Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

[*N*,*N*′-Bis(3-aminopropyl)ethylenediamine]disaccharinatozinc(II) monohydrate

Hümeyra Paşaoğlu,[a] Gökhan Kaştaş,[a]* Okan Z. Yeşilel,[b] Onur Şahin[a] and Orhan Büyükgüngör[a]

[a]Department of Physics, Faculty of Arts and Sciences, Ondokuz Mayıs University, 55139 Kurupelit Samsun, Turkey, and [b]Department of Chemistry, Faculty of Arts and Sciences, Eskişehir Osmangazi University, 26480 Eskişehir, Turkey
Correspondence e-mail: gkastas@omu.edu.tr

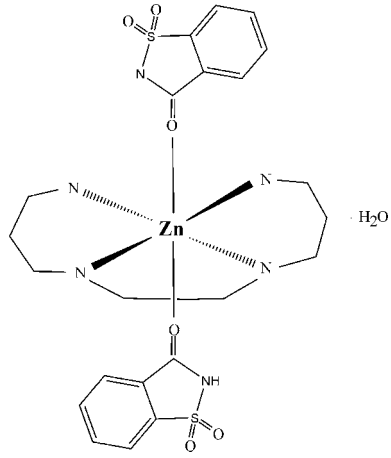
Received 4 October 2007; accepted 9 October 2007

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.008 Å; disorder in main residue; R factor = 0.068; wR factor = 0.122; data-to-parameter ratio = 18.1.

In the title complex, $[Zn(C_7H_4NO_3S)_2(C_8H_{22}N_4)]\cdot H_2O$ or $[Zn(sac)_2(paen)]\cdot H_2O$ [sac = saccharinate and paen = *N*,*N*′-bis(3-aminopropyl)ethylenediamine], the zinc cation is octahedrally coordinated. The equatorial plane of the octahedron is formed by N atoms of the paen ligand, whereas the axial positions are occupied by the carbonyl O atoms of the two sac ligands. One of the sulfonyl groups of the sac ligands shows disorder and was modelled with two different orientations and site occupancies of 0:38 (1):0.62 (1). The molecular packing is stabilized by intermolecular O—H···O and N—H···O hydrogen bonds between water molecules and neighbouring $[Zn(sac)_2(paen)]$ molecules, which form chains running parallel to [010]. The crystal used was an inversion twin.

Related literature

For the synthesis of $[Zn(sac)_2(H_2O)_4]\cdot 2H_2O$ used as an educt for crystal growth of the title compound, see: Koksal *et al.* (2001). For related structures containing sac ligands, see: Yeşilel *et al.* (2006); Yilmaz *et al.* (2002, 2006).



Experimental

Crystal data

$[Zn(C_7H_4NO_3S)_2(C_8H_{22}N_4)]\cdot H_2O$
M_r = 622.03
Orthorhombic, $P2_12_12_1$
a = 10.1025 (5) Å
b = 14.3045 (10) Å
c = 19.2547 (11) Å
V = 2782.5 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.08 mm^{-1}
T = 296 K
0.32 × 0.30 × 0.25 mm

Data collection

Stoe IPDSII diffractometer
Absorption correction: numerical (*X-RED32*; Stoe, 2002)
T_{min} = 0.710, T_{max} = 0.811
49189 measured reflections
6894 independent reflections
3763 reflections with $I > 2\sigma(I)$
R_{int} = 0.148

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.068
$wR(F^2)$ = 0.122
S = 0.92
6894 reflections
381 parameters
29 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}
Absolute structure: Flack (1983), 3014 Friedel pairs
Flack parameter: 0.449 (17)

Table 1
Selected geometric parameters (Å, °).

N3—Zn1	2.100 (4)	N6—Zn1	2.124 (4)
N4—Zn1	2.180 (5)	O1—Zn1	2.204 (4)
N5—Zn1	2.174 (5)	O2—Zn1	2.197 (3)
N6—Zn1—N5	171.1 (2)	O2—Zn1—O1	179.72 (18)
N3—Zn1—N4	171.58 (18)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*A*···N1	0.90	2.56	3.282 (7)	137
N6—H6*A*···N2	0.90	2.52	3.224 (6)	135
N5—H5*A*···N1	1.00 (6)	2.60 (6)	3.411 (7)	138 (4)
N4—H4*A*···N2	0.76 (7)	2.53 (7)	3.278 (6)	166 (7)
O1*W*—H1*W*···O5	0.86 (2)	2.01 (3)	2.833 (8)	161 (8)
N3—H3*B*···O1*W*^{i}	0.90	2.22	3.052 (7)	154
N6—H6*B*···O1*W*^{i}	0.90	2.22	3.071 (9)	157

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

  doi:10.1107/S1600536807049458

Data collection: *X-AREA* (Stoe, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The authors thank the Faculty of Arts and Sciences, Ondokuz Mayıs University, Turkey, for the use of the Stoe IPDS II diffractometer (purchased under grant No. F279 of the University Research Fund).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2151).

