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Bis{μ-2-[(2-oxidobenzylidene)aminomethyl]phenolato-κ^3O,N,O'}-bis[(pyridine-κN)zinc(II)]

Chun Yang,‡ Ou-Yang Yan, Qing-Lun Wang* and Dai-Zheng Liao

Department of Chemistry, Nankai University, Tianjin 300071, People's Republic of China
Correspondence e-mail: wangql@nankai.edu.cn

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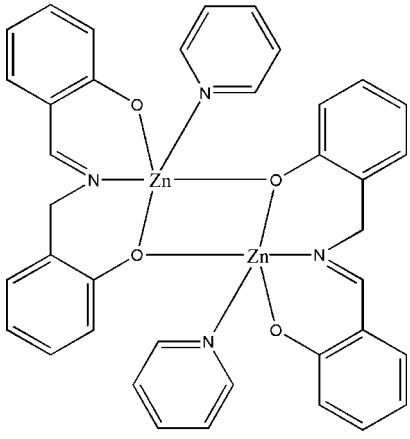
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.004 Å; R factor = 0.037; wR factor = 0.084; data-to-parameter ratio = 16.4.

In the title centrosymmetric zinc(II) complex, $[Zn_2(C_4H_{13}NO_2)_2(C_6H_5N)_2]$, each Zn^{II} atom is coordinated by two 2-[(2-oxidobenzylidene)aminomethyl]phenolate (L) ligands and one pyridine (py) molecule in a distorted trigonal-bipyramidal geometry. Each L ligand behaves as a tridentate ligand and provides a phenolate oxygen bridge which links the two Zn^{II} atoms. The ZnL(py) units are linked by π–π interactions between adjacent pyridine molecules, with a centroid–centroid distance of 3.724 Å, resulting in a two-dimensional structure.

Related literature

For the biochemical and catalytic activity of zinc(II) complexes, see: Marco *et al.* (2004); Kim *et al.* (2000). Zn^{II} ions in phenoxide-bridged dinuclear complexes provide flexible coordination numbers, see: Atakol *et al.* (1999); Huang *et al.* (2006). For the preparation of H_2L, see: Moustafa (2003).



Experimental

Crystal data

$[Zn_2(C_{14}H_{13}NO_2)_2(C_5H_5N)_2]$
M_r = 739.42
Monoclinic, $P2_1/c$
a = 10.145 (6) Å
b = 13.806 (8) Å
c = 12.671 (8) Å
β = 102.063 (10)°
V = 1735.5 (18) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.43 mm^{-1}
T = 294 (2) K
0.20 × 0.18 × 0.12 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.693, T_{max} = 1.000
(expected range = 0.584–0.843)
9656 measured reflections
3561 independent reflections
2440 reflections with $I > 2\sigma(I)$
R_{int} = 0.056

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.084
S = 1.00
3561 reflections
217 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

Data collection: *SMART-NT* (Bruker, 1998); cell refinement: *SAINT-NT* (Bruker, 1998); data reduction: *SAINT-NT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL-NT* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL-NT*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2532).

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‡ Permanent address: School of Chemical Engineering and Technology, Hebei University of Technology, Tianjin 300130, People's Republic of China.

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supporting information

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Bis{μ-2-[(2-oxidobenzylidene)aminomethyl]phenolato-κ^3O,N,O'}bis[(pyridine-κN)zinc(II)]

Chun Yang, Ou-Yang Yan, Qing-Lun Wang and Dai-Zheng Liao

S1. Comment

The schiff base ligand, *N*, *N*′ -bis-salicylidene-1,3-diaminopropane give a phenoxide-bridged homodinuclear complex with $ZnCl_2$. In the crystal structure, one Zn^{II} center has a distorted square pyramid geometry, the other one has a distorted tetrahedral geometry (Atakol *et al.*, 1999). In the tetra-imine macrocyclic cavity of dinuclear zinc(II) complexes, both of the two Zn^{II} ions take a distorted square pyramid geometry with an apical position occupied by a water molecule (Huang *et al.*, 2006).

