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# Bis[ $\mu$ -1,3-bis(1*H*-imidazol-1-yl)propane- $\kappa^2N^3:N^{3'}$ ]bis(dichloridozinc) dihydrate

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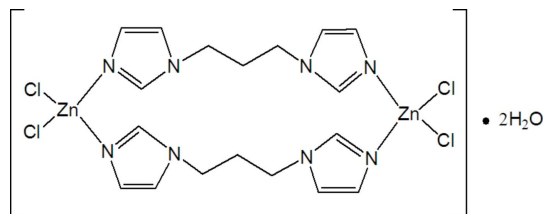
Received 24 March 2014; accepted 11 April 2014

Key indicators: single-crystal X-ray study;  $T = 296$  K; mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å;  $R$  factor = 0.030;  $wR$  factor = 0.087; data-to-parameter ratio = 20.5.

The title hydrated complex,  $[\text{Zn}_2\text{Cl}_4(\text{C}_9\text{H}_{12}\text{N}_4)_2] \cdot 2\text{H}_2\text{O}$ , is a discrete dinuclear zinc complex with 1,3-bis(1*H*-imidazol-1-yl)propane as the bridging ligand. The complex molecule lies about a crystallographic inversion centre. The  $\text{Zn}^{\text{II}}$  atom exhibits a distorted tetrahedral coordination geometry defined by two imidazole N atoms and two Cl atoms. O—H...Cl hydrogen bonding between the lattice water molecules and the terminal Cl atoms of the molecule lead to a two-dimensional structure extending parallel to (100).

## Related literature

For related structures containing the 1,3-bis(imidazol)propane ligand, see: Ma *et al.* (2012); Kan *et al.* (2012); Jiang *et al.* (2011); Shen & Lin (2012).



## Experimental

### Crystal data

 $[\text{Zn}_2\text{Cl}_4(\text{C}_9\text{H}_{12}\text{N}_4)_2] \cdot 2\text{H}_2\text{O}$  $M_r = 661.02$ 

Monoclinic,  $P2_1/c$   
 $a = 10.1378$  (4) Å  
 $b = 9.7173$  (4) Å  
 $c = 13.8801$  (6) Å  
 $\beta = 93.704$  (2)°  
 $V = 1364.50$  (10) Å<sup>3</sup>

$Z = 2$   
 Mo  $K\alpha$  radiation  
 $\mu = 2.18$  mm<sup>-1</sup>  
 $T = 296$  K  
 $0.25 \times 0.18 \times 0.12$  mm

### Data collection

Bruker APEXII CCD diffractometer  
 Absorption correction: multi-scan (SADABS; Bruker, 2006)  
 $T_{\text{min}} = 0.631$ ,  $T_{\text{max}} = 0.770$

21184 measured reflections  
 3162 independent reflections  
 2473 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.033$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.030$   
 $wR(F^2) = 0.087$   
 $S = 1.04$   
 3162 reflections

154 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.55$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.23$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1W—H1WA...Cl1	0.85	2.47	3.282 (3)	160
O1W—H1WB...Cl2 <sup>i</sup>	0.85	2.76	3.473 (4)	143

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

Data collection: APEX2 (Bruker, 2006); cell refinement: SAINT (Bruker, 2006); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: DIAMOND (Brandenburg, 1999); software used to prepare material for publication: publCIF (Westrip, 2010).

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Supporting information for this paper is available from the IUCr electronic archives (Reference: WM5017).