References

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Koksal, F., Karabulut, B. & &Yerli, Y. (2001). *Int. J. Inorg. Mater.* **3**, 413–416.
Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Stoe (2002). *X-AREA* (Version 1.18) and *X-RED32* (Version 1.04). Stoe & Cie, Darmstadt, Germany.
Yeşilel, O. Z., Ölmez, H., Paşaoğlu, H. & Kaştaş, G. (2006). *Z. Naturforsch. Teil B*, **61**, 153–158.
Yilmaz, V. T., Guney, S., Andac, O. & Harrison, W. T. A. (2002). *Acta Cryst.* C**58**, m427–m430.
Yilmaz, V. T., Kars, V. & Kazak, C. (2006). *J. Coord. Chem.* **59**, 1937–1944.

supplementary materials

Acta Cryst. (2007). E**63**, m2710-m2711 [**doi:10.1107/S1600536807049458**]

[*N*,*N*'-Bis(3-aminopropyl)ethylenediamine]disaccharinatozinc(II) monohydrate

H. Pasaoglu, G. Kastas, O. Z. Yesilel, O. Sahin and O. Büyükgüngör

Comment

In title compound the Zn(II) cation is octahedrally coordinated by two sac and one paen ligands. Most interestingly, the two sac ligands are bonded to the Zn site *via* the carbonyl O atoms although N-coordination is usually preferred by this ligand with first-row transition metals. The equatorial plane, defined by the atoms (N3, N4, N5, N6), of the octahedron is formed by N atoms of paen ligands, whereas the axial positions belong to the carbonyl O atoms of the sac ligands. The Zn—N bonds are slightly shorter than the Zn—O bonds (Table 1). Whereas the (O–Zn–O) angle is nearly linear, the *trans*- (N–Zn–N) angles of the equatorial plane deviate significantly from linearity. One of the sulfonyl groups of the sac ligands shows disorder, and was refined with two different orientations (see Fig. 1). However, the bond lengths and angles of the sac ligands are similiar to those observed in related structures (Yeşilel *et al.*, 2006; Yilmaz *et al.*, 2002; 2006).

The molecular packing is stabilized by intermolecular hydrogen bonds (Table2, Fig. 2). Neighbouring $Zn(sac)_2(paen)$ moieties are linked *via* water molecules through O—H···O and N—H···O hydrogen bonds, which leads to chains extending parallel to (010).

Experimental

Under continous stirring at room temperature, an aqueous solution of paen (350 mg, 2.0 mmol) was added dropwise to 50 ml of an aqueous solution of $[Zn(sac)_2(H_2O)_4]\cdot 2H_2O$ (548 mg, 1.0 mmol). For synthesis of the latter compound, see: Koksal *et al.* (2001). The resulting solution was heated up to 353 K and stirred continuously for 4 h. Then the solution was allowed to cool to room temperature slowly. Small colourless crystals grown in the solution were filtered off and washed with cold distilled water and ethanol, and were finally dried.

Refinement

The disordered sulfonyl group of the sacharinate ligand was modelled with two different orientations (S1A—S1B; O3A—O3B, O4A—O4B) and with refined occupancy factors of 0.383 (11) and 0.617 (11), respectively. All H atoms except those bonded to N4 and N5 atoms were placed in geometrically idealized positions with distances of d(N–H) = 0.90 Å; d(C–H) = 0.93 − 0.97 Å, and were refined as riding atoms with $U_{iso}(H) = 1.2U_{eq}$ of the parent atoms. H atoms bonded to N4 and N5 were found from difference maps and were refined freely. H atoms of the water molecule were constrained to O—H distances of 0.85 (2) Å and were refined with $U_{iso}(H) = 1.2U_{eq}(OW)$. The measured crystal was racemically twinned with an approximate twin ratio of 1:1.

Figures

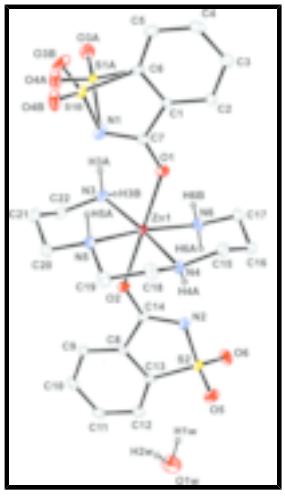


Fig. 1. The molecular structure of (I), with atom labels and displacement ellipsoids displayed at the 30% probability level. H atoms not involved in hydrogen bonding have been omitted for clarity.

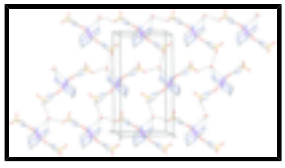


Fig. 2. The packing diagram of the title compound showing the chain structure parallel to (010). Benzene rings and some hydrogen bonds have been omitted for clarity.

[*N*,*N*'-Bis(3-aminopropyl)ethylendiamine]disaccharinatozinc(II) monohydrate

Crystal data

$[Zn(C_7H_4NO_3S)_2(C_8H_{22}N_4)]\cdot H_2O$	F_{000} = 1296
M_r = 622.03	D_x = 1.485 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71069 Å
Hall symbol: P 2ac 2ab	Cell parameters from 23038 reflections
a = 10.1025 (5) Å	θ = 1.4–26.6º
b = 14.3045 (10) Å	μ = 1.08 mm^{-1}
c = 19.2547 (11) Å	T = 296 K
V = 2782.5 (3) Å^3	Rectangular prism, colorless
Z = 4	0.32 × 0.30 × 0.25 mm

Data collection

Stoe IPDSII diffractometer	6894 independent reflections
Radiation source: fine-focus sealed tube	3763 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.148
T = 296 K	θ_{max} = 28.5º
ω scans	θ_{min} = 1.8º
Absorption correction: numerical (X-RED32; Stoe, 2002)	h = −12→13
T_{min} = 0.710, T_{max} = 0.811	k = −19→18

