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Bis[2-(2-pyridylmethylamino)ethane-sulfonato- $\kappa^3 N, N', O$]zinc(II)

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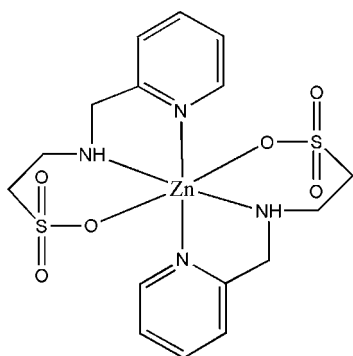
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Key indicators: single-crystal X-ray study; $T = 291$ K; mean $\sigma(C-C) = 0.003$ Å; R factor = 0.023; wR factor = 0.062; data-to-parameter ratio = 17.7.

The title mononuclear complex, $[Zn(C_8H_{11}N_2O_3S)_2]$, is a zinc salt of 2-(2-pyridylmethylamino)ethanesulfonic acid (Hpmt). The Zn^{II} ion is located on an inversion centre and is octahedrally surrounded by four N and two O atoms. The deprotonated pmt⁻ anion coordinates in a facial arrangement through its two N atoms and one of the sulfonate O atoms. The crystal packing is determined by intermolecular N—H...O and C—H...O hydrogen bonds.

Related literature

For the structures of the Co(II) and Ni(II) analogues, see: Li *et al.* (2008); Liao *et al.* (2007). For the preparation of the Hpmt ligand, see: Li *et al.* (2006).



Experimental

Crystal data

 $[Zn(C_8H_{11}N_2O_3S)_2]$ $M_r = 495.87$ Monoclinic, $P2_1/c$ $a = 9.6288$ (13) Å $b = 10.0047$ (13) Å $c = 11.3624$ (15) Å $\beta = 105.965$ (1)° $V = 1052.4$ (2) Å³ $Z = 2$ Mo $K\alpha$ radiation $\mu = 1.41$ mm⁻¹ $T = 291$ K

0.50 × 0.39 × 0.29 mm

Data collection

Bruker APEXII CCD area-detector diffractometer

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

 $T_{\min} = 0.540$, $T_{\max} = 0.689$

6318 measured reflections

2419 independent reflections

2221 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.012$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.023$ $wR(F^2) = 0.062$ $S = 1.07$

2419 reflections

137 parameters

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 0.23$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.38$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Zn1—N2	2.1336 (12)	Zn1—N1	2.2130 (13)
Zn1—O1	2.1465 (11)		
N2—Zn1—O1	92.40 (5)	O1—Zn1—N1 ⁱ	89.78 (5)
N2 ⁱ —Zn1—O1	87.60 (4)	N2—Zn1—N1	78.06 (5)
N2—Zn1—N1 ⁱ	101.93 (5)	O1—Zn1—N1	90.22 (5)

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

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H1N ⁱⁱ ···O2 ⁱⁱⁱ	0.855 (18)	2.079 (18)	2.9259 (17)	170.6 (16)
C1—H1···O2 ⁱⁱⁱ	0.93	2.47	3.388 (2)	169
C4—H4···O3 ^{iv}	0.93	2.49	3.324 (2)	150
C6—H6B···O1 ⁱ	0.97	2.56	3.056 (2)	112
C8—H8B···O2 ^v	0.97	2.56	3.265 (2)	130

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

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

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2794).

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supplementary materials

Acta Cryst. (2009). E65, m706 [doi:10.1107/S1600536809019990]

Bis[2-(2-pyridylmethylamino)ethanesulfonato- κ^3N,N',O]zinc(II)

Z.-X. Du and G.-Y. Zhang

Comment

The complex (I) is isostructural with its analogues [Co(C₈H₁₁N₂O₃S)₂] (Li *et al.*, 2006), [Ni(C₈H₁₁N₂O₃S)₂] (Liao *et al.*, 2007) and [Cu(C₈H₁₁N₂O₃S)₂].2H₂O (Li *et al.*, 2008), whose structures have been described in detail. The six-coordinate Zn^{II} ion locates on a centre of symmetry with two deprotonated pmt⁻ anions coordinate in a tridentate facial arrangement with its three donor atoms (Fig.1). The bond lengths and angles of (I) are in good agreement with its Co(II) and Ni(II) analogues (Table 1).

The N—H donor and S=O acceptor groups of the pmt ions are involved in hydrogen bonding interactions and forms a two-dimensional network in the *bc* plane (Table 2 and Fig. 2).

