Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

Di-μ-chlorido-bis{[2-(2-pyridylmethylamino)ethanesulfonato-κ^3N,N',O]copper(II)}

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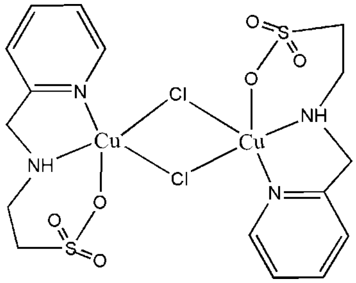
Received 30 November 2007; accepted 21 December 2007

Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.004 Å; R factor = 0.024; wR factor = 0.068; data-to-parameter ratio = 16.0.

In the title compound, $[Cu_2(C_8H_{11}N_2O_3S)_2Cl_2]$, the Cu atoms are five-coordinated in a distorted square-pyramidal geometry by three donor atoms of the deprotonated anionic 2-(2-pyridylmethylamino)ethanesulfonate (pmt) ligand and two Cl atoms. The Cl atoms bridge two Cu atoms, giving a binuclear structure; the centroid of the Cu_2Cl_2 ring lies on a crystallographic center of inversion. The complex is stabilized by hydrogen bonds and π–π stacking interactions [average interplanar distance = 3.4969 (1) Å and ring-centroid separation distance = 4.1068 (4) Å].

Related literature

For related literature, see: Li *et al.* (2006, 2007*a*,*b*).



Experimental

Crystal data

$[Cu_2(C_8H_{11}N_2O_3S)_2Cl_2]$
M_r = 628.48
Triclinic, $P\bar{1}$
a = 8.294 (1) Å
b = 8.362 (1) Å
c = 9.110 (1) Å
α = 103.773 (2)°
β = 98.118 (2)°
γ = 113.043 (2)°
V = 544.9 (1) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 2.43 mm^{-1}
T = 291 (2) K
0.33 × 0.20 × 0.09 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.504, T_{max} = 0.811
3270 measured reflections
2385 independent reflections
2224 reflections with $I > 2\sigma(I)$
R_{int} = 0.009

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.067
S = 1.10
2385 reflections
149 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H1N···O3^{i}	0.95 (3)	2.20 (3)	2.966 (2)	137 (2)

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2004); 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*.

This work was supported by the National Natural Science Foundation of China (No. 20471026) and the Natural Science Foundation of Henan Province (No. 0311021200).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2051).

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doi:10.1107/S1600536808000779

supporting information

Acta Cryst. (2008). E**64**, m341   [doi:10.1107/S1600536808000779]

Di-μ-chlorido-bis{[2-(2-pyridylmethylamino)ethanesulfonato-κ^3*N*,*N*′,*O*]copper(II)}

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S1. Comment

Previously, one Co (Li *et al.*, 2006) and two Cu complexes (Li *et al.*, 2007*a*,b) containing the reduced schiff base ligand 2-pyridine-2-ylmethylamino-ethanesulfonic acid (Hpmt) have been reported. Herein we describe the structure of another dinuclear Cu compound, Figure 1.

The title compound is a binuclear Cu complex and each Cu center has square-pyramidal geometry formed by two N and one O atoms of an anionic pmt ligand and two chlorine atoms. The plane N1/N2/O1/Cl1A defines the base of the pyramid while Cl1 occupies the apical position. Cu1 is situated 0.168 (1) Å above the N1/N2/O1/Cl1A plane. The chlorine atoms bridge the Cu atoms to form this binuclear structure, with a Cu1···Cu1(-*x*, -*y* + 2, -*z* + 1) distance of 3.461 (2) Å.

The N—H donor and S?O acceptor (Table 1) groups of the pmt ligand participate in hydrogen bonding and they join the dinuclear complex units into a one-dimensional chain structure along *b* axis (Figure 2 and Table 1). These chains are further expanded into a two-dimensional network *via* π-π stacking between the pyridine rings of adjacent parallel chains. The interplanar average distance and ring-centroid separation disstance are 3.4969 (1) Å and 4.1068 (4) Å, respectively.

S2. Experimental

The ligand 2-pyridin-2-ylmethylamino-ethanesulfonic acid (Hpmt) was prepared according to the method of Li *et al.* (2006). 10 ml of an aqueous solution of $CuCl_2 \times 2 H_2O$ (0.171 g, 1 mmol) was dropped into 10 ml of an methanolic solution of Hpmt (0.216 g, 1 mmol). Then the mixture was stirred for 6 h at 343 K.The filtrate was left to stand under air for about one week to obtain blue block-shaped crystals. Analysis, found (%): C, 30.51; H, 3.50; N, 8.91; S, 10.18. [$C_{16}H_{22}N_4O_6S_2Cl_2Cu_2$] requires (%): C,30.55; H,5.55; N,12.96; S,14.81.