49189 measured reflections	$l = -25 \rightarrow 25$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.068$	$w = 1/[\sigma^2(F_o^2) + (0.0535P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.122$	$(\Delta/\sigma)_{max} = 0.001$
$S = 0.92$	$\Delta\rho_{max} = 0.37$ e Å^{-3}
6894 reflections	$\Delta\rho_{min} = -0.36$ e Å^{-3}
381 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
29 restraints	Extinction coefficient: 0.0015 (4)
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 3014 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: 0.449 (17)

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.1672 (4)	0.4017 (3)	0.1425 (2)	0.0451 (10)	
C2	0.1978 (5)	0.3115 (3)	0.1605 (3)	0.0590 (13)	
H2	0.2750	0.2830	0.1449	0.071*	
C3	0.1087 (6)	0.2641 (4)	0.2031 (3)	0.0728 (15)	
H3	0.1278	0.2035	0.2175	0.087*	
C4	−0.0065 (6)	0.3058 (4)	0.2241 (3)	0.0749 (15)	
H4	−0.0662	0.2717	0.2507	0.090*	
C5	−0.0361 (6)	0.3954 (4)	0.2072 (3)	0.0755 (17)	
H5	−0.1127	0.4243	0.2232	0.091*	
C6	0.0520 (5)	0.4412 (4)	0.1656 (3)	0.0624 (14)	
C7	0.2450 (5)	0.4672 (3)	0.0984 (3)	0.0608 (13)	
C8	0.8111 (4)	0.6093 (3)	−0.1499 (2)	0.0429 (10)	
C9	0.7907 (5)	0.7016 (3)	−0.1649 (3)	0.0526 (12)	

H9	0.7208	0.7345	−0.1451	0.063*	
C10	0.8768 (6)	0.7441 (4)	−0.2101 (3)	0.0694 (15)	
H10	0.8649	0.8068	−0.2212	0.083*	
C11	0.9808 (7)	0.6953 (4)	−0.2394 (3)	0.0725 (16)	
H11	1.0372	0.7255	−0.2703	0.087*	
C12	1.0022 (6)	0.6024 (4)	−0.2234 (3)	0.0682 (13)	
H12	1.0724	0.5691	−0.2424	0.082*	
C13	0.9140 (4)	0.5615 (3)	−0.1776 (2)	0.0501 (11)	
C14	0.7301 (4)	0.5471 (3)	−0.1025 (2)	0.0468 (10)	
C15	0.4820 (6)	0.2962 (3)	−0.0276 (3)	0.0731 (15)	
H15A	0.4710	0.2454	−0.0605	0.088*	
H15B	0.4096	0.2931	0.0054	0.088*	
C16	0.6118 (6)	0.2837 (4)	0.0107 (3)	0.0718 (15)	
H16A	0.6182	0.2191	0.0257	0.086*	
H16B	0.6837	0.2950	−0.0216	0.086*	
C17	0.6310 (6)	0.3457 (4)	0.0730 (3)	0.0771 (17)	
H17A	0.5550	0.3393	0.1034	0.093*	
H17B	0.7083	0.3246	0.0985	0.093*	
C18	0.3549 (6)	0.3961 (4)	−0.1050 (4)	0.0820 (17)	
H18A	0.2796	0.3785	−0.0766	0.098*	
H18B	0.3576	0.3547	−0.1449	0.098*	
C19	0.3383 (6)	0.4956 (4)	−0.1292 (3)	0.0810 (16)	
H19A	0.4118	0.5128	−0.1590	0.097*	
H19B	0.2572	0.5014	−0.1559	0.097*	
C20	0.3312 (6)	0.6581 (4)	−0.0891 (3)	0.0747 (16)	
H20A	0.2514	0.6705	−0.1155	0.090*	
H20B	0.4065	0.6710	−0.1188	0.090*	
C21	0.3355 (6)	0.7216 (4)	−0.0276 (4)	0.084 (2)	
H21A	0.2642	0.7040	0.0035	0.101*	
H21B	0.3175	0.7847	−0.0435	0.101*	
C22	0.4633 (6)	0.7233 (3)	0.0139 (3)	0.0760 (18)	
H22A	0.5375	0.7310	−0.0176	0.091*	
H22B	0.4623	0.7765	0.0451	0.091*	
N1	0.1887 (5)	0.5510 (3)	0.0913 (3)	0.0946 (19)	
N2	0.7764 (4)	0.4591 (3)	−0.0980 (2)	0.0628 (12)	
N3	0.4813 (5)	0.6373 (3)	0.0542 (2)	0.0702 (12)	
H3A	0.4140	0.6331	0.0847	0.084*	
H3B	0.5563	0.6432	0.0791	0.084*	
N4	0.4754 (5)	0.3859 (3)	−0.0651 (3)	0.0616 (12)	
N5	0.3334 (4)	0.5583 (3)	−0.0687 (3)	0.0658 (12)	
N6	0.6482 (4)	0.4462 (3)	0.0551 (2)	0.0693 (12)	
H6A	0.7227	0.4519	0.0298	0.083*	
H6B	0.6606	0.4782	0.0948	0.083*	
O1	0.3520 (4)	0.4442 (3)	0.0723 (2)	0.0758 (11)	
O2	0.6306 (3)	0.5774 (2)	−0.07302 (19)	0.0600 (9)	
O1W	0.8016 (5)	0.3978 (5)	−0.3387 (3)	0.1273 (19)	
O3A	0.107 (2)	0.6125 (9)	0.2157 (8)	0.101 (5)	0.383 (11)
O3B	0.0292 (14)	0.6268 (5)	0.1665 (10)	0.130 (6)	0.617 (11)
O4A	−0.0316 (15)	0.5925 (16)	0.1166 (15)	0.115 (4)	0.383 (11)