Experimental

The ligand Hpmt was prepared according to the method of Li *et al.*, 2006. To the solution of Hpmt (2.0 mmol, 0.43 g) in water (25 ml), solid ZnCl₂ (1 mmol, 0.14 g) was added. The resulting mixture was stirred at 333 K for 5 h, then cooled to room temperature. After filtration, the filtrate was left to stand at room temperature for slow evaporation. Colourless block-shaped crystals suitable for X-ray diffraction were obtained in a yield of 42%. Analysis, found: C 38.66, H 4.37, N 11.32, S 12.95%; C₁₆H₂₂N₄O₆S₂Zn requires: C 38.72, H 4.44, N 11.29, S 12.90%. IR (KBr, ν , cm⁻¹): 771.3 [γ (C=C—H)], 746.5(γ CH₂); 1190.3, 1151.4, 1038.8(ν SO₃⁻); 1607.2, 1572.3(ν C=C + ν C=N); 3198.2(ν N—H).

Refinement

H atoms bonded to C were positioned geometrically with C—H distance of 0.93–0.97 Å, and treated as riding atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The N—H hydrogen atom was located in a difference Fourier map and refined isotropically.

Figures

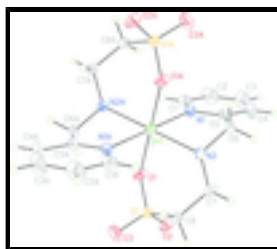


Fig. 1. Molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level. Atoms with the suffix A are at the symmetry position ($-x, -y, -z$).

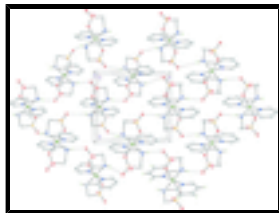


Fig. 2. The hydrogen bonding interactions in (I) (dashed lines) projected in *bc* plane. H atoms on C atoms have been omitted.

Bis[2-(2-pyridylmethylamino)ethanesulfonato- κ^3N,N',O]zinc(II)

Crystal data

[Zn(C₈H₁₁N₂O₃S)₂]

M_r = 495.87

Monoclinic, *P*2₁/*c*

Hall symbol: -*P* 2ybc

a = 9.6288 (13) Å

b = 10.0047 (13) Å

c = 11.3624 (15) Å

β = 105.965 (1)°

V = 1052.4 (2) Å³

Z = 2

*F*₀₀₀ = 512

D_x = 1.565 Mg m⁻³

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 4168 reflections

θ = 2.8–28.2°

μ = 1.41 mm⁻¹

T = 291 K

Block, colourless

0.50 × 0.39 × 0.29 mm

Data collection

Bruker APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

T = 291 K

φ and ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

*T*_{min} = 0.540, *T*_{max} = 0.689

6318 measured reflections

2419 independent reflections

2221 reflections with *I* > 2σ(*I*)