S3. Refinement

H atoms bonded to C and N were positioned geometrically with C—H distances of 0.93 or 0.97 Å and a N—H distance of 0.95 Å, respectively, and treated as riding atoms with U_{iso}(H) = 1.2 U_{eq}(C or N).

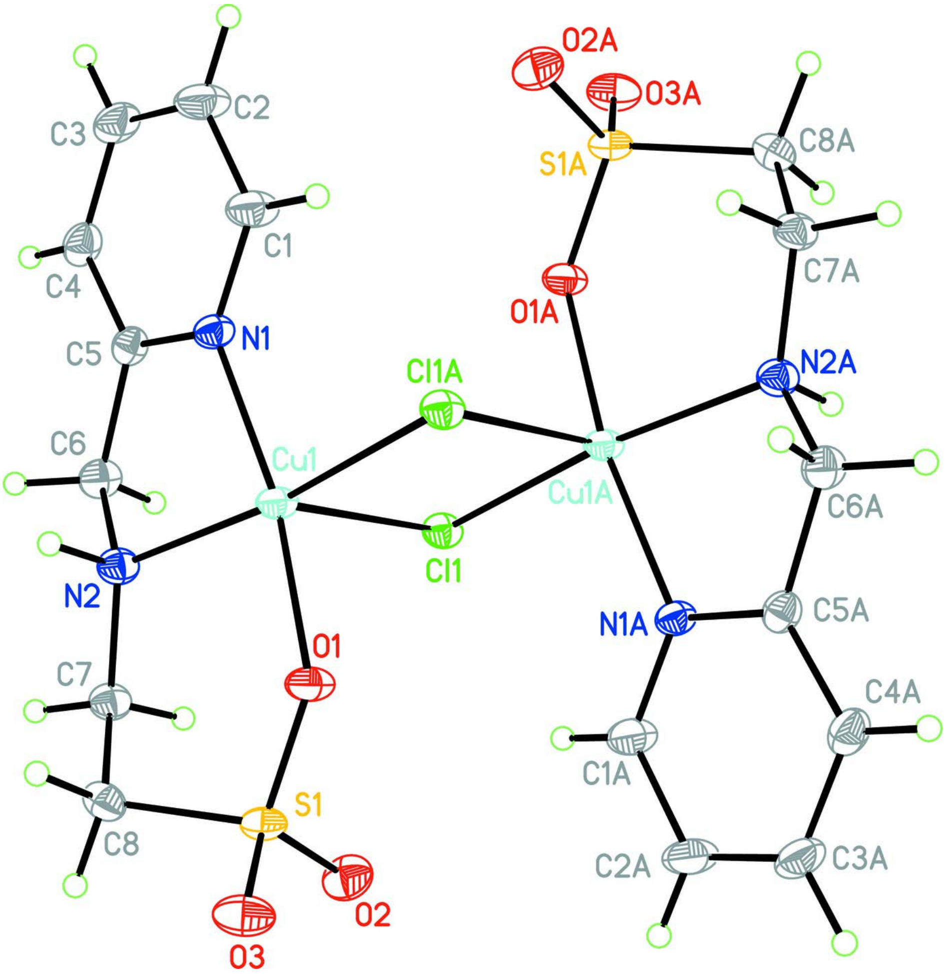

Figure 1

The molecular structure of (I) with atom-numbering scheme. Atoms with the suffix A are at the symmetry position (-*x*, -*y* + 2, -*z* + 1).

