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(2,2′-Bipyridine-κ^2N,N')(4-hydroxy-2-oxidobenzaldehyde thiosemicarbazonato-κ^3O^2,N^1,S)zinc(II)

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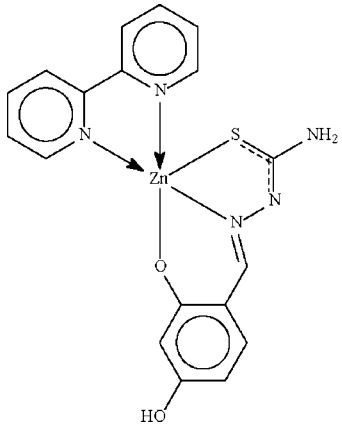
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.009 Å; R factor = 0.062; wR factor = 0.195; data-to-parameter ratio = 17.1.

The Zn^{II} atom in the title compound, $[Zn(C_8H_7N_3O_2S)(C_{10}H_8N_2)]$, is N,N'-chelated by the heterocycle and N,O,S-chelated by the doubly deprotonated Schiff base ligand in a distorted square-pyramidal environment. O—H···O and N—H···N hydrogen bonds link adjacent molecules into a layer structure.

Related literature

For the square-pyramidal 1,10-phenanthroline adduct, which exists as a monohydrated DMSO disolvate, see: Tan *et al.* (2009).



Experimental

Crystal data

$[Zn(C_8H_7N_3O_2S)(C_{10}H_8N_2)]$
M_r = 430.78
Monoclinic, $P2_1/c$
a = 16.1256 (4) Å
b = 7.0478 (2) Å
c = 17.6387 (5) Å
β = 113.646 (2)°
V = 1836.33 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.48 mm^{-1}
T = 100 (2) K
0.10 × 0.04 × 0.02 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.867, T_{max} = 0.971
15773 measured reflections
4191 independent reflections
2919 reflections with $I > 2\sigma(I)$
R_{int} = 0.086

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.195
S = 1.04
4191 reflections
245 parameters
24 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.88 e Å^{-3}
$\Delta\rho_{min}$ = −0.96 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O1^{i}	0.84	1.85	2.625 (5)	153
N3—H32···N2^{ii}	0.88	2.15	2.936 (7)	148

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2348).

References

Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Bruker (2007). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Tan, K. W., Ng, C. H., Maah, M. J. & Ng, S. W. (2009). *Acta Cryst.* E**65**, m61–m62.
Westrip, S. P. (2009). *publCIF*. In preparation.

supplementary materials

Acta Cryst. (2009). E**65**, m143 [**doi:10.1107/S1600536808043973**]

(2,2'-Bipyridine-κ^2N,N')(4-hydroxy-2-oxidobenzaldehyde thiosemicarbazonato-κ^3O^2,N^1,S)zinc(II)

K. W. Tan, C. H. Ng, M. J. Maah and S. W. Ng

Comment

(type here to add)

Experimental

Zinc acetate monohydrate (0.22 g, 1 mmol), 2,4-dihydroxybenzaldehyde thiosemicarbazone (0.21 g, 1 mmol) and 2,2'-bipyridine (0.16 g, 1 mmol) were heated in ethanol (40 ml). The compound that precipitated upon heating for 30 min was collected and recrystallized from DMF.

Refinement

Hydrogen atoms were placed in calculated positions (C–H 0.95, N–H 0.88, O–H 0.84 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2–1.5*U*(*C*,*N*,*O*).

The four carbon atoms of one of the two rings of the 2,2'-bipyridine molecule showed somewhat large anisotropic temperature factors. These were restrained to be nearly isotropic.

Figures

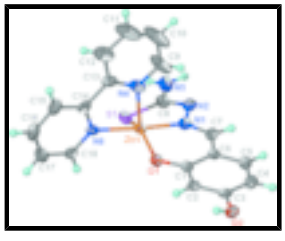


Fig. 1. Thermal ellipsoid (Barbour, 2001) plot of $Zn(C_8H_7N_3O_2S)(C_{10}H_8N_2)$ at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radii.