O4B	−0.0713 (8)	0.5555 (7)	0.0682 (7)	0.115 (4)	0.617 (11)
O5	0.8858 (5)	0.3817 (3)	−0.1988 (2)	0.1005 (16)	
O6	1.0226 (4)	0.4277 (3)	−0.1006 (2)	0.1084 (17)	
S1A	0.0774 (9)	0.5583 (5)	0.1553 (5)	0.079 (3)	0.383 (11)
S1B	0.0367 (4)	0.5542 (3)	0.1183 (3)	0.0725 (14)	0.617 (11)
S2	0.90760 (14)	0.44672 (10)	−0.14354 (8)	0.0665 (4)	
Zn1	0.49146 (5)	0.51064 (3)	−0.00068 (4)	0.05359 (16)	
H5A	0.252 (6)	0.548 (4)	−0.040 (3)	0.082 (16)*	
H4A	0.548 (7)	0.393 (5)	−0.073 (4)	0.10 (3)*	
H1W	0.816 (7)	0.405 (7)	−0.2951 (15)	0.154*	
H2W	0.720 (2)	0.405 (7)	−0.341 (4)	0.154*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.049 (3)	0.044 (2)	0.042 (2)	−0.0024 (19)	0.009 (2)	−0.0002 (19)
C2	0.055 (3)	0.062 (3)	0.060 (3)	0.005 (2)	0.001 (3)	0.008 (2)
C3	0.077 (4)	0.067 (3)	0.075 (4)	−0.002 (3)	0.004 (3)	0.019 (3)
C4	0.074 (4)	0.082 (4)	0.068 (3)	−0.021 (4)	0.019 (3)	0.007 (3)
C5	0.072 (4)	0.070 (3)	0.084 (4)	−0.015 (3)	0.038 (3)	−0.005 (3)
C6	0.063 (3)	0.057 (3)	0.067 (3)	−0.003 (2)	0.037 (3)	−0.001 (3)
C7	0.053 (3)	0.059 (3)	0.070 (3)	−0.002 (2)	0.022 (2)	0.008 (3)
C8	0.037 (2)	0.047 (2)	0.045 (3)	−0.0072 (19)	−0.0025 (19)	0.001 (2)
C9	0.061 (3)	0.042 (2)	0.054 (3)	−0.003 (2)	−0.002 (2)	0.004 (2)
C10	0.089 (4)	0.052 (3)	0.068 (4)	−0.014 (3)	−0.008 (3)	0.016 (3)
C11	0.084 (4)	0.077 (3)	0.056 (3)	−0.021 (3)	0.018 (3)	0.013 (2)
C12	0.055 (3)	0.081 (3)	0.068 (3)	−0.003 (3)	0.016 (3)	0.007 (3)
C13	0.047 (3)	0.059 (3)	0.045 (2)	0.000 (2)	0.015 (2)	0.005 (2)
C14	0.033 (2)	0.055 (3)	0.052 (3)	−0.003 (2)	0.0064 (19)	0.003 (2)
C15	0.063 (4)	0.058 (3)	0.098 (4)	−0.002 (3)	0.021 (3)	0.010 (2)
C16	0.069 (3)	0.066 (3)	0.080 (4)	0.015 (2)	0.012 (3)	0.013 (3)
C17	0.066 (4)	0.085 (4)	0.080 (4)	0.020 (3)	0.006 (3)	0.030 (3)
C18	0.074 (4)	0.072 (4)	0.100 (5)	−0.012 (3)	−0.013 (4)	0.007 (3)
C19	0.067 (3)	0.087 (4)	0.089 (4)	−0.004 (3)	−0.015 (3)	0.011 (3)
C20	0.061 (3)	0.072 (4)	0.091 (4)	0.009 (3)	0.004 (3)	0.024 (3)
C21	0.073 (4)	0.059 (3)	0.121 (6)	0.016 (3)	0.029 (4)	0.030 (3)
C22	0.080 (4)	0.059 (3)	0.089 (5)	0.003 (2)	0.033 (3)	−0.006 (3)
N1	0.080 (3)	0.049 (2)	0.155 (5)	0.007 (2)	0.069 (3)	0.022 (3)
N2	0.053 (2)	0.046 (2)	0.090 (3)	0.0027 (18)	0.035 (2)	0.016 (2)
N3	0.069 (3)	0.068 (2)	0.074 (3)	0.012 (2)	0.020 (2)	0.002 (2)
N4	0.045 (3)	0.053 (2)	0.088 (3)	−0.0020 (19)	0.010 (2)	0.003 (2)
N5	0.050 (2)	0.063 (3)	0.084 (3)	0.005 (2)	0.014 (2)	0.022 (3)
N6	0.061 (3)	0.079 (3)	0.068 (3)	0.003 (2)	0.009 (2)	0.008 (2)
O1	0.057 (2)	0.075 (2)	0.095 (3)	0.008 (2)	0.037 (2)	0.021 (2)
O2	0.0461 (19)	0.059 (2)	0.075 (2)	0.0036 (15)	0.0220 (17)	0.0056 (17)
O1W	0.088 (4)	0.187 (5)	0.107 (4)	0.000 (4)	−0.009 (3)	0.013 (4)
O3A	0.120 (13)	0.070 (8)	0.113 (11)	0.009 (8)	0.033 (10)	0.006 (8)
O3B	0.114 (9)	0.048 (4)	0.227 (15)	−0.009 (5)	0.085 (9)	−0.047 (7)

