Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis(diethylenetriamine-κ^3N)copper(II) bis(sulfadiazinate)

G. M. Golzar Hossain[a]* and A. J. Amoroso[b]

[a]Department of Chemistry, University of Dhaka, Dhaka 1000, Bangladesh, and [b]School of Chemistry, Cardiff University, Cardiff CF10 3AT, Wales

Correspondence e-mail: acsbd@yahoo.com

Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.004 Å
R factor = 0.043
wR factor = 0.112
Data-to-parameter ratio = 17.4

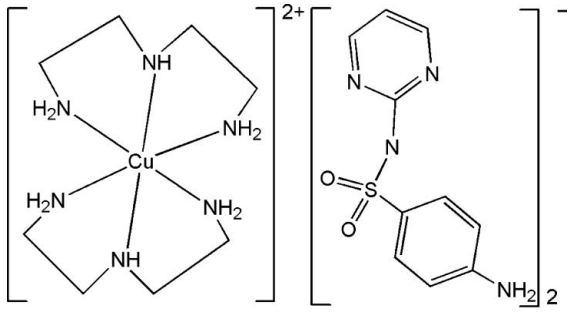
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 1 February 2007
Accepted 7 February 2007

In the title compound, $[Cu(C_4H_{13}N_3)_2](C_{10}H_9N_4O_2S)_2$, the Cu atom (site symmetry $\overline{1}$) displays a Jahn–Teller distorted octahedral CuN_6 geometry arising from the two tridentate diethylenetriamine ligands. The cation and anion interact by way of N—H···N and N—H···O hydrogen bonds.

Comment

We have attempted to show the coordination behaviour of sulfadiazine with the copper(II) ion in the presence of diethylenetriamine. In the title complex, (I), the diethylenetriamine molecule coordinates directly with the Cu atom and the sulfadiazine molecule acts as counter-ion. The crystal structure of (I) contains $[Cu(dien)_2]^{2+}$ cations and sdz^- counter-ions (dien = diethylenetriamine and sdzH = sulfadiazine), forming a salt.



(I)

The Cu^{II} centre (site symmetry $\overline{1}$) is octahedrally coordinated by two tridentate dien molecules, with the Cu—N bond distances (Table 1) showing a typical Jahn–Teller distortion (Ye *et al.*, 1998). The central N atom of the ligand displays the shortest Cu—N bond. The *cis* N—Cu—N angles vary from 80.49 (9) to 99.51 (9)°. The dihedral angle between the aromatic rings of the anion is 71.10 (14)°.

The cation and anion interact by way of N—H···N and N—H···O hydrogen bonds (Table 2), resulting in a three-dimensional framework (Fig. 2). A weak N—H···N bond between the anions also occurs. Compound (I) is the first copper complex containing sulfadiazine acting as a counter-ion.

Experimental

The sodium salt of sulfadiazine (Nasdz, 5.446 g, 2 mmol) was dissolved in hot methanol (50 ml) and a methanol solution (10 ml) of $CuCl_2{\cdot}2H_2O$ (1.705 g, 1 mmol) was added slowly with constant stirring on a hot plate. A red precipitate was formed and the mixture was

stirred for a futher 6 h. The precipitate was filtered off and dried over silica gel; it was then dissolved in dimethylformamide solution (50 ml), diethylenetriamine (5 ml) was added and the mixture stirred for 30 min. A week later, blue block-shaped crystals of (I) were filtered off and dried over silica gel.

Crystal data

$[Cu(C_4H_{13}N_3)_2](C_{10}H_9N_4O_2S)_2$
M_r = 768.43
Monoclinic, $P2_1/c$
a = 14.5949 (3) Å
b = 7.8231 (2) Å
c = 15.9672 (5) Å
β = 111.065 (1)°
V = 1701.26 (8) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.82 mm^{-1}
T = 150 (2) K
0.18 × 0.15 × 0.12 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
T_{min} = 0.866, T_{max} = 0.908
12097 measured reflections
3882 independent reflections
2736 reflections with $I > 2\sigma(I)$
R_{int} = 0.061

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.112
S = 1.04
3882 reflections
223 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.61 e Å^{-3}

Table 1
Selected bond lengths (Å).