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## supporting information

*Acta Cryst.* (2014). E70, m184 [doi:10.1107/S1600536814008162]

**Bis[ $\mu$ -1,3-bis(1*H*-imidazol-1-yl)propane- $\kappa^2$ N<sup>3</sup>:N<sup>3'</sup>]bis(dichloridozinc) dihydrate****Xiao-Juan Wang and Yun-Long Feng****S1. Comment**

In the past few years, complexes based on the 1,3-bis(imidazol)propane (1,3-bip) ligand have been reported, such as [Mn<sub>4</sub>(tbip)<sub>4</sub>(1,3-bip)]<sub>n</sub>·2nH<sub>2</sub>O (H<sub>2</sub>tbip = (5-*tert*-butyl isophthalic acid) (Ma *et al.*, 2012), [Cd(HL)(1,3-bip)]<sub>n</sub>·5nH<sub>2</sub>O (H<sub>3</sub>L = 5-(2-carboxybenzyloxy)isophthalic acid) (Kan *et al.*, 2012), [Zn(L)(1,3-bip)]<sub>n</sub> (H<sub>2</sub>L = 5-methylisophthalic acid) (Jiang *et al.*, 2011), [Cd(1,3-bip)Cl<sub>2</sub>]<sub>n</sub> (Shen *et al.*, 2012). In order to extend our knowledge in this field, we report here the syntheses and structure of a new complex, [ZnCl<sub>2</sub>(C<sub>9</sub>H<sub>12</sub>N<sub>4</sub>)<sub>2</sub>·2H<sub>2</sub>O], (I).

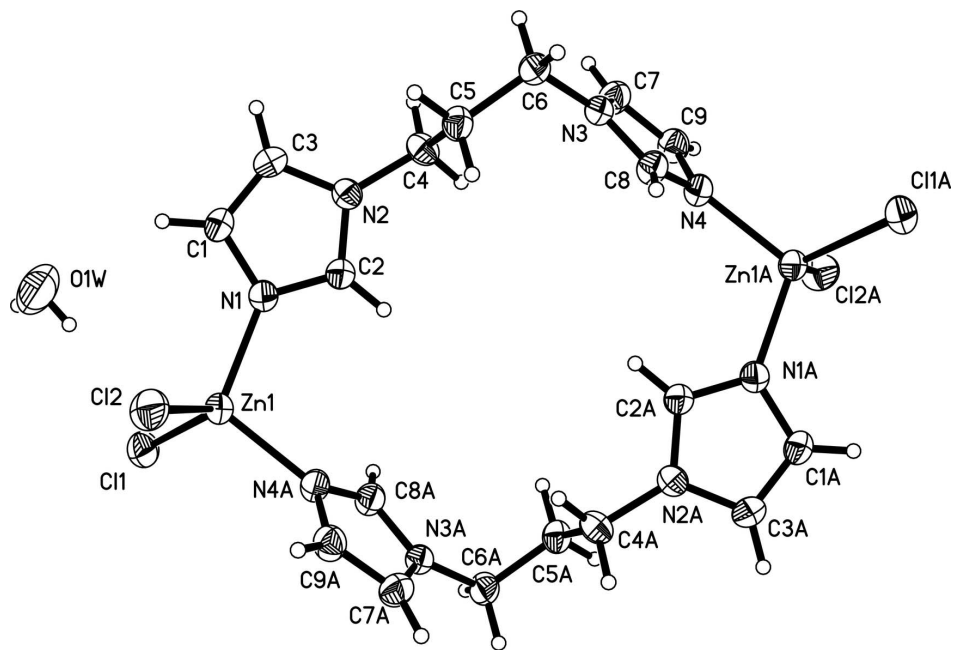
The asymmetric unit of (I) consists of one Zn<sup>2+</sup> ion, one 1,3-bip ligand, two Cl<sup>-</sup> ions, and one lattice water molecules. A perspective view of the molecular entities of complex (I) is presented in Fig. 1. The complex contains centrosymmetric dimers with bridging 1,3-bip ligands. The Zn(II) atom is four-coordinated in a distorted tetrahedral coordination. O—H···Cl hydrogen bonds between the lattice water molecules and Cl atoms lead to a layered structure extending parallel to (100) (Fig. 2).

**S2. Experimental**

A mixture of 1,3-bis(imidazol)propane (0.088 g, 0.5 mmol), ZnCl<sub>2</sub> (0.204 g, 1.5 mmol), and Na<sub>2</sub>CO<sub>3</sub> (0.060 g, 0.5 mmol) in H<sub>2</sub>O (16 ml)/C<sub>2</sub>H<sub>5</sub>OH (2 ml) was placed in a 25 ml Teflon-lined stainless steel vessel and heated at 433 K for 72 h, then cooled to room temperature over a period of 24 h. Colourless crystals suitable for X-ray analysis were obtained.

