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Dichlorido{2-hydroxyimino-*N*′-[1-(2-pyridyl)ethylidene]propanohydrazide-κ^3N,N',O}zinc(II) hemihydrate

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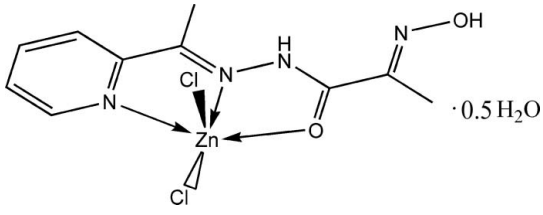
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.037; wR factor = 0.070; data-to-parameter ratio = 17.7.

The title compound, $[ZnCl_2(C_{10}H_{12}N_4O_2)]\cdot 0.5H_2O$, was readily prepared by the reaction between $ZnCl_2$ and 2-hydroxyimino-*N*′-[1-(2-pyridyl)ethylidene]propanohydrazide. The Zn atom has a distorted trigonal–bipyramidal geometry with two Cl atoms and one azomethine N atom in the equatorial plane and one pyridine N atom and one amide O atom in the axial positions. In the crystal structure, complex molecules are connected in pairs by N—H···Cl hydrogen bonds, formed between the amide NH of one molecule and the Cl atom of a neighboring one. Molecular pairs are connected by hydrogen bonds involving the uncoordinated water molecule, which lies on a twofold axis.

Related literature

For details of the structure and biological activity of zinc(II) complexes, see: Canary *et al.* (1998); Comba *et al.* (2002); Kasuga *et al.* (2003); Panosyan *et al.* (2003); Rodriuez-Argüelles *et al.* (1995); Sousa *et al.* (2003). For the preparation of 2-(oximato)propanehydrazide, see: Fritsky *et al.* (1998).



Experimental

Crystal data

$[ZnCl_2(C_{10}H_{12}N_4O_2)]\cdot 0.5H_2O$
M_r = 365.51
Monoclinic, $C2/c$
a = 14.5666 (8) Å
b = 12.9898 (8) Å
c = 15.8671 (8) Å
β = 109.873 (5)°
V = 2823.5 (3) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 2.13 mm^{-1}
T = 100 (2) K
0.5 × 0.1 × 0.05 mm

Data collection

Kuma KM-4 CCD area-detector diffractometer
Absorption correction: multi-scan (*PLATON*; Spek, 2003)
T_{min} = 0.774, T_{max} = 0.891
17382 measured reflections
3373 independent reflections
3069 reflections with $I > 2\sigma(I)$
R_{int} = 0.043

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.070
S = 1.21
3373 reflections
191 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Zn1—N1	2.1409 (19)	Zn1—Cl2	2.2513 (6)
Zn1—N2	2.1142 (18)	Zn1—Cl3	2.2195 (6)
Zn1—O1	2.2348 (16)		
N2—Zn1—N1	74.71 (7)	N2—Zn1—O1	72.57 (6)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N3—H2···Cl3^{i}	0.80 (3)	2.59 (3)	3.310 (2)	150 (3)
O1W—H1W···Cl2^{ii}	0.81 (3)	2.44 (3)	3.182 (2)	153 (3)
O2—H1···O1W^{iii}	0.88 (3)	1.89 (4)	2.7015 (19)	153 (3)

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

Data collection: *KM-4 CCD Software* (Kuma, 1999); 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: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2108).

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Dichlorido{2-hydroxyimino-*N*′-[1-(2-pyridyl)ethylidene]propanohydrazide-κ^3N,N',O}zinc(II) hemihydrate

Yurii S. Moroz, Tetyana Yu. Sliva, Kinga Kulon, Henryk Kozłowski and Igor O. Fritsky

S1. Comment

In the past few decades mononuclear zinc(II) complexes attract a lot of attention because of their biological and catalytical activity (Kasuga *et al.*, 2003; Panosyan *et al.*, 2003; Rodriuez-Argüelles *et al.*, 1995; Sousa *et al.*, 2003). On the other hand, many zinc mononuclear complexes containing additional vacant donor sets and chelate centers are taken as ligands for building of homo- and heteropolynuclear systems, which are widely used in bioinorganic modeling and catalysis. The properties of the obtained compounds are closely connected with their structure, so the best results one could obtain using well designed ligand systems. We report here the synthesis and structure of a zinc complex, $[ZnCl_2(C_{10}H_{12}N_4O_2)].0.5H_2O$, based on a novel polynucleative ligand 2-(oximato)-*N*′-(1-(pyridin-2-yl)ethylidene)propanehydrazide (*L*).