The title complex $[Zn_2(py)_2(L)_2]$, (I), is the first dinuclear zinc(II) complex containing 2-hydroxybenzylamine-2′-hydroxybenzylidene(H_2L) and pyridine(py). In (I), there are two phenoxide bridges between two Zn^{II} atoms (Fig. 1). As can be seen from Table 1, in this complex the Zn—*N*(pyridine type) distance (2.129 Å) is similar to the average value of the reported structures, which is 2.132Å (Marco *et al.*, 2004). The Zn—O bond lengths of 2.155(Zn1—O1A), 1.988(Zn1—O2A) and 1.9814 (Zn1—O1)Å in the title complex are comparable to those found in previously reported cadmium and zinc phenoxide complexes (1.864–2.170 Å) (Kim *et al.*, 2000). The bond lengths and angles show that the five coordinations around Zn^{II} atoms are not ideal square pyramid or trigonal bipyramid. The evalution between the two geometrics can be quantified using the τ parameter (Atakol *et al.*, 1999), the τ value is calculated as 0.81, indicating a distorted trigonal bipyramid geometry. The Zn—O—Zn—O ring is planar (360°). Caculation indicates that Zn···Zn distance is 3.3 Å. The packing diagram of complex (I) is shown in Fig.2. The distance between adjacent pyridines is 3.724 Å. The $[Zn_2(py)_2(L)_2]$ units are thus linked by $\pi\cdots\pi$ interaction between pyridine molecules, resulting in the formation of a two-dimensional structure.

S2. Experimental

The schiff base ligand H_2L was prepared by a published procedure (Moustafa, 2003). H_2L(0.1135 g, 0.5 mmol) was dissolved in methanol (20 ml). After a hot solution of zinc(II) acetate dihydrate (0.1097 g, 0.5 mmol) in water (10 ml) was added dropwise to the solution with continuous stirring, yellow precipitation came into being. When py (5 ml) was added to the turbid solution, the yellow precipitation was dissolved quickly and completely. After stirring further for 1 h with heating, the resulting solution was cooled and filtered. After about 1 week, single crystals of (I), suitable for X-ray structure determination, were obtained by slow evaporation of the solution at room temperature. Analysis calculated for $C_{38}H_{32}N_4O_4Zn_2$: C 61.73, N 7.58, H 4.36%. Found: C 62.39, N 7.59, H 4.30%.

S3. Refinement

All H atoms were positioned geometrically, with C—H = 0.93 and 0.97 Å for methylene and methyl H, and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C).