(2,2'-Bipyridine-κ^2N,N')(4-hydroxy-2-oxidobenzaldehyde thiosemicarbazonato-κ^3O^2,N^1,S)zinc(II)

Crystal data

$[Zn(C_8H_7N_3O_2S)(C_{10}H_8N_2)]$	F_{000} = 880
M_r = 430.78	D_x = 1.558 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 3053 reflections
a = 16.1256 (4) Å	θ = 2.3–26.2°
b = 7.0478 (2) Å	μ = 1.48 mm^{-1}

c = 17.6387 (5) Å	T = 100 (2) K
β = 113.646 (2)º	Prism, yellow
V = 1836.33 (9) Å^3	0.10 × 0.04 × 0.02 mm
Z = 4	

Data collection

Bruker SMART APEX diffractometer	4191 independent reflections
Radiation source: fine-focus sealed tube	2919 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.086
T = 100(2) K	θ_{max} = 27.5º
ω scans	θ_{min} = 1.4º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −20→20
T_{min} = 0.867, T_{max} = 0.971	k = −9→9
15773 measured reflections	l = −21→22

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.062	H-atom parameters constrained
$wR(F^2)$ = 0.195	$w = 1/[\sigma^2(F_o^2) + (0.1071P)^2 + 3.3408P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{max}$ = 0.001
4191 reflections	$\Delta\rho_{max}$ = 0.88 e Å^{-3}
245 parameters	$\Delta\rho_{min}$ = −0.96 e Å^{-3}
24 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Zn1	0.26010 (4)	0.61341 (9)	0.78222 (4)	0.0186 (2)
S1	0.35671 (10)	0.8297 (2)	0.75159 (9)	0.0239 (3)
O1	0.1393 (2)	0.4912 (5)	0.7423 (2)	0.0221 (8)
O2	−0.0804 (3)	−0.0017 (6)	0.6388 (2)	0.0238 (8)
H2	−0.0941	0.0325	0.6780	0.036*
N1	0.2953 (3)	0.4436 (7)	0.7058 (3)	0.0213 (10)
N2	0.3774 (3)	0.4754 (7)	0.6981 (3)	0.0226 (10)
N3	0.4867 (3)	0.6892 (7)	0.7112 (3)	0.0276 (11)
H31	0.5135	0.6021	0.6932	0.033*
H32	0.5107	0.8029	0.7242	0.033*
N4	0.3299 (3)	0.4781 (7)	0.8970 (3)	0.0276 (11)
N6	0.2340 (3)	0.7942 (6)	0.8656 (3)	0.0186 (9)