The title compound consists of neutral complex molecules and solvated water molecules (Fig. 1). The Zn atom has a distorted trigonal-bipyramidal geometry, defined by two Cl atoms and one azomethine N atom at the equatorial plane and a pyridine N atom and an amide O atom at the axial positions. The Zn—N, Zn—O and Zn—Cl bond lengths are comparable to previously reported zinc complexes with thiosemicarbasone and semicarbasone derivatives (Kasuga *et al.*, 2003) and with pyridine complexed to the metal ion (Canary *et al.*, 1998; Comba *et al.*, 2002) (Table 1). The bite angles around the central atom deviate from an ideal square-planar configuration [*e.g.* N2—Zn1—O1 = 72.57 (6)°, N2—Zn1—N1 = 74.71 (7)°], that is a consequence of the formation of two almost flat five-membered chelate rings. In the crystal packing, the molecules are connected in pairs by N—H···Cl hydrogen bonds (Fig. 2), where the protonated amide group from one molecule acts as a donor and the Cl atom from the neighboring molecule as an acceptor (Table 2). The weak π-stacking interaction is observed between the pyridine ring and oximic group. The molecular pairs are connected to each other by hydrogen bonds involving the solvated water molecules, resulting in an extensive three-dimensional system (Fig. 3).

S2. Experimental

2-(Oximato)-*N*′-(1-(pyridin-2-yl)ethylidene)propanehydrazide (*L*) was prepared according to early reported method (Fritsky *et al.*, 1998). 2-Acetylpyridine (1.2 ml, 0.0107 mol) was added to a stirred warm ethanol/water solution (20 ml) of 2-(oximato)propanehydrazide (1.17 g, 0.01 mol). After stirring at 333 k for 6 h, the solution was cooled and a white precipitate was formed immediately. It was filtered off, washed with water and acetone and dried under vacuum (yield 72%, 1.59 g). ^{1}H NMR, 400.13 MHz, (DMSO-d6): 12.118 (s, 1H, N—OH), 10.242 (s, 1H, NH), 8.608 (dt, 1H, py-6, J6,5 = 4.8 Hz, J6,4 = 1.8 Hz), 8.066 (d, 1H, py-3, J3,4 = 7.8 Hz), 7.861 (td, 1H, py-4, J4,5,3 = 7.8 Hz, J4,6 = 1.8 Hz), 7.415 (ddd, 1H, py-5, J5,6 = 4.8 Hz, J5,4 = 7.8 Hz, J = 1.2 Hz), 2.376 (s, 3H, CH_3(py)), 1.985(s, 3H, CH_3). IR (KBr, cm^{-1}): 1660 (CO_{amid}), 1030 (NO_{oxim}), 3340 (NH_{as}). Analysis calculated for $C_{10}H_{12}N_4O_2$: C 54.54, H 5.49, N 25.44%; found: C 54.41, H 5.62, N 25.42%.

Zinc(II) chloride (0.014 g, 0.1 mmol) in H_2O (5 ml) was added to 10 ml of hot methanol solution of *L* (0.022 g, 0.1 mmol). The solution was left for slow evaporation at room temperature. After 5 days cubic crystals of the title compound suitable for X-ray analysis were obtained.

S3. Refinement

H atoms on C atoms were positioned geometrically and refined as riding atoms, with C—H = 0.93Å (CH) and 0.96Å (CH_3), and with U_{iso}(H) = 1.2U_{eq}(C) for aromatic and U_{iso}(H) = 1.5U_{eq}(C) for methyl group. H atoms on N and O atoms were located from a difference Fourier map and were refined isotropically.