**S3. Refinement**

The carbon-bound H-atoms were positioned geometrically and included in the refinement using a riding model [aromatic C—H 0.93 Å and aliphatic C—H 0.97 Å,  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ ]. The oxygen-bound H-atoms were located in a difference Fourier map and were refined with the O—H distance restraint of 0.85 Å [ $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{O})$ ].

**Figure 1**

Perspective view of the molecular entities of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. [Symmetry code: (A)  $-x + 1, -y + 1, -z + 1$ .]

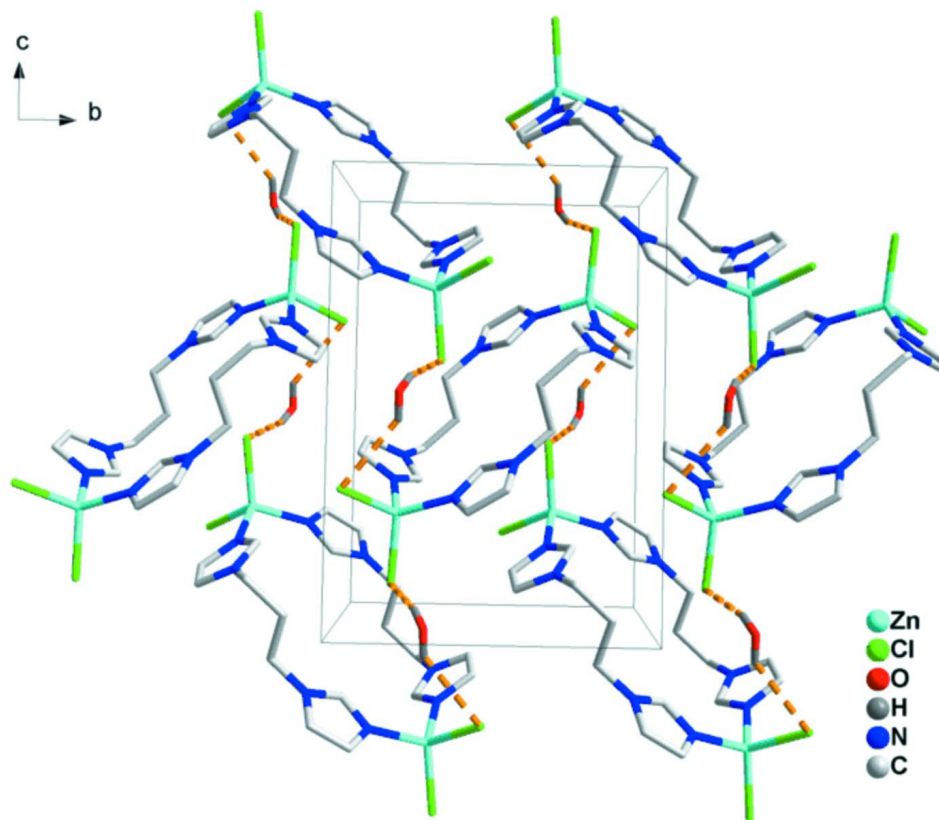


Figure 2

The layer structure of (I) viewed along [100]. Dashed lines indicate O—H···Cl hydrogen bonds.

### Bis[ $\mu$ -1,3-bis(1*H*-imidazol-1-yl)propane- $\kappa^2N^3:N^3$ ]bis(dichloridozinc) dihydrate

#### Crystal data

[Zn<sub>2</sub>Cl<sub>4</sub>(C<sub>9</sub>H<sub>12</sub>N<sub>4</sub>)<sub>2</sub>] $\cdot$ 2H<sub>2</sub>O