C1	0.1130 (4)	0.3300 (8)	0.7005 (3)	0.0198 (11)
C2	0.0305 (3)	0.2459 (8)	0.6918 (3)	0.0192 (11)
H2A	−0.0049	0.3053	0.7170	0.023*
C3	−0.0009 (4)	0.0790 (8)	0.6476 (3)	0.0199 (11)
C4	0.0493 (4)	−0.0115 (8)	0.6105 (3)	0.0250 (12)
H4	0.0276	−0.1248	0.5798	0.030*
C5	0.1305 (4)	0.0642 (8)	0.6187 (3)	0.0246 (12)
H5	0.1644	0.0017	0.5927	0.030*
C6	0.1660 (3)	0.2315 (7)	0.6641 (3)	0.0182 (10)
C7	0.2514 (4)	0.2930 (8)	0.6672 (3)	0.0210 (11)
H7	0.2788	0.2180	0.6388	0.025*
C8	0.4083 (4)	0.6481 (8)	0.7189 (3)	0.0233 (12)
C9	0.3807 (6)	0.3222 (11)	0.9082 (5)	0.0486 (19)
H9	0.3852	0.2632	0.8615	0.058*
C10	0.4268 (7)	0.2446 (14)	0.9857 (5)	0.068 (3)
H10	0.4631	0.1346	0.9924	0.082*
C11	0.4195 (6)	0.3282 (13)	1.0529 (5)	0.059 (2)
H11	0.4503	0.2752	1.1065	0.071*
C12	0.3680 (5)	0.4878 (11)	1.0429 (4)	0.0390 (16)
H12	0.3628	0.5480	1.0890	0.047*
C13	0.3235 (4)	0.5594 (9)	0.9635 (3)	0.0246 (12)
C14	0.2655 (4)	0.7348 (8)	0.9444 (3)	0.0235 (12)
C15	0.2450 (5)	0.8306 (10)	1.0037 (4)	0.0345 (15)
H15	0.2672	0.7861	1.0592	0.041*
C16	0.1917 (5)	0.9915 (11)	0.9804 (4)	0.0429 (18)
H16	0.1754	1.0573	1.0194	0.051*
C17	0.1624 (5)	1.0556 (9)	0.9000 (4)	0.0356 (15)
H17	0.1276	1.1684	0.8831	0.043*
C18	0.1848 (4)	0.9525 (8)	0.8448 (4)	0.0242 (12)
H18	0.1643	0.9961	0.7893	0.029*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.0165 (3)	0.0207 (3)	0.0182 (3)	0.0020 (2)	0.0065 (2)	0.0029 (2)
S1	0.0208 (7)	0.0196 (7)	0.0340 (8)	0.0012 (5)	0.0139 (6)	0.0039 (5)
O1	0.0178 (19)	0.021 (2)	0.028 (2)	−0.0017 (15)	0.0101 (16)	−0.0024 (16)
O2	0.0186 (19)	0.027 (2)	0.025 (2)	−0.0039 (16)	0.0079 (16)	−0.0022 (16)
N1	0.018 (2)	0.026 (2)	0.021 (2)	−0.0014 (19)	0.0095 (19)	0.0022 (18)
N2	0.014 (2)	0.030 (3)	0.024 (2)	−0.0033 (19)	0.0094 (19)	−0.002 (2)
N3	0.022 (2)	0.025 (2)	0.043 (3)	−0.006 (2)	0.021 (2)	−0.005 (2)
N4	0.027 (3)	0.033 (3)	0.024 (2)	0.009 (2)	0.012 (2)	0.012 (2)
N6	0.016 (2)	0.019 (2)	0.019 (2)	0.0003 (17)	0.0052 (18)	0.0038 (17)
C1	0.020 (3)	0.019 (3)	0.019 (3)	0.002 (2)	0.007 (2)	0.004 (2)
C2	0.016 (2)	0.024 (3)	0.018 (2)	0.002 (2)	0.007 (2)	0.002 (2)
C3	0.015 (2)	0.023 (3)	0.018 (2)	0.001 (2)	0.002 (2)	0.006 (2)
C4	0.027 (3)	0.026 (3)	0.022 (3)	−0.002 (2)	0.010 (2)	0.000 (2)
C5	0.024 (3)	0.028 (3)	0.023 (3)	0.003 (2)	0.010 (2)	−0.003 (2)

C6	0.018 (3)	0.018 (3)	0.018 (2)	0.002 (2)	0.006 (2)	0.0031 (19)
C7	0.022 (3)	0.025 (3)	0.018 (3)	0.000 (2)	0.009 (2)	−0.001 (2)
C8	0.022 (3)	0.026 (3)	0.025 (3)	0.001 (2)	0.013 (2)	0.003 (2)
C9	0.059 (4)	0.049 (4)	0.048 (4)	0.032 (4)	0.032 (4)	0.022 (3)
C10	0.085 (6)	0.075 (5)	0.054 (5)	0.054 (5)	0.037 (4)	0.031 (4)
C11	0.065 (5)	0.076 (5)	0.039 (4)	0.037 (4)	0.024 (4)	0.032 (4)
C12	0.039 (4)	0.051 (4)	0.032 (3)	0.010 (3)	0.019 (3)	0.015 (3)
C13	0.020 (3)	0.031 (3)	0.021 (3)	0.000 (2)	0.006 (2)	0.007 (2)
C14	0.022 (3)	0.027 (3)	0.019 (3)	−0.007 (2)	0.005 (2)	−0.003 (2)
C15	0.042 (4)	0.036 (4)	0.021 (3)	0.001 (3)	0.008 (3)	−0.007 (2)
C16	0.053 (5)	0.041 (4)	0.029 (3)	0.004 (3)	0.011 (3)	−0.014 (3)
C17	0.040 (4)	0.028 (3)	0.034 (3)	0.010 (3)	0.010 (3)	−0.002 (3)
C18	0.020 (3)	0.023 (3)	0.023 (3)	−0.001 (2)	0.002 (2)	−0.001 (2)