O4A	0.050 (5)	0.086 (6)	0.209 (12)	0.012 (4)	0.004 (5)	0.067 (7)
O4B	0.050 (5)	0.086 (6)	0.209 (12)	0.012 (4)	0.004 (5)	0.067 (7)
O5	0.140 (4)	0.059 (2)	0.103 (3)	−0.002 (3)	0.048 (3)	−0.014 (2)
O6	0.071 (3)	0.136 (4)	0.118 (4)	0.045 (3)	0.018 (3)	0.063 (3)
S1A	0.087 (5)	0.057 (3)	0.095 (5)	0.031 (3)	0.059 (4)	0.021 (4)
S1B	0.064 (2)	0.0434 (13)	0.110 (3)	0.0058 (14)	0.038 (2)	0.008 (2)
S2	0.0651 (8)	0.0589 (7)	0.0756 (9)	0.0126 (6)	0.0299 (7)	0.0147 (7)
Zn1	0.0456 (3)	0.0536 (3)	0.0616 (3)	0.0076 (2)	0.0124 (3)	0.0074 (3)

Geometric parameters (Å, °)

C1—C6	1.368 (6)	C18—C19	1.507 (7)
C1—C2	1.372 (6)	C18—H18A	0.9700
C1—C7	1.490 (6)	C18—H18B	0.9700
C2—C3	1.393 (8)	C19—N5	1.471 (8)
C2—H2	0.9300	C19—H19A	0.9700
C3—C4	1.369 (8)	C19—H19B	0.9700
C3—H3	0.9300	C20—N5	1.481 (6)
C4—C5	1.356 (8)	C20—C21	1.492 (8)
C4—H4	0.9300	C20—H20A	0.9700
C5—C6	1.364 (7)	C20—H20B	0.9700
C5—H5	0.9300	C21—C22	1.519 (8)
C6—S1A	1.706 (9)	C21—H21A	0.9700
C6—S1B	1.861 (7)	C21—H21B	0.9700
C7—O1	1.237 (6)	C22—N3	1.466 (6)
C7—N1	1.333 (6)	C22—H22A	0.9700
C8—C13	1.355 (6)	C22—H22B	0.9700
C8—C9	1.367 (6)	N1—S1B	1.623 (6)
C8—C14	1.514 (6)	N1—S1A	1.671 (7)
C9—C10	1.372 (7)	N2—S2	1.599 (4)
C9—H9	0.9300	N3—Zn1	2.100 (4)
C10—C11	1.382 (8)	N3—H3A	0.9000
C10—H10	0.9300	N3—H3B	0.9000
C11—C12	1.381 (8)	N4—Zn1	2.180 (5)
C11—H11	0.9300	N4—H4A	0.76 (7)
C12—C13	1.384 (6)	N5—Zn1	2.174 (5)
C12—H12	0.9300	N5—H5A	1.00 (6)
C13—S2	1.769 (5)	N6—Zn1	2.124 (4)
C14—O2	1.233 (5)	N6—H6A	0.9000
C14—N2	1.346 (6)	N6—H6B	0.9000
C15—N4	1.473 (6)	O1—Zn1	2.204 (4)
C15—C16	1.515 (8)	O2—Zn1	2.197 (3)
C15—H15A	0.9700	O1W—H1W	0.86 (2)
C15—H15B	0.9700	O1W—H2W	0.84 (2)
C16—C17	1.505 (8)	O3A—S1A	1.430 (14)
C16—H16A	0.9700	O3B—S1B	1.395 (11)
C16—H16B	0.9700	O4A—S1A	1.417 (14)
C17—N6	1.489 (7)	O4B—S1B	1.456 (11)
C17—H17A	0.9700	O5—S2	1.431 (5)