$M_r = 661.02$

Monoclinic,  $P2_1/c$

Hall symbol: -P 2ybc

$a = 10.1378$  (4) Å

$b = 9.7173$  (4) Å

$c = 13.8801$  (6) Å

$\beta = 93.704$  (2)°

$V = 1364.50$  (10) Å<sup>3</sup>

$Z = 2$

$F(000) = 672$

$D_x = 1.609$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 6173 reflections

$\theta = 2.0$ – $27.6$ °

$\mu = 2.18$  mm<sup>-1</sup>

$T = 296$  K

Block, colourless

$0.25 \times 0.18 \times 0.12$  mm

#### Data collection

Bruker APEXII CCD  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\omega$  and  $\phi$ -scans

Absorption correction: multi-scan  
(*SADABS*; Bruker, 2006)

$T_{\min} = 0.631$ ,  $T_{\max} = 0.770$

21184 measured reflections

3162 independent reflections

2473 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.033$

$\theta_{\max} = 27.6$ °,  $\theta_{\min} = 2.0$ °

$h = -13 \rightarrow 13$

$k = -11 \rightarrow 12$

$l = -18 \rightarrow 18$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.030$

$wR(F^2) = 0.087$

$S = 1.04$

3162 reflections

154 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

$w = 1/[\sigma^2(F_o^2) + (0.0432P)^2 + 0.5084P]$

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

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.55$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.23$  e Å<sup>-3</sup>

#### 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<sup>2</sup>)

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
Zn1	0.34226 (3)	0.80572 (3)	0.74784 (2)	0.04641 (11)

Cl1	0.39773 (7)	0.82340 (8)	0.90676 (5)	0.0634 (2)
Cl2	0.19959 (7)	0.97104 (7)	0.69087 (6)	0.0684 (2)
O1W	0.1166 (3)	0.7064 (4)	0.9784 (2)	0.1265 (12)
H1WA	0.1788	0.7355	0.9456	0.152*
H1WB	0.1429	0.7043	1.0377	0.152*
N1	0.25814 (18)	0.6260 (2)	0.70857 (13)	0.0443 (4)
N2	0.21115 (19)	0.43655 (19)	0.62798 (13)	0.0430 (4)
N3	0.29331 (19)	0.1944 (2)	0.37271 (14)	0.0452 (4)
N4	0.49316 (19)	0.1728 (2)	0.32351 (14)	0.0458 (5)
C1	0.1371 (2)	0.5783 (3)	0.73318 (17)	0.0474 (6)
H1A	0.0841	0.6199	0.7771	0.057*
C2	0.2989 (2)	0.5376 (2)	0.64438 (16)	0.0449 (5)
H2A	0.3782	0.5449	0.6146	0.054*
C3	0.1072 (2)	0.4622 (3)	0.68377 (17)	0.0481 (6)
H3A	0.0310	0.4096	0.6869	0.058*
C4	0.2215 (3)	0.3244 (2)	0.55803 (18)	0.0505 (6)
H4A	0.1746	0.2443	0.5797	0.061*
H4B	0.3137	0.2993	0.5542	0.061*
C5	0.1646 (2)	0.3666 (3)	0.45951 (16)	0.0489 (5)
H5A	0.2165	0.4420	0.4363	0.059*
H5B	0.0752	0.3999	0.4651	0.059*
C6	0.1616 (2)	0.2514 (3)	0.3861 (2)	0.0555 (6)
H6A	0.1046	0.1785	0.4068	0.067*
H6B	0.1241	0.2858	0.3247	0.067*
C7	0.3435 (3)	0.0752 (3)	0.41050 (19)	0.0571 (6)
H7A	0.3011	0.0141	0.4498	0.069*
C8	0.3855 (2)	0.2498 (3)	0.32038 (17)	0.0489 (6)
H8A	0.3753	0.3318	0.2862	0.059*
C9	0.4668 (3)	0.0622 (3)	0.38023 (18)	0.0533 (6)
H9A	0.5245	-0.0101	0.3955	0.064*