Geometric parameters (Å, °)

Zn1—O1	1.983 (4)	C4—C5	1.367 (8)
Zn1—N1	2.045 (5)	C4—H4	0.9500
Zn1—N4	2.109 (5)	C5—C6	1.412 (8)
Zn1—N6	2.112 (5)	C5—H5	0.9500
Zn1—S1	2.3911 (15)	C6—C7	1.423 (7)
S1—C8	1.746 (6)	C7—H7	0.9500
O1—C1	1.329 (7)	C9—C10	1.381 (10)
O2—C3	1.354 (6)	C9—H9	0.9500
O2—H2	0.8400	C10—C11	1.370 (12)
N1—C7	1.306 (7)	C10—H10	0.9500
N1—N2	1.403 (6)	C11—C12	1.367 (10)
N2—C8	1.310 (7)	C11—H11	0.9500
N3—C8	1.355 (7)	C12—C13	1.387 (8)
N3—H31	0.8800	C12—H12	0.9500
N3—H32	0.8800	C13—C14	1.505 (8)
N4—C9	1.338 (8)	C14—C15	1.393 (8)
N4—C13	1.346 (8)	C15—C16	1.382 (10)
N6—C18	1.333 (7)	C15—H15	0.9500
N6—C14	1.340 (7)	C16—C17	1.380 (9)
C1—C2	1.408 (7)	C16—H16	0.9500
C1—C6	1.437 (7)	C17—C18	1.373 (9)
C2—C3	1.389 (8)	C17—H17	0.9500
C2—H2A	0.9500	C18—H18	0.9500
C3—C4	1.385 (8)		
O1—Zn1—N1	90.29 (16)	C6—C5—H5	118.7
O1—Zn1—N4	102.37 (18)	C5—C6—C7	116.2 (5)
N1—Zn1—N4	100.70 (19)	C5—C6—C1	118.6 (5)
O1—Zn1—N6	93.82 (16)	C7—C6—C1	125.1 (5)
N1—Zn1—N6	175.77 (18)	N1—C7—C6	125.5 (5)
N4—Zn1—N6	77.46 (19)	N1—C7—H7	117.2
O1—Zn1—S1	146.42 (12)	C6—C7—H7	117.2
N1—Zn1—S1	81.14 (13)	N2—C8—N3	115.8 (5)
N4—Zn1—S1	111.10 (15)	N2—C8—S1	126.5 (4)