Cu1—N2	2.030 (2)	Cu1—N1	2.339 (3)
Cu1—N3	2.116 (3)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1B···O12	0.92	2.06	2.887 (3)	149
N1—H1A···N11^{i}	0.92	2.24	3.121 (3)	161
N2—H2···N12^{ii}	0.93	2.14	3.068 (3)	174
N3—H3B···N11^{i}	0.92	2.44	3.283 (3)	152
N3—H3A···O12^{iii}	0.92	2.20	3.071 (3)	157
N14—H14A···N13^{iv}	0.88	2.47	3.161 (3)	136

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

The H atoms were positioned geometrically (C—H = 0.95–0.99 and N—H = 0.88–0.93 Å) and refined as riding, with U_{iso}(H) = 1.2U_{eq}(C,N).

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

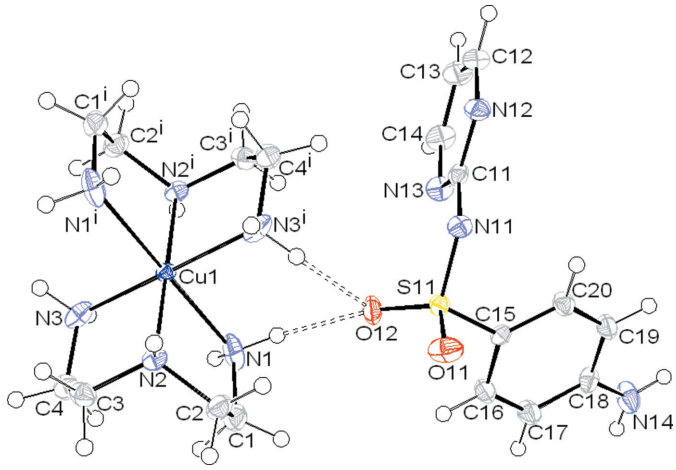


Figure 1
View of the molecular structure of (I), showing 50% displacement ellipsoids (arbitrary spheres for the H atoms) [symmetry code: (i) $-x, -y, -z$]. Hydrogen bonds are indicated by dashed lines.

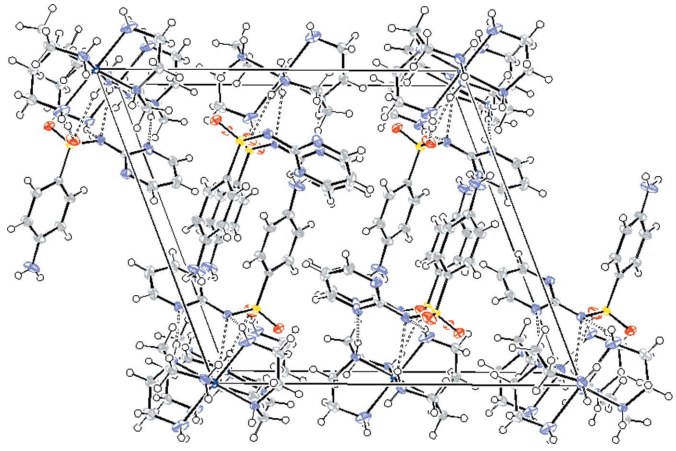


Figure 2
The packing of (I), viewed along the b axis. Dashed lines indicate the hydrogen-bonding interactions.

The authors acknowledge the School of Chemistry, Cardiff University, Wales.