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Zn1	0.04298 (17)	0.04543 (18)	0.05161 (18)	0.00076 (12)	0.00928 (12)	-0.00741 (12)
Cl1	0.0657 (4)	0.0750 (5)	0.0500 (4)	-0.0032 (3)	0.0081 (3)	-0.0131 (3)
Cl2	0.0546 (4)	0.0569 (4)	0.0943 (5)	0.0137 (3)	0.0087 (4)	0.0021 (4)
O1W	0.0764 (17)	0.190 (4)	0.116 (2)	0.0006 (18)	0.0274 (16)	-0.022 (2)
N1	0.0451 (10)	0.0451 (11)	0.0431 (10)	0.0013 (9)	0.0051 (8)	-0.0024 (8)
N2	0.0450 (10)	0.0398 (10)	0.0437 (10)	0.0030 (8)	0.0000 (8)	-0.0004 (8)
N3	0.0391 (10)	0.0488 (12)	0.0480 (11)	-0.0025 (8)	0.0062 (8)	-0.0095 (9)
N4	0.0453 (11)	0.0478 (12)	0.0451 (11)	0.0026 (9)	0.0088 (8)	-0.0006 (8)
C1	0.0415 (12)	0.0553 (15)	0.0462 (12)	0.0047 (11)	0.0082 (10)	0.0008 (10)
C2	0.0439 (12)	0.0435 (13)	0.0477 (12)	-0.0006 (10)	0.0070 (10)	-0.0029 (10)
C3	0.0403 (12)	0.0522 (15)	0.0518 (13)	-0.0034 (10)	0.0029 (10)	0.0065 (11)
C4	0.0585 (15)	0.0388 (13)	0.0536 (14)	0.0029 (11)	-0.0008 (11)	-0.0051 (10)
C5	0.0466 (13)	0.0520 (14)	0.0486 (13)	0.0060 (11)	0.0062 (10)	-0.0028 (11)
C6	0.0366 (12)	0.0696 (17)	0.0604 (15)	0.0003 (12)	0.0040 (11)	-0.0170 (13)

C7	0.0603 (16)	0.0513 (15)	0.0614 (15)	-0.0057 (12)	0.0171 (12)	0.0044 (12)
C8	0.0467 (13)	0.0493 (14)	0.0514 (14)	0.0031 (11)	0.0094 (11)	0.0013 (11)
C9	0.0574 (15)	0.0461 (14)	0.0570 (14)	0.0063 (11)	0.0088 (12)	0.0011 (11)

*Geometric parameters (Å, °)*

Zn1—N1	2.0038 (19)	C1—C3	1.345 (3)
Zn1—N4 <sup>i</sup>	2.0053 (19)	C1—H1A	0.9300
Zn1—C11	2.2476 (7)	C2—H2A	0.9300
Zn1—C12	2.2694 (7)	C3—H3A	0.9300
O1W—H1WA	0.8500	C4—C5	1.507 (3)
O1W—H1WB	0.8500	C4—H4A	0.9700
N1—C2	1.323 (3)	C4—H4B	0.9700
N1—C1	1.375 (3)	C5—C6	1.513 (3)
N2—C2	1.334 (3)	C5—H5A	0.9700
N2—C3	1.370 (3)	C5—H5B	0.9700
N2—C4	1.468 (3)	C6—H6A	0.9700
N3—C8	1.333 (3)	C6—H6B	0.9700
N3—C7	1.357 (3)	C7—C9	1.350 (4)
N3—C6	1.468 (3)	C7—H7A	0.9300
N4—C8	1.322 (3)	C8—H8A	0.9300
N4—C9	1.369 (3)	C9—H9A	0.9300
N4—Zn1 <sup>i</sup>	2.0053 (19)		
N1—Zn1—N4 <sup>i</sup>	108.03 (8)	N2—C4—C5	111.01 (19)
N1—Zn1—C11	114.12 (6)	N2—C4—H4A	109.4
N4 <sup>i</sup> —Zn1—C11	108.26 (6)	C5—C4—H4A	109.4
N1—Zn1—C12	105.77 (6)	N2—C4—H4B	109.4
N4 <sup>i</sup> —Zn1—C12	106.63 (6)	C5—C4—H4B	109.4
C11—Zn1—C12	113.66 (3)	H4A—C4—H4B	108.0
H1WA—O1W—H1WB	109.3	C4—C5—C6	113.6 (2)
C2—N1—C1	105.7 (2)	C4—C5—H5A	108.8
C2—N1—Zn1	127.09 (16)	C6—C5—H5A	108.8
C1—N1—Zn1	126.64 (16)	C4—C5—H5B	108.8
C2—N2—C3	107.35 (19)	C6—C5—H5B	108.8
C2—N2—C4	125.7 (2)	H5A—C5—H5B	107.7
C3—N2—C4	126.8 (2)	N3—C6—C5	112.65 (19)
C8—N3—C7	107.2 (2)	N3—C6—H6A	109.1
C8—N3—C6	126.3 (2)	C5—C6—H6A	109.1
C7—N3—C6	126.5 (2)	N3—C6—H6B	109.1
C8—N4—C9	105.8 (2)	C5—C6—H6B	109.1
C8—N4—Zn1 <sup>i</sup>	129.55 (17)	H6A—C6—H6B	107.8
C9—N4—Zn1 <sup>i</sup>	124.41 (16)	C9—C7—N3	106.9 (2)
C3—C1—N1	109.3 (2)	C9—C7—H7A	126.5
C3—C1—H1A	125.3	N3—C7—H7A	126.5
N1—C1—H1A	125.3	N4—C8—N3	111.1 (2)
N1—C2—N2	111.0 (2)	N4—C8—H8A	124.4
N1—C2—H2A	124.5	N3—C8—H8A	124.4