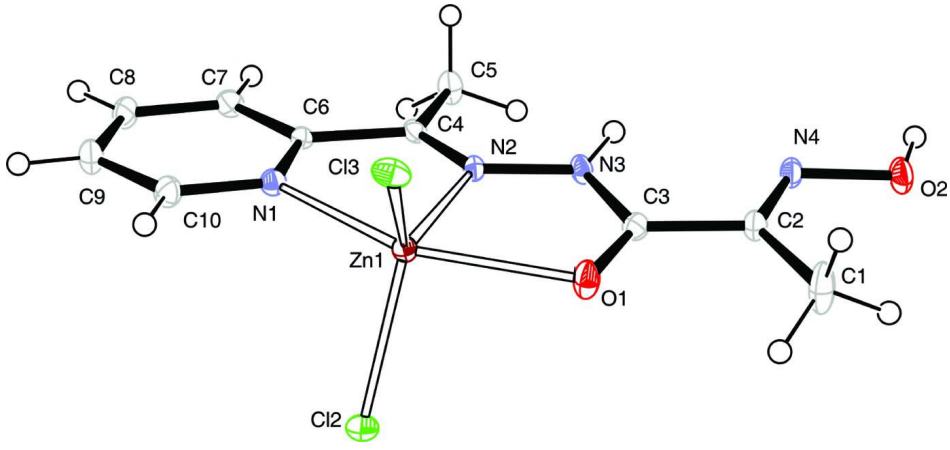

Figure 1

The molecular structure of the title compound. Displacement ellipsoids are shown at the 40% probability level. Water molecule is not shown.

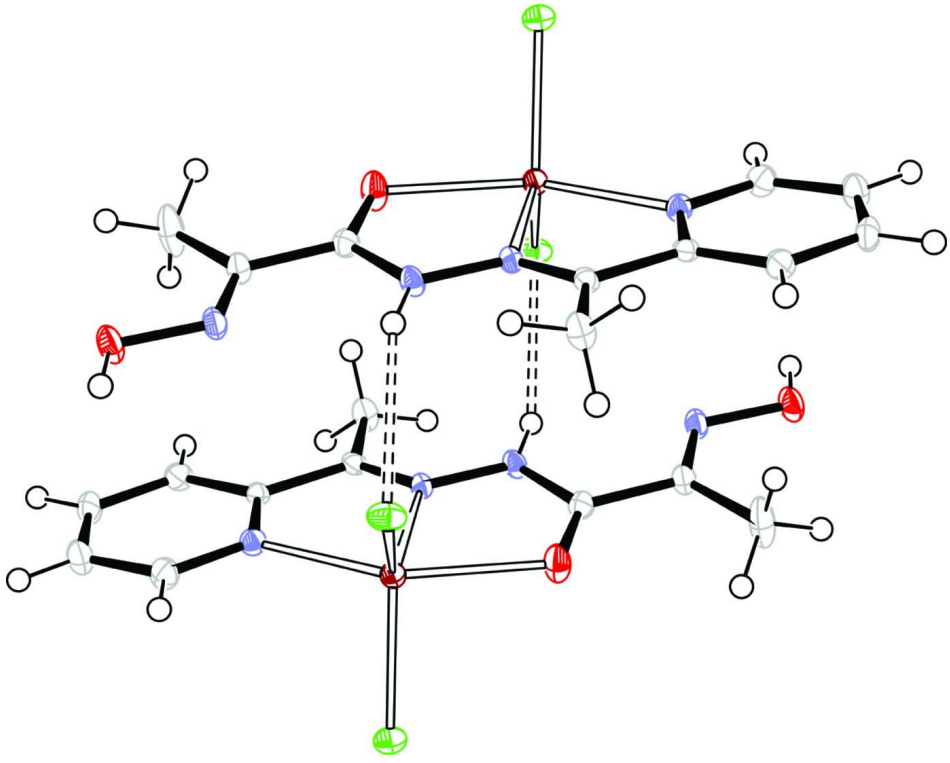

Figure 2

A view of a pair of the complex molecules. Displacement ellipsoids are shown at the 40% probability level. Hydrogen bonds are indicated by dashed lines.

