Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

[μ-10,21-Dimethyl-3,6,14,17-tetraazatricyclo[17.3.1^{8,12}]tetracosa-1(23),8,10,12(24),19,21-hexaene-23,24-diolato-$\kappa^4N^3,N^6,O^{23},O^{24}$:$\kappa^4N^{14},N^{17},O^{23},O^{24}$]-bis(nitrato-$\kappa O$)zinc(II)

Jie Liu, Jian-Fang Ma,* Shun-Li Li and Guang-Ju Ping

Department of Chemistry, Northeast Normal University, Changchun 130024, People's Republic of China
Correspondence e-mail: majf247nenu@yahoo.com.cn

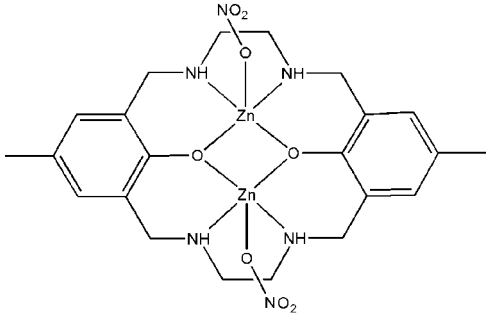
Received 13 June 2007; accepted 14 June 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.028; wR factor = 0.080; data-to-parameter ratio = 17.0.

The title compound, $[Zn_2(NO_3)_2(C_{22}H_{30}N_4O_2)]$, is a centrosymmetric dinuclear zinc compound. Each Zn atom has a square-pyramidal geometry with N_2O_3 donors, being coordinated by two N atoms and two O atoms from the macrocyclic 10,21-dimethyl-3,6,14,17-tetraazatricyclo[17.3.1^{8,12}]tetracosa-1(23),8,10,12(24),19,21-hexaene-23,24-diolate ligand and one O atom from a nitrate anion. In the structure, two Zn atoms are linked by two phenolate O atoms to give a four-membered Zn_2O_2 ring.

Related literature

For related literature, see: Dealwis *et al.* (1995); Burley *et al.* (1990); Roderick & Mathews (1993); Dutta *et al.* (2005); Mandal & Nag (1986); Bazzicalupi *et al.* (1997).



Experimental

Crystal data

$[Zn_2(NO_3)_2(C_{22}H_{30}N_4O_2)]$
M_r = 637.26
Monoclinic, $P2_1/c$
a = 8.2190 (5) Å
b = 14.1450 (8) Å
c = 11.0550 (6) Å
β = 103.744 (9)°
V = 1248.43 (12) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.98 mm^{-1}
T = 293 (2) K
0.40 × 0.30 × 0.25 mm

Data collection

Bruker APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.495, T_{max} = 0.609
7558 measured reflections
3027 independent reflections
2524 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.080
S = 1.07
3027 reflections
178 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Zn1—O1^{i}	2.0057 (13)	Zn1—N2	2.1051 (19)
Zn1—O1	2.0068 (14)	Zn1—N1	2.1216 (16)
Zn1—O2	2.0079 (18)		
O1^{i}—Zn1—O1	74.53 (6)	O2—Zn1—N2	114.32 (8)
O1^{i}—Zn1—O2	104.21 (8)	O1^{i}—Zn1—N1	143.63 (7)
O1—Zn1—O2	100.80 (8)	O1—Zn1—N1	88.27 (6)
O1^{i}—Zn1—N2	89.50 (6)	O2—Zn1—N1	110.44 (8)
O1—Zn1—N2	144.15 (7)	N2—Zn1—N1	86.19 (7)

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990); software used to prepare material for publication: *SHELXL97*.

The authors thank the National Natural Science Foundation of China (No. 20471014), Program for New Century Excellent Talents in Chinese Universities (NCET-05-0320), the Fok Ying Tung Education Foundation and the Analysis and Testing Foundation of Northeast Normal University for support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WK2063).

