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Bis{μ-2-[2-(2-pyridyl)ethyliminomethyl]-phenolato}bis[azidozinc(II)]

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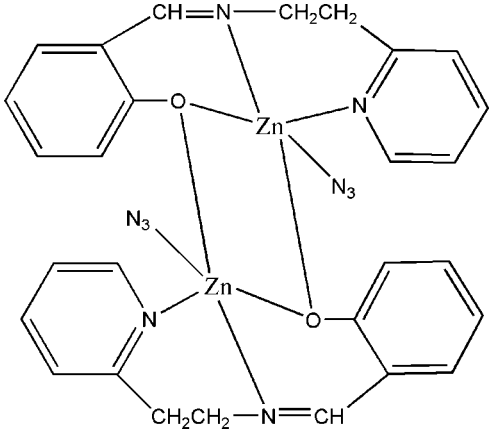
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.009 Å; R factor = 0.062; wR factor = 0.154; data-to-parameter ratio = 12.9.

In the centrosymmetric title dinuclear zinc(II) compound, $[Zn_2(C_{14}H_{13}N_2O)_2(N_3)_2]$, each Zn^{II} ion has a slightly distorted trigonal bipyramidal geometry and is coordinated by two N atoms and one O atom from one Schiff base ligand, an O atom from the other Schiff base ligand, and another N atom from an azide ligand. The crystal structure involves intermolecular C—H···N hydrogen bonds.

Related literature

For related literature, see: Tandon *et al.* (2000); Fu & Ye (2007); Li & Zhang (2004); You & Zhu (2004).



Experimental

Crystal data

$[Zn_2(C_{14}H_{13}N_2O)_2(N_3)_2]$
M_r = 665.37
Monoclinic, $P2_1/c$
a = 9.523 (9) Å
b = 9.466 (9) Å
c = 15.853 (14) Å
β = 100.664 (17)°
V = 1404 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.75 mm^{-1}
T = 293 (2) K
0.05 × 0.05 × 0.05 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.915, T_{max} = 0.915
11424 measured reflections
2460 independent reflections
1824 reflections with $I > 2\sigma(I)$
R_{int} = 0.117

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.154
S = 1.00
2460 reflections
190 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9A···N5^{i}	0.97	2.58	3.198 (9)	122

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

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; 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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2072).

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supporting information

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Bis{μ-2-[2-(2-pyridyl)ethyliminomethyl]phenolato}bis[azidozinc(II)]

Xiang-Rong Zhou, Zhong-Shu Li, Lei Zhang and Zhi-Hong Zhou

S1. Comment

Transition metal compounds containing Schiff base ligands are of great interest since many years. These compounds play an important role in the development of coordination chemistry related to their potential applications in catalysis and enzymatic reactions, magnetism and molecular architecture (You & Zhu, 2004; Li & Zhang, 2004).

We have focused on the synthesis of Schiff base complex which is formed by $Cu(CH_3COO)_2.H_2O$, schiff base ligand 2-(pyridin-2-ylethyliminomethyl)phenol and sodium azide. The title dinuclear zinc(II) complex is reported here.

As shown in Fig. 1, the molecule of the title compound is composed of two Zn^{II} atoms, two schiff base ligand 2-(pyridin-2-ylethyliminomethyl)phenol and two azido. Each Zn^{II} atom shows a slightly distorted trigonal-bipyramidal geometry (Fu & Ye, 2007) formed by two N atoms and one O atom from one schiff base ligand (S.S. Tandon *et al.*, 2000), the another O atom of the second schiff base, together with another N atom from azido. There are intermolecular C—H···N hydrogen bonds in the crystal structure leading to a one-dimensional supramolecular structure (Fig. 2 and Table 1).

S2. Experimental

The title compound was synthesized by $Cu(CH_3COO)_2.H_2O$, schiff base ligand 2-(pyridin-2-ylethyliminomethyl)phenol and sodium azide. All chemicals used (reagent grade) were commercially available. Salicylaldehyde (0.122 g, 1 mmol) was dissolved in ethanol (5 mL) and ethanol solution (5 mL) containing 2-aminoethylpyridine (0.108 g, 1 mmol) was added slowly with stirring. The resulting yellow solution was continuously stirred for about 30 min. at room temperature, and then $Cu(CH_3COO)_2.H_2O$ (0.200 g, 1 mmol) and sodium azide (0.13 g 2 mmol) in aqueous solution (5 mL) was added with stirring homogeneously. Colorless crystals suitable for X-ray analysis were obtained by slow evaporation at room temperature over several days.