References

Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Nonius (2000). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Ye, B.-H., Ji, L.-N., Xue, F. & Mak, T. C. W. (1998). *Polyhedron*, **17**, 2687–2692.

supporting information

Acta Cryst. (2007). E**63**, m759–m760 [https://doi.org/10.1107/S1600536807006551]

Bis(diethylenetriamine-κ^3N)copper(II) bis(sulfadiazinate)

G. M. Golzar Hossain and A. J. Amoroso

Bis(diethylenetriamine-κ^3N)copper(II) bis(disulfadiazinate)

Crystal data

$[Cu(C_4H_{13}N_3)_2](C_{10}H_9N_4O_2S)_2$
$M_r = 768.43$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 14.5949$ (3) Å
$b = 7.8231$ (2) Å
$c = 15.9672$ (5) Å
$\beta = 111.065$ (1)°
$V = 1701.26$ (8) Å^3
$Z = 2$
$F(000) = 806$
$D_x = 1.500$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3882 reflections
$\theta = 2.9$–$27.5°$
$\mu = 0.82$ mm^{-1}
$T = 150$ K
Block, blue
0.18 × 0.15 × 0.12 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: multi-scan (SORTAV; Blessing, 1995)
$T_{min} = 0.866$, $T_{max} = 0.908$
12097 measured reflections
3882 independent reflections
2736 reflections with $I > 2\sigma(I)$
$R_{int} = 0.061$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.9°$
$h = -18\rightarrow15$
$k = -9\rightarrow8$
$l = -20\rightarrow18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.112$
$S = 1.04$
3882 reflections
223 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0363P)^2 + 1.8984P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.36$ e Å^{-3}
$\Delta\rho_{min} = -0.61$ 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	0.0000	0.0000	0.0000	0.01426 (13)
S11	0.22755 (5)	0.49540 (8)	0.17688 (4)	0.01740 (17)
O11	0.16332 (15)	0.5537 (3)	0.22203 (14)	0.0298 (5)
O12	0.21618 (15)	0.3150 (2)	0.15310 (14)	0.0284 (5)
N11	0.20855 (17)	0.6196 (3)	0.09376 (15)	0.0193 (5)
N12	0.23381 (18)	0.7153 (3)	−0.03169 (16)	0.0236 (5)
N13	0.32361 (18)	0.4708 (3)	0.04694 (16)	0.0233 (5)
N14	0.63905 (19)	0.5729 (3)	0.42756 (18)	0.0361 (7)
H14A	0.6738	0.6611	0.4220	0.043*
H14B	0.6662	0.4941	0.4683	0.043*
C11	0.2580 (2)	0.5981 (3)	0.03619 (18)	0.0178 (6)
C12	0.2792 (2)	0.6999 (4)	−0.09041 (19)	0.0270 (7)
H12	0.2634	0.7787	−0.1388	0.032*
C13	0.3484 (2)	0.5741 (4)	−0.0842 (2)	0.0318 (7)
H13	0.3805	0.5657	−0.1263	0.038*
C14	0.3677 (2)	0.4622 (4)	−0.0134 (2)	0.0293 (7)
H14	0.4148	0.3745	−0.0070	0.035*
C15	0.34896 (19)	0.5224 (3)	0.25256 (17)	0.0160 (5)
C16	0.3897 (2)	0.4008 (4)	0.31956 (19)	0.0244 (6)
H16	0.3517	0.3052	0.3242	0.029*
C17	0.4846 (2)	0.4186 (4)	0.3790 (2)	0.0267 (7)
H17	0.5113	0.3357	0.4248	0.032*
C18	0.5426 (2)	0.5580 (4)	0.37272 (19)	0.0235 (6)
C19	0.4993 (2)	0.6831 (4)	0.30793 (19)	0.0238 (6)
H19	0.5359	0.7818	0.3048	0.029*
C20	0.4042 (2)	0.6649 (3)	0.24866 (19)	0.0216 (6)
H20	0.3762	0.7505	0.2047	0.026*
N1	0.13525 (19)	−0.0258 (3)	0.13378 (19)	0.0310 (6)
H1A	0.1653	−0.1300	0.1362	0.037*
H1B	0.1803	0.0591	0.1377	0.037*
N2	−0.06940 (17)	0.0168 (3)	0.08940 (15)	0.0184 (5)
H2	−0.1193	0.0972	0.0675	0.022*
N3	−0.02299 (19)	−0.2660 (3)	0.00658 (16)	0.0273 (6)
H3A	−0.0704	−0.3026	−0.0459	0.033*
H3B	0.0342	−0.3244	0.0142	0.033*
C1	0.0981 (2)	−0.0106 (4)	0.2073 (2)	0.0272 (7)
H1C	0.1462	0.0529	0.2576	0.033*
H1D	0.0901	−0.1260	0.2292	0.033*
C2	0.0005 (2)	0.0817 (4)	0.17680 (19)	0.0253 (7)
H2A	−0.0294	0.0684	0.2231	0.030*