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  doi:10.1107/S1600536807029285

supplementary materials

Acta Cryst. (2007). E**63**, m1954 [**doi:10.1107/S1600536807029285**]

[μ-10,21-Dimethyl-3,6,14,17-tetraazatricyclo[17.3.1^{8,12}]tetracosa-1(23),8,10,12(24),19,21-hexaene-23,24-diolato-$\kappa^4N^3,N^6,O^{23},O^{24}$:$\kappa^4N^{14},N^{17},O^{23},O^{24}$]bis(nitrato-$\kappa O$)zinc(II)

J. Liu, J.-F. Ma, S.-L. Li and G.-J. Ping

Comment

Dinuclear zinc(II) cores have attracted much interest as a result of their significance in biological systems (Dealwis *et al.*, 1995; Burley *et al.*, 1990; Roderick & Mathews, 1993). In addition, some synthetic dinuclear zinc(II) compounds are found to have functions in dephosphorylation (Bazzicalupi *et al.*, 1997). To further widen the scope of application of such zinc compounds, there is a need to prepare new series of dinuclear zinc compounds. In this work, a new dinuclear zinc(II) compound has been synthesized, and its structure (I) is reported here.

As shown in Fig. 1, $[Zn_2L(NO_3)_2]$ is a centrosymmetric dinuclear zinc compound. The coordination environment around zinc is a square-pyramid with two N atoms and two O atoms from *L* ligand occupying the basal positions and one O atom from NO_3^- anion occupying the apical position. In the crystal structure two zinc atoms are bridged by two phenolate O atoms to generate a four-membered Zn_2O_2 ring. The Zn—O and Zn—N distances are normal (Dutta *et al.*, 2005).

Experimental

The ligand $C_{22}H_{32}N_4O_2$ (H_2L) was prepared by the reported procedure (Mandal & Nag, 1986). A mixture of H_2L (0.10 g, 0.26 mmol) and $Zn(NO_3)_2.6H_2O$ (0.15 g, 0.52 mmol) in methanol (20 ml) was stirred for 10 min. The resulting solution was filtered. Colorless single crystals were obtained by slow evaporation of the filtrate at room temperature (yield 56%).

Refinement

All H-atoms bound to carbon were refined using a riding model with d(C—H) = 0.93 Å, $U_{iso} = 1.2U_{eq}$ (C) for aromatic and 0.96 Å, $U_{iso} = 1.5U_{eq}$ (C) for CH_3 atoms. The imino H atoms were located in a difference Fourier map and refined isotropically with U_{iso}(H) = 1.5 U_{eq}(N).

Figures

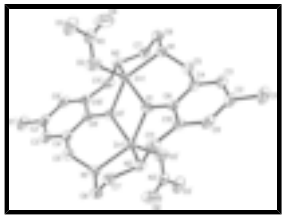


Fig. 1. A view of the molecule of (I). Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity.

[μ-10,21-Dimethyl-3,6,14,17-tetraazatricyclo[17.3.1^{8,12}]tetracosa- 1(23),8,10,12 (24),19,21-hexaene-23,24-diolato- κ^4*N*^{3},*N*^{6},*O*^{23},*O*^{24}:κ^4*N*^{14},*N*^{17},*O*^{23},*O*^{24}]bis(nitrato-κ*O*)zinc(II)

Crystal data

$[Zn_2(NO_3)_2(C_{22}H_{30}N_4O_2)]$	F_{000} = 656
M_r = 637.26	D_x = 1.695 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71069 Å
Hall symbol: -P 2ybc	Cell parameters from 3000 reflections
a = 8.2190 (5) Å	θ = 2.4–28.4º
b = 14.1450 (8) Å	μ = 1.98 mm^{-1}
c = 11.0550 (6) Å	T = 293 (2) K
β = 103.744 (9)º	Block, colorless
V = 1248.43 (12) Å^3	0.40 × 0.30 × 0.25 mm
Z = 2	

Data collection

Bruker APEX CCD area-detector diffractometer	3027 independent reflections
Radiation source: fine-focus sealed tube	2524 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.019
T = 293(2) K	θ_{max} = 28.4º
ω scans	θ_{min} = 2.4º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −10→10
T_{min} = 0.495, T_{max} = 0.609	k = −10→18
7558 measured reflections	l = −14→14