S3. Refinement

Positional parameters of all H atoms were calculated geometrically.

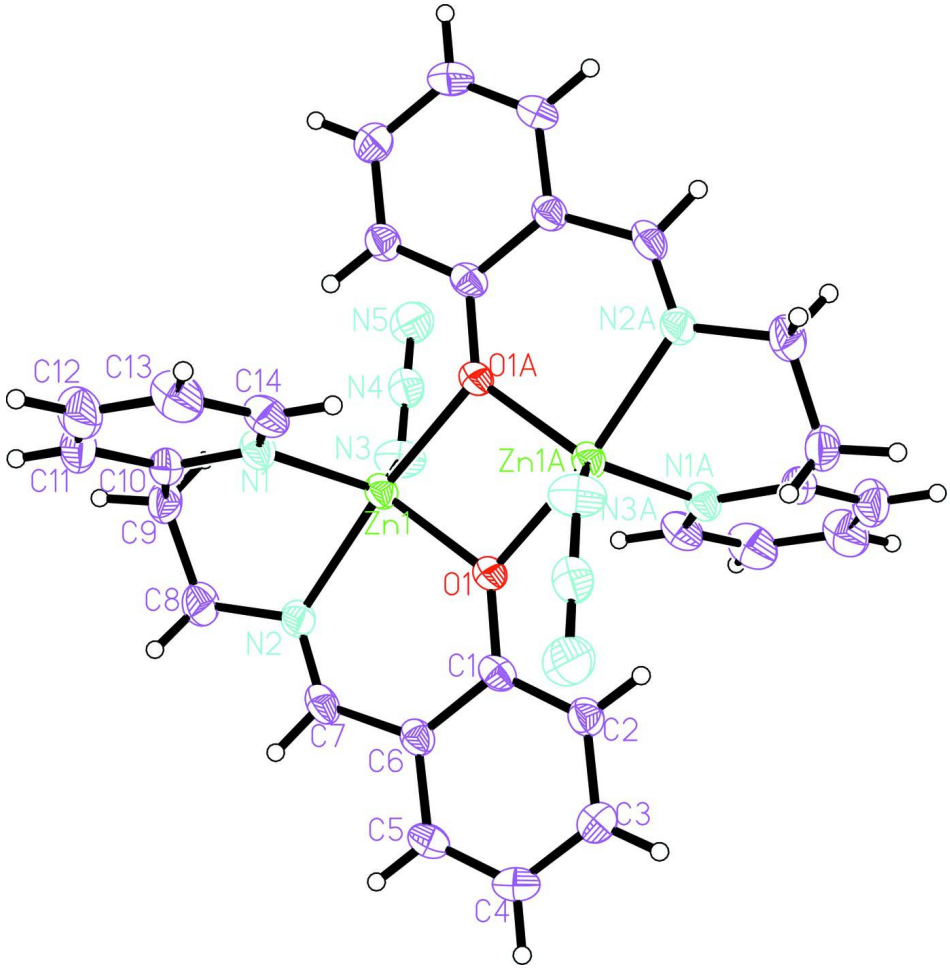

Figure 1

The molecular structure of the title compound with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. [Symmetry code i : -x, -y, -z]