*R*_{int} = 0.012

θ_{max} = 27.5°

θ_{min} = 2.8°

h = -11→12

k = -9→12

l = -14→14

Refinement

Refinement on *F*²

Least-squares matrix: full

R [*F*² > 2σ(*F*²)] = 0.023

wR(*F*²) = 0.062

S = 1.07

2419 reflections

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

$w = 1/[\sigma^2(F_o^2) + (0.0322P)^2 + 0.3355P]$

where $P = (F_o^2 + 2F_c^2)/3$

(Δ/σ)_{max} = 0.001

Δρ_{max} = 0.23 e Å⁻³

137 parameters

$$\Delta\rho_{\min} = -0.38 \text{ e } \text{\AA}^{-3}$$

Primary atom site location: structure-invariant direct methods

Extinction correction: none

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Zn1	0.5000	0.0000	0.5000	0.02437 (8)
S1	0.63235 (4)	0.29294 (4)	0.45567 (3)	0.02858 (10)
O1	0.57188 (13)	0.16226 (11)	0.40792 (10)	0.0375 (3)
O2	0.55332 (14)	0.40018 (12)	0.37950 (10)	0.0428 (3)
O3	0.78676 (14)	0.30037 (16)	0.47547 (13)	0.0573 (4)
N1	0.27433 (14)	0.03618 (13)	0.39160 (12)	0.0309 (3)
N2	0.42452 (13)	0.13247 (12)	0.61609 (11)	0.0274 (3)
C1	0.2143 (2)	0.02195 (18)	0.27020 (16)	0.0412 (4)
H1	0.2677	-0.0185	0.2232	0.049*
C2	0.0760 (2)	0.0655 (3)	0.2137 (2)	0.0633 (6)
H2	0.0370	0.0549	0.1297	0.076*
C3	-0.0031 (2)	0.1249 (3)	0.2834 (2)	0.0746 (7)
H3	-0.0961	0.1558	0.2468	0.090*
C4	0.05670 (19)	0.1385 (2)	0.4085 (2)	0.0584 (5)
H4	0.0044	0.1775	0.4571	0.070*
C5	0.19633 (16)	0.09248 (16)	0.45981 (15)	0.0350 (3)
C6	0.26892 (17)	0.10136 (17)	0.59524 (15)	0.0360 (3)
H6A	0.2233	0.1707	0.6313	0.043*
H6B	0.2584	0.0171	0.6342	0.043*
C7	0.44409 (17)	0.27835 (15)	0.59923 (13)	0.0303 (3)
H7A	0.4184	0.3273	0.6639	0.036*
H7B	0.3796	0.3066	0.5217	0.036*
C8	0.59850 (16)	0.31189 (15)	0.60131 (13)	0.0292 (3)
H8A	0.6637	0.2542	0.6599	0.035*

supplementary materials

H8B	0.6190	0.4034	0.6287	0.035*
H1N	0.4634 (18)	0.1133 (17)	0.6913 (16)	0.032 (4)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.02484 (13)	0.02218 (13)	0.02641 (12)	0.00349 (8)	0.00760 (9)	-0.00102 (8)
S1	0.03239 (19)	0.02599 (19)	0.02848 (17)	0.00050 (14)	0.01027 (14)	0.00143 (13)
O1	0.0591 (7)	0.0267 (6)	0.0308 (5)	-0.0037 (5)	0.0194 (5)	-0.0023 (4)
O2	0.0672 (8)	0.0301 (6)	0.0319 (5)	0.0094 (5)	0.0148 (5)	0.0068 (5)
O3	0.0353 (7)	0.0786 (11)	0.0625 (8)	-0.0049 (6)	0.0208 (6)	-0.0068 (7)
N1	0.0272 (6)	0.0305 (6)	0.0330 (6)	0.0024 (5)	0.0051 (5)	0.0010 (5)
N2	0.0307 (6)	0.0268 (6)	0.0253 (6)	0.0022 (5)	0.0087 (5)	0.0003 (5)
C1	0.0404 (9)	0.0424 (10)	0.0366 (8)	-0.0027 (7)	0.0035 (7)	0.0020 (7)
C2	0.0453 (11)	0.0799 (16)	0.0501 (11)	-0.0037 (11)	-0.0114 (9)	0.0067 (11)
C3	0.0310 (10)	0.097 (2)	0.0817 (16)	0.0137 (11)	-0.0080 (10)	0.0102 (15)
C4	0.0270 (8)	0.0691 (14)	0.0786 (14)	0.0112 (9)	0.0137 (9)	0.0001 (11)
C5	0.0259 (7)	0.0329 (8)	0.0474 (9)	0.0013 (6)	0.0118 (6)	0.0005 (7)
C6	0.0327 (8)	0.0387 (9)	0.0430 (8)	0.0013 (7)	0.0210 (7)	-0.0030 (7)
C7	0.0367 (8)	0.0245 (7)	0.0313 (7)	0.0042 (6)	0.0120 (6)	-0.0028 (6)
C8	0.0348 (7)	0.0268 (7)	0.0241 (6)	-0.0009 (6)	0.0048 (5)	-0.0029 (5)

Geometric parameters (\AA , $^\circ$)