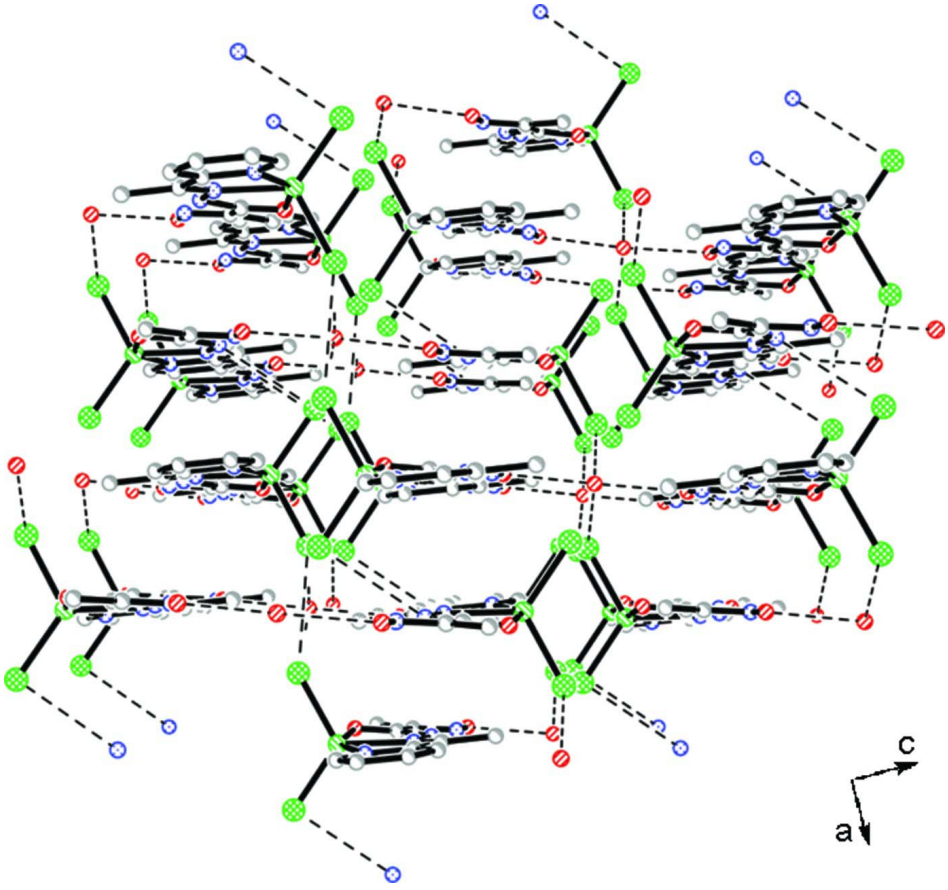

Figure 3

A packing diagram of the title compound along the *b*-axis direction. Hydrogen bonds are indicated by dashed lines. H atoms have been omitted for clarity.

Dichlorido[2-hydroxyimino-*N*′-[1-(2-pyridyl)ethylidene]propanohydrazide- κ^3N,N',O]zinc(II) monohydrate

Crystal data

$[ZnCl_2(C_{10}H_{12}N_4O_2)]\cdot 0.5H_2O$
$M_r = 365.51$
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
$a = 14.5666\ (8)$ Å
$b = 12.9898\ (8)$ Å
$c = 15.8671\ (8)$ Å
$\beta = 109.873\ (5)°$
$V = 2823.5\ (3)$ Å^3
$Z = 8$
$F(000) = 1480$
$D_x = 1.720$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3373 reflections
$\theta = 3.1–28.5°$
$\mu = 2.13$ mm^{-1}
$T = 100$ K
Cubic, yellow
0.5 × 0.1 × 0.05 mm

Data collection

Kum KM4 CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*PLATON*; Spek, 2003)
$T_{min} = 0.774$, $T_{max} = 0.891$
17382 measured reflections
3373 independent reflections
3069 reflections with $I > 2\sigma(I)$