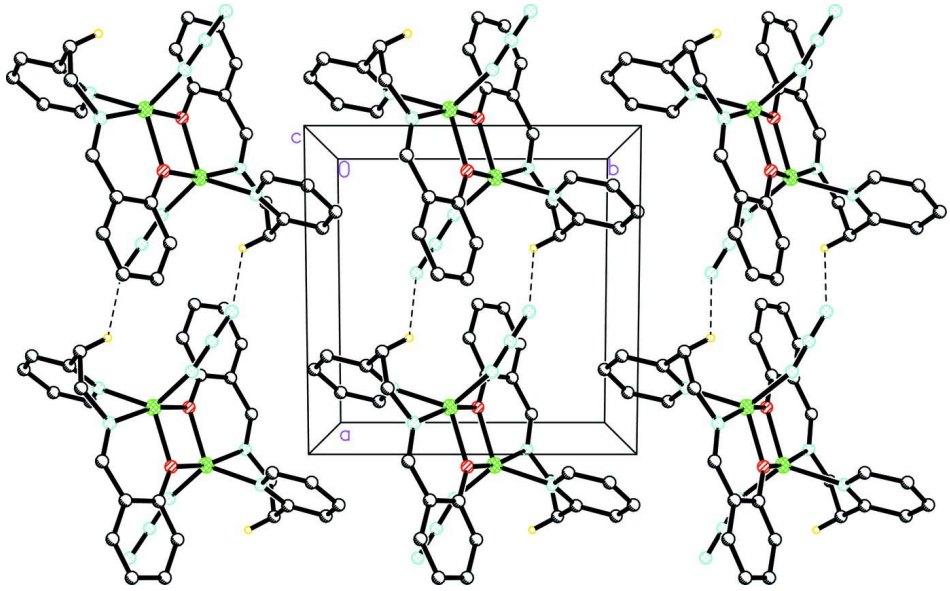

Figure 2

One-dimensional structure in the title compound. Hydrogen bonds are shown as dashed lines. Displacement ellipsoids are drawn at the 30% probability level and all hydrogen atoms, except H9a was omitted for clarity.

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$[Zn_2(C_{14}H_{13}N_2O)_2(N_3)_2]$
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Hall symbol: -P 2ybc
$a = 9.523$ (9) Å
$b = 9.466$ (9) Å
$c = 15.853$ (14) Å
$\beta = 100.664$ (17)°
$V = 1404$ (2) Å^3
$Z = 2$
$F(000) = 680$
$D_x = 1.575$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2415 reflections
$\theta = 3.1$–$27.5°$
$\mu = 1.75$ mm^{-1}
$T = 293$ K
Block, colorless
0.05 × 0.05 × 0.05 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.192 pixels mm^{-1}
thin–slice ω scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
$T_{min} = 0.915$, $T_{max} = 0.915$
11424 measured reflections
2460 independent reflections
1824 reflections with $I > 2\sigma(I)$
$R_{int} = 0.117$
$\theta_{max} = 25.0°$, $\theta_{min} = 3.1°$
$h = -11 \rightarrow 11$
$k = -11 \rightarrow 11$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.062$
$wR(F^2) = 0.154$
$S = 1.00$
2460 reflections
190 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map	$w = 1/[\sigma^2(F_o^2) + (0.0733P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
Hydrogen site location: inferred from neighbouring sites	$(\Delta/\sigma)_{max} < 0.001$
H-atom parameters constrained	$\Delta\rho_{max} = 0.32$ e Å^{-3}
	$\Delta\rho_{min} = -0.37$ 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}
Zn1	0.89278 (7)	0.07610 (6)	0.05279 (4)	0.0388 (3)
O1	1.0963 (4)	0.0175 (4)	0.0700 (2)	0.0443 (10)
N2	0.9352 (5)	0.1930 (5)	0.1691 (3)	0.0437 (12)
C2	1.3382 (6)	−0.0122 (6)	0.1341 (4)	0.0439 (14)
H2A	1.3492	−0.0743	0.0904	0.053*
N1	0.8313 (5)	0.2740 (5)	−0.0024 (3)	0.0437 (12)
C1	1.2057 (6)	0.0467 (5)	0.1339 (3)	0.0387 (13)
C7	1.0588 (7)	0.2101 (6)	0.2129 (4)	0.0460 (14)
H7A	1.0666	0.2750	0.2576	0.055*
C6	1.1919 (6)	0.1403 (6)	0.2022 (3)	0.0407 (14)
C5	1.3113 (7)	0.1695 (6)	0.2653 (4)	0.0513 (16)
H5A	1.3017	0.2302	0.3099	0.062*
N4	0.6662 (7)	−0.1265 (5)	0.0553 (3)	0.0491 (13)
C10	0.7506 (6)	0.3590 (6)	0.0385 (4)	0.0448 (14)
N3	0.7675 (7)	−0.0641 (6)	0.0914 (4)	0.0615 (15)
C8	0.8169 (7)	0.2774 (7)	0.1912 (4)	0.0578 (17)
H8A	0.8531	0.3685	0.2133	0.069*
H8B	0.7782	0.2296	0.2360	0.069*
N5	0.5662 (7)	−0.1902 (6)	0.0247 (4)	0.0684 (16)
C3	1.4564 (7)	0.0186 (7)	0.1979 (4)	0.0528 (16)
H3B	1.5443	−0.0231	0.1964	0.063*
C11	0.7189 (8)	0.4943 (7)	0.0084 (5)	0.0626 (19)
H11A	0.6620	0.5516	0.0358	0.075*
C14	0.8829 (7)	0.3237 (6)	−0.0705 (4)	0.0536 (16)
H14A	0.9394	0.2653	−0.0975	0.064*
C9	0.6990 (7)	0.2995 (7)	0.1140 (4)	0.0527 (16)
H9A	0.6526	0.2096	0.0980	0.063*
H9B	0.6280	0.3628	0.1298	0.063*
C4	1.4422 (8)	0.1111 (7)	0.2632 (4)	0.0618 (18)
H4A	1.5207	0.1335	0.3053	0.074*