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.028	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.080	$w = 1/[\sigma^2(F_o^2) + (0.0446P)^2 + 0.1778P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.07	$(\Delta/\sigma)_{max}$ = 0.001
3027 reflections	$\Delta\rho_{max}$ = 0.40 e Å^{-3}
178 parameters	$\Delta\rho_{min}$ = −0.29 e Å^{-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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Zn1	0.52604 (3)	0.398830 (14)	0.56583 (2)	0.03572 (9)
C1	1.0529 (3)	0.3634 (2)	0.1572 (3)	0.0680 (7)
H1A	1.1433	0.3280	0.2081	0.102*
H1B	1.0967	0.4190	0.1262	0.102*
H1C	0.9960	0.3251	0.0885	0.102*
C2	0.9306 (3)	0.39238 (14)	0.2345 (3)	0.0509 (6)
C3	0.9619 (3)	0.37071 (14)	0.3605 (2)	0.0478 (5)
H3	1.0562	0.3352	0.3964	0.057*
C4	0.8564 (3)	0.40063 (12)	0.4347 (2)	0.0419 (5)
C5	0.9026 (3)	0.38988 (14)	0.5739 (2)	0.0463 (5)
H5A	1.0041	0.3525	0.5969	0.056*
H5B	0.9277	0.4520	0.6108	0.056*
C6	0.8087 (3)	0.36047 (16)	0.7661 (2)	0.0487 (5)
H6A	0.8335	0.4267	0.7845	0.058*
H6B	0.9060	0.3237	0.8069	0.058*
C7	0.6586 (3)	0.33110 (16)	0.8152 (2)	0.0495 (5)
H7A	0.6401	0.2637	0.8027	0.059*
H7B	0.6816	0.3436	0.9039	0.059*
C8	0.7136 (2)	0.45295 (13)	0.3782 (2)	0.0402 (4)
C9	0.7880 (3)	0.44471 (16)	0.1820 (2)	0.0493 (5)
H9	0.7665	0.4604	0.0980	0.059*
C10	0.6769 (3)	0.47418 (14)	0.2512 (2)	0.0437 (5)
C11	0.5148 (3)	0.52395 (15)	0.1905 (2)	0.0483 (5)
H11A	0.5111	0.5339	0.1031	0.058*
H11B	0.4214	0.4835	0.1956	0.058*
N1	0.7725 (2)	0.34492 (11)	0.62923 (17)	0.0400 (4)
N2	0.5050 (2)	0.38322 (13)	0.75088 (18)	0.0435 (4)
N3	0.3579 (2)	0.22836 (14)	0.4825 (2)	0.0524 (5)
O1	0.61249 (19)	0.48312 (10)	0.44910 (15)	0.0504 (4)
O2	0.3842 (3)	0.30868 (13)	0.44489 (18)	0.0728 (5)
O3	0.2602 (3)	0.17463 (14)	0.4146 (2)	0.0801 (6)
O4	0.4378 (4)	0.20400 (16)	0.5846 (2)	0.1122 (9)
H1N	0.773 (7)	0.290 (3)	0.614 (5)	0.168*