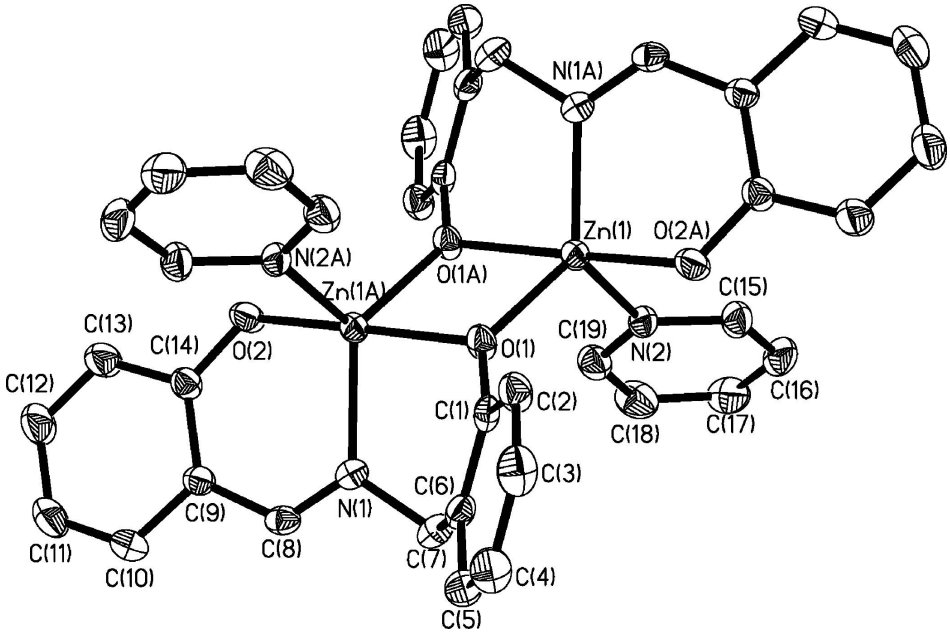

Figure 1

The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity.

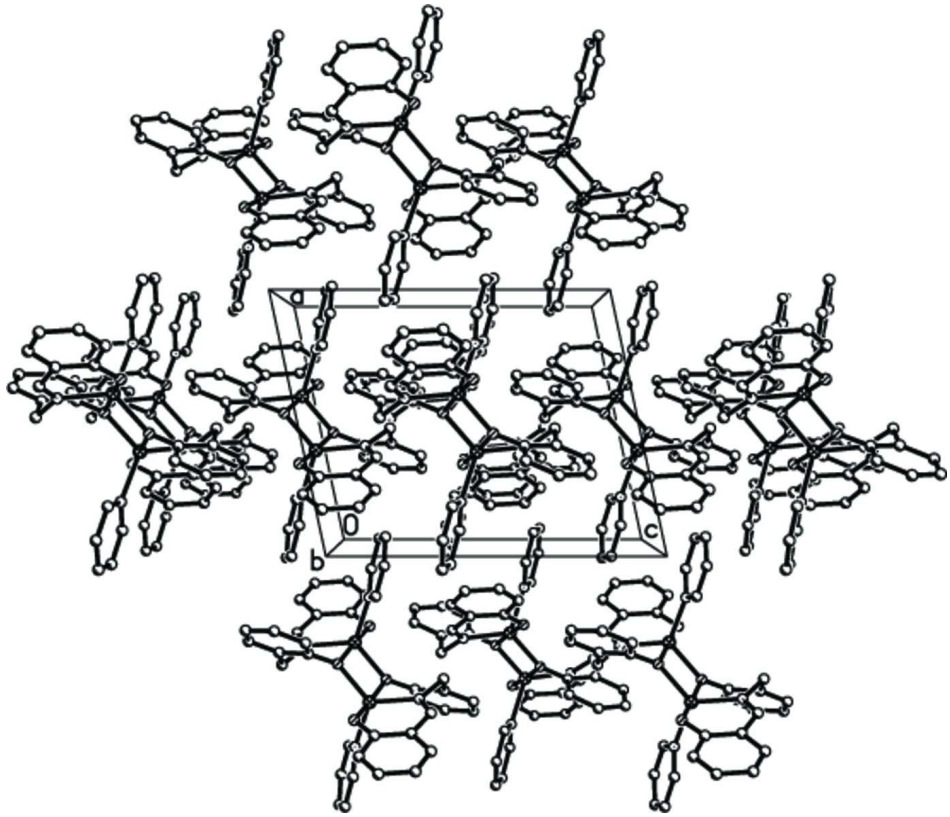

Figure 2

A view of the crystal packing along the *b* axis.

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Crystal data

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$M_r = 739.42$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 10.145\ (6)$ Å
$b = 13.806\ (8)$ Å
$c = 12.671\ (8)$ Å
$\beta = 102.063\ (10)°$
$V = 1735.5\ (18)$ Å^3
$Z = 2$
$F(000) = 760$
$D_x = 1.415$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2807 reflections
$\theta = 2.5–24.3°$
$\mu = 1.43$ mm^{-1}
$T = 294$ K
Block, yellow
0.20 × 0.18 × 0.12 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.693$, $T_{max} = 1.000$
9656 measured reflections
3561 independent reflections
2440 reflections with $I > 2\sigma(I)$
$R_{int} = 0.056$
$\theta_{max} = 26.5°$, $\theta_{min} = 2.1°$
$h = -12\rightarrow12$
$k = -17\rightarrow14$
$l = -15\rightarrow14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.084$
$S = 1.00$
3561 reflections
217 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
$w = 1/[\sigma^2(F_o^2) + (0.0341P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.40$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Zn1	0.60866 (3)	1.08479 (2)	0.49115 (2)	0.03166 (11)