C13	0.8535 (9)	0.4595 (7)	−0.1009 (5)	0.071 (2)
H13A	0.8901	0.4920	−0.1477	0.085*
C12	0.7695 (9)	0.5460 (7)	−0.0612 (5)	0.076 (2)
H12A	0.7476	0.6373	−0.0810	0.091*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.0443 (4)	0.0393 (4)	0.0344 (4)	0.0026 (3)	0.0112 (3)	−0.0035 (3)
O1	0.041 (2)	0.051 (2)	0.040 (2)	0.0056 (19)	0.0049 (19)	−0.0138 (18)
N2	0.052 (3)	0.047 (3)	0.033 (3)	0.009 (2)	0.010 (2)	−0.001 (2)
C2	0.044 (4)	0.045 (3)	0.045 (4)	−0.001 (3)	0.013 (3)	−0.007 (3)
N1	0.056 (3)	0.039 (3)	0.037 (3)	0.002 (2)	0.013 (2)	−0.001 (2)
C1	0.054 (4)	0.037 (3)	0.027 (3)	−0.004 (3)	0.011 (3)	0.002 (2)
C7	0.066 (4)	0.040 (3)	0.035 (3)	0.004 (3)	0.016 (3)	−0.006 (2)
C6	0.053 (4)	0.035 (3)	0.034 (3)	0.002 (3)	0.008 (3)	0.000 (2)
C5	0.059 (4)	0.056 (4)	0.036 (4)	−0.002 (3)	0.003 (3)	−0.012 (3)
N4	0.066 (4)	0.043 (3)	0.041 (3)	0.007 (3)	0.017 (3)	0.008 (2)
C10	0.049 (4)	0.037 (3)	0.046 (4)	0.006 (3)	0.004 (3)	−0.002 (3)
N3	0.067 (4)	0.072 (4)	0.045 (3)	−0.022 (3)	0.009 (3)	0.009 (3)
C8	0.060 (4)	0.067 (4)	0.051 (4)	0.013 (3)	0.022 (3)	−0.009 (3)
N5	0.066 (4)	0.060 (4)	0.076 (5)	−0.003 (3)	0.004 (3)	0.005 (3)
C3	0.044 (4)	0.056 (4)	0.057 (4)	0.003 (3)	0.007 (3)	−0.002 (3)
C11	0.077 (5)	0.047 (4)	0.065 (5)	0.017 (4)	0.016 (4)	0.003 (3)
C14	0.069 (4)	0.052 (4)	0.041 (4)	−0.008 (3)	0.014 (3)	−0.005 (3)
C9	0.053 (4)	0.053 (4)	0.055 (4)	0.013 (3)	0.017 (3)	−0.003 (3)
C4	0.054 (4)	0.072 (5)	0.052 (4)	−0.004 (4)	−0.009 (3)	−0.011 (3)
C13	0.107 (6)	0.048 (4)	0.058 (5)	−0.016 (4)	0.016 (4)	0.007 (3)
C12	0.115 (7)	0.038 (4)	0.070 (6)	0.003 (4)	0.005 (5)	0.005 (3)