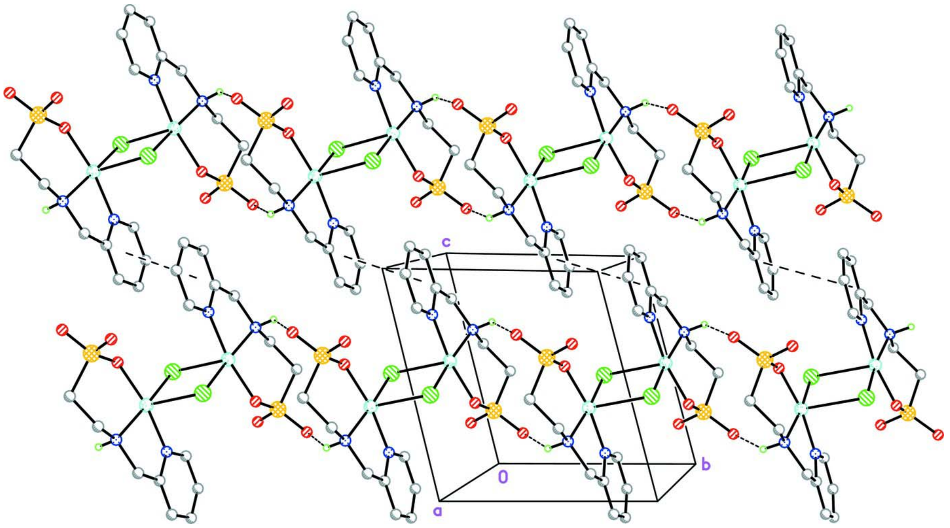

Figure 2

Packing diagram, projected on the *bc* plane, showing the hydrogen bonding and π-π stacking interactions. H atoms have partially been omitted for the sake of clarity.

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$c = 9.110$ (1) Å
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$\beta = 98.118$ (2)°
$\gamma = 113.043$ (2)°
$V = 544.9$ (1) Å^3

$Z = 1$
$F(000) = 318$
$D_x = 1.915$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2667 reflections
$\theta = 2.4$–28.3°
$\mu = 2.43$ mm^{-1}
$T = 291$ K
Block, blue
0.33 × 0.20 × 0.09 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.505$, $T_{max} = 0.811$

3270 measured reflections
2385 independent reflections
2224 reflections with $I > 2\sigma(I)$
$R_{int} = 0.009$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.4°$
$h = -10 \rightarrow 10$
$k = -10 \rightarrow 10$
$l = -9 \rightarrow 11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.023$
$wR(F^2) = 0.067$
$S = 1.10$
2385 reflections
149 parameters

0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement	$(\Delta/\sigma)_{max} = 0.001$
$w = 1/[\sigma^2(F_o^2) + (0.037P)^2 + 0.2091P]$ where $P = (F_o^2 + 2F_c^2)/3$	$\Delta\rho_{max} = 0.48$ e Å^{-3}
	$\Delta\rho_{min} = -0.34$ e Å^{-3}

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cu1	−0.00815 (3)	0.79189 (3)	0.40050 (2)	0.02480 (9)
Cl1	0.21525 (6)	1.15056 (7)	0.48221 (5)	0.02695 (12)
S1	0.25729 (6)	0.72960 (7)	0.64825 (6)	0.02540 (12)
O1	0.09412 (19)	0.7556 (2)	0.59265 (16)	0.0311 (3)
O2	0.4160 (2)	0.9013 (2)	0.72554 (19)	0.0385 (4)
O3	0.2140 (2)	0.6050 (2)	0.74013 (19)	0.0381 (4)
N1	−0.1223 (2)	0.7825 (2)	0.18742 (18)	0.0257 (3)
N2	0.1380 (2)	0.6952 (2)	0.2771 (2)	0.0276 (3)
C1	−0.2789 (3)	0.7931 (4)	0.1445 (3)	0.0382 (5)
H1	−0.3426	0.8078	0.2188	0.046*
C2	−0.3480 (3)	0.7828 (4)	−0.0065 (3)	0.0443 (6)
H2	−0.4578	0.7878	−0.0340	0.053*
C3	−0.2519 (3)	0.7652 (3)	−0.1151 (2)	0.0391 (5)
H3	−0.2961	0.7581	−0.2174	0.047*
C4	−0.0881 (3)	0.7580 (3)	−0.0712 (2)	0.0332 (5)
H4	−0.0203	0.7481	−0.1430	0.040*
C5	−0.0275 (3)	0.7659 (3)	0.0813 (2)	0.0264 (4)
C6	0.1491 (3)	0.7631 (3)	0.1417 (2)	0.0314 (4)
H6A	0.1726	0.6835	0.0600	0.038*
H6B	0.2478	0.8857	0.1731	0.038*
C7	0.3187 (3)	0.7178 (3)	0.3579 (2)	0.0287 (4)
H7A	0.3988	0.8472	0.4099	0.034*
H7B	0.3725	0.6699	0.2812	0.034*
C8	0.2998 (3)	0.6170 (3)	0.4776 (2)	0.0296 (4)
H8A	0.4104	0.6049	0.5074	0.035*
H8B	0.2011	0.4944	0.4297	0.035*
H1N	0.051 (4)	0.570 (4)	0.233 (3)	0.045 (7)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.02148 (14)	0.03819 (16)	0.02055 (13)	0.01626 (11)	0.00702 (9)	0.01323 (10)
Cl1	0.0222 (2)	0.0363 (3)	0.0269 (2)	0.01426 (19)	0.01029 (18)	0.01360 (18)
S1	0.0178 (2)	0.0297 (3)	0.0285 (2)	0.00845 (19)	0.00306 (18)	0.01438 (19)
O1	0.0270 (7)	0.0504 (9)	0.0255 (7)	0.0230 (7)	0.0082 (6)	0.0174 (6)
O2	0.0268 (8)	0.0346 (8)	0.0401 (8)	0.0044 (6)	0.0010 (6)	0.0087 (6)
O3	0.0324 (8)	0.0455 (9)	0.0432 (9)	0.0154 (7)	0.0091 (7)	0.0296 (7)
N1	0.0243 (8)	0.0332 (9)	0.0211 (7)	0.0132 (7)	0.0053 (6)	0.0109 (6)
N2	0.0229 (8)	0.0328 (9)	0.0281 (8)	0.0125 (7)	0.0071 (7)	0.0112 (7)
C1	0.0324 (11)	0.0617 (15)	0.0289 (10)	0.0259 (11)	0.0085 (9)	0.0196 (10)
C2	0.0384 (13)	0.0680 (17)	0.0339 (11)	0.0295 (12)	0.0035 (10)	0.0217 (11)
C3	0.0485 (14)	0.0455 (13)	0.0227 (10)	0.0210 (11)	0.0021 (9)	0.0139 (9)
C4	0.0422 (12)	0.0353 (11)	0.0224 (9)	0.0166 (10)	0.0103 (9)	0.0096 (8)
C5	0.0294 (10)	0.0245 (9)	0.0236 (9)	0.0101 (8)	0.0073 (8)	0.0080 (7)
C6	0.0297 (10)	0.0429 (12)	0.0262 (9)	0.0174 (9)	0.0123 (8)	0.0139 (8)
C7	0.0204 (9)	0.0343 (11)	0.0319 (10)	0.0118 (8)	0.0072 (8)	0.0115 (8)
C8	0.0246 (10)	0.0280 (10)	0.0388 (11)	0.0132 (8)	0.0078 (8)	0.0130 (8)