R_{int} = 0.043
θ_{max} = 28.5°, θ_{min} = 3.1°
h = −19→19
k = −16→16
l = −20→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.070
S = 1.21
3373 reflections
191 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0282P)^2 + 3.185P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.8446 (2)	0.45584 (19)	0.90460 (16)	0.0300 (6)
H1A	0.8849	0.3958	0.9225	0.045*
H1B	0.8500	0.4827	0.8501	0.045*
H1C	0.7778	0.4378	0.8949	0.045*
C2	0.87720 (17)	0.53560 (17)	0.97658 (14)	0.0155 (4)
C3	0.86291 (17)	0.64543 (17)	0.95017 (14)	0.0149 (4)
C4	0.89866 (16)	0.89516 (17)	1.03894 (14)	0.0127 (4)
C5	0.96032 (18)	0.89213 (18)	1.13564 (14)	0.0187 (5)
H5A	0.9937	0.8272	1.1490	0.028*
H5B	0.9197	0.9003	1.1718	0.028*
H5C	1.0073	0.9469	1.1483	0.028*
C6	0.86661 (16)	0.99579 (17)	0.99301 (14)	0.0124 (4)
C7	0.89136 (17)	1.08943 (18)	1.03628 (15)	0.0163 (5)
H7	0.9293	1.0921	1.0967	0.020*
C8	0.85855 (19)	1.17985 (18)	0.98807 (16)	0.0194 (5)
H8	0.8745	1.2437	1.0157	0.023*
C9	0.80194 (19)	1.17314 (18)	0.89857 (16)	0.0203 (5)
H9	0.7788	1.2323	0.8650	0.024*
C10	0.78027 (17)	1.07659 (18)	0.85973 (15)	0.0181 (5)
H10	0.7424	1.0722	0.7994	0.022*
N1	0.81153 (14)	0.98950 (15)	0.90538 (12)	0.0140 (4)
N2	0.86837 (14)	0.81687 (14)	0.98712 (12)	0.0127 (4)
N3	0.88998 (14)	0.71758 (14)	1.01651 (13)	0.0144 (4)
N4	0.91703 (14)	0.51964 (14)	1.06044 (12)	0.0161 (4)
O1	0.82753 (14)	0.67055 (12)	0.87067 (10)	0.0213 (4)
O2	0.92836 (13)	0.41557 (13)	1.08008 (11)	0.0211 (4)
Cl2	0.88241 (4)	0.85938 (4)	0.75864 (3)	0.01726 (13)
Cl3	0.63307 (4)	0.81753 (5)	0.78506 (4)	0.01874 (13)
Zn1	0.793127 (19)	0.838156 (19)	0.848619 (16)	0.01239 (8)
H1	0.954 (3)	0.413 (3)	1.139 (2)	0.048 (10)*

O1W	0.0000	0.34448 (19)	0.2500	0.0206 (5)
H1W	0.045 (2)	0.308 (3)	0.250 (2)	0.048 (11)*
H2	0.905 (2)	0.699 (2)	1.068 (2)	0.024 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0471 (17)	0.0123 (12)	0.0180 (12)	−0.0014 (11)	−0.0052 (11)	−0.0005 (9)
C2	0.0178 (11)	0.0114 (11)	0.0162 (11)	−0.0004 (9)	0.0045 (9)	0.0004 (8)
C3	0.0163 (11)	0.0135 (11)	0.0150 (10)	−0.0008 (9)	0.0054 (9)	−0.0011 (8)
C4	0.0134 (11)	0.0124 (11)	0.0129 (10)	−0.0022 (8)	0.0054 (8)	−0.0001 (8)
C5	0.0235 (13)	0.0145 (12)	0.0138 (10)	−0.0020 (9)	0.0005 (9)	0.0002 (9)
C6	0.0122 (10)	0.0130 (11)	0.0135 (10)	0.0006 (8)	0.0063 (8)	0.0016 (8)
C7	0.0181 (12)	0.0163 (11)	0.0141 (10)	−0.0019 (9)	0.0050 (9)	−0.0016 (8)
C8	0.0256 (13)	0.0108 (11)	0.0220 (12)	−0.0017 (9)	0.0086 (10)	−0.0027 (9)
C9	0.0243 (13)	0.0140 (12)	0.0211 (12)	0.0019 (9)	0.0058 (10)	0.0033 (9)
C10	0.0196 (12)	0.0179 (12)	0.0152 (11)	0.0031 (9)	0.0039 (9)	0.0019 (9)
N1	0.0153 (9)	0.0132 (9)	0.0130 (9)	−0.0003 (7)	0.0042 (7)	−0.0008 (7)
N2	0.0150 (9)	0.0103 (9)	0.0130 (9)	0.0011 (7)	0.0051 (7)	0.0016 (7)
N3	0.0202 (10)	0.0115 (9)	0.0097 (9)	−0.0001 (8)	0.0028 (8)	0.0021 (7)
N4	0.0208 (10)	0.0103 (9)	0.0169 (9)	0.0011 (8)	0.0062 (8)	0.0019 (7)
O1	0.0333 (10)	0.0136 (8)	0.0122 (7)	0.0026 (7)	0.0015 (7)	0.0002 (6)
O2	0.0322 (10)	0.0126 (8)	0.0164 (8)	0.0014 (7)	0.0054 (7)	0.0043 (6)
Cl2	0.0152 (3)	0.0210 (3)	0.0172 (3)	−0.0004 (2)	0.0076 (2)	−0.0007 (2)
Cl3	0.0154 (3)	0.0255 (3)	0.0158 (3)	−0.0057 (2)	0.0059 (2)	−0.0026 (2)
Zn1	0.01436 (14)	0.01195 (13)	0.01015 (12)	−0.00066 (10)	0.00323 (9)	−0.00064 (9)
O1W	0.0272 (14)	0.0137 (12)	0.0202 (12)	0.000	0.0070 (11)	0.000