O1	0.46079 (18)	1.05680 (13)	0.56701 (14)	0.0360 (5)
O2	0.3413 (2)	0.78111 (13)	0.45886 (14)	0.0444 (5)
N1	0.4099 (2)	0.87742 (16)	0.66733 (16)	0.0326 (5)
N2	0.7965 (2)	1.02458 (17)	0.57069 (17)	0.0356 (6)
C1	0.4150 (3)	1.09767 (19)	0.6492 (2)	0.0330 (6)
C2	0.3621 (3)	1.1911 (2)	0.6419 (2)	0.0422 (7)
H2	0.3611	1.2278	0.5802	0.051*
C3	0.3106 (3)	1.2300 (2)	0.7263 (3)	0.0531 (9)
H3	0.2740	1.2920	0.7201	0.064*
C4	0.3136 (3)	1.1771 (2)	0.8189 (3)	0.0550 (9)
H4	0.2799	1.2035	0.8754	0.066*
C5	0.3670 (3)	1.0849 (2)	0.8273 (2)	0.0492 (8)
H5	0.3695	1.0497	0.8903	0.059*
C6	0.4171 (3)	1.0433 (2)	0.7438 (2)	0.0351 (7)
C7	0.4791 (3)	0.9441 (2)	0.7531 (2)	0.0429 (7)
H7A	0.5732	0.9494	0.7489	0.051*
H7B	0.4753	0.9170	0.8230	0.051*
C8	0.3665 (3)	0.7963 (2)	0.6971 (2)	0.0351 (7)
H8	0.3792	0.7859	0.7710	0.042*
C9	0.3000 (3)	0.7197 (2)	0.6279 (2)	0.0340 (6)
C10	0.2446 (3)	0.6435 (2)	0.6794 (2)	0.0503 (8)
H10	0.2511	0.6465	0.7536	0.060*
C11	0.1821 (4)	0.5660 (2)	0.6240 (3)	0.0643 (10)
H11	0.1460	0.5170	0.6597	0.077*
C12	0.1733 (4)	0.5613 (2)	0.5133 (3)	0.0640 (10)
H12	0.1308	0.5087	0.4745	0.077*
C13	0.2264 (3)	0.6333 (2)	0.4608 (2)	0.0552 (9)
H13	0.2191	0.6281	0.3866	0.066*
C14	0.2920 (3)	0.7152 (2)	0.5145 (2)	0.0365 (7)
C15	0.9071 (3)	1.0806 (2)	0.5933 (2)	0.0446 (7)
H15	0.8991	1.1462	0.5762	0.054*
C16	1.0325 (3)	1.0443 (3)	0.6410 (2)	0.0561 (9)
H16	1.1073	1.0849	0.6558	0.067*
C17	1.0448 (3)	0.9487 (3)	0.6659 (3)	0.0607 (10)
H17	1.1287	0.9229	0.6970	0.073*
C18	0.9335 (3)	0.8906 (3)	0.6452 (3)	0.0603 (9)
H18	0.9401	0.8251	0.6630	0.072*
C19	0.8101 (3)	0.9309 (2)	0.5970 (2)	0.0461 (8)
H19	0.7343	0.8913	0.5826	0.055*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.02991 (19)	0.03587 (19)	0.03086 (18)	−0.00479 (15)	0.01014 (13)	−0.00201 (15)
O1	0.0333 (11)	0.0390 (11)	0.0407 (11)	−0.0073 (8)	0.0192 (9)	−0.0102 (9)
O2	0.0649 (14)	0.0390 (12)	0.0338 (10)	−0.0139 (10)	0.0206 (10)	−0.0039 (9)
N1	0.0330 (13)	0.0348 (13)	0.0293 (12)	0.0030 (10)	0.0044 (10)	−0.0020 (10)
N2	0.0286 (13)	0.0446 (15)	0.0342 (13)	−0.0056 (11)	0.0076 (10)	−0.0026 (11)