Geometric parameters (Å, º)

Zn1—N3	1.957 (6)	N4—N5	1.155 (7)
Zn1—O1	1.986 (4)	N4—N3	1.185 (7)
Zn1—N1	2.104 (5)	C10—C11	1.380 (8)
Zn1—N2	2.125 (5)	C10—C9	1.487 (8)
Zn1—O1^{i}	2.158 (4)	C8—C9	1.515 (8)
O1—C1	1.342 (6)	C8—H8A	0.9700
O1—Zn1^{i}	2.158 (4)	C8—H8B	0.9700
N2—C7	1.262 (7)	C3—C4	1.381 (9)
N2—C8	1.475 (7)	C3—H3B	0.9300
C2—C1	1.378 (8)	C11—C12	1.373 (10)
C2—C3	1.398 (8)	C11—H11A	0.9300
C2—H2A	0.9300	C14—C13	1.383 (9)
N1—C14	1.350 (7)	C14—H14A	0.9300
N1—C10	1.358 (7)	C9—H9A	0.9700
C1—C6	1.423 (7)	C9—H9B	0.9700
C7—C6	1.467 (8)	C4—H4A	0.9300

C7—H7A	0.9300	C13—C12	1.375 (10)
C6—C5	1.396 (8)	C13—H13A	0.9300
C5—C4	1.369 (9)	C12—H12A	0.9300
C5—H5A	0.9300		

N3—Zn1—O1	113.8 (2)	N1—C10—C11	119.5 (6)
N3—Zn1—N1	126.5 (2)	N1—C10—C9	117.4 (5)
O1—Zn1—N1	119.68 (18)	C11—C10—C9	123.1 (6)
N3—Zn1—N2	96.3 (2)	N4—N3—Zn1	132.5 (5)
O1—Zn1—N2	90.16 (17)	N2—C8—C9	111.6 (5)
N1—Zn1—N2	83.74 (18)	N2—C8—H8A	109.3
N3—Zn1—O1^{i}	97.9 (2)	C9—C8—H8A	109.3
O1—Zn1—O1^{i}	78.48 (16)	N2—C8—H8B	109.3
N1—Zn1—O1^{i}	92.66 (17)	C9—C8—H8B	109.3
N2—Zn1—O1^{i}	164.47 (18)	H8A—C8—H8B	108.0
C1—O1—Zn1	130.4 (3)	C4—C3—C2	119.8 (6)
C1—O1—Zn1^{i}	127.3 (3)	C4—C3—H3B	120.1
Zn1—O1—Zn1^{i}	101.52 (16)	C2—C3—H3B	120.1
C7—N2—C8	118.4 (5)	C10—C11—C12	121.4 (6)
C7—N2—Zn1	123.5 (4)	C10—C11—H11A	119.3
C8—N2—Zn1	117.2 (4)	C12—C11—H11A	119.3
C1—C2—C3	122.1 (5)	N1—C14—C13	121.5 (6)
C1—C2—H2A	118.9	N1—C14—H14A	119.3
C3—C2—H2A	118.9	C13—C14—H14A	119.3
C14—N1—C10	119.8 (5)	C10—C9—C8	113.4 (5)
C14—N1—Zn1	121.9 (4)	C10—C9—H9A	108.9
C10—N1—Zn1	117.9 (4)	C8—C9—H9A	108.9
O1—C1—C2	120.0 (5)	C10—C9—H9B	108.9
O1—C1—C6	122.4 (5)	C8—C9—H9B	108.9
C2—C1—C6	117.6 (5)	H9A—C9—H9B	107.7
N2—C7—C6	128.1 (5)	C5—C4—C3	119.3 (6)
N2—C7—H7A	116.0	C5—C4—H4A	120.3
C6—C7—H7A	116.0	C3—C4—H4A	120.3
C5—C6—C1	119.4 (5)	C12—C13—C14	119.4 (7)
C5—C6—C7	115.7 (5)	C12—C13—H13A	120.3
C1—C6—C7	124.9 (5)	C14—C13—H13A	120.3
C4—C5—C6	121.8 (6)	C13—C12—C11	118.4 (6)
C4—C5—H5A	119.1	C13—C12—H12A	120.8
C6—C5—H5A	119.1	C11—C12—H12A	120.8
N5—N4—N3	175.9 (7)		