H2N	0.414 (7)	0.353 (4)	0.749 (5)	0.168*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.03569 (14)	0.03053 (13)	0.04245 (15)	0.00473 (8)	0.01229 (10)	0.00441 (8)
C1	0.0588 (15)	0.0631 (15)	0.095 (2)	−0.0046 (12)	0.0434 (15)	−0.0265 (15)
C2	0.0465 (12)	0.0400 (11)	0.0748 (16)	−0.0088 (9)	0.0316 (11)	−0.0194 (10)
C3	0.0346 (10)	0.0321 (9)	0.0811 (16)	−0.0013 (8)	0.0227 (10)	−0.0089 (10)
C4	0.0371 (10)	0.0275 (9)	0.0650 (13)	−0.0025 (7)	0.0199 (9)	−0.0024 (8)
C5	0.0352 (10)	0.0368 (10)	0.0672 (14)	−0.0018 (8)	0.0129 (10)	0.0041 (9)
C6	0.0458 (11)	0.0436 (11)	0.0527 (12)	0.0066 (9)	0.0035 (9)	0.0043 (9)
C7	0.0573 (13)	0.0453 (11)	0.0444 (11)	0.0103 (9)	0.0092 (10)	0.0099 (9)
C8	0.0410 (10)	0.0279 (9)	0.0583 (12)	0.0013 (7)	0.0247 (9)	−0.0004 (8)
C9	0.0513 (12)	0.0462 (12)	0.0554 (13)	−0.0061 (9)	0.0226 (10)	−0.0136 (9)
C10	0.0442 (11)	0.0341 (10)	0.0565 (12)	−0.0024 (8)	0.0193 (9)	−0.0071 (8)
C11	0.0479 (12)	0.0478 (12)	0.0492 (12)	0.0017 (9)	0.0117 (9)	−0.0077 (9)
N1	0.0371 (8)	0.0299 (8)	0.0531 (10)	0.0014 (6)	0.0107 (7)	0.0034 (7)
N2	0.0460 (10)	0.0402 (9)	0.0450 (9)	0.0038 (7)	0.0124 (8)	0.0023 (7)
N3	0.0438 (10)	0.0504 (11)	0.0658 (13)	−0.0012 (8)	0.0185 (9)	−0.0124 (9)
O1	0.0557 (9)	0.0413 (8)	0.0654 (10)	0.0207 (6)	0.0365 (8)	0.0171 (7)
O2	0.0836 (13)	0.0563 (11)	0.0666 (11)	−0.0142 (9)	−0.0057 (10)	0.0032 (8)
O3	0.0671 (12)	0.0692 (12)	0.1050 (16)	−0.0187 (9)	0.0225 (11)	−0.0374 (11)
O4	0.144 (2)	0.0728 (14)	0.0964 (17)	−0.0331 (15)	−0.0171 (17)	0.0208 (13)

Geometric parameters (Å, °)

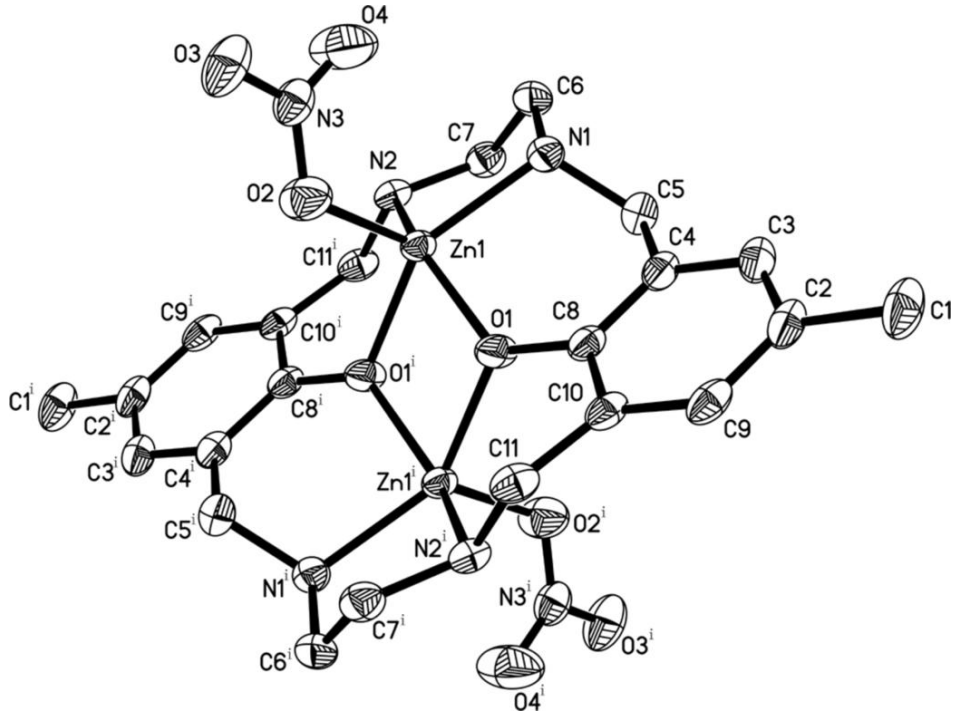
Zn1—O1^{i}	2.0057 (13)	C6—H6A	0.9700
Zn1—O1	2.0068 (14)	C6—H6B	0.9700
Zn1—O2	2.0079 (18)	C7—N2	1.489 (3)
Zn1—N2	2.1051 (19)	C7—H7A	0.9700
Zn1—N1	2.1216 (16)	C7—H7B	0.9700
C1—C2	1.523 (3)	C8—O1	1.340 (2)
C1—H1A	0.9600	C8—C10	1.397 (3)
C1—H1B	0.9600	C9—C10	1.388 (3)
C1—H1C	0.9600	C9—H9	0.9300
C2—C3	1.389 (4)	C10—C11	1.515 (3)
C2—C9	1.391 (3)	C11—N2^{i}	1.490 (3)
C3—C4	1.394 (3)	C11—H11A	0.9700
C3—H3	0.9300	C11—H11B	0.9700
C4—C8	1.402 (3)	N1—H1N	0.80 (5)
C4—C5	1.503 (3)	N2—C11^{i}	1.490 (3)
C5—N1	1.494 (3)	N2—H2N	0.86 (5)
C5—H5A	0.9700	N3—O4	1.213 (3)
C5—H5B	0.9700	N3—O3	1.224 (3)
C6—N1	1.488 (3)	N3—O2	1.246 (3)
C6—C7	1.519 (3)	O1—Zn1^{i}	2.0057 (13)
O1^{i}—Zn1—O1	74.53 (6)	C6—C7—H7A	109.4