N6—Zn1—S1	95.92 (13)	N3—C8—S1	117.6 (4)
C8—S1—Zn1	92.73 (19)	N4—C9—C10	121.7 (7)
C1—O1—Zn1	128.1 (3)	N4—C9—H9	119.1
C3—O2—H2	109.5	C10—C9—H9	119.1
C7—N1—N2	114.5 (5)	C11—C10—C9	119.2 (7)
C7—N1—Zn1	125.7 (4)	C11—C10—H10	120.4
N2—N1—Zn1	119.5 (3)	C9—C10—H10	120.4
C8—N2—N1	112.9 (5)	C10—C11—C12	120.0 (7)
C8—N3—H31	120.0	C10—C11—H11	120.0
C8—N3—H32	120.0	C12—C11—H11	120.0
H31—N3—H32	120.0	C11—C12—C13	118.1 (7)
C9—N4—C13	118.5 (5)	C11—C12—H12	121.0
C9—N4—Zn1	124.9 (5)	C13—C12—H12	121.0
C13—N4—Zn1	116.5 (4)	N4—C13—C12	122.4 (6)
C18—N6—C14	118.9 (5)	N4—C13—C14	114.3 (5)
C18—N6—Zn1	125.2 (4)	C12—C13—C14	123.2 (6)
C14—N6—Zn1	115.8 (4)	N6—C14—C15	121.5 (6)
O1—C1—C2	119.8 (5)	N6—C14—C13	115.6 (5)
O1—C1—C6	123.2 (5)	C15—C14—C13	122.9 (5)
C2—C1—C6	117.0 (5)	C16—C15—C14	118.7 (6)
C3—C2—C1	122.2 (5)	C16—C15—H15	120.6
C3—C2—H2A	118.9	C14—C15—H15	120.6
C1—C2—H2A	118.9	C17—C16—C15	119.3 (6)
O2—C3—C4	117.3 (5)	C17—C16—H16	120.3
O2—C3—C2	122.4 (5)	C15—C16—H16	120.3
C4—C3—C2	120.3 (5)	C18—C17—C16	118.5 (6)
C5—C4—C3	119.2 (5)	C18—C17—H17	120.8
C5—C4—H4	120.4	C16—C17—H17	120.8
C3—C4—H4	120.4	N6—C18—C17	123.0 (5)
C4—C5—C6	122.6 (5)	N6—C18—H18	118.5
C4—C5—H5	118.7	C17—C18—H18	118.5
O1—Zn1—S1—C8	−95.8 (3)	C4—C5—C6—C1	−2.5 (8)
N1—Zn1—S1—C8	−18.8 (2)	O1—C1—C6—C5	−178.1 (5)
N4—Zn1—S1—C8	79.3 (2)	C2—C1—C6—C5	3.3 (7)
N6—Zn1—S1—C8	158.1 (2)	O1—C1—C6—C7	−0.7 (8)
N1—Zn1—O1—C1	15.3 (4)	C2—C1—C6—C7	−179.2 (5)
N4—Zn1—O1—C1	−85.8 (4)	N2—N1—C7—C6	−178.8 (5)
N6—Zn1—O1—C1	−163.8 (4)	Zn1—N1—C7—C6	7.3 (8)
S1—Zn1—O1—C1	89.5 (5)	C5—C6—C7—N1	180.0 (5)
O1—Zn1—N1—C7	−13.1 (5)	C1—C6—C7—N1	2.5 (9)
N4—Zn1—N1—C7	89.5 (5)	N1—N2—C8—N3	−178.7 (5)
S1—Zn1—N1—C7	−160.5 (5)	N1—N2—C8—S1	−1.0 (7)
O1—Zn1—N1—N2	173.2 (4)	Zn1—S1—C8—N2	17.3 (5)
N4—Zn1—N1—N2	−84.1 (4)	Zn1—S1—C8—N3	−165.0 (4)
S1—Zn1—N1—N2	25.8 (4)	C13—N4—C9—C10	−0.5 (12)
C7—N1—N2—C8	163.9 (5)	Zn1—N4—C9—C10	178.0 (7)
Zn1—N1—N2—C8	−21.8 (6)	N4—C9—C10—C11	0.7 (15)
O1—Zn1—N4—C9	92.1 (6)	C9—C10—C11—C12	−0.8 (16)
N1—Zn1—N4—C9	−0.5 (6)	C10—C11—C12—C13	0.6 (13)