Geometric parameters (Å, º)

Cu1—O1	1.9775 (14)	C1—H1	0.9300
Cu1—N1	2.0051 (16)	C2—C3	1.371 (4)
Cu1—N2	2.0268 (17)	C2—H2	0.9300
Cu1—Cl1^{i}	2.2901 (5)	C3—C4	1.390 (3)
Cu1—Cl1	2.6796 (7)	C3—H3	0.9300
Cl1—Cu1^{i}	2.2901 (5)	C4—C5	1.386 (3)
S1—O2	1.4380 (16)	C4—H4	0.9300
S1—O3	1.4568 (15)	C5—C6	1.501 (3)
S1—O1	1.4916 (14)	C6—H6A	0.9700
S1—C8	1.774 (2)	C6—H6B	0.9700
N1—C5	1.345 (3)	C7—C8	1.519 (3)
N1—C1	1.346 (3)	C7—H7A	0.9700
N2—C6	1.475 (3)	C7—H7B	0.9700
N2—C7	1.492 (2)	C8—H8A	0.9700
N2—H1N	0.95 (3)	C8—H8B	0.9700
C1—C2	1.382 (3)		
O1—Cu1—N1	169.95 (7)	C3—C2—C1	118.9 (2)
O1—Cu1—N2	92.73 (6)	C3—C2—H2	120.5
N1—Cu1—N2	81.18 (7)	C1—C2—H2	120.5
O1—Cu1—Cl1^{i}	89.03 (4)	C2—C3—C4	119.45 (19)
N1—Cu1—Cl1^{i}	95.57 (5)	C2—C3—H3	120.3
N2—Cu1—Cl1^{i}	169.68 (5)	C4—C3—H3	120.3
O1—Cu1—Cl1	96.00 (5)	C5—C4—C3	118.9 (2)
N1—Cu1—Cl1	92.76 (5)	C5—C4—H4	120.5
N2—Cu1—Cl1	97.82 (5)	C3—C4—H4	120.5

