	S4—C5—H5	122.8
N1—Cu1—N5	92.10 (17)	N5—C6—C7	176.0 (10)
N5^{iii}—Cu1—N5	180.0	C6—C7—H7A	109.5
C1—S1—C2	86.5 (3)	C6—C7—H7B	109.5
C2—S2—C3	98.9 (2)	H7A—C7—H7B	109.5
C4—S3—C3	99.0 (2)	C6—C7—H7C	109.5

C5—S4—C4	86.6 (2)	H7A—C7—H7C	109.5
C1—N1—N2	112.7 (4)	H7B—C7—H7C	109.5
C1—N1—Cu1	127.6 (4)	N6—C8—C9	180.0
N4^{i}—Cu1—N1—C1	63.0 (5)	C2—S1—C1—N1	−0.1 (5)
N4^{ii}—Cu1—N1—C1	−117.0 (5)	N1—N2—C2—S1	0.3 (5)
N5^{iii}—Cu1—N1—C1	−26.8 (5)	N1—N2—C2—S2	179.9 (3)
N5—Cu1—N1—C1	153.2 (5)	C1—S1—C2—N2	−0.1 (4)
N4^{i}—Cu1—N1—N2	−109.4 (3)	C1—S1—C2—S2	−179.8 (4)
N4^{ii}—Cu1—N1—N2	70.6 (3)	C3—S2—C2—N2	6.9 (5)
N5^{iii}—Cu1—N1—N2	160.8 (3)	C3—S2—C2—S1	−173.5 (3)
N5—Cu1—N1—N2	−19.2 (3)	C2—S2—C3—S3	71.7 (3)
C1—N1—N2—C2	−0.4 (6)	C4—S3—C3—S2	78.6 (3)
Cu1—N1—N2—C2	173.1 (3)	N4—N3—C4—S4	0.2 (5)
C4—N3—N4—C5	−0.6 (6)	N4—N3—C4—S3	179.3 (4)
C4—N3—N4—Cu1^{iv}	−177.6 (3)	C5—S4—C4—N3	0.2 (4)
N4^{i}—Cu1—N5—C6	−53.6 (14)	C5—S4—C4—S3	−179.0 (4)
N4^{ii}—Cu1—N5—C6	126.4 (14)	C3—S3—C4—N3	6.7 (5)
N1^{iii}—Cu1—N5—C6	37.7 (14)	C3—S3—C4—S4	−174.2 (3)
N1—Cu1—N5—C6	−142.3 (14)	N3—N4—C5—S4	0.8 (6)
N2—N1—C1—S1	0.3 (7)	Cu1^{iv}—N4—C5—S4	177.3 (3)
Cu1—N1—C1—S1	−172.5 (3)	C4—S4—C5—N4	−0.6 (5)

Symmetry codes: (i) −*x*+1/2, *y*−1/2, −*z*+1/2; (ii) *x*, −*y*+2, *z*−1/2; (iii) −*x*+1/2, −*y*+3/2, −*z*; (iv) −*x*+1/2, *y*+1/2, −*z*+1/2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···O3^{v}	0.93	2.35	2.955 (8)	123
C3—H3*A*···O1^{vi}	0.97	2.41	3.277 (9)	149
C5—H5···O1^{vii}	0.93	2.45	3.169 (9)	135

Symmetry codes: (v) −*x*+1, −*y*+1, −*z*+1; (vi) −*x*+1/2, −*y*+3/2, −*z*+1; (vii) −*x*+1/2, *y*+1/2, −*z*+3/2.