N3—Zn1—O1—C1	−96.2 (5)	O1—C1—C6—C5	−178.0 (5)
N1—Zn1—O1—C1	83.7 (5)	C2—C1—C6—C5	0.6 (8)
N2—Zn1—O1—C1	0.8 (4)	O1—C1—C6—C7	2.6 (8)
O1^{i}—Zn1—O1—C1	170.2 (5)	C2—C1—C6—C7	−178.8 (5)
N3—Zn1—O1—Zn1^{i}	93.6 (2)	N2—C7—C6—C5	−174.0 (6)
N1—Zn1—O1—Zn1^{i}	−86.5 (2)	N2—C7—C6—C1	5.4 (10)
N2—Zn1—O1—Zn1^{i}	−169.34 (19)	C1—C6—C5—C4	0.4 (9)

O1^{i}—Zn1—O1—Zn1^{i}	0.0	C7—C6—C5—C4	179.8 (6)
N3—Zn1—N2—C7	120.0 (5)	C14—N1—C10—C11	1.8 (9)
O1—Zn1—N2—C7	6.0 (5)	Zn1—N1—C10—C11	174.3 (5)
N1—Zn1—N2—C7	−113.8 (5)	C14—N1—C10—C9	−178.9 (5)
O1^{i}—Zn1—N2—C7	−36.6 (9)	Zn1—N1—C10—C9	−6.4 (7)
N3—Zn1—N2—C8	−70.6 (4)	O1—Zn1—N3—N4	−120.4 (7)
O1—Zn1—N2—C8	175.4 (4)	N1—Zn1—N3—N4	59.7 (8)
N1—Zn1—N2—C8	55.6 (4)	N2—Zn1—N3—N4	146.6 (7)
O1^{i}—Zn1—N2—C8	132.9 (6)	O1^{i}—Zn1—N3—N4	−39.5 (7)
N3—Zn1—N1—C14	−137.7 (5)	C7—N2—C8—C9	153.0 (6)
O1—Zn1—N1—C14	42.4 (5)	Zn1—N2—C8—C9	−16.9 (7)
N2—Zn1—N1—C14	129.0 (5)	C1—C2—C3—C4	−0.3 (10)
O1^{i}—Zn1—N1—C14	−35.9 (5)	N1—C10—C11—C12	−1.3 (11)
N3—Zn1—N1—C10	49.9 (5)	C9—C10—C11—C12	179.5 (7)
O1—Zn1—N1—C10	−129.9 (4)	C10—N1—C14—C13	−1.1 (9)
N2—Zn1—N1—C10	−43.3 (4)	Zn1—N1—C14—C13	−173.3 (5)
O1^{i}—Zn1—N1—C10	151.8 (4)	N1—C10—C9—C8	70.0 (7)
Zn1—O1—C1—C2	176.8 (4)	C11—C10—C9—C8	−110.8 (7)
Zn1^{i}—O1—C1—C2	−15.3 (7)	N2—C8—C9—C10	−53.1 (7)
Zn1—O1—C1—C6	−4.6 (7)	C6—C5—C4—C3	−1.3 (11)
Zn1^{i}—O1—C1—C6	163.2 (4)	C2—C3—C4—C5	1.2 (10)
C3—C2—C1—O1	178.0 (5)	N1—C14—C13—C12	−0.2 (11)
C3—C2—C1—C6	−0.6 (8)	C14—C13—C12—C11	0.7 (12)
C8—N2—C7—C6	−179.0 (5)	C10—C11—C12—C13	0.0 (12)
Zn1—N2—C7—C6	−9.7 (9)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9*A*···N5^{ii}	0.97	2.58	3.198 (9)	122

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