Cl1^{i}—Cu1—Cl1	92.091 (19)	N1—C5—C4	121.49 (19)
Cu1^{i}—Cl1—Cu1	87.910 (19)	N1—C5—C6	115.27 (16)
O2—S1—O3	114.61 (10)	C4—C5—C6	123.21 (18)
O2—S1—O1	112.23 (10)	N2—C6—C5	108.83 (16)
O3—S1—O1	109.38 (9)	N2—C6—H6A	109.9
O2—S1—C8	107.16 (10)	C5—C6—H6A	109.9
O3—S1—C8	106.96 (10)	N2—C6—H6B	109.9
O1—S1—C8	105.98 (9)	C5—C6—H6B	109.9
S1—O1—Cu1	134.45 (9)	H6A—C6—H6B	108.3
C5—N1—C1	119.10 (17)	N2—C7—C8	110.64 (16)
C5—N1—Cu1	114.53 (13)	N2—C7—H7A	109.5
C1—N1—Cu1	126.36 (14)	C8—C7—H7A	109.5
C6—N2—C7	111.18 (16)	N2—C7—H7B	109.5
C6—N2—Cu1	108.32 (13)	C8—C7—H7B	109.5
C7—N2—Cu1	120.32 (13)	H7A—C7—H7B	108.1
C6—N2—H1N	104.2 (16)	C7—C8—S1	113.05 (14)
C7—N2—H1N	112.8 (16)	C7—C8—H8A	109.0
Cu1—N2—H1N	98.3 (16)	S1—C8—H8A	109.0
N1—C1—C2	122.1 (2)	C7—C8—H8B	109.0
N1—C1—H1	119.0	S1—C8—H8B	109.0
C2—C1—H1	119.0	H8A—C8—H8B	107.8

O1—Cu1—Cl1—Cu1^{i}	−89.25 (4)	N1—Cu1—N2—C7	158.15 (16)
N1—Cu1—Cl1—Cu1^{i}	95.68 (5)	Cl1^{i}—Cu1—N2—C7	−129.6 (2)
N2—Cu1—Cl1—Cu1^{i}	177.15 (5)	Cl1—Cu1—N2—C7	66.55 (14)
Cl1^{i}—Cu1—Cl1—Cu1^{i}	0.0	C5—N1—C1—C2	1.6 (4)
O2—S1—O1—Cu1	86.40 (15)	Cu1—N1—C1—C2	−179.12 (19)
O3—S1—O1—Cu1	−145.24 (13)	N1—C1—C2—C3	−1.4 (4)
C8—S1—O1—Cu1	−30.26 (16)	C1—C2—C3—C4	0.0 (4)
N1—Cu1—O1—S1	70.6 (4)	C2—C3—C4—C5	1.1 (4)
N2—Cu1—O1—S1	18.24 (15)	C1—N1—C5—C4	−0.5 (3)
Cl1^{i}—Cu1—O1—S1	−171.93 (14)	Cu1—N1—C5—C4	−179.82 (16)
Cl1—Cu1—O1—S1	−79.93 (14)	C1—N1—C5—C6	177.54 (19)
O1—Cu1—N1—C5	−68.7 (4)	Cu1—N1—C5—C6	−1.8 (2)
N2—Cu1—N1—C5	−15.48 (14)	C3—C4—C5—N1	−0.9 (3)
Cl1^{i}—Cu1—N1—C5	174.39 (13)	C3—C4—C5—C6	−178.7 (2)
Cl1—Cu1—N1—C5	82.02 (14)	C7—N2—C6—C5	−170.83 (17)
O1—Cu1—N1—C1	112.1 (4)	Cu1—N2—C6—C5	−36.51 (19)
N2—Cu1—N1—C1	165.2 (2)	N1—C5—C6—N2	25.8 (2)
Cl1^{i}—Cu1—N1—C1	−4.89 (19)	C4—C5—C6—N2	−156.21 (19)
Cl1—Cu1—N1—C1	−97.27 (19)	C6—N2—C7—C8	−169.70 (17)
O1—Cu1—N2—C6	−159.28 (14)	Cu1—N2—C7—C8	62.2 (2)
N1—Cu1—N2—C6	28.76 (14)	N2—C7—C8—S1	−74.07 (19)
Cl1^{i}—Cu1—N2—C6	101.0 (3)	O2—S1—C8—C7	−65.44 (17)
Cl1—Cu1—N2—C6	−62.84 (13)	O3—S1—C8—C7	171.21 (14)
O1—Cu1—N2—C7	−29.89 (15)	O1—S1—C8—C7	54.58 (17)

Symmetry code: (i) $-x$, $-y+2$, $-z+1$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H1*N*···O3^{ii}	0.95 (3)	2.20 (3)	2.966 (2)	137 (2)

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