Zn1—N2	2.1336 (12)	C1—H1	0.9300
Zn1—N2 ⁱ	2.1336 (12)	C2—C3	1.376 (4)
Zn1—O1	2.1465 (11)	C2—H2	0.9300
Zn1—O1 ⁱ	2.1465 (11)	C3—C4	1.386 (3)
Zn1—N1 ⁱ	2.2130 (13)	C3—H3	0.9300
Zn1—N1	2.2130 (13)	C4—C5	1.388 (2)
S1—O3	1.4431 (13)	C4—H4	0.9300
S1—O2	1.4549 (12)	C5—C6	1.508 (2)
S1—O1	1.4727 (11)	C6—H6A	0.9700
S1—C8	1.7825 (15)	C6—H6B	0.9700
N1—C5	1.342 (2)	C7—C8	1.518 (2)
N1—C1	1.349 (2)	C7—H7A	0.9700
N2—C6	1.4841 (19)	C7—H7B	0.9700
N2—C7	1.4908 (19)	C8—H8A	0.9700
N2—H1N	0.856 (17)	C8—H8B	0.9700
C1—C2	1.380 (3)		
N2—Zn1—N2 ⁱ	180.0	N1—C1—H1	119.0
N2—Zn1—O1	92.40 (5)	C2—C1—H1	119.0
N2 ⁱ —Zn1—O1	87.60 (4)	C3—C2—C1	118.96 (19)
N2—Zn1—O1 ⁱ	87.60 (5)	C3—C2—H2	120.5
N2 ⁱ —Zn1—O1 ⁱ	92.40 (5)	C1—C2—H2	120.5
O1—Zn1—O1 ⁱ	180.0	C2—C3—C4	119.55 (18)
N2—Zn1—N1 ⁱ	101.93 (5)	C2—C3—H3	120.2

N2 ⁱ —Zn1—N1 ⁱ	78.06 (5)	C4—C3—H3	120.2
O1—Zn1—N1 ⁱ	89.78 (5)	C3—C4—C5	118.7 (2)
O1 ⁱ —Zn1—N1 ⁱ	90.22 (5)	C3—C4—H4	120.7
N2—Zn1—N1	78.06 (5)	C5—C4—H4	120.7
N2 ⁱ —Zn1—N1	101.94 (5)	N1—C5—C4	121.82 (16)
O1—Zn1—N1	90.22 (5)	N1—C5—C6	115.98 (13)
O1 ⁱ —Zn1—N1	89.78 (5)	C4—C5—C6	122.19 (16)
N1 ⁱ —Zn1—N1	180.0	N2—C6—C5	109.85 (12)
O3—S1—O2	113.74 (9)	N2—C6—H6A	109.7
O3—S1—O1	112.90 (8)	C5—C6—H6A	109.7
O2—S1—O1	110.27 (7)	N2—C6—H6B	109.7
O3—S1—C8	107.06 (8)	C5—C6—H6B	109.7
O2—S1—C8	105.93 (7)	H6A—C6—H6B	108.2
O1—S1—C8	106.36 (7)	N2—C7—C8	111.86 (12)
S1—O1—Zn1	129.76 (6)	N2—C7—H7A	109.2
C5—N1—C1	118.96 (14)	C8—C7—H7A	109.2
C5—N1—Zn1	111.53 (10)	N2—C7—H7B	109.2
C1—N1—Zn1	129.14 (12)	C8—C7—H7B	109.2
C6—N2—C7	110.03 (12)	H7A—C7—H7B	107.9
C6—N2—Zn1	105.77 (9)	C7—C8—S1	112.98 (10)
C7—N2—Zn1	116.88 (9)	C7—C8—H8A	109.0
C6—N2—H1N	104.9 (12)	S1—C8—H8A	109.0
C7—N2—H1N	108.2 (12)	C7—C8—H8B	109.0
Zn1—N2—H1N	110.4 (11)	S1—C8—H8B	109.0
N1—C1—C2	122.03 (19)	H8A—C8—H8B	107.8
O3—S1—O1—Zn1	103.94 (10)	N1—Zn1—N2—C7	-90.09 (10)
O2—S1—O1—Zn1	-127.61 (9)	C5—N1—C1—C2	1.1 (3)
C8—S1—O1—Zn1	-13.19 (11)	Zn1—N1—C1—C2	-171.19 (16)
N2—Zn1—O1—S1	33.74 (10)	N1—C1—C2—C3	-0.2 (3)
N2 ⁱ —Zn1—O1—S1	-146.26 (10)	C1—C2—C3—C4	-0.7 (4)
N1 ⁱ —Zn1—O1—S1	-68.19 (10)	C2—C3—C4—C5	0.7 (4)
N1—Zn1—O1—S1	111.81 (10)	C1—N1—C5—C4	-1.1 (3)
N2—Zn1—N1—C5	-14.41 (11)	Zn1—N1—C5—C4	172.45 (15)
N2 ⁱ —Zn1—N1—C5	165.59 (11)	C1—N1—C5—C6	178.25 (15)
O1—Zn1—N1—C5	-106.82 (11)	Zn1—N1—C5—C6	-8.16 (17)
O1 ⁱ —Zn1—N1—C5	73.18 (11)	C3—C4—C5—N1	0.3 (3)
N2—Zn1—N1—C1	158.35 (15)	C3—C4—C5—C6	-179.1 (2)
N2 ⁱ —Zn1—N1—C1	-21.65 (15)	C7—N2—C6—C5	80.21 (15)
O1—Zn1—N1—C1	65.94 (14)	Zn1—N2—C6—C5	-46.87 (14)
O1 ⁱ —Zn1—N1—C1	-114.06 (14)	N1—C5—C6—N2	37.7 (2)
O1—Zn1—N2—C6	122.46 (10)	C4—C5—C6—N2	-142.93 (17)
O1 ⁱ —Zn1—N2—C6	-57.54 (10)	C6—N2—C7—C8	-171.67 (11)
N1 ⁱ —Zn1—N2—C6	-147.26 (9)	Zn1—N2—C7—C8	-51.08 (14)
N1—Zn1—N2—C6	32.74 (9)	N2—C7—C8—S1	85.42 (13)
O1—Zn1—N2—C7	-0.37 (10)	O3—S1—C8—C7	-168.32 (11)
O1 ⁱ —Zn1—N2—C7	179.63 (10)	O2—S1—C8—C7	69.97 (12)