C1	0.0239 (14)	0.0372 (16)	0.0392 (16)	−0.0080 (12)	0.0096 (12)	−0.0133 (13)
C2	0.0455 (19)	0.0363 (17)	0.0480 (18)	−0.0079 (14)	0.0171 (14)	−0.0074 (14)
C3	0.051 (2)	0.0375 (18)	0.073 (2)	0.0010 (15)	0.0197 (18)	−0.0152 (17)
C4	0.060 (2)	0.055 (2)	0.059 (2)	−0.0088 (17)	0.0321 (18)	−0.0267 (18)
C5	0.056 (2)	0.056 (2)	0.0395 (17)	−0.0135 (17)	0.0188 (15)	−0.0146 (16)
C6	0.0332 (16)	0.0377 (16)	0.0336 (15)	−0.0057 (13)	0.0050 (12)	−0.0114 (13)
C7	0.0418 (18)	0.0504 (18)	0.0330 (15)	0.0001 (14)	0.0000 (13)	−0.0070 (14)
C8	0.0325 (16)	0.0437 (18)	0.0291 (14)	0.0082 (13)	0.0064 (12)	0.0048 (13)
C9	0.0332 (16)	0.0392 (16)	0.0306 (14)	0.0015 (13)	0.0086 (12)	0.0030 (13)
C10	0.057 (2)	0.056 (2)	0.0392 (17)	−0.0067 (17)	0.0138 (15)	0.0105 (16)
C11	0.073 (3)	0.060 (2)	0.064 (2)	−0.0266 (19)	0.026 (2)	0.0044 (19)
C12	0.079 (3)	0.054 (2)	0.064 (2)	−0.0310 (19)	0.025 (2)	−0.0107 (18)
C13	0.075 (2)	0.053 (2)	0.0420 (18)	−0.0196 (18)	0.0224 (17)	−0.0087 (16)
C14	0.0387 (17)	0.0359 (16)	0.0376 (16)	−0.0031 (13)	0.0142 (13)	−0.0022 (13)
C15	0.0351 (17)	0.054 (2)	0.0463 (17)	−0.0098 (15)	0.0123 (14)	−0.0063 (15)
C16	0.0306 (18)	0.086 (3)	0.050 (2)	−0.0117 (17)	0.0054 (15)	−0.008 (2)
C17	0.0317 (19)	0.098 (3)	0.051 (2)	0.0096 (19)	0.0062 (15)	0.012 (2)
C18	0.052 (2)	0.067 (2)	0.062 (2)	0.0123 (18)	0.0121 (17)	0.0219 (18)
C19	0.0376 (18)	0.053 (2)	0.0483 (18)	−0.0027 (15)	0.0095 (14)	0.0083 (15)

Geometric parameters (Å, º)