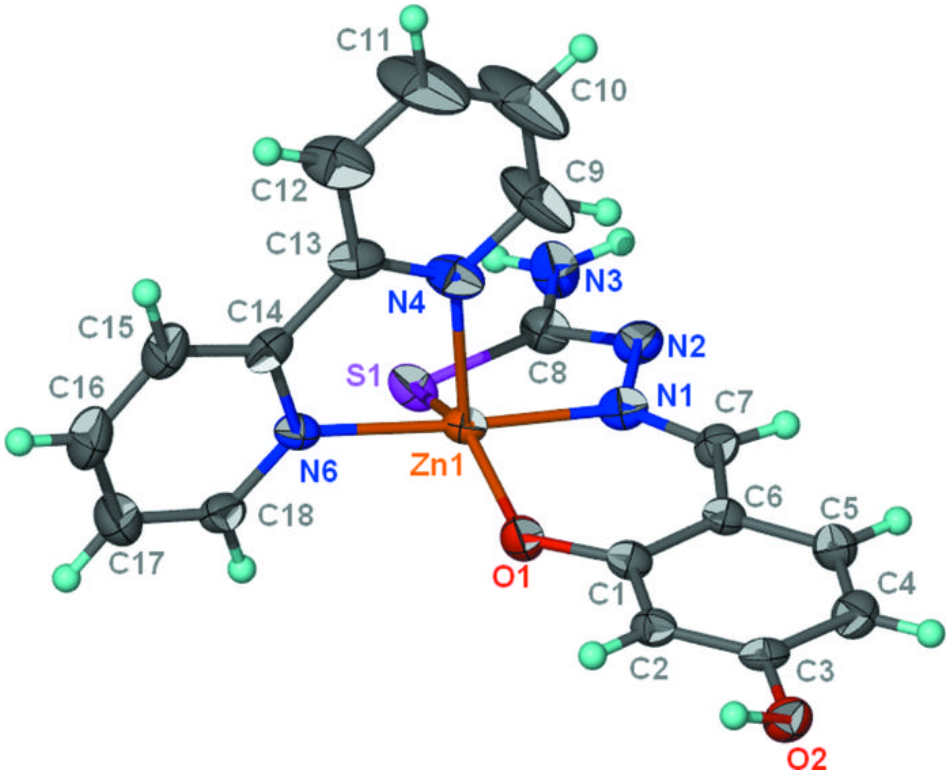
N6—Zn1—N4—C9	−176.7 (6)	C9—N4—C13—C12	0.3 (10)
S1—Zn1—N4—C9	−85.1 (6)	Zn1—N4—C13—C12	−178.3 (5)
O1—Zn1—N4—C13	−89.4 (4)	C9—N4—C13—C14	179.5 (6)
N1—Zn1—N4—C13	177.9 (4)	Zn1—N4—C13—C14	0.9 (6)
N6—Zn1—N4—C13	1.8 (4)	C11—C12—C13—N4	−0.4 (10)
S1—Zn1—N4—C13	93.4 (4)	C11—C12—C13—C14	−179.5 (7)
O1—Zn1—N6—C18	−78.4 (4)	C18—N6—C14—C15	2.6 (8)
N4—Zn1—N6—C18	179.8 (5)	Zn1—N6—C14—C15	−173.4 (5)
S1—Zn1—N6—C18	69.4 (4)	C18—N6—C14—C13	−177.6 (5)
O1—Zn1—N6—C14	97.3 (4)	Zn1—N6—C14—C13	6.4 (6)
N4—Zn1—N6—C14	−4.6 (4)	N4—C13—C14—N6	−4.9 (7)
S1—Zn1—N6—C14	−114.9 (4)	C12—C13—C14—N6	174.3 (6)
Zn1—O1—C1—C2	167.2 (4)	N4—C13—C14—C15	174.9 (6)
Zn1—O1—C1—C6	−11.3 (7)	C12—C13—C14—C15	−5.9 (9)
O1—C1—C2—C3	179.0 (5)	N6—C14—C15—C16	−0.7 (10)
C6—C1—C2—C3	−2.4 (8)	C13—C14—C15—C16	179.5 (6)
C1—C2—C3—O2	−179.6 (5)	C14—C15—C16—C17	−1.7 (11)
C1—C2—C3—C4	0.3 (8)	C15—C16—C17—C18	2.3 (11)
O2—C3—C4—C5	−179.3 (5)	C14—N6—C18—C17	−2.0 (9)
C2—C3—C4—C5	0.7 (8)	Zn1—N6—C18—C17	173.5 (5)
C3—C4—C5—C6	0.4 (9)	C16—C17—C18—N6	−0.5 (10)
C4—C5—C6—C7	179.9 (5)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O1^{i}	0.84	1.85	2.625 (5)	153
N3—H32···N2^{ii}	0.88	2.15	2.936 (7)	148

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

Fig. 1


C11
C10
C12
C9
C13
N4
N3
C15
C14
N2
S1
C8
C16
N1
C7
N6
Zn1
C6
C5
C18
O1
C4
C17
C1
C2
C3
O2