Geometric parameters (Å, º)

C1—C2	1.495 (3)	C7—H7	0.9300
C1—H1A	0.9600	C8—C9	1.381 (3)
C1—H1B	0.9600	C8—H8	0.9300
C1—H1C	0.9600	C9—C10	1.386 (3)
C2—N4	1.275 (3)	C9—H9	0.9300
C2—C3	1.482 (3)	C10—N1	1.337 (3)
C3—O1	1.233 (3)	C10—H10	0.9300
C3—N3	1.364 (3)	Zn1—N1	2.1409 (19)
C4—N2	1.288 (3)	N2—N3	1.371 (3)
C4—C5	1.492 (3)	Zn1—N2	2.1142 (18)
C4—C6	1.492 (3)	N3—H2	0.80 (3)
C5—H5A	0.9600	N4—O2	1.384 (2)
C5—H5B	0.9600	Zn1—O1	2.2348 (16)
C5—H5C	0.9600	O2—H1	0.88 (3)
C6—N1	1.351 (3)	Zn1—Cl2	2.2513 (6)
C6—C7	1.383 (3)	Zn1—Cl3	2.2195 (6)
C7—C8	1.394 (3)	O1W—H1W	0.81 (3)

C2—C1—H1A	109.5	C7—C8—H8	120.5
C2—C1—H1B	109.5	C8—C9—C10	118.7 (2)
H1A—C1—H1B	109.5	C8—C9—H9	120.6
C2—C1—H1C	109.5	C10—C9—H9	120.6
H1A—C1—H1C	109.5	N1—C10—C9	122.7 (2)
H1B—C1—H1C	109.5	N1—C10—H10	118.6
N4—C2—C3	115.0 (2)	C9—C10—H10	118.6
N4—C2—C1	126.8 (2)	C10—N1—C6	118.69 (19)
C3—C2—C1	118.27 (19)	C10—N1—Zn1	125.33 (15)
O1—C3—N3	121.2 (2)	C6—N1—Zn1	115.73 (15)
O1—C3—C2	120.9 (2)	C4—N2—N3	122.44 (18)
N3—C3—C2	117.88 (19)	C4—N2—Zn1	120.30 (15)
N2—C4—C5	126.3 (2)	N3—N2—Zn1	117.03 (13)
N2—C4—C6	113.42 (18)	C3—N3—N2	114.28 (18)
C5—C4—C6	120.29 (19)	C3—N3—H2	119 (2)
C4—C5—H5A	109.5	N2—N3—H2	125 (2)
C4—C5—H5B	109.5	C2—N4—O2	111.77 (18)
H5A—C5—H5B	109.5	C3—O1—Zn1	114.29 (14)
C4—C5—H5C	109.5	N4—O2—H1	104 (2)
H5A—C5—H5C	109.5	N2—Zn1—N1	74.71 (7)
H5B—C5—H5C	109.5	N2—Zn1—Cl3	123.36 (5)
N1—C6—C7	121.8 (2)	N1—Zn1—Cl3	105.30 (5)
N1—C6—C4	115.30 (19)	N2—Zn1—O1	72.57 (6)
C7—C6—C4	122.86 (19)	N1—Zn1—O1	147.07 (6)
C6—C7—C8	119.1 (2)	Cl3—Zn1—O1	95.66 (5)
C6—C7—H7	120.5	N2—Zn1—Cl2	117.91 (5)
C8—C7—H7	120.5	N1—Zn1—Cl2	97.91 (5)
C9—C8—C7	119.0 (2)	Cl3—Zn1—Cl2	118.08 (2)
C9—C8—H8	120.5	O1—Zn1—Cl2	94.10 (5)