C17—H17B	0.9700	O6—S2	1.452 (4)
C18—N4	1.446 (7)		
C6—C1—C2	119.9 (4)	C21—C20—H20B	109.2
C6—C1—C7	112.0 (4)	H20A—C20—H20B	107.9
C2—C1—C7	128.1 (4)	C20—C21—C22	116.8 (5)
C1—C2—C3	117.4 (5)	C20—C21—H21A	108.1
C1—C2—H2	121.3	C22—C21—H21A	108.1
C3—C2—H2	121.3	C20—C21—H21B	108.1
C4—C3—C2	120.8 (5)	C22—C21—H21B	108.1
C4—C3—H3	119.6	H21A—C21—H21B	107.3
C2—C3—H3	119.6	N3—C22—C21	111.7 (4)
C5—C4—C3	121.9 (5)	N3—C22—H22A	109.3
C5—C4—H4	119.1	C21—C22—H22A	109.3
C3—C4—H4	119.1	N3—C22—H22B	109.3
C4—C5—C6	116.8 (5)	C21—C22—H22B	109.3
C4—C5—H5	121.6	H22A—C22—H22B	107.9
C6—C5—H5	121.6	C7—N1—S1B	113.3 (4)
C5—C6—C1	123.2 (5)	C7—N1—S1A	105.5 (4)
C5—C6—S1A	129.6 (4)	C14—N2—S2	110.9 (3)
C1—C6—S1A	103.9 (4)	C22—N3—Zn1	117.6 (3)
C5—C6—S1B	130.5 (4)	C22—N3—H3A	107.9
C1—C6—S1B	105.7 (4)	Zn1—N3—H3A	107.9
O1—C7—N1	124.8 (4)	C22—N3—H3B	107.9
O1—C7—C1	121.7 (4)	Zn1—N3—H3B	107.9
N1—C7—C1	113.5 (4)	H3A—N3—H3B	107.2
C13—C8—C9	121.4 (4)	C18—N4—C15	112.7 (4)
C13—C8—C14	110.8 (4)	C18—N4—Zn1	106.4 (3)
C9—C8—C14	127.8 (4)	C15—N4—Zn1	115.5 (4)
C8—C9—C10	117.8 (5)	C18—N4—H4A	134 (6)
C8—C9—H9	121.1	C15—N4—H4A	100 (5)
C10—C9—H9	121.1	Zn1—N4—H4A	86 (5)
C9—C10—C11	121.2 (5)	C19—N5—C20	112.2 (5)
C9—C10—H10	119.4	C19—N5—Zn1	105.2 (3)
C11—C10—H10	119.4	C20—N5—Zn1	118.2 (4)
C12—C11—C10	120.9 (5)	C19—N5—H5A	112 (3)
C12—C11—H11	119.5	C20—N5—H5A	106 (3)
C10—C11—H11	119.5	Zn1—N5—H5A	103 (3)
C11—C12—C13	116.6 (5)	C17—N6—Zn1	116.7 (4)
C11—C12—H12	121.7	C17—N6—H6A	108.1
C13—C12—H12	121.7	Zn1—N6—H6A	108.1
C8—C13—C12	122.1 (5)	C17—N6—H6B	108.1
C8—C13—S2	107.1 (3)	Zn1—N6—H6B	108.1
C12—C13—S2	130.7 (4)	H6A—N6—H6B	107.3
O2—C14—N2	125.6 (4)	C7—O1—Zn1	134.7 (3)
O2—C14—C8	120.8 (4)	C14—O2—Zn1	131.4 (3)
N2—C14—C8	113.7 (4)	H1W—O1W—H2W	102 (7)
N4—C15—C16	112.4 (4)	O4A—S1A—O3A	113.8 (13)
N4—C15—H15A	109.1	O4A—S1A—N1	99.0 (11)
C16—C15—H15A	109.1	O3A—S1A—N1	119.6 (9)

N4—C15—H15B	109.1	O4A—S1A—C6	106.5 (10)
C16—C15—H15B	109.1	O3A—S1A—C6	117.9 (7)
H15A—C15—H15B	107.9	N1—S1A—C6	97.2 (4)
C17—C16—C15	115.5 (5)	O3B—S1B—O4B	113.0 (8)
C17—C16—H16A	108.4	O3B—S1B—N1	106.6 (6)
C15—C16—H16A	108.4	O4B—S1B—N1	119.8 (5)
C17—C16—H16B	108.4	O3B—S1B—C6	109.0 (7)
C15—C16—H16B	108.4	O4B—S1B—C6	113.4 (4)
H16A—C16—H16B	107.5	N1—S1B—C6	93.1 (3)
N6—C17—C16	113.5 (5)	O5—S2—O6	115.1 (3)
N6—C17—H17A	108.9	O5—S2—N2	110.7 (3)
C16—C17—H17A	108.9	O6—S2—N2	111.8 (2)
N6—C17—H17B	108.9	O5—S2—C13	109.5 (2)
C16—C17—H17B	108.9	O6—S2—C13	110.9 (3)
H17A—C17—H17B	107.7	N2—S2—C13	97.5 (2)
N4—C18—C19	110.7 (5)	N3—Zn1—N6	99.03 (19)
N4—C18—H18A	109.5	N3—Zn1—N5	89.83 (19)
C19—C18—H18A	109.5	N6—Zn1—N5	171.1 (2)
N4—C18—H18B	109.5	N3—Zn1—N4	171.58 (18)
C19—C18—H18B	109.5	N6—Zn1—N4	89.30 (18)
H18A—C18—H18B	108.1	N5—Zn1—N4	81.88 (19)
N5—C19—C18	109.5 (5)	N3—Zn1—O2	88.58 (15)
N5—C19—H19A	109.8	N6—Zn1—O2	91.83 (16)
C18—C19—H19A	109.8	N5—Zn1—O2	87.23 (15)
N5—C19—H19B	109.8	N4—Zn1—O2	92.45 (16)
C18—C19—H19B	109.8	N3—Zn1—O1	91.14 (17)
H19A—C19—H19B	108.2	N6—Zn1—O1	88.13 (15)
N5—C20—C21	112.1 (5)	N5—Zn1—O1	92.84 (17)
N5—C20—H20A	109.2	N4—Zn1—O1	87.83 (17)
C21—C20—H20A	109.2	O2—Zn1—O1	179.72 (18)
N5—C20—H20B	109.2		
C6—C1—C2—C3	−0.8 (8)	C1—C6—S1A—O4A	127.3 (13)
C7—C1—C2—C3	179.6 (5)	S1B—C6—S1A—O4A	29.9 (12)
C1—C2—C3—C4	2.0 (9)	C5—C6—S1A—O3A	56.1 (12)
C2—C3—C4—C5	−3.0 (10)	C1—C6—S1A—O3A	−103.4 (10)
C3—C4—C5—C6	2.7 (10)	S1B—C6—S1A—O3A	159.2 (13)
C4—C5—C6—C1	−1.6 (9)	C5—C6—S1A—N1	−174.9 (6)
C4—C5—C6—S1A	−157.6 (7)	C1—C6—S1A—N1	25.7 (7)
C4—C5—C6—S1B	168.0 (5)	S1B—C6—S1A—N1	−71.8 (6)
C2—C1—C6—C5	0.7 (9)	C7—N1—S1B—O3B	125.5 (9)
C7—C1—C6—C5	−179.6 (5)	S1A—N1—S1B—O3B	45.7 (10)
C2—C1—C6—S1A	161.8 (6)	C7—N1—S1B—O4B	−104.7 (7)
C7—C1—C6—S1A	−18.5 (7)	S1A—N1—S1B—O4B	175.6 (10)
C2—C1—C6—S1B	−171.1 (5)	C7—N1—S1B—C6	14.6 (6)
C7—C1—C6—S1B	8.6 (6)	S1A—N1—S1B—C6	−65.1 (7)
C6—C1—C7—O1	−179.7 (5)	C5—C6—S1B—O3B	67.1 (9)
C2—C1—C7—O1	0.0 (9)	C1—C6—S1B—O3B	−122.0 (7)
C6—C1—C7—N1	1.1 (7)	S1A—C6—S1B—O3B	−32.4 (8)
C2—C1—C7—N1	−179.3 (6)	C5—C6—S1B—O4B	−59.8 (8)