H2B	0.0119	0.2052	0.1710	0.030*
C3	−0.1161 (2)	−0.1497 (4)	0.0942 (2)	0.0236 (6)
H3C	−0.1819	−0.1547	0.0464	0.028*
H3D	−0.1247	−0.1591	0.1528	0.028*
C4	−0.0547 (2)	−0.2989 (4)	0.0834 (2)	0.0243 (6)
H4A	0.0035	−0.3135	0.1389	0.029*
H4B	−0.0939	−0.4054	0.0728	0.029*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.0160 (3)	0.0133 (2)	0.0130 (2)	0.00058 (18)	0.00470 (18)	−0.00037 (17)
S11	0.0140 (3)	0.0193 (3)	0.0172 (3)	−0.0019 (3)	0.0035 (3)	0.0007 (3)
O11	0.0179 (11)	0.0489 (13)	0.0250 (11)	0.0028 (10)	0.0106 (9)	0.0044 (9)
O12	0.0305 (12)	0.0177 (10)	0.0285 (12)	−0.0083 (9)	0.0002 (10)	0.0009 (8)
N11	0.0192 (12)	0.0186 (11)	0.0189 (12)	0.0042 (9)	0.0055 (10)	0.0015 (9)
N12	0.0275 (14)	0.0209 (12)	0.0198 (13)	0.0031 (10)	0.0055 (11)	0.0043 (9)
N13	0.0225 (13)	0.0234 (13)	0.0246 (13)	0.0086 (10)	0.0094 (11)	0.0022 (9)
N14	0.0225 (15)	0.0298 (14)	0.0405 (17)	−0.0073 (12)	−0.0073 (13)	0.0074 (12)
C11	0.0150 (14)	0.0181 (13)	0.0169 (14)	−0.0003 (11)	0.0015 (11)	−0.0011 (10)
C12	0.0321 (18)	0.0295 (16)	0.0187 (15)	−0.0017 (13)	0.0080 (14)	0.0035 (12)
C13	0.038 (2)	0.0379 (18)	0.0262 (17)	−0.0002 (15)	0.0191 (15)	−0.0013 (14)
C14	0.0300 (18)	0.0324 (16)	0.0277 (17)	0.0088 (13)	0.0130 (14)	−0.0005 (12)
C15	0.0144 (13)	0.0179 (13)	0.0149 (13)	0.0012 (10)	0.0044 (11)	−0.0018 (10)
C16	0.0244 (16)	0.0211 (15)	0.0255 (16)	−0.0053 (12)	0.0064 (13)	0.0034 (11)
C17	0.0250 (17)	0.0250 (15)	0.0251 (16)	−0.0018 (13)	0.0029 (13)	0.0064 (12)
C18	0.0203 (16)	0.0213 (14)	0.0247 (16)	−0.0022 (12)	0.0029 (13)	−0.0040 (11)
C19	0.0225 (16)	0.0200 (14)	0.0260 (16)	−0.0054 (12)	0.0051 (13)	0.0011 (11)
C20	0.0213 (15)	0.0172 (13)	0.0239 (15)	0.0005 (11)	0.0052 (13)	0.0029 (11)
N1	0.0255 (15)	0.0162 (12)	0.0562 (19)	−0.0038 (10)	0.0207 (14)	−0.0067 (11)
N2	0.0160 (12)	0.0220 (12)	0.0162 (12)	0.0038 (9)	0.0045 (10)	0.0007 (9)
N3	0.0226 (14)	0.0356 (15)	0.0189 (13)	0.0044 (11)	0.0016 (11)	−0.0055 (10)
C1	0.0263 (16)	0.0243 (15)	0.0243 (16)	−0.0032 (13)	0.0010 (13)	0.0024 (12)
C2	0.0255 (17)	0.0315 (16)	0.0195 (15)	−0.0023 (13)	0.0088 (13)	−0.0049 (12)
C3	0.0174 (15)	0.0298 (16)	0.0235 (15)	−0.0002 (12)	0.0071 (12)	0.0057 (12)
C4	0.0203 (16)	0.0248 (15)	0.0230 (15)	0.0001 (12)	0.0020 (13)	0.0043 (11)