Zn1—O1	1.9814 (19)	C6—C7	1.501 (4)
Zn1—O2^{i}	1.988 (2)	C7—H7A	0.9700
Zn1—N1^{i}	2.045 (2)	C7—H7B	0.9700
Zn1—N2	2.129 (2)	C8—C9	1.448 (4)
Zn1—O1^{i}	2.155 (2)	C8—H8	0.9300
O1—C1	1.349 (3)	C9—C10	1.415 (4)
O1—Zn1^{i}	2.155 (2)	C9—C14	1.423 (4)
O2—C14	1.313 (3)	C10—C11	1.362 (4)
O2—Zn1^{i}	1.988 (2)	C10—H10	0.9300
N1—C8	1.288 (3)	C11—C12	1.389 (4)
N1—C7	1.484 (3)	C11—H11	0.9300
N1—Zn1^{i}	2.045 (2)	C12—C13	1.368 (4)
N2—C19	1.335 (3)	C12—H12	0.9300
N2—C15	1.344 (3)	C13—C14	1.413 (4)
C1—C2	1.394 (4)	C13—H13	0.9300
C1—C6	1.411 (4)	C15—C16	1.383 (4)
C2—C3	1.392 (4)	C15—H15	0.9300
C2—H2	0.9300	C16—C17	1.357 (5)
C3—C4	1.376 (4)	C16—H16	0.9300
C3—H3	0.9300	C17—C18	1.366 (5)
C4—C5	1.379 (4)	C17—H17	0.9300
C4—H4	0.9300	C18—C19	1.390 (4)
C5—C6	1.389 (4)	C18—H18	0.9300
C5—H5	0.9300	C19—H19	0.9300
O1—Zn1—O2^{i}	101.29 (8)	C6—C7—H7A	109.1

O1—Zn1—N1^{i}	127.04 (8)	N1—C7—H7B	109.1
O2^{i}—Zn1—N1^{i}	92.31 (8)	C6—C7—H7B	109.1
O1—Zn1—N2	112.80 (9)	H7A—C7—H7B	107.8
O2^{i}—Zn1—N2	93.97 (9)	N1—C8—C9	127.0 (3)
N1^{i}—Zn1—N2	117.01 (8)	N1—C8—H8	116.5
O1—Zn1—O1^{i}	76.26 (8)	C9—C8—H8	116.5
O2^{i}—Zn1—O1^{i}	175.72 (8)	C10—C9—C14	119.2 (3)
N1^{i}—Zn1—O1^{i}	86.51 (8)	C10—C9—C8	116.3 (2)
N2—Zn1—O1^{i}	90.23 (8)	C14—C9—C8	124.4 (2)
C1—O1—Zn1	135.25 (16)	C11—C10—C9	122.3 (3)
C1—O1—Zn1^{i}	120.19 (15)	C11—C10—H10	118.9
Zn1—O1—Zn1^{i}	103.74 (8)	C9—C10—H10	118.9
C14—O2—Zn1^{i}	125.11 (17)	C10—C11—C12	118.8 (3)
C8—N1—C7	117.6 (2)	C10—C11—H11	120.6
C8—N1—Zn1^{i}	122.84 (19)	C12—C11—H11	120.6
C7—N1—Zn1^{i}	119.56 (18)	C13—C12—C11	120.6 (3)
C19—N2—C15	117.8 (3)	C13—C12—H12	119.7
C19—N2—Zn1	122.13 (19)	C11—C12—H12	119.7
C15—N2—Zn1	120.0 (2)	C12—C13—C14	122.7 (3)
O1—C1—C2	121.8 (2)	C12—C13—H13	118.6
O1—C1—C6	119.1 (2)	C14—C13—H13	118.6
C2—C1—C6	119.1 (2)	O2—C14—C13	119.3 (2)
C3—C2—C1	120.5 (3)	O2—C14—C9	124.3 (2)
C3—C2—H2	119.8	C13—C14—C9	116.4 (3)
C1—C2—H2	119.8	N2—C15—C16	122.3 (3)
C4—C3—C2	120.4 (3)	N2—C15—H15	118.8
C4—C3—H3	119.8	C16—C15—H15	118.8
C2—C3—H3	119.8	C17—C16—C15	119.1 (3)
C3—C4—C5	119.6 (3)	C17—C16—H16	120.5
C3—C4—H4	120.2	C15—C16—H16	120.5
C5—C4—H4	120.2	C16—C17—C18	119.6 (3)
C4—C5—C6	121.5 (3)	C16—C17—H17	120.2
C4—C5—H5	119.2	C18—C17—H17	120.2
C6—C5—H5	119.2	C17—C18—C19	118.8 (3)
C5—C6—C1	119.0 (3)	C17—C18—H18	120.6
C5—C6—C7	122.1 (3)	C19—C18—H18	120.6
C1—C6—C7	118.9 (2)	N2—C19—C18	122.3 (3)
N1—C7—C6	112.5 (2)	N2—C19—H19	118.8
N1—C7—H7A	109.1	C18—C19—H19	118.8