C13—C8—C9—C10	1.1 (7)	C1—C6—S1B—O4B	111.2 (6)
C14—C8—C9—C10	−179.4 (4)	S1A—C6—S1B—O4B	−159.2 (9)
C8—C9—C10—C11	−0.1 (8)	C5—C6—S1B—N1	175.8 (6)
C9—C10—C11—C12	−0.7 (9)	C1—C6—S1B—N1	−13.3 (5)
C10—C11—C12—C13	0.6 (8)	S1A—C6—S1B—N1	76.4 (6)
C9—C8—C13—C12	−1.2 (7)	C14—N2—S2—O5	−115.8 (4)
C14—C8—C13—C12	179.2 (4)	C14—N2—S2—O6	114.4 (4)
C9—C8—C13—S2	178.4 (4)	C14—N2—S2—C13	−1.7 (4)
C14—C8—C13—S2	−1.2 (5)	C8—C13—S2—O5	116.8 (4)
C11—C12—C13—C8	0.3 (8)	C12—C13—S2—O5	−63.7 (6)
C11—C12—C13—S2	−179.2 (4)	C8—C13—S2—O6	−115.1 (4)
C13—C8—C14—O2	−178.9 (5)	C12—C13—S2—O6	64.4 (6)
C9—C8—C14—O2	1.6 (7)	C8—C13—S2—N2	1.7 (4)
C13—C8—C14—N2	0.1 (6)	C12—C13—S2—N2	−178.8 (5)
C9—C8—C14—N2	−179.4 (5)	C22—N3—Zn1—N6	138.1 (4)
N4—C15—C16—C17	70.4 (6)	C22—N3—Zn1—N5	−40.7 (4)
C15—C16—C17—N6	−68.9 (6)	C22—N3—Zn1—O2	46.5 (4)
N4—C18—C19—N5	−59.4 (7)	C22—N3—Zn1—O1	−133.6 (4)
N5—C20—C21—C22	68.2 (7)	C17—N6—Zn1—N3	136.5 (4)
C20—C21—C22—N3	−72.1 (6)	C17—N6—Zn1—N4	−42.2 (4)
O1—C7—N1—S1B	168.7 (5)	C17—N6—Zn1—O2	−134.6 (4)
C1—C7—N1—S1B	−12.1 (8)	C17—N6—Zn1—O1	45.6 (4)
O1—C7—N1—S1A	−161.8 (7)	C19—N5—Zn1—N3	163.8 (3)
C1—C7—N1—S1A	17.4 (8)	C20—N5—Zn1—N3	37.6 (4)
O2—C14—N2—S2	−179.9 (4)	C19—N5—Zn1—N4	−17.7 (3)
C8—C14—N2—S2	1.2 (5)	C20—N5—Zn1—N4	−143.8 (5)
C21—C22—N3—Zn1	60.2 (6)	C19—N5—Zn1—O2	75.2 (3)
C19—C18—N4—C15	167.5 (5)	C20—N5—Zn1—O2	−51.0 (4)
C19—C18—N4—Zn1	40.0 (6)	C19—N5—Zn1—O1	−105.1 (3)
C16—C15—N4—C18	176.7 (5)	C20—N5—Zn1—O1	128.8 (4)
C16—C15—N4—Zn1	−60.7 (5)	C18—N4—Zn1—N6	169.4 (4)
C18—C19—N5—C20	174.1 (5)	C15—N4—Zn1—N6	43.5 (4)
C18—C19—N5—Zn1	44.3 (5)	C18—N4—Zn1—N5	−11.9 (4)
C21—C20—N5—C19	−176.1 (5)	C15—N4—Zn1—N5	−137.8 (4)
C21—C20—N5—Zn1	−53.4 (6)	C18—N4—Zn1—O2	−98.8 (4)
C16—C17—N6—Zn1	58.9 (6)	C15—N4—Zn1—O2	135.3 (4)
N1—C7—O1—Zn1	−6.3 (10)	C18—N4—Zn1—O1	81.3 (4)
C1—C7—O1—Zn1	174.6 (4)	C15—N4—Zn1—O1	−44.6 (4)
N2—C14—O2—Zn1	2.1 (8)	C14—O2—Zn1—N3	142.0 (5)
C8—C14—O2—Zn1	−179.1 (3)	C14—O2—Zn1—N6	43.0 (4)
C7—N1—S1A—O4A	−133.4 (12)	C14—O2—Zn1—N5	−128.1 (5)
S1B—N1—S1A—O4A	−23.0 (11)	C14—O2—Zn1—N4	−46.4 (5)
C7—N1—S1A—O3A	102.6 (9)	C7—O1—Zn1—N3	46.4 (6)
S1B—N1—S1A—O3A	−147.1 (12)	C7—O1—Zn1—N6	145.4 (6)
C7—N1—S1A—C6	−25.4 (8)	C7—O1—Zn1—N5	−43.5 (6)
S1B—N1—S1A—C6	85.0 (9)	C7—O1—Zn1—N4	−125.3 (6)
C5—C6—S1A—O4A	−73.2 (14)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3A···N1	0.90	2.56	3.282 (7)	137
N6—H6A···N2	0.90	2.52	3.224 (6)	135
N5—H5A···N1	1.00 (6)	2.60 (6)	3.411 (7)	138 (4)
N4—H4A···N2	0.76 (7)	2.53 (7)	3.278 (6)	166 (7)
O1W—H1W···O5	0.86 (2)	2.01 (3)	2.833 (8)	161 (8)
N3—H3B···O1W^{i}	0.90	2.22	3.052 (7)	154
N6—H6B···O1W^{i}	0.90	2.22	3.071 (9)	157