N2—C2—H2A	124.5	C7—C9—N4	108.9 (2)
C1—C3—N2	106.6 (2)	C7—C9—H9A	125.5
C1—C3—H3A	126.7	N4—C9—H9A	125.5
N2—C3—H3A	126.7		
N4 <sup>i</sup> —Zn1—N1—C2	-2.4 (2)	C2—N2—C4—C5	-86.8 (3)
Cl1—Zn1—N1—C2	-122.86 (18)	C3—N2—C4—C5	88.8 (3)
Cl2—Zn1—N1—C2	111.46 (19)	N2—C4—C5—C6	-175.0 (2)
N4 <sup>i</sup> —Zn1—N1—C1	-172.98 (18)	C8—N3—C6—C5	-78.9 (3)
Cl1—Zn1—N1—C1	66.57 (19)	C7—N3—C6—C5	101.8 (3)
Cl2—Zn1—N1—C1	-59.12 (19)	C4—C5—C6—N3	-58.6 (3)
C2—N1—C1—C3	0.2 (3)	C8—N3—C7—C9	0.5 (3)
Zn1—N1—C1—C3	172.38 (16)	C6—N3—C7—C9	180.0 (2)
C1—N1—C2—N2	-0.6 (3)	C9—N4—C8—N3	0.6 (3)
Zn1—N1—C2—N2	-172.77 (14)	Zn1 <sup>i</sup> —N4—C8—N3	174.77 (15)
C3—N2—C2—N1	0.8 (3)	C7—N3—C8—N4	-0.7 (3)
C4—N2—C2—N1	177.2 (2)	C6—N3—C8—N4	179.8 (2)
N1—C1—C3—N2	0.3 (3)	N3—C7—C9—N4	-0.2 (3)
C2—N2—C3—C1	-0.7 (2)	C8—N4—C9—C7	-0.2 (3)
C4—N2—C3—C1	-177.0 (2)	Zn1 <sup>i</sup> —N4—C9—C7	-174.79 (17)

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

*Hydrogen-bond geometry* ( $\text{\AA}$ ,  $^\circ$ )

<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>
O1 <i>W</i> —H1 <i>WA</i> $\cdots$ C11	0.85	2.47	3.282 (3)	160
O1 <i>W</i> —H1 <i>WB</i> $\cdots$ Cl2 <sup>ii</sup>	0.85	2.76	3.473 (4)	143

Symmetry code: (ii)  $x, -y+3/2, z+1/2$ .