O2^{i}—Zn1—O1—C1	−14.5 (3)	C2—C1—C6—C7	−177.7 (2)
N1^{i}—Zn1—O1—C1	−116.1 (2)	C8—N1—C7—C6	−125.5 (3)
N2—Zn1—O1—C1	84.8 (3)	Zn1^{i}—N1—C7—C6	56.0 (3)
O1^{i}—Zn1—O1—C1	169.1 (3)	C5—C6—C7—N1	122.3 (3)
O2^{i}—Zn1—O1—Zn1^{i}	176.41 (8)	C1—C6—C7—N1	−60.5 (3)
N1^{i}—Zn1—O1—Zn1^{i}	74.75 (12)	C7—N1—C8—C9	−178.2 (2)
N2—Zn1—O1—Zn1^{i}	−84.36 (10)	Zn1^{i}—N1—C8—C9	0.3 (4)
O1^{i}—Zn1—O1—Zn1^{i}	0.0	N1—C8—C9—C10	−171.4 (3)

O1—Zn1—N2—C19	53.3 (2)	N1—C8—C9—C14	11.1 (4)
O2^{i}—Zn1—N2—C19	157.3 (2)	C14—C9—C10—C11	−0.8 (5)
N1^{i}—Zn1—N2—C19	−108.1 (2)	C8—C9—C10—C11	−178.5 (3)
O1^{i}—Zn1—N2—C19	−21.9 (2)	C9—C10—C11—C12	0.4 (5)
O1—Zn1—N2—C15	−128.6 (2)	C10—C11—C12—C13	0.0 (6)
O2^{i}—Zn1—N2—C15	−24.6 (2)	C11—C12—C13—C14	−0.1 (6)
N1^{i}—Zn1—N2—C15	70.0 (2)	Zn1^{i}—O2—C14—C13	162.6 (2)
O1^{i}—Zn1—N2—C15	156.2 (2)	Zn1^{i}—O2—C14—C9	−17.7 (4)
Zn1—O1—C1—C2	63.6 (4)	C12—C13—C14—O2	179.6 (3)
Zn1^{i}—O1—C1—C2	−128.6 (2)	C12—C13—C14—C9	−0.2 (5)
Zn1—O1—C1—C6	−118.3 (2)	C10—C9—C14—O2	−179.1 (3)
Zn1^{i}—O1—C1—C6	49.4 (3)	C8—C9—C14—O2	−1.6 (4)
O1—C1—C2—C3	177.4 (3)	C10—C9—C14—C13	0.6 (4)
C6—C1—C2—C3	−0.6 (4)	C8—C9—C14—C13	178.1 (3)
C1—C2—C3—C4	1.1 (5)	C19—N2—C15—C16	0.8 (4)
C2—C3—C4—C5	−0.5 (5)	Zn1—N2—C15—C16	−177.4 (2)
C3—C4—C5—C6	−0.6 (5)	N2—C15—C16—C17	0.1 (5)
C4—C5—C6—C1	1.0 (4)	C15—C16—C17—C18	−1.1 (5)
C4—C5—C6—C7	178.2 (3)	C16—C17—C18—C19	1.1 (5)
O1—C1—C6—C5	−178.5 (2)	C15—N2—C19—C18	−0.7 (4)
C2—C1—C6—C5	−0.4 (4)	Zn1—N2—C19—C18	177.5 (2)
O1—C1—C6—C7	4.2 (4)	C17—C18—C19—N2	−0.3 (5)

Symmetry code: (i) −*x*+1, −*y*+2, −*z*+1.