O1^{i}—Zn1—O2	104.21 (8)	N2—C7—H7B	109.4
O1—Zn1—O2	100.80 (8)	C6—C7—H7B	109.4
O1^{i}—Zn1—N2	89.50 (6)	H7A—C7—H7B	108.0
O1—Zn1—N2	144.15 (7)	O1—C8—C10	120.25 (18)
O2—Zn1—N2	114.32 (8)	O1—C8—C4	118.52 (19)
O1^{i}—Zn1—N1	143.63 (7)	C10—C8—C4	121.23 (18)
O1—Zn1—N1	88.27 (6)	C10—C9—C2	121.9 (2)
O2—Zn1—N1	110.44 (8)	C10—C9—H9	119.0
N2—Zn1—N1	86.19 (7)	C2—C9—H9	119.0
C2—C1—H1A	109.5	C9—C10—C8	118.5 (2)
C2—C1—H1B	109.5	C9—C10—C11	121.3 (2)
H1A—C1—H1B	109.5	C8—C10—C11	120.12 (18)
C2—C1—H1C	109.5	N2^{i}—C11—C10	113.11 (18)
H1A—C1—H1C	109.5	N2^{i}—C11—H11A	109.0
H1B—C1—H1C	109.5	C10—C11—H11A	109.0
C3—C2—C9	118.26 (19)	N2^{i}—C11—H11B	109.0
C3—C2—C1	120.9 (2)	C10—C11—H11B	109.0
C9—C2—C1	120.8 (2)	H11A—C11—H11B	107.8
C2—C3—C4	122.0 (2)	C6—N1—C5	111.31 (17)
C2—C3—H3	119.0	C6—N1—Zn1	103.15 (12)
C4—C3—H3	119.0	C5—N1—Zn1	115.58 (12)
C3—C4—C8	118.1 (2)	C6—N1—H1N	111 (4)
C3—C4—C5	122.1 (2)	C5—N1—H1N	107 (4)
C8—C4—C5	119.36 (18)	Zn1—N1—H1N	110 (4)
N1—C5—C4	115.52 (18)	C7—N2—C11^{i}	113.39 (18)
N1—C5—H5A	108.4	C7—N2—Zn1	105.06 (13)
C4—C5—H5A	108.4	C11^{i}—N2—Zn1	111.88 (14)
N1—C5—H5B	108.4	C7—N2—H2N	113 (4)
C4—C5—H5B	108.4	C11^{i}—N2—H2N	105 (4)
H5A—C5—H5B	107.5	Zn1—N2—H2N	108 (3)
N1—C6—C7	109.84 (18)	O4—N3—O3	121.7 (2)
N1—C6—H6A	109.7	O4—N3—O2	118.1 (2)
C7—C6—H6A	109.7	O3—N3—O2	120.1 (2)
N1—C6—H6B	109.7	C8—O1—Zn1^{i}	129.47 (12)
C7—C6—H6B	109.7	C8—O1—Zn1	123.38 (12)
H6A—C6—H6B	108.2	Zn1^{i}—O1—Zn1	105.47 (6)
N2—C7—C6	111.27 (17)	N3—O2—Zn1	118.45 (15)
N2—C7—H7A	109.4		
C9—C2—C3—C4	−0.7 (3)	O2—Zn1—N1—C5	104.10 (15)
C1—C2—C3—C4	176.62 (19)	N2—Zn1—N1—C5	−141.34 (15)
C2—C3—C4—C8	0.9 (3)	C6—C7—N2—C11^{i}	−86.8 (2)
C2—C3—C4—C5	−170.78 (18)	C6—C7—N2—Zn1	35.7 (2)
C3—C4—C5—N1	−129.7 (2)	O1^{i}—Zn1—N2—C7	−152.22 (14)
C8—C4—C5—N1	58.7 (2)	O1—Zn1—N2—C7	−90.10 (16)
N1—C6—C7—N2	−57.0 (2)	O2—Zn1—N2—C7	102.38 (15)
C3—C4—C8—O1	−179.12 (17)	N1—Zn1—N2—C7	−8.36 (14)