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

Fig. 1

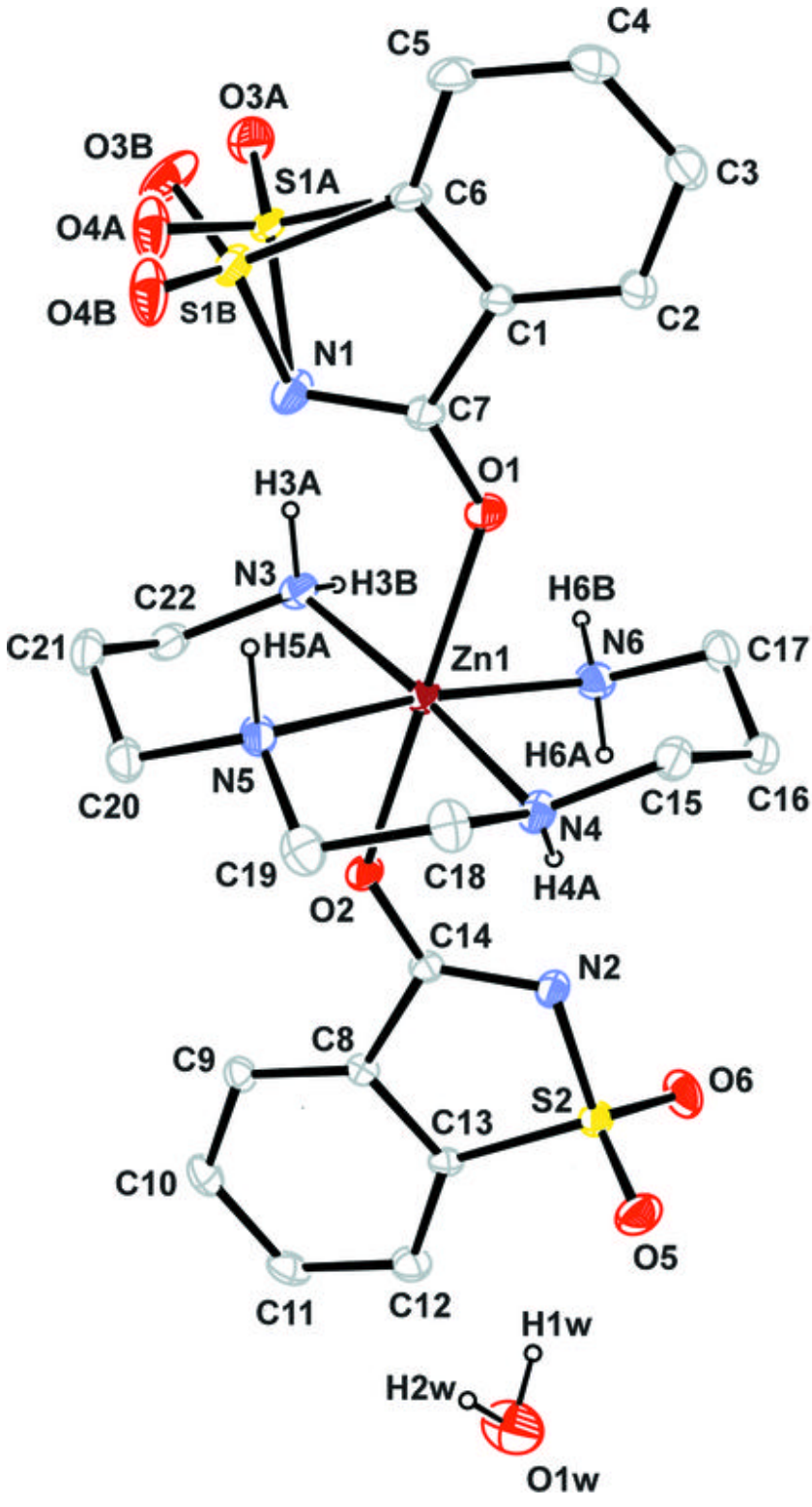

C5
C4
O3A
O3B
S1A
C6
C3
O4A
O4B
S1B
C1
C2
N1
C7
O1
H3A
N3
H3B
C22
H6B
C21
H5A
Zn1
N6
C17
N5
H6A
C20
C15
C16
N4
C19
C18
H4A
O2
C14
N2
C8
C9
S2
O6
C13
C10
O5
C11
C12
H1w
H2w
O1w

Fig. 2