supplementary materials

N1ⁱ—Zn1—N2—C7 89.91 (10) O1—S1—C8—C7 -47.37 (12)
Symmetry codes: (i) $-x+1, -y, -z+1$.

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N2—H1N \cdots O2 ⁱⁱ	0.855 (18)	2.079 (18)	2.9259 (17)	170.6 (16)
C1—H1 \cdots O2 ⁱⁱⁱ	0.93	2.47	3.388 (2)	169
C4—H4 \cdots O3 ^{iv}	0.93	2.49	3.324 (2)	150
C6—H6B \cdots O1 ⁱ	0.97	2.56	3.056 (2)	112
C8—H8B \cdots O2 ^v	0.97	2.56	3.265 (2)	130

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

Fig. 1

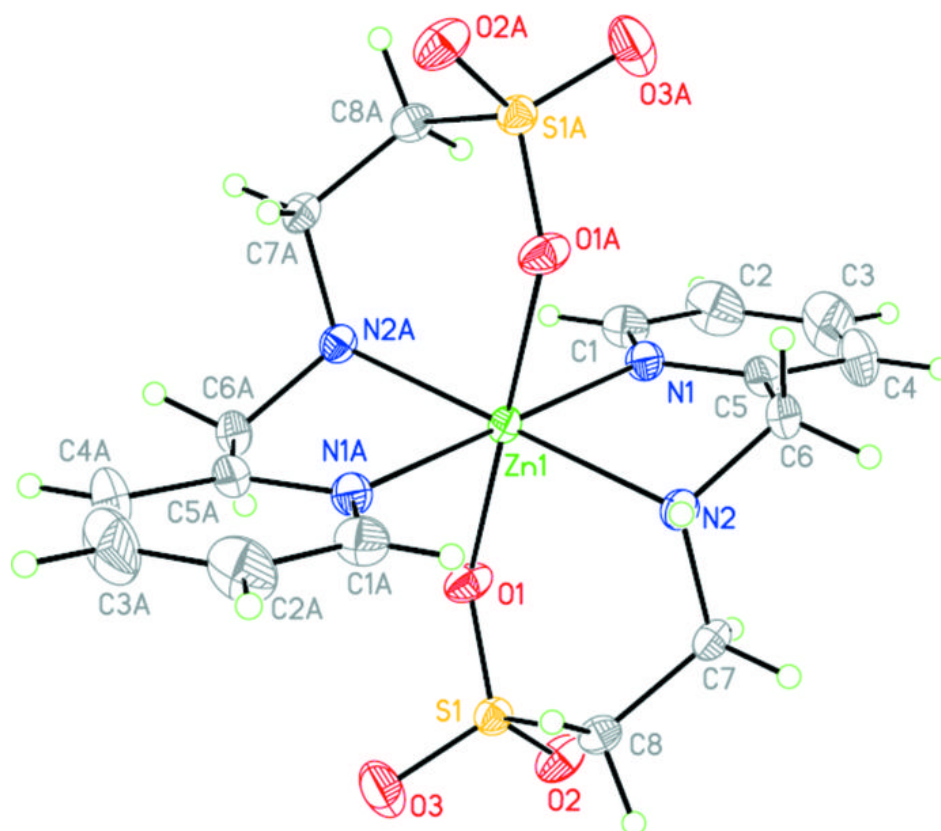


Fig. 2