Geometric parameters (Å, º)

Cu1—N2	2.030 (2)	C16—H16	0.9500
Cu1—N3	2.116 (3)	C17—C18	1.406 (4)
Cu1—N1	2.339 (3)	C17—H17	0.9500
Cu1—N1^{i}	2.339 (3)	C18—C19	1.399 (4)
Cu1—N2^{i}	2.030 (2)	C19—C20	1.377 (4)
Cu1—N3^{i}	2.116 (3)	C19—H19	0.9500
S11—O11	1.446 (2)	C20—H20	0.9500
S11—O12	1.456 (2)	N1—C1	1.464 (4)
S11—N11	1.587 (2)	N1—H1A	0.9200

S11—C15	1.762 (3)	N1—H1B	0.9200
N11—C11	1.368 (3)	N2—C2	1.491 (4)
N12—C12	1.334 (4)	N2—C3	1.485 (3)
N12—C11	1.366 (3)	N2—H2	0.9300
N13—C14	1.339 (4)	N3—C4	1.481 (4)
N13—C11	1.349 (3)	N3—H3A	0.9200
N14—C18	1.370 (4)	N3—H3B	0.9200
N14—H14A	0.8800	C1—C2	1.512 (4)
N14—H14B	0.8800	C1—H1C	0.9900
C12—C13	1.387 (4)	C1—H1D	0.9900
C12—H12	0.9500	C2—H2A	0.9900
C13—C14	1.376 (4)	C2—H2B	0.9900
C13—H13	0.9500	C3—C4	1.519 (4)
C14—H14	0.9500	C3—H3C	0.9900
C15—C20	1.390 (4)	C3—H3D	0.9900
C15—C16	1.395 (4)	C4—H4A	0.9900
C16—C17	1.376 (4)	C4—H4B	0.9900