N4—C2—C3—O1	178.0 (2)	Zn1—N2—N3—C3	2.7 (2)
C1—C2—C3—O1	−2.0 (4)	C3—C2—N4—O2	−179.79 (19)
N4—C2—C3—N3	−2.1 (3)	C1—C2—N4—O2	0.2 (4)
C1—C2—C3—N3	177.9 (2)	N3—C3—O1—Zn1	−8.3 (3)
N2—C4—C6—N1	0.0 (3)	C2—C3—O1—Zn1	171.56 (16)
C5—C4—C6—N1	179.6 (2)	C4—N2—Zn1—N1	−6.55 (17)
N2—C4—C6—C7	−179.8 (2)	N3—N2—Zn1—N1	178.85 (17)
C5—C4—C6—C7	−0.1 (3)	C4—N2—Zn1—Cl3	−105.03 (17)
N1—C6—C7—C8	0.0 (3)	N3—N2—Zn1—Cl3	80.36 (16)
C4—C6—C7—C8	179.7 (2)	C4—N2—Zn1—O1	169.73 (19)
C6—C7—C8—C9	0.3 (4)	N3—N2—Zn1—O1	−4.88 (15)
C7—C8—C9—C10	−0.4 (4)	C4—N2—Zn1—Cl2	84.41 (17)
C8—C9—C10—N1	0.3 (4)	N3—N2—Zn1—Cl2	−90.20 (15)
C9—C10—N1—C6	−0.1 (3)	C10—N1—Zn1—N2	−179.8 (2)
C9—C10—N1—Zn1	−173.99 (18)	C6—N1—Zn1—N2	6.09 (15)
C7—C6—N1—C10	−0.1 (3)	C10—N1—Zn1—Cl3	−58.74 (19)
C4—C6—N1—C10	−179.9 (2)	C6—N1—Zn1—Cl3	127.17 (14)
C7—C6—N1—Zn1	174.42 (17)	C10—N1—Zn1—O1	173.63 (16)

C4—C6—N1—Zn1	−5.4 (2)	C6—N1—Zn1—O1	−0.5 (2)
C5—C4—N2—N3	0.4 (3)	C10—N1—Zn1—Cl2	63.31 (19)
C6—C4—N2—N3	179.97 (19)	C6—N1—Zn1—Cl2	−110.78 (15)
C5—C4—N2—Zn1	−173.95 (18)	C3—O1—Zn1—N2	6.96 (17)
C6—C4—N2—Zn1	5.7 (3)	C3—O1—Zn1—N1	13.6 (2)
O1—C3—N3—N2	4.1 (3)	C3—O1—Zn1—Cl3	−116.27 (17)
C2—C3—N3—N2	−175.80 (19)	C3—O1—Zn1—Cl2	124.96 (17)
C4—N2—N3—C3	−171.8 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H2···Cl3^{i}	0.80 (3)	2.59 (3)	3.310 (2)	150 (3)
O1*W*—H1*W*···Cl2^{ii}	0.81 (3)	2.44 (3)	3.182 (2)	153 (3)
O2—H1···O1*W*^{iii}	0.88 (3)	1.89 (4)	2.7015 (19)	153 (3)

Symmetry codes: (i) −*x*+3/2, −*y*+3/2, −*z*+2; (ii) −*x*+1, −*y*+1, −*z*+1; (iii) *x*+1, *y*, *z*+1.