C5—C4—C8—O1	−7.2 (3)	O1^{i}—Zn1—N2—C11^{i}	−28.78 (14)
C3—C4—C8—C10	0.4 (3)	O1—Zn1—N2—C11^{i}	33.3 (2)
C5—C4—C8—C10	172.34 (17)	O2—Zn1—N2—C11^{i}	−134.18 (14)
C3—C2—C9—C10	−0.9 (3)	N1—Zn1—N2—C11^{i}	115.08 (15)
C1—C2—C9—C10	−178.2 (2)	C10—C8—O1—Zn1^{i}	−32.2 (3)
C2—C9—C10—C8	2.1 (3)	C4—C8—O1—Zn1^{i}	147.37 (15)
C2—C9—C10—C11	−174.79 (19)	C10—C8—O1—Zn1	130.89 (16)
O1—C8—C10—C9	177.62 (18)	C4—C8—O1—Zn1	−49.6 (2)
C4—C8—C10—C9	−1.9 (3)	O1^{i}—Zn1—O1—C8	−166.5 (2)
O1—C8—C10—C11	−5.4 (3)	O2—Zn1—O1—C8	−64.57 (18)
C4—C8—C10—C11	175.08 (18)	N2—Zn1—O1—C8	126.99 (17)
C9—C10—C11—N2^{i}	−125.4 (2)	N1—Zn1—O1—C8	45.92 (17)
C8—C10—C11—N2^{i}	57.7 (3)	O1^{i}—Zn1—O1—Zn1^{i}	0.0
C7—C6—N1—C5	169.00 (16)	O2—Zn1—O1—Zn1^{i}	101.92 (9)
C7—C6—N1—Zn1	44.46 (18)	N2—Zn1—O1—Zn1^{i}	−66.52 (14)
C4—C5—N1—C6	−164.74 (17)	N1—Zn1—O1—Zn1^{i}	−147.59 (8)
C4—C5—N1—Zn1	−47.5 (2)	O4—N3—O2—Zn1	−10.1 (3)
O1^{i}—Zn1—N1—C6	64.33 (16)	O3—N3—O2—Zn1	173.52 (17)
O1—Zn1—N1—C6	124.92 (13)	O1^{i}—Zn1—O2—N3	−128.41 (18)
O2—Zn1—N1—C6	−134.20 (13)	O1—Zn1—O2—N3	155.00 (18)
N2—Zn1—N1—C6	−19.64 (13)	N2—Zn1—O2—N3	−32.4 (2)
O1^{i}—Zn1—N1—C5	−57.37 (19)	N1—Zn1—O2—N3	62.8 (2)
O1—Zn1—N1—C5	3.22 (14)		

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

Fig. 1


O4
O3
N3
C6
C7
N1
N2
O2
C5
C4
C3
C11i
Zn1
C2
C1
C9i
C10i
O1
C8
O1i
C1i
C8i
C10
C2i
C9
C3i
C4i
C11
Zn1i
C5i
O2i
N2i
N1i
C7i
N3i
C6i
O3i
O4i