N1—Cu1—N1^{i}	180.0	C19—C18—C17	118.2 (3)
N2^{i}—Cu1—N2	180.0	C20—C19—C18	120.7 (3)
N3^{i}—Cu1—N3	180.0	C20—C19—H19	119.6
N1^{i}—Cu1—N2	99.56 (9)	C18—C19—H19	119.6
N1—Cu1—N2	80.44 (9)	C19—C20—C15	120.6 (3)
N1^{i}—Cu1—N2^{i}	80.44 (9)	C19—C20—H20	119.7
N1—Cu1—N2^{i}	99.56 (9)	C15—C20—H20	119.7
N1^{i}—Cu1—N3	91.92 (9)	C1—N1—Cu1	106.90 (18)
N1—Cu1—N3	88.08 (9)	C1—N1—H1A	110.3
N1^{i}—Cu1—N3^{i}	88.08 (9)	Cu1—N1—H1A	110.3
N1—Cu1—N3^{i}	91.92 (9)	C1—N1—H1B	110.3
N2^{i}—Cu1—N3^{i}	84.30 (9)	Cu1—N1—H1B	110.3
N2—Cu1—N3^{i}	95.70 (9)	H1A—N1—H1B	108.6
N2^{i}—Cu1—N3	95.70 (9)	C2—N2—C3	115.0 (2)
N2—Cu1—N3	84.30 (9)	C2—N2—Cu1	109.48 (17)
O11—S11—O12	113.71 (13)	C3—N2—Cu1	109.50 (16)
O11—S11—N11	105.91 (12)	C2—N2—H2	107.5
O12—S11—N11	113.97 (12)	C3—N2—H2	107.5
O11—S11—C15	107.01 (12)	Cu1—N2—H2	107.5
O12—S11—C15	106.70 (12)	C4—N3—Cu1	108.35 (17)
N11—S11—C15	109.30 (12)	C4—N3—H3A	110.0
C11—N11—S11	120.72 (18)	Cu1—N3—H3A	110.0
C12—N12—C11	116.4 (2)	C4—N3—H3B	110.0
C14—N13—C11	116.6 (2)	Cu1—N3—H3B	110.0
C18—N14—H14A	120.0	H3A—N3—H3B	108.4
C18—N14—H14B	120.0	N1—C1—C2	111.0 (2)
H14A—N14—H14B	120.0	N1—C1—H1C	109.4
N13—C11—N12	124.5 (3)	C2—C1—H1C	109.4
N13—C11—N11	121.8 (2)	N1—C1—H1D	109.4
N12—C11—N11	113.7 (2)	C2—C1—H1D	109.4

N12—C12—C13	123.1 (3)	H1C—C1—H1D	108.0
N12—C12—H12	118.4	N2—C2—C1	112.8 (2)
C13—C12—H12	118.4	N2—C2—H2A	109.0
C14—C13—C12	116.0 (3)	C1—C2—H2A	109.0
C14—C13—H13	122.0	N2—C2—H2B	109.0
C12—C13—H13	122.0	C1—C2—H2B	109.0
N13—C14—C13	123.3 (3)	H2A—C2—H2B	107.8
N13—C14—H14	118.3	N2—C3—C4	111.6 (2)
C13—C14—H14	118.3	N2—C3—H3C	109.3
C20—C15—C16	119.1 (3)	C4—C3—H3C	109.3
C20—C15—S11	121.5 (2)	N2—C3—H3D	109.3
C16—C15—S11	119.4 (2)	C4—C3—H3D	109.3
C17—C16—C15	120.4 (3)	H3C—C3—H3D	108.0
C17—C16—H16	119.8	N3—C4—C3	109.5 (2)
C15—C16—H16	119.8	N3—C4—H4A	109.8
C16—C17—C18	120.8 (3)	C3—C4—H4A	109.8
C16—C17—H17	119.6	N3—C4—H4B	109.8
C18—C17—H17	119.6	C3—C4—H4B	109.8
N14—C18—C19	120.0 (3)	H4A—C4—H4B	108.2
N14—C18—C17	121.8 (3)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*B*···O12	0.92	2.06	2.887 (3)	149
N1—H1*A*···N11^{ii}	0.92	2.24	3.121 (3)	161
N2—H2···N12^{iii}	0.93	2.14	3.068 (3)	174
N3—H3*B*···N11^{ii}	0.92	2.44	3.283 (3)	152
N3—H3*A*···O12^{i}	0.92	2.20	3.071 (3)	157
N14—H14*A*···N13^{iv}	0.88	2.47	3.161 (3)	136

Symmetry codes: (i) $-x, -y, -z$; (ii) $x, y-1, z$; (iii) $-x, -y+1, -z$; (iv) $-x+1, y+1/2